

**NEAR-INFRARED TUNABLE
DIODE LASER ABSORPTION
SPECTROMETER FOR
TRACE GAS DETECTION**

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Dedicated to my parents, for their love, support and encouragement.

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Abstract

A ground based near-infrared tunable diode laser absorption spectrometer (TDLAS), capable of measuring methane (CH₄) and nitrous oxide (N₂O) in their natural abundance's within the atmosphere, has been designed and constructed. The spectrometer has high resolution (0.0003 cm⁻¹), high precision (equivalent to a minimum detectable absorption of ~ 2 x 10⁻⁷) and uses the latest developments in near-infrared technology to provide similar sensitivities to mid-infrared laser spectrometers.

A theoretical analysis of the noise performance of different detection schemes has been performed. It was concluded that near optimum performance could be achieved using second harmonic wavelength modulation spectroscopy in the 50-100 kHz region.

Instrument sensitivity was enhanced through the use of a Herriott multipass cell. A computer model of the behaviour of a paraxial ray passing through such a cell has been developed, allowing suitable choice of cell design and spot pattern to be made. The model was modified to allow the behaviour of an astigmatic Herriott cell to be investigated. The model has shown that the astigmatic cell has all the inherent stability properties of the conventional cell. An astigmatic cell capable of supporting a 100 m pathlength in a 2 litre mode volume was designed, built and tested.

Performance assessment of the spectrometer was carried out. Detection limits equivalent to 560 pptV of methane and 22 ppbV of nitrous oxide were obtained. A field test and intercomparison with methane monitors (Flame Ionisation Detectors), showed that the retrieved concentrations from the spectrometer agreed to within the measurement uncertainty of the other instruments.

Laboratory support for high resolution spectroscopic measurements of gases of atmospheric interest, including, methane, nitrous oxide and ammonia has been carried out. A rotational analysis of nitrous oxide was performed.

A coolable Herriott cell capable of operating between 77 and 298 K has been designed, built and tested.

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Chapter 1 -

Introduction

1 - Introduction

In the last two decades there has been increasing interest and concern about man's effect on the environment. It is clear that the Earth's atmosphere is changing. The concentrations of trace gases are (generally) increasing, not on geological timescales, but at rates as large as 1 % per year. The largest changes in concentrations are being observed in methane (CH₄), carbon monoxide (CO) and nitrous oxide (N₂O). The timescale of these changes point to man's activities as being responsible [1]. Further evidence that that these changes are anthropogenic include the observation that the increase in CO is largely confined to the highly populated and industrialised northern hemisphere. Industrialisation, however, is not only responsible for these changes. Changes in land use and agricultural practices, including the heavy use of nitrogen based fertilisers and animal husbandry, are also thought to contribute to the release of trace gases [2].

These anthropogenic changes are thought to be responsible for two phenomenon being observed in the Earth's current climate, ozone depletion and global warming. Both are described below.

1.1 Global Warming

The ultimate energy source for all weather and climate is radiation from the sun. Averaged globally and annually, about a third of incoming solar radiation is reflected back to space. Of the remainder, some is absorbed by the atmosphere, but most is absorbed by the land, ocean and ice surfaces. The solar radiation absorbed by the Earth's surface and atmosphere ($\sim 240 \text{ Wm}^{-2}$) is balanced at the top of the atmosphere by outgoing radiation at infrared wavelengths. Some of the outgoing infrared radiation is trapped by the naturally occurring greenhouse gases (principally water vapour, but also carbon dioxide, ozone, methane and nitrous oxide) and by clouds, which keep the troposphere about 33°C warmer than it would otherwise be. This is known as the natural greenhouse effect. In an unperturbed state, the net incoming solar radiation at

the top of the atmosphere, averaged over the globe over long periods of time, must be balanced by net outgoing radiation

A change in average net radiation at the top of the troposphere, because of a change in either solar or infrared radiation is defined as radiative forcing. A radiative forcing perturbs the balance between incoming and outgoing radiation. Over time, climate responds to the perturbation to re-establish the radiative balance. A positive radiative forcing tends on average to warm the surface, while a negative forcing will tend to cool it.

For example, an increase in the concentration of any of the greenhouse gases would bring about a reduction in outgoing radiation. This would result in a positive radiative forcing and a warming of the Earth's surface. When the concentration of such naturally occurring greenhouse gases increase it is referred to as the enhanced greenhouse effect. It is estimated that the global mean temperature has increased by over two degrees, over the past two centuries, as a direct consequence of the enhanced greenhouse effect [3].

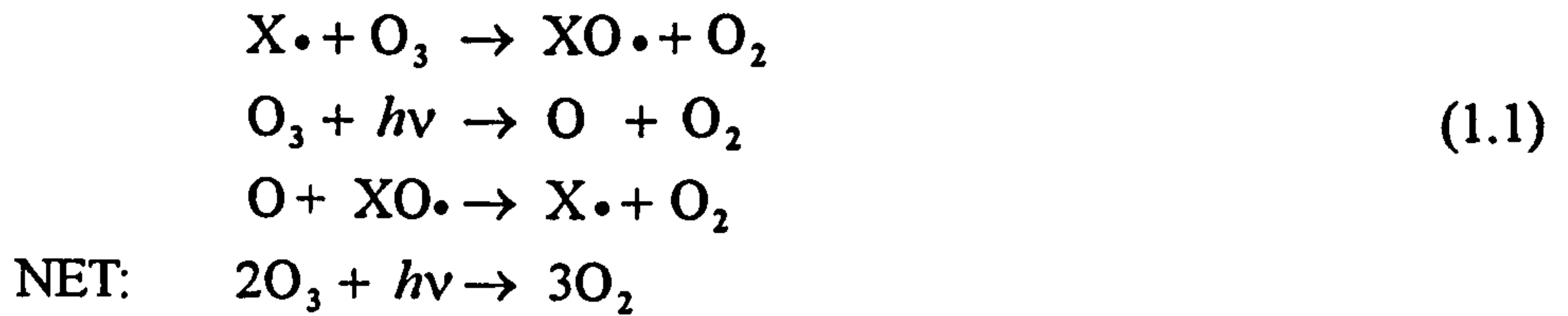
1.2 Ozone Depletion

Ozone is a key species in stratospheric chemistry. It is extremely important to the Earth's ecosystem since it absorbs harmful radiation ($\lambda \approx 230\text{nm}$) which sterilises biological life on a nuclei level.

Decreases in stratospheric ozone have occurred since the 1970s. In 1988 the International Ozone Trends Panel reported a maximum decline of 6.2% in ozone between 30°N and 64°N in the period from 1969 to 1986 [4]. The most startling feature was the discovery of the Antarctic Ozone hole by Farman in 1984 [5]. The October average total ozone values over Antarctica are 50-70 % lower than those observed in the 1960s. The ozone loss occurs at altitudes between 14 km and 24 km. The chemistry responsible for this ozone depletion will now be described.

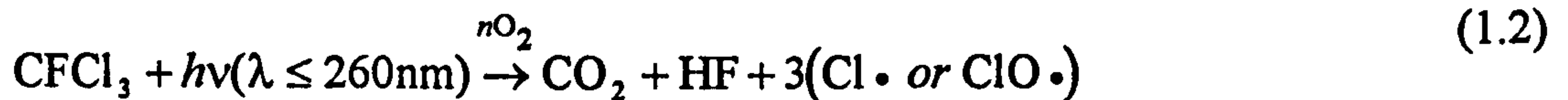
1.2.1 Ozone Chemistry

The destruction of ozone is brought about by catalytic reaction cycles that may be summarised as:



In this set of reactions, $X\cdot$ and $XO\cdot$ are radicals that catalyse the conversion of O_3 to O_2 . The most important catalysts in the atmosphere are the oxides of nitrogen, NO and NO_2 . Ground level emissions of N_2O are responsible for the bulk of oxides of nitrogen in the stratosphere.

The large reduction in ozone observed during the winter months is caused by the enhanced CFC-ozone chemistry that occurs in the cold stratosphere over the poles. Above about 17-25 km, the available solar radiation is energetic enough to destroy the CFC's releasing chlorine and chlorine monoxide molecules:



These molecules are extremely powerful ozone destroying catalysts. The relevant ozone destruction cycle that results from the release of these molecules is obtained by substituting Cl and ClO for $X\cdot$ and $XO\cdot$ in the reactions given in (1.1). CFC concentrations are increasing at approximately 6% per year [6].

1.3 Atmospheric Monitoring

Accurate and precise monitoring of atmospheric trace gases and industrial pollutants has become increasingly important as a consequence of the concerns over the causes and effects of atmospheric pollution as detailed previously. Many techniques exist for

monitoring trace gases and pollutants, ranging from ground based spectroscopic techniques to aircraft, balloon and satellite observations. The retrieved data from such measurements is being used in theoretical models to predict current and long term trends. In addition, legislation concerning gaseous emissions from industrial sources and motor vehicles, and the requirement for safety monitors capable of long term unattended operation has led to the search for new and inexpensive instrumentation to accurately monitor gases such as N_2O , CH_4 , CO and NH_3 [7]. A review of different detection techniques and instrumentation currently available is given below.

1.3.1 Measurement Technique and Instrumentation Review

Fourier transform Infrared (FTIR) spectroscopy has been used extensively for trace gas detection. Spectral coverage is very broad (2-10 μm) with all wavelengths being detected simultaneously. Solar spectra have successfully been recorded on a variety of platforms. The Atmospheric Trace Molecule Spectroscopy (ATMOS) instrument was part of the ATLAS payload on the Space Shuttle and was used to record solar occultation spectra at sunrise and sunset [8]. Toon *et al* [9] have used FTIR on board an aircraft and measured key trace species across the polar vortex boundary. The disadvantage of FTIR is that sensitivity is limited by the low intensity of the IR light sources, the instrumentation is very expensive, not particularly compact and has slow response times (typically 1-2 minutes). Instrument resolution is typically 0.01 cm^{-1} .

Tunable Diode Laser Heterodyne Spectrometers (TDLHS) operate by heterodyning tunable diode laser radiation, at wavelengths around $10\text{ }\mu\text{m}$, with solar radiation. By measuring the power generated in a narrow window (5-50 MHz) in the RF region, as the laser is tuned, resolutions of 0.001 cm^{-1} (30 MHz) can be obtained. Martin has used this technique for retrievals of O_3 , HNO_3 and ClONO_2 [10]. This technique precludes point monitoring.

LIDAR utilise two IR laser wavelengths, one where the molecule absorbs and the other where there is very little absorption. Both are back-scattered, but the signal with no absorption will be stronger providing a differential spectrum. This technique has been

used to measure a wide variety of gases, including NO, NO₂, O₃ and CO₂. Sensitivity with this technique is limited by pressure broadening.

Laser Induced Fluorescence (LIF) measures the fluorescence from molecules that have been electronically excited by laser radiation. This technique is one of the most sensitive, but is not widely applicable because a different excitation scheme is needed for each species and many polyatomic molecules photodissociate rather than fluoresce.

Optoacoustic techniques have only recently been investigated. Modulated light absorption in a resonant cell results in the formation of pressure waves which are detected with a microphone. An advantage of this technique is that the sensitivity is not dependent on the absorption pathlength, meaning that instrumentation can be kept compact. Good sensitivities can be obtained but high laser powers are needed. This technique is one of the most promising for future development.

Analytical techniques such as Mass Spectroscopy (MS), Gas Chromatography and Titration can provide exceptional sensitivity and accuracy, but are not suitable for *in situ* real time measurements.

Tunable Diode Laser Absorption Spectroscopy (TDLAS) measures the absorption resulting from the interaction of light with molecules. A semiconductor laser, usually operating in the infrared, is used as a source. A variety of detection techniques exist to allow the absorption to be detected. Of all the techniques discussed so far, TDLAS has been shown to provide some of the best sensitivities. Consequently, it has become one of the most widely used techniques for trace gas and pollutant detection.

At the outset of this research virtually all TDLAS operated in the mid-infrared. Instruments such as the Airborne Laser Infrared Absorption Spectrometer (ALIAS) [11] used lead salt lasers operating in the 3-10 μm region to record spectra of N₂O, CH₄, NO₂ and HNO₃. Absorptions as small as 1×10^{-5} could be detected giving detection limits in the parts per trillion (ppt - 1 part in 10^{12}) to parts per billion (ppb - 1 part in 10^9) for the species mentioned.

The mid-infrared has conventionally been chosen because the strong fundamental transitions of most molecular species are found in this region. However, the mid-infrared lead salt lasers used in these spectrometers have low power outputs, suffer from poor mode and beam quality and require cryogenic cooling. This limits the

sensitivity that mid-infrared instruments can achieve. It also prevents long term unattended operation and inhibits their use on aircraft.

In the near-infrared, where the first or second overtone bands are found, the transitions are usually two to three orders of magnitude weaker than the fundamentals. In 1992, Cooper and Martineli [12] suggested that the latest advances in near-infrared lasers (high bandwidths, excellent mode purity and high output powers), developed for the communication bands at 1.3 and 1.5 μm , could compensate for the weaker linestrengths associated with overtone transitions and provide sensitivities close or equivalent to those obtained with mid-infrared lasers. The main advantage, however, would arise from the fact that these lasers and the accompanying detectors would operate at room temperature. This would significantly reduce the weight, size and cost of the instrumentation.

The main aim of this study has been to design, construct and assess the performance of a ground based near-infrared tunable diode laser absorption spectrometer. This spectrometer would utilise the latest advances in near-infrared technology to provide sensitivities equivalent to mid-infrared instrumentation.

The remainder of this thesis has been split up into six chapters, organised as follows:

Chapter 2 contains the background theory relating to infrared spectroscopy, the semiconductor laser and the physics necessary for the interpretation of atmospheric spectra.

Chapter 3 describes work carried out in the laboratory aimed at measuring spectroscopic parameters (linestrengths, line positions, broadening coefficients etc.) of gas species of interest to this study (CH_4 , N_2O , NH_3). The operating characteristics of the lasers used are also described.

Chapter 4 contains a detailed description and analysis of the multipass Herriott cell used in the instrument to provide pathlength and sensitivity enhancement. Design and testing of an astigmatic Herriott cell, capable of providing a 100m pathlength in a 2 litre mode volume is described.

Chapter 5 covers the design of the near-infrared tunable diode laser absorption spectrometer capable of detecting methane and nitrous oxide. A theoretical sensitivity

analysis of different detection schemes has been performed so as to assess which is best suited to this particular application, with an emphasis on high sensitivity, practicality and cost effectiveness. The optical and electronic layout of the instrument is described and a study into optical fringe reduction techniques is discussed.

Chapter 6 details the steps taken to calibrate and assess the performance of the instrument. A field test, measuring methane at a landfill site is described and the results of an intercomparison with other methane monitors are given. An overall error budget for methane and nitrous oxide measurements has been determined.

Chapter 7 covers the design, construction and operation of a coolable Herriott cell, capable of operating between 77 and 298 K. This cell has important applications in simplifying congested spectra and allowing the temperature dependence of molecular line parameters to be determined.

Chapter 2-

Background Theory

2.1 Spectroscopic Theory

2.1.1 Classification of Molecules

For the purpose of spectroscopic study, molecules are classified according to their principal moment of inertia. The moment of inertia, I , of any molecule about any axis through the centre of gravity is given by [13]:

$$I = \sum_i m_i r_i^2 \quad (2.1)$$

where m_i and r_i are the mass and distance of atom i from the axis. Convention has the axis with the maximum moment of inertia labelled the c axis, and the axis with the minimum moment of inertia, the a axis. A third axis, perpendicular to the other two, is called the b axis. According to convention:

$$I_c \geq I_b \geq I_a \quad (2.2)$$

Molecules can be classified into four distinct groups by their moment of inertia. Linear, Spherical Top, Symmetric Top and Asymmetric Top. The last two classes can be further differentiated into oblate and prolate symmetric tops and near oblate and near prolate asymmetric tops. Table 2.1 lists the different classes, the relationship of their principle moments of inertia, and a typical molecular example.

Moment of Inertia	Class	Example
$I_c = I_b > I_a = 0$	Linear	N_2O
$I_c = I_b = I_a$	Spherical Top	CH_4
	Symmetric Top	
$I_c > I_b = I_a$	- Oblate	$CHCl_3$
$I_c = I_b > I_a$	- Prolate	CH_3Cl
	Asymmetric Top	
$I_c > I_b \approx I_a$	- Near Oblate	$HONO_2$
$I_c \approx I_b > I_a$	- Near Prolate	H_2O

Table 2.1 - Classification of Molecules

2.1.2 The Harmonic Oscillator-Rigid Rotor Model

A molecule in space will possess vibrational, rotational and electronic motion. The combined energy of the molecule will be quantized into discrete states which can be determined by solving the Schrödinger equation:

$$H\Psi = E\Psi \quad (2.3)$$

The complexity involved in solving this directly for a system of interacting nuclei and electrons, however, precludes this approach from being used in all but the simplest of molecules.

The Born-Oppenheimer approximation assumes that the rotational, vibrational and electronic motions can be treated as if they are independent. As such, the combined energy of the molecule will simply be the sum of the energies associated with these motions:

$$E = E_e + E_v + E_r \quad (2.4)$$

This allows the Hamiltonian and wavefunction to be resolved into corresponding components, i.e.:

$$H = H_e + H_v + H_r \quad (2.5)$$

$$\Psi = \psi_e \psi_v \psi_r$$

Solutions for the energy levels of the independent motions are much more easily obtained.

Molecular spectroscopy may be defined as the study of the interaction of electromagnetic waves with matter. Absorption or emission of electromagnetic radiation occurs when there is a transition between two different states of either the rotational, vibrational or electronic energy levels. In the infrared, where this study is concerned, the electromagnetic radiation does not have the required energy to bring

about electronic transitions and spectra obtained in this region are a result of rotational and vibrational transitions only.

A simplified analysis of the rotational and vibrational motions of a diatomic molecule can go along way to explaining the form the absorption spectrum takes. Such an analysis is given below.

Vibration - Harmonic Oscillator Approximation

The vibration of a diatomic molecule may be approximated by assuming that the compression and extension of the bond between the two atoms can be likened to the behaviour of a spring. In this case the bond will obey Hooke's law:

$$f = -k(r - r_{eq}) \quad (2.6)$$

where f is the restoring force, k the force constant and r the internuclear distance. The energy curve resulting will be parabolic and will have the form:

$$E = \frac{1}{2}k(r - r_{eq})^2 \quad (2.7)$$

This model for the vibrating diatomic molecule is called the simple harmonic oscillator. Continuing the spring analogy, the bond will exhibit a particular oscillation frequency dependent upon the mass of the system and the force constant. Classically this oscillation frequency will be:

$$\bar{\omega}_{osc} = \frac{1}{2\pi c} \sqrt{\frac{k}{\mu}} \quad (2.8)$$

where μ is the reduced mass.

Solving the Schrödinger equation for the simple harmonic oscillator, with potential energy given by (2.7), gives the allowed vibrational energies. They can be shown to be [14]:

$$\epsilon_v = \left(v + \frac{1}{2} \right) \bar{\omega}_{osc} \quad (2.9)$$

where v is the vibrational quantum number. Equation (2.9) shows that vibrational energy levels are equally spaced for a harmonic oscillator.

It can be shown that the selection rule for the harmonic oscillator is:

$$\Delta v = \pm 1 \quad (2.10)$$

The selection rule states that transitions can only occur between neighbouring states. This would give rise to a series of superimposed transitions at a frequency given by $\bar{\omega}_{osc}$.

Later we will see that real molecules do not obey exactly the simple laws of harmonic motion.

Rotation - Rigid Rotor Approximation

A rigid rotor is a useful model for the end over end rotation of a diatomic molecule. The bond joining the nuclei is regarded as a rigid, weightless rod. The moment of inertia about the centre of gravity can be shown to be [13]:

$$I = \frac{m_1 m_2}{m_1 + m_2} r_o^2 = \mu r_o^2 \quad (2.11)$$

where m_1 and m_2 are the masses of the two nuclei, r_o is the bond length and μ , as before, is the reduced mass. The Schrödinger equation can be used to determine the allowed rotational energy levels for the rigid rotor approximation. They can be shown to be [14]:

$$\epsilon_J = \frac{h}{8\pi^2 I c} J(J+1) \quad (2.12)$$

J is the rotational quantum number and can take any integer value from zero upwards.

Equation (2.12) is usually given as:

$$\epsilon_J = BJ(J + 1) \quad (2.13)$$

where B, the rotational constant, is:

$$B = \frac{h}{8\pi^2 I c} \quad (2.14)$$

The separation of the energy levels increases with increasing J.

Selection rules for rotational transitions can be shown to be:

$$\Delta J = \pm 1 \quad (2.15)$$

The rigid rotor model predicts that transitions will occur at frequencies given by 2B, 4B, 6B, 8B, ..., cm^{-1} . In other words a series of equally spaced lines with separation 2B.

Rovibrational Transitions

So far, rotational and vibrational transitions have been treated separately. However, lines resulting from both vibrational and rotational transitions are allowed. Using the Born-Oppenheimer approximation, the energy of the combined transition, is just the sum of the energies associated with the individual transitions, i.e.:

$$\epsilon_{\text{Tot}} = \epsilon_v + \epsilon_r \quad (2.16)$$

Selection rules for rovibrational transitions are the same as for individual transitions, i.e.:

$$\Delta J = \pm 1, \Delta v = \pm 1 \quad (2.17)$$

Rovibrational transitions would give rise to a spectrum, centred at a frequency given by $\bar{\omega}_{\text{osc}}$, with equally spaced lines on either side, with separation 2B. The centre frequency is usually referred to as the band centre. Transitions with $\Delta J = + 1$ are

known as R branch transitions while those with $\Delta J = -1$ are referred to as P branch transitions.

2.1.3 Anharmonicity and Centrifugal Distortion

Anharmonicity

Real molecules do not obey the laws of simple harmonic motion. For example, if the molecule is stretched, there will come a point where it will eventually break, and the molecule will dissociate into atoms. An empirical expression which fits the potential energy curve associated with real vibrating molecules was derived by Morse. If this expression is used in the Schrödinger equation, the pattern of allowed vibrational energy levels becomes [14]:

$$\epsilon_v = \left(v + \frac{1}{2}\right)\bar{\omega}_e + \left(v + \frac{1}{2}\right)^2 \bar{\omega}_e \chi_e \quad (2.18)$$

where $\bar{\omega}_e$ is the oscillation frequency and χ_e is the anharmonicity constant which will always be positive. Instead of the vibrational levels being equally spaced, as was the case with the harmonic oscillator, the separation decreases with increasing v . This anharmonicity also leads to changes in the selection rules. They can be shown to be:

$$\Delta v = \pm 1, \pm 2, \pm 3, \dots \quad (2.19)$$

The transitions associated with $\Delta v = \pm 2, \pm 3$ are known as the first and second overtones. Each additional overtone transition will have rapidly diminishing probability. As a rule of thumb, for each additional vibrational quanta, the intensity of the lines will be ~ 100 times weaker. The first and second overtones will to a first approximation be at $2\bar{\omega}_e$ and $3\bar{\omega}_e$.

At room temperature, virtually all molecules will be in the ground state, because $h\bar{\omega}_e \geq kT$. If the molecule is very anharmonic or has a very low vibrational frequency, then some molecules will be in the $v = 1$ state, giving rise to the possibility of *hot band*

transitions, i.e. $\nu = 1 \rightarrow \nu = 2$. The low population of the $\nu = 1$ state means that such transitions will be extremely weak.

Centrifugal Distortion

Real atomic bonds are not rigid, and as the rotational energy increases (high J), centrifugal distortion will lead to an increasing bond length. From the expression for the moment of inertia in (2.11), it can be seen that the increased bond length will lead to an increased inertia and ultimately in a reduced rotational constant, B . Thus the rotational transitions will no longer have equal spacing, but will gradually start to converge as the rotational energy is increased. Solving the Schrödinger equation for a non rigid rotor gives the allowed rotational energies:

$$\epsilon_J = BJ(J + 1) - DJ^2(J + 1)^2 \quad (2.20)$$

D is known as the centrifugal distortion constant, and is given by:

$$D = \frac{h^3}{32\pi^4 I^2 r^2 k c} \quad (2.21)$$

This implies a harmonic force field. If this is not the case, then the rotational energies are given by:

$$\epsilon_J = BJ(J + 1) - DJ^2(J + 1)^2 + HJ^3(J + 1)^3 + KJ^4(J + 1)^4 \quad (2.22)$$

where H and K are small constants depending on the molecular geometry.

The selection rule for the non rigid rotor are the same as the rigid rotor.

2.1.4 Polyatomic Molecules

So far only an analysis of the rotational and vibrational motions of a diatomic molecule has been considered. The effect of vibration and rotation on polyatomic molecules will now be discussed.

Vibration

A molecule consisting of N atoms will have $3N$ degrees of freedom. Specifying the translational movement of the molecule requires three of these $3N$ degrees of freedom and another three are used to specify the three perpendicular components of the rotation. The only other type of motion allowed is internal vibration. Therefore, $3N-6$ modes of vibration are possible in a N atom non linear molecule. If the molecule is linear, only two degrees of freedom are required to specify the rotation, giving $3N-5$ modes of vibration. Thus, while a diatomic molecule only had one fundamental oscillation frequency, $\bar{\omega}_e$, polyatomic molecules will have $3N-6$ or $3N-5$.

Vibrational modes are further distinguished by the change in the electric dipole they produce. If the change in the electric dipole is along the symmetry axis, the vibration is referred to as being parallel, while if it is at right angles to the symmetry axis it is known as a perpendicular transition

Polyatomic selection rules are also changed, with combination bands and difference bands now permitted i.e. $\nu_1 + \nu_2$, $2\nu_1 + \nu_3$, $\nu_1 - \nu_2$. These bands will normally be very weak, but can significantly complicate spectra.

Rotation

The discussion on diatomic molecules can equally be applied to polyatomic linear molecules, with the rotational energy levels given by the expression in (2.20). Polyatomic linear molecules will generally have greater moments of inertia than diatomic's leading to spectral lines that are more closely spaced.

Symmetric top molecule's rotational energy levels become more complicated. There are now two directions of rotation and consequently two quantum numbers are used to describe the degree of rotation. J , is chosen to represent the total angular momentum and K is used to represent the rotation about the top axis. The energy levels for the symmetric top can be shown to be:

$$\epsilon_{J,K} = BJ(J+1) + (A-B)K^2 - D_J J^2(J+1)^2 - D_{JK} J(J+1)K^2 - D_K K^4 \quad (2.23)$$

A and B are the rotational constants for the two moments of inertia, i.e.:

$$A = \frac{h}{8\pi^2 I_a c}, B = \frac{h}{8\pi^2 I_b c} \quad (2.24)$$

J, as before, can take any positive integer value while K can take values:

$$K = J, J-1, J-2, \dots, 0, \dots, -(J-1), -J \quad (2.25)$$

The selection rules for this molecule may be shown to be:

$$\Delta J = \pm 1, \Delta K = 0 \quad (2.26)$$

The spectrum will be similar to that obtained for a linear molecule except that, for each rotational transition, J+1 additional lines will be associated with K.

Rovibrational Transitions - Selection rules

For rovibrational transitions, the selection rules are found to depend on whether the vibrational transition is parallel or perpendicular. The selection rules are summarised below:

Linear Molecule - Parallel

$$\Delta J = \pm 1, \Delta v = \pm 1, (\pm 2, \pm 3, \dots), \Delta l = 0 \quad (2.27)$$

Linear Molecule - Perpendicular

$$\Delta J = 0, \pm 1, \Delta v = \pm 1, (\pm 2, \pm 3, \dots), \Delta l = \pm 1 \quad (2.28)$$

For the first time, selection rules now say that a vibrational transition can take place without a simultaneous rotational transition. This gives rise to a set of lines between the P and R branches. These lines are commonly referred to as Q branch transitions. In addition, the orbital angular momentum quantum number, l, has been included. A

component of the orbital angular momentum along the internuclear axis allows $\Delta l = \pm 1$ for perpendicular transitions.

Symmetric Top - Parallel

$$\Delta v = \pm 1, \Delta J = 0, \pm 1, \Delta K = 0 \quad (2.29)$$

Symmetric Top - Perpendicular

$$\Delta v = \pm 1, \Delta J = 0, \pm 1, \Delta K = \pm 1 \quad (2.30)$$

Again, for perpendicular vibration, Q branch transitions will be allowed.

For more complicated molecular shapes the complexity of the spectrum increases. This is outwith the scope of work covered in this thesis but an analysis can be found in Herzberg's book [15].

2.1.5 Thermal Populations

It will be shown later that one of the main factors governing the strength of a transition is the population of the energy level from which the excitation is occurring. The fractional population of the mth level is given by the Boltzmann distribution [16]:

$$F_m = \frac{N_m}{\sum_i N_i} = \frac{g_m \exp(-E_m/kT)}{\sum_i g_i \exp(-E_i/kT)} \quad (2.31)$$

where g_m is known as the statistical weight of the state and E_m is the energy of the state. The statistical weight will comprise of the degeneracies associated with vibration, rotation and nuclear spin. The denominator is known as the partition function, and is the sum of all the states accessible to the molecule at a temperature T. The partition sum, Q, will be the product of the internal partition functions associated with vibration and rotation. i.e.

$$Q = Q_v Q_r \quad (2.32)$$

The vibrational partition sum, Q_v , can be written as the product of the $3N-6$ normal modes of vibration for a N atom molecule:

$$Q_v = Q_v(1)Q_v(2)\dots Q_v(3n-6) \quad (2.33)$$

where for each mode:

$$Q_v(j) = \frac{1}{[1 - \exp(-hc\nu_j/kT)]} \quad (2.34)$$

The fractional population of a given level will be given by:

$$F_v = \frac{g_v}{Q_v} \exp\left(-\frac{(E_v - E_0)}{kT}\right) \quad (2.35)$$

The rotational partition function, Q_r , will be the sum of all the rotational levels. It can be shown to be:

$$Q_r = \sum_J (2J + 1) \exp(-E_J/kT) \quad (2.36)$$

The $(2J + 1)$ term represents the statistical weight of each rotational level, and arises from the $(2J+1)$ values that M_J can take.

2.1.6 Spin Degeneracy

The discussion so far about the thermal population of a given level has not considered the effect of nuclear spin degeneracy. Spin degeneracy does not affect the vibrational population levels, but can be an important factor in the population of rotational levels.

Particles with spin of half integer value (Fermi particles), that exchange position with an identical particle equidistant from the centre of the molecule, will cause the overall sign of the wavefunction ($\Psi = \psi_e \psi_v \psi_r \psi_{ns}$) to change (a consequence of the Pauli Exclusion principle). In such a case it is said that the overall functions are antisymmetrical to nuclear exchange. If the particle has integer spin (Bose particles), the exchange will not lead to a change in sign of the overall wavefunction and the function is said to be symmetrical to nuclear exchange. Determining all the possible spin combinations allows the number of symmetric and antisymmetric nuclear spin wavefunctions for a given molecule to be determined. Taking the example of acetylene, it is found that the ratio of symmetric to antisymmetric functions is three to one. In order to preserve the antisymmetry of the overall wavefunction, it is found that the symmetrical nuclear spin functions can only exist for even J and that the antisymmetrical nuclear spin functions can only exist for odd J. This leads to the populations of the odd J energy levels being three times greater than the even J energy levels. Consequently, rotational line intensities of acetylene exhibit a characteristic strong - weak - strong - weak signature. Calculating the number of symmetrical and antisymmetrical wavefunctions becomes increasingly complicated as the symmetry of the molecule becomes lower.

2.1.7 Absorption Coefficients and Line Intensities

The Beer-Lambert Law describes the homogeneous attenuation of radiation travelling through a homogeneous absorbing medium. It takes the form:

$$I(\nu, x) = I_0 \exp(-\sigma(\nu)x) \quad (2.37)$$

where $I(\nu, x)$ is the emergent intensity, I_0 is the incident intensity, x is the concentration pathlength product and $\sigma(\nu)$ is the absolute absorption coefficient. x represents the number of absorbing molecules per unit area and will have units of $\text{molecules}\cdot\text{cm}^{-2}$. The absolute absorption coefficient will be given in units of $\text{cm}^2\cdot\text{molecule}^{-1}$. For the absorption of monochromatic light by one molecular gas in the atmosphere, the Beer-Lambert law becomes:

$$I(\nu, l) = I_0 \exp(-\alpha(\nu)P_a l) \quad (2.38)$$

where $\alpha(\nu)$ is the linear absorption coefficient defined in $\text{cm}^{-1} \text{atmospheres}^{-1}$, P_a is the partial pressure (atm.) of the gas in the atmosphere and l is the optical pathlength in cm.

The absorption coefficient can be related to the molecular line intensity, S , by:

$$\alpha(\nu) = Sg(\nu - \nu_0) * N \quad (2.39)$$

where $g(\nu - \nu_0)$ is the normalized lineshape function (units of cm) and ν_0 is the frequency at line centre. The molecular line intensity, S , is given in units of cm molecule^{-1} . N is the total number of absorbing molecules per cm^3 per atmosphere. The value of N at 296 K is known as Loschmidt's number ($N_L = 2.479 \times 10^{19} \text{ molecules cm}^{-3} \text{ atm}^{-1}$).

The line intensity can also be related to the Einstein coefficient for stimulated absorption, B_{ji} , between levels i and j by:

$$S = \frac{h\bar{\nu}_{ji}}{c} B_{ji} (N_i - N_j) \quad (2.40)$$

where $\bar{\nu}_{ji}$ is in Hz and N_i and N_j are the population of the two levels under consideration. Following Duxbury [17] and substituting the expressions for the Einstein coefficients it can be shown that the line intensity (cm molecule^{-1}) is given by:

$$S = \frac{CN\nu_{ji}}{N_0 Q_v Q_r} \exp\left(\frac{-E_i}{kT}\right) \left[1 - \exp\left(\frac{-h\nu_{ji}}{kT}\right) \right] \left| \langle i | \mu_F | j \rangle \right|^2 \quad (2.41)$$

where $C = 8\pi^3/3hc$, N is the number of absorbing gas molecules per cm^3 per atmosphere, Q_v and Q_r are the vibrational and rotational partition functions

and $\left| \langle i | \mu_F | j \rangle \right|^2$ is the square of the dipole transition moment with units Coulomb per cm ($\text{C}\cdot\text{cm}^{-1}$). μ_F is the electric dipole moment operator and is given by [13]:

$$\mu_F = \sum_i q_i \mathbf{r}_i \quad (2.42)$$

where q_i and \mathbf{r}_i are the charge and position vector of the i th particle. The transition moment can be thought of as an oscillating electric dipole moment due to the transition. The square of the transition moment gives the transition probability.

2.1.8 Lineshapes

So far, nothing has been said about the form of the lineshape function, $g(\nu - \nu_0)$. For atmospheric measurements, the lineshapes fall into three regimes, namely Doppler broadened, Lorentzian pressure broadened and an intermediate case, Voigt.

All radiative transitions have a characteristic lifetime, Δt . As a consequence of Heisenberg's uncertainty principle, there will be an uncertainty in the energy of the transition, i.e.:

$$\Delta E \Delta t \geq \hbar \quad (2.43)$$

This leads to all transitions having a characteristic natural linewidth (HWHM), that can be shown to be [13]:

$$\Delta \nu \geq \frac{32\pi^3 \nu^3}{(4\pi\epsilon_0)3hc^3} \left| \langle i | \mu_F | j \rangle \right|^2 \quad (2.44)$$

When collisions occur between atoms or molecules there is an exchange of energy which leads to a broadening of the energy levels. The more collisions that occur the greater the broadening effect. This broadening mechanism dominates at high pressure. For this reason, this broadening mechanism is usually referred to as pressure

broadening. It has a characteristic Lorentzian lineshape given by the following expression:

$$g_L(\nu - \nu_0) = \frac{1/\gamma_L}{[(\nu - \nu_0)^2 + \gamma_L^2]} \quad (2.45)$$

where γ_L the Lorentzian HWHM, is given by:

$$\gamma_L = [\gamma_a P_a + \gamma_b P_b] \left(\frac{T_0}{T} \right)^{\frac{1}{2}} \quad (2.46)$$

where γ_a and γ_b are the self and foreign gas broadening coefficients and P_a and P_b are partial pressures of the respective gases.

At low sample pressures Doppler broadening is found to dominate. Doppler broadening arises from the motion of absorbing or emitting molecules relative to the source or the detector. It can be shown that the characteristic lineshape resulting from Doppler broadening takes the form:

$$g_D(\nu - \nu_0) = \frac{1}{\gamma_D} \left(\frac{\ln(2)}{\pi} \right)^{\frac{1}{2}} \exp\left(\frac{-(\nu - \nu_0)^2 \cdot \ln(2)}{\gamma_D} \right) \quad (2.47)$$

where the Doppler width, γ_D , is defined as

$$\gamma_D = \frac{\nu_0}{c} \left(\frac{2kT \cdot \ln(2)}{m} \right)^{\frac{1}{2}} \quad (2.48)$$

At intermediate pressures, the lineshape is a convolution of both the Lorentzian and Gaussian lineshapes. It is known as the Voigt lineshape and can be expressed as:

$$g_v(v - v_o) = \left(\frac{Bx}{\pi} \right) \int_{-\infty}^{+\infty} \left(\frac{\exp(-y^2)}{[x^2 + (z - y)^2]} \right) dy \quad (2.49)$$

where

$$\begin{aligned} B &= \left(\frac{1}{\gamma_D} \right) \left[\frac{\ln(2)}{\pi} \right]^{\frac{1}{2}} \\ x &= \left(\frac{\gamma_L}{\gamma_D} \right) \left[\ln(2) \right]^{\frac{1}{2}} \\ z &= \left[\frac{(v - v_o)}{\gamma_D} \right] \left[\ln(2) \right]^{\frac{1}{2}} \end{aligned} \quad (2.50)$$

There are many approximations to this integral. A commonly used one is that derived by Humlicek [18]. The width of the Voigt function can be determined empirically by the following expression:

$$\gamma_v = 0.536\gamma_L + \left(0.2166\gamma_L^2 + \gamma_D^2 \right)^{\frac{1}{2}} \quad (2.51)$$

2.2 Diode Laser Theory

2.2.1 Theory of Operation

At the heart of the semiconductor laser is the p-n junction. The p-n junction is formed from a single-crystal material in which one region is doped with acceptor impurity atoms to form the p-region and the adjacent region is doped with donor atoms to form the n-region. Majority carrier electrons in the n-region will begin diffusing into the p-region and majority carrier holes in the p-region will diffuse into the n-region. Eventually a steady state is reached, in such a way that further diffusion of holes and electrons is opposed by the electric field across the p-n junction, arising from the negatively charged acceptors on the p side and the positively charged donors on the n side. The Fermi level, defined as the energy at which the probability of occupation is exactly one-half, will be continuous across the junction.

If an external voltage is applied, the electric field across the junction will be reduced, allowing further diffusion of electrons and holes. A narrow depletion region forms where electrons and holes are present simultaneously. These electron-hole pairs can undergo several processes by which they can recombine either radiatively or non radiatively. Radiative recombination involves the emission of a photon with energy, $h\nu \cong E_g$, where E_g is the bandgap of the material. This photon can be reabsorbed, forming another electron hole pair. By suitable choice of doping material and bias voltage, a situation known as population inversion can occur. The rate of photon emission exceeds that of absorption and the junction is then said to exhibit optical gain. The wavelength of the stimulated emission in μm is [19]:

$$\lambda \cong \frac{1.24}{E_g} \tag{2.52}$$

A second requirement for lasing is that the emission is coherent. This is achieved by forming an optical cavity about the junction. Cleaving the crystal, so that the ends act

as two partially reflecting parallel mirrors, forms a Fabry-Perot resonant cavity. The cavity will cause a build-up of optical intensity from positive feedback. The resonant modes of the cavity can be shown to be:

$$N\left(\frac{\lambda}{2}\right) = \mu L \quad (2.53)$$

where N is an integer, μ is the refractive index of the material concerned, L is the cavity length, and λ is the wavelength of the emission. The broad gain curve, typical of a semiconductor laser, ensures that several longitudinal cavity modes can lase at one time. The laser output is referred to as being multimode. This basic p-n junction diode laser configuration is known as a homojunction laser.

2.2.2 Confinement

In the homojunction laser both the charge carriers and photons can diffuse out from the active region of the diode. This leads to poor internal and external quantum efficiencies, resulting in high threshold currents. To improve the quantum efficiency, these charge carriers and photons must be confined within the active region. This is achieved by sandwiching the active material between two passive layers of material that have wider bandgaps. This is known as a double heterostructure. The potential barriers formed at both junctions ensures that the charge carriers are confined. The passive layers also confine the photons via index guiding. The layers are of lower refractive index than the active region, resulting in total internal reflection occurring at the interface.

2.2.3 Single mode operation

For spectroscopic purposes it is advantageous if the laser output is single mode. Several techniques exist to enable single mode operation. Two of these techniques

incorporate a grating into the semiconductor structure. In the distributed feedback laser (DFB) the grating is adjacent to the active region where the current flows. The grating layer is etched with a first order grating of period Λ . This provides additional feedback by backscattering. Coherent coupling can only take place when the following condition is met:

$$\Lambda = \frac{m\lambda}{2\mu} \quad (2.54)$$

where Λ is the grating period, μ is the refractive index, λ is the lasing wavelength and m is the order of Bragg diffraction. Any cavity modes that do not meet this condition will destructively interfere ensuring single mode operation. A consequence of this additional feedback mechanism is a reduced linewidth. Typically linewidths of the order 10-50 MHz are achieved.

The second technique operates on the same principle, but the grating is in the passive part of the laser. These devices are known as distributed Bragg reflection (DBR) lasers. These lasers, however, are not as efficient as DFB devices.

A third technique used to achieve single mode operation is to use an external cavity. The front facet of the diode is anti reflection coated and an external grating and the back facet of the diode form the cavity surfaces. The grating selects one particular wavelength to feedback into the laser, ensuring that only one mode will lase. The higher Q factor, associated with the longer cavity length gives linewidths in the 100 kHz region. However, the external grating is alignment sensitive, making these devices less practical to operate.

2.2.4 Tuning Mechanism

The operating wavelength of the laser can be tuned by either changing the laser current or the diode temperature. In both cases, the tuning mechanism is thermal. Change in the junction temperature changes the bandgap of the material, and from equation (2.52), the wavelength of the diode. It is strictly more correct to say that current tuning

is dominated by the thermal mechanism, because the change in the current density also causes the wavelength to change. The current density tunes the laser in the opposite sense to thermal tuning. Thermal tuning changes the refractive index of the material and hence the cavity length.

The conflict in the tuning rates of the broad band gain curve of the laser and the longitudinal modes leads to *mode hops*. In DFB lasers the tuning rates are similar, giving continuous tuning over 3-5 nm.

2.2.5 Near and Mid-infrared Lasers - Comparison of Characteristics

In the mid-infrared, laser diodes are made from group II-group VI elements such as PbS, PbSe and PbTe. For this reason they are normally referred to as lead salt lasers. The bandgaps of these materials allow coverage between 2.5 - 20 μm . Lead salt materials have poor electrical and quantum efficiencies, in addition, growing these materials is difficult, which has resulted in homostructure devices being the dominant type of lead salt laser commercially produced. The poor efficiency, means that cryogenic operation ($\sim 30\text{K}$) and high threshold currents of 0.5 - 1 amp are required. This has limited the practicality of these devices in field instruments. Laser output is multimode, but at low currents is *dominant mode*. The lack of confinement in the homostructure device produces inferior near and far field beam quality. Typical power outputs of 100 - 500 μW are obtained. Recent developments in material growth techniques has allowed double heterostructure devices to be manufactured. The increased efficiency associated with confinement allows the lasers to be operated at liquid nitrogen temperatures (77K).

In the near-infrared, commercial production of lasers for the communications industry, has led to superior production techniques. The lasers are made of group III - group V elements such as GaAs. In practice the structure of commercial lasers is significantly more complicated. High quantum efficiency allows low threshold currents (20-30 mA), high output power (1-10 mW) and room temperature operation. Confinement and distributed feedback gives superior beam quality and single mode operation. A recent development has been the strained quantum well laser. In the quantum well laser, the

active layer is so thin ($<20\text{nm}$), that carriers are confined in quantum states. This improves efficiency and threshold currents as low as a 2-5 mA can be achieved.

The near-infrared lasers used for communication purposes are usually packaged with inbuilt thermoelectric coolers and optical isolators to further improve noise performance.

Chapter 3 -

Spectroscopic Measurements

3.1 Introduction

This chapter describes work undertaken in the laboratory aimed at measuring near-infrared spectroscopic parameters (line positions, line intensity, broadening coefficients etc.) of different gas species of interest, including methane (CH_4), nitrous oxide (N_2O), acetylene ($^{12}\text{C}_2\text{H}_2$, $^{13}\text{C}_2\text{H}_2$) and ammonia ($^{14}\text{NH}_3$, $^{15}\text{NH}_3$). Spectroscopic measurement was carried out using near-infrared distributed feed back (DFB) lasers and a multipass absorption Herriott cell (Chapter 4), allowing absorptions as small as $\sim 1 \times 10^{-4}$ to be observed. Before any measurements were taken, the operating characteristics of the lasers (wavelength coverage, side mode suppression ratio, power output etc.) were determined. This work is described first.

3.2 Laser Characteristics

Three lasers were used in this study. All were single mode distributed feedback devices operating in the near-infrared. The operating characteristics of each device was determined before being used for spectroscopic measurement.

3.2.1 Anritsu 1650 nm DFB (SD3B254F)

This device came in a butterfly package, had inbuilt thermoelectric coolers for temperature control, an optical isolator to prevent feedback and was fibre coupled (SMF 10/125 μm). An internal photodiode was provided to allow the power of the device to be monitored. The laser had a spectral halfwidth of ~ 10 MHz. The specification sheet for this device is given in Appendix 1. The device was bought to measure the $2\nu_3$ Q branch of CH_4 .

The wavelength coverage of this laser was measured with a Burleigh wavemeter (WA-1000, 0.001 cm^{-1} resolution). The temperature of the diode was incremented in 2.5°C steps between 2.5°C and 40°C . At each temperature, the wavelength of the device was recorded at laser currents of 40, 80 and 120 mA. **Figure 3.1** shows the wavelength of the laser against temperature for these different current values. It is seen that a linear relationship between the temperature of the device and the operating wavelength was found. Equations representing this linear relationship are given for each current setting. They are listed in **Table 3.1**. The tuning rate with temperature was $\sim -0.376\text{ cm}^{-1}/^\circ\text{C}$. The diode could be tuned between 6014 and 5999 cm^{-1} by varying the temperature between 2.5 and 40°C . At higher laser currents, the tuning rate with temperature was shown to increase.

Scanning the laser current between 40 and 120 mA typically changed the wavelength by -0.7 cm^{-1} . As the temperature of the laser was decreased, the tuning rate with current exhibited a slight increase. **Figure 3.2** shows how the laser wavelength changed with current at a temperature of $\sim 25^\circ\text{C}$. The wavelength was recorded at 10 mA intervals between 30 and 120 mA. It is seen that the wavelength does not have a linear dependence with current. The average tuning rate is $\sim -0.008\text{ cm}^{-1}/\text{mA}$. A polynomial fit to the observed behaviour was made. **Table 3.2** lists the polynomial coefficients

obtained from this fit. The lasing threshold occurred at ~26 mA. The threshold current went down as the temperature was reduced.

Laser power was recorded with an HP 8153A lightwave multimeter. Typical power levels of ~ 1mW were obtained at 100 mA (25 °C).

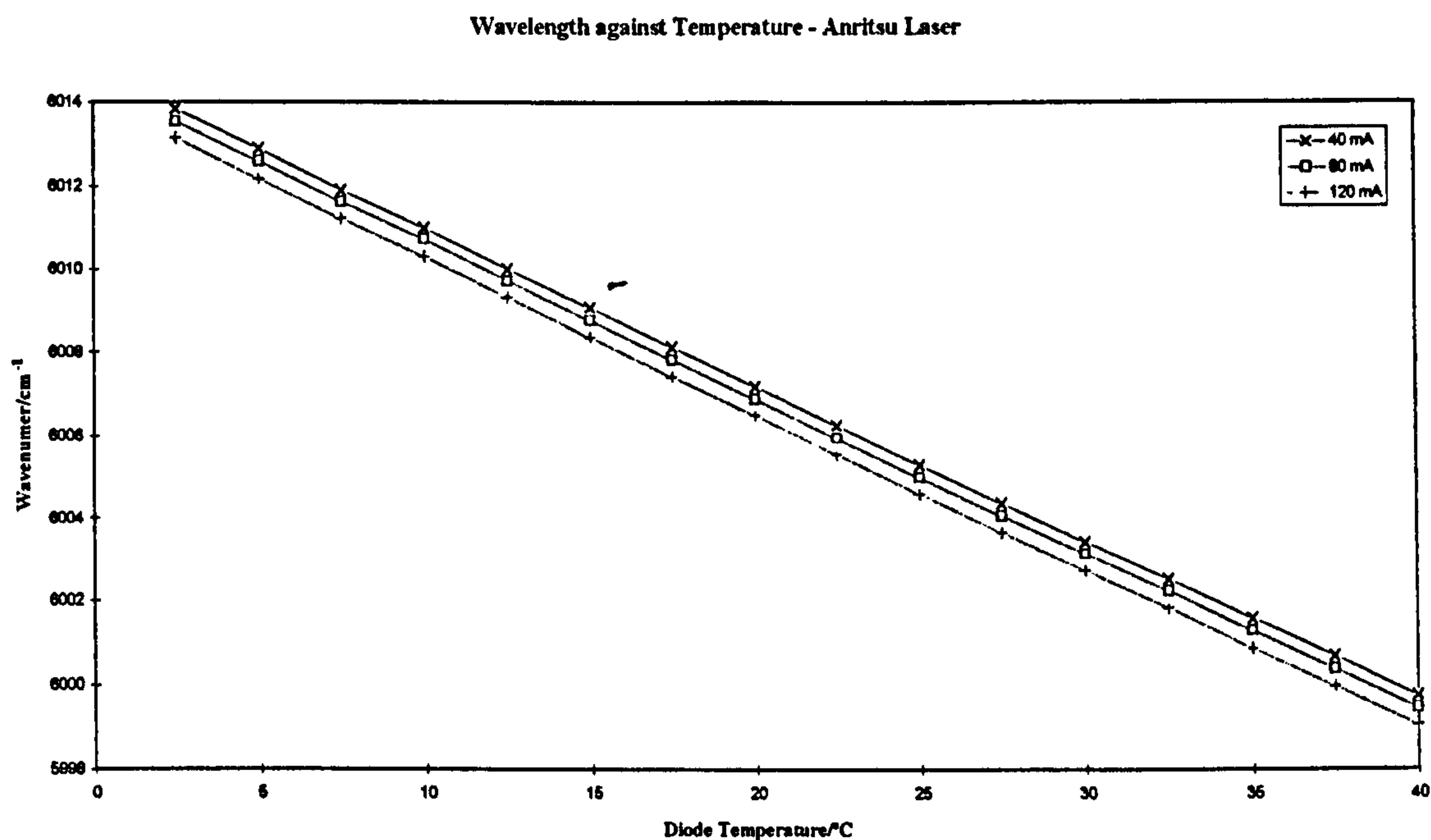


Figure 3.1 - Temperature Dependence of Laser Operating Frequency -Anritsu Laser

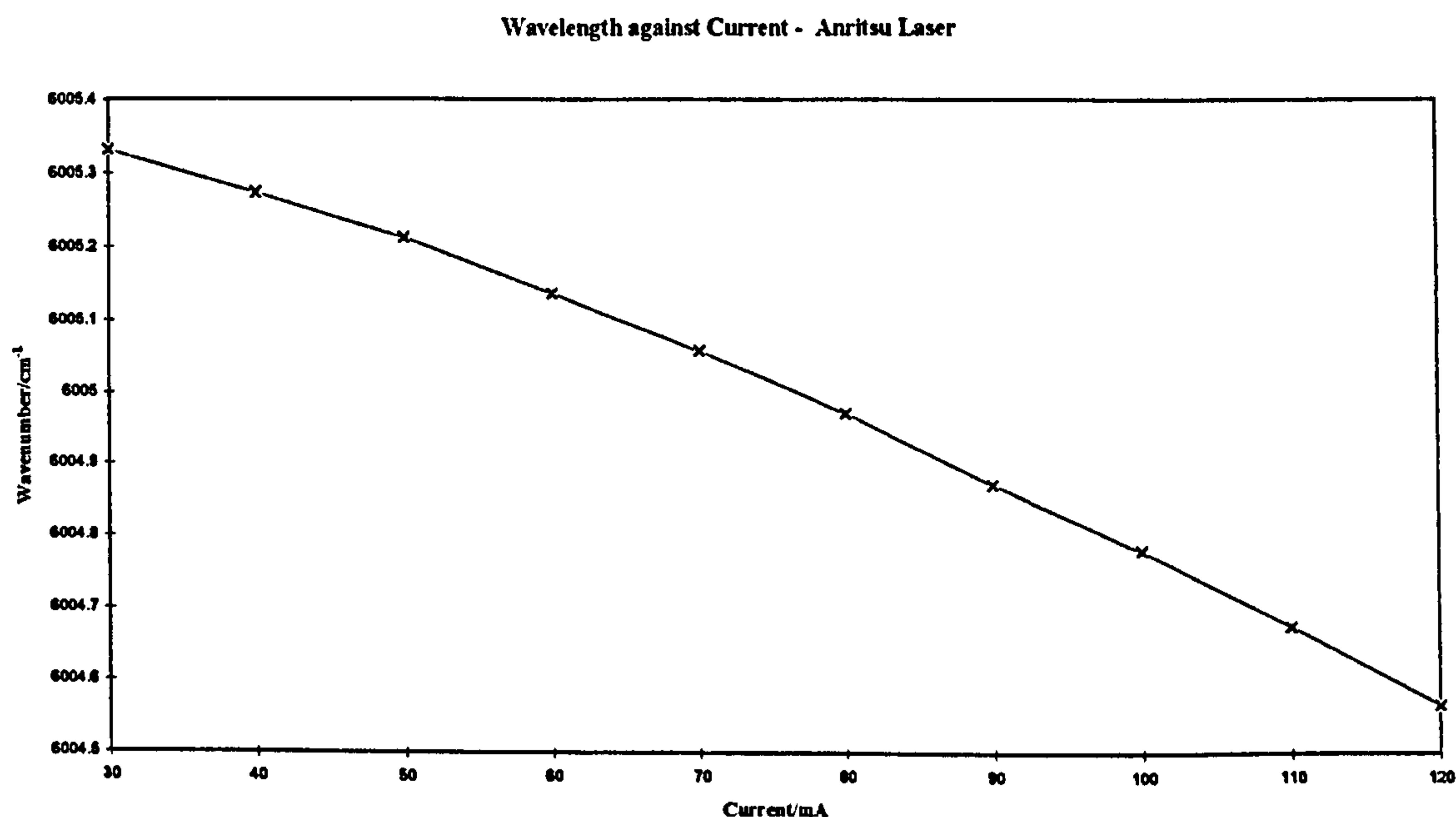


Figure 3.2 - Laser operating frequency against laser current - Anritsu Laser

<u>Current</u>	<u>Equation</u>
40 mA	$y (\text{cm}^{-1}) = -0.4036x (\text{°C}) + 6597.9$
80 mA	$y (\text{cm}^{-1}) = -0.4108x (\text{°C}) + 6596.4$
120 mA	$y (\text{cm}^{-1}) = -0.4165x (\text{°C}) + 6594.3$

Table 3.1 - Equations representing linear dependence of the laser operating frequency with temperature - Anritsu Laser

<u>Polynomial Coefficient</u>	<u>Value</u>
$y (\text{cm}^{-1}) = a_0X (\text{mA})^3 + a_1X (\text{mA})^2 + a_2X (\text{mA}) + a_3$	
a0	1.416×10^{-7}
a1	-6.353×10^{-5}
a2	-1.625×10^{-3}
a3	6005.43

Table 3.2 - Polynomial coefficients for laser frequency dependence with current (25 °C) - Anritsu Laser

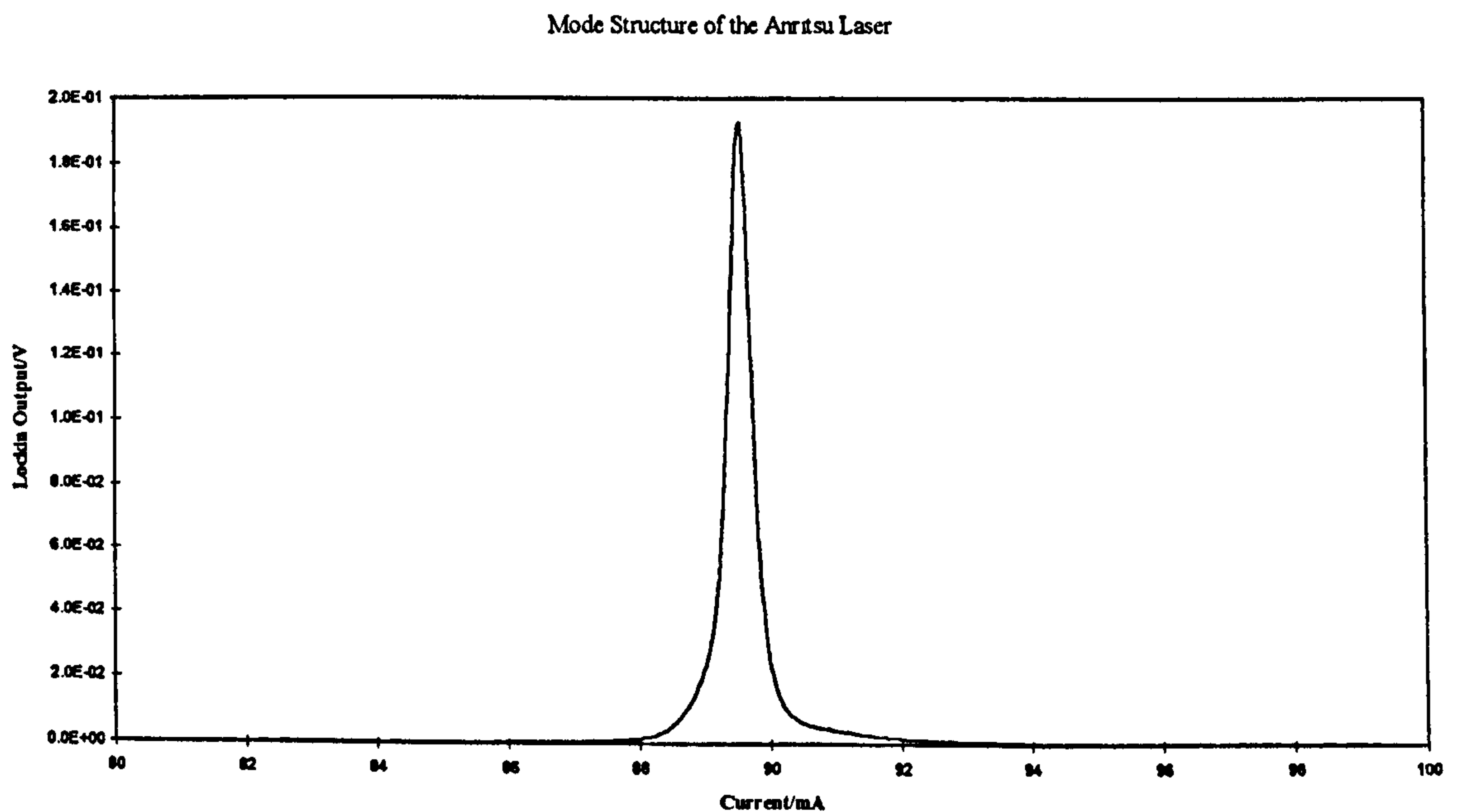


Figure 3.3 - Mode Structure of the Anritsu Laser

The mode structure of the diode was also investigated using a spherical mirror High Q Fabry-Perot scanning Interferometer (TecOptics FSR 7.5 GHz). **Figure 3.3** shows the mode structure. No side modes are apparent. The slight asymmetry is caused by misalignment of the laser beam going into the interferometer. By expanding the baseline it was possible to observe the side modes. They were approximately 40000 times weaker than the main mode. This would give a side mode suppression ratio of ~ 46 dB.

3.2.2 Thomson 1520 nm DFB (MKL 1200DFB XII/12)

The Thomson laser was packaged in a standard TO-18 can. No thermoelectric cooler or optical isolator was provided. The laser had a quoted spectral halfwidth of ~ 40 MHz. The specification sheet for this laser can be found in Appendix 1. This device was bought to measure the $3 \nu_3$ band of nitrous oxide at 1520 nm.

The wavelength coverage of this laser was measured with the Bruker 120M Fourier Transform Spectrometer at the National Physical Laboratory (NPL). The laser was passed through a ground glass scatter plate ensuring that the coherence of the beam was destroyed. A X20 beam expander produced a collimated beam ~ 10 cm in diameter which was directed into the Bruker side port.

Figure 3.4 shows the wavelength dependence with temperature. The temperature was incremented in 2.5 °C steps over the range 5 to 35 °C. At each temperature the wavelength was recorded at 40 , 80 and 120 mA. It is seen that a linear relationship between the temperature of the device and the operating wavelength was found. Equations representing this linear relationship are given for each current setting. They are listed in **Table 3.3**. The tuning rate with temperature was ~ -0.41 $\text{cm}^{-1}/\text{°C}$. The diode could be tuned between 6596 and 6578 cm^{-1} by varying the temperature between 5 and 35 °C. At higher laser currents the tuning rate with temperature increased.

Scanning the laser current between 40 and 120 mA typically changed the wavelength by -3.5 cm^{-1} . The tuning rate with current is significantly bigger than was observed with the Anritsu device. As the temperature of the laser was decreased the tuning rate with current exhibited a slight increase. **Figure 3.5** shows how the laser wavelength changed

with current at a temperature of ~ 25 °C. The wavelength was recorded at 2.5 mA intervals between 40 and 120 mA. It is seen that the wavelength does not have a linear dependence with current. The average tuning rate was ~ 0.044 cm^{-1}/mA . At higher currents this tuning rate increased. A polynomial fit to the observed behaviour was made. Table 3.4 gives the polynomial coefficients obtained from this fit. The laser threshold occurred at ~ 33 mA. The threshold current went down as the temperature was reduced.

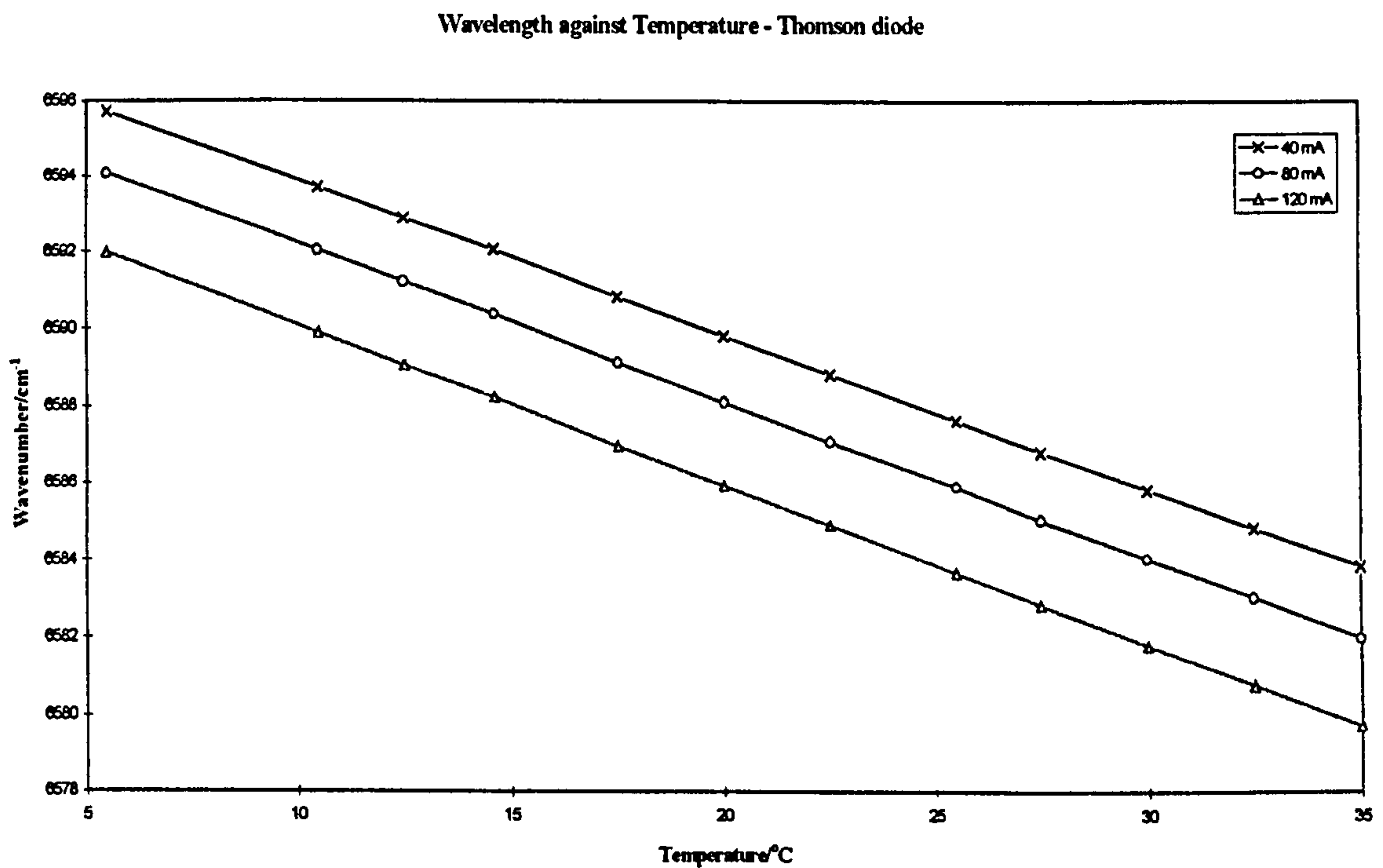


Figure 3.4 - Temperature Dependence of Laser Operating Frequency -Thomson Laser

<u>Current</u>	<u>Equation</u>
40 mA	$y (\text{cm}^{-1}) = -0.3759x (\text{°C}) + 6014.7$
80 mA	$y (\text{cm}^{-1}) = -0.3760x (\text{°C}) + 6014.4$
120 mA	$y (\text{cm}^{-1}) = -0.3762x (\text{°C}) + 6014.0$

Table 3.3 - Equations representing linear dependence of the laser operating frequency with temperature - Thomson Laser

Wavelength against Laser Current - Thomson Diode

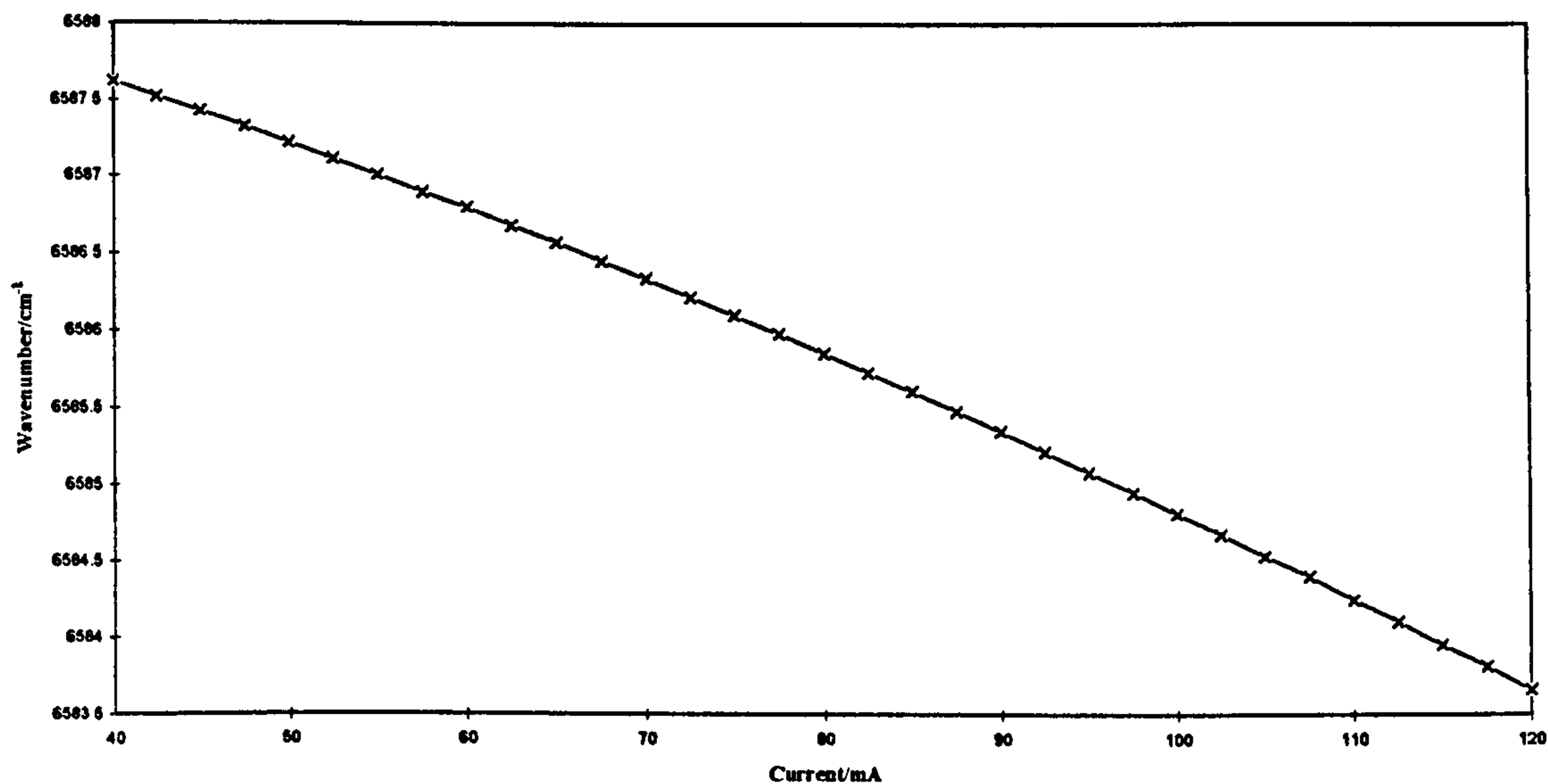


Figure 3.5 - Laser operating frequency against laser current - Thomson Laser

<u>Polynomial Coefficient</u>	<u>Value</u>
$y_{(cm-1)} = a_0X_{(mA)}^3 + a_1X_{(mA)}^2 + a_2X_{(mA)} + a_3$	
a0	8.477×10^{-8}
a1	-1.596×10^{-4}
a2	-2.569×10^{-2}
a3	6588.90

Table 3.4 - Polynomial coefficients for laser frequency dependence with current (25 °C) - Thomson Laser

The laser power was measured with a Ge detector (Hamamatsu B1720-02) At 100 mA, the typical power output was ~ 7 mW (25 °C).

The spherical mirror Fabry-Perot scanning Interferometer (TecOptics FSR 7.5 GHz) was again used to study the mode structure of the laser. Figure 3.6 shows the main

mode, with no side modes apparent. The side mode suppression ratio was determined to be ~ 37 dB.

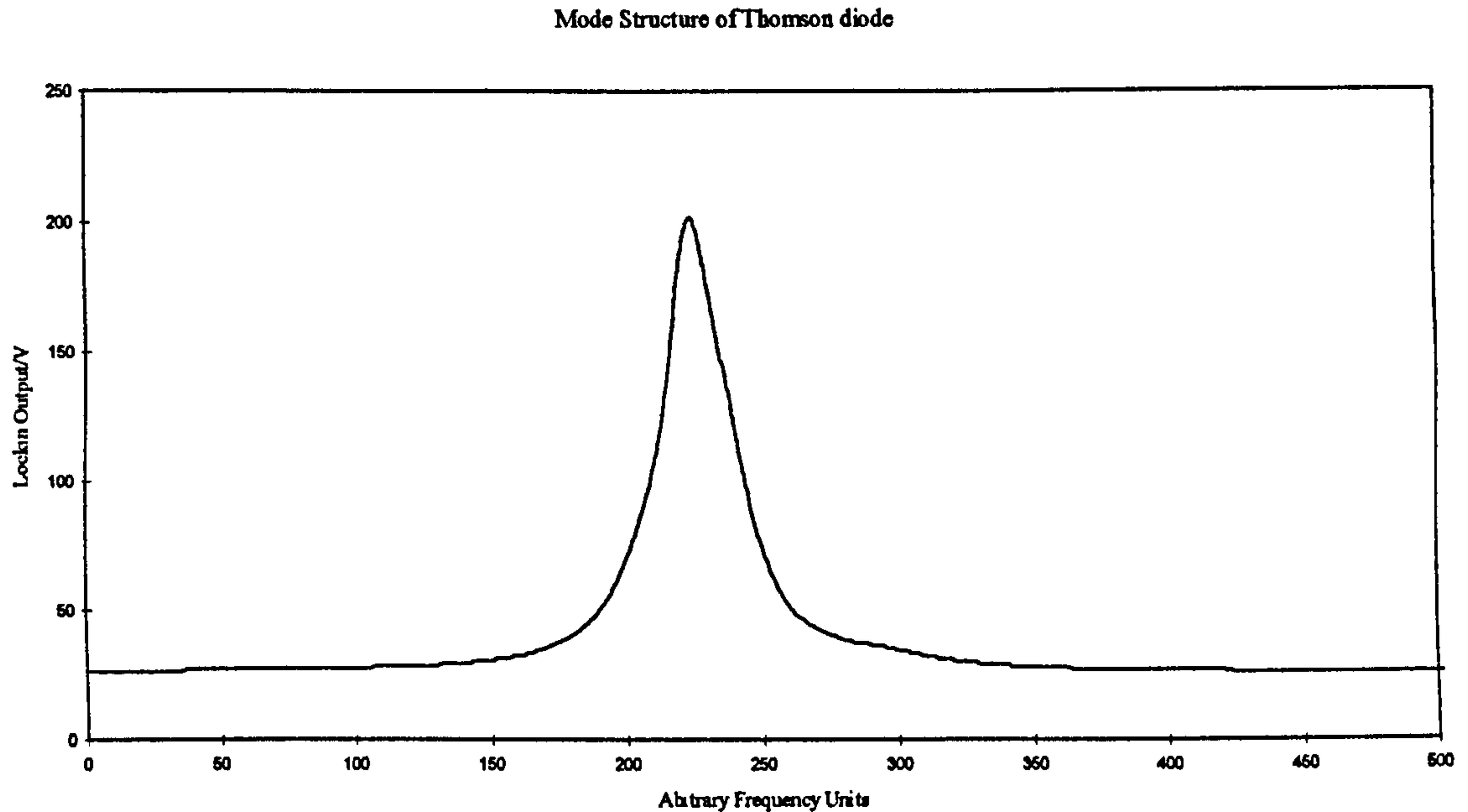


Figure 3.6 - Mode Structure of the Thomson Laser

3.2.1 NEC 1550 nm DFB (NDL5650)

This device was packaged in a standard can configuration. Like the Thomson diode, no optical isolator or inbuilt thermoelectric cooler were provided. The laser has a spectral halfwidth of ~ 50 MHz. This device operated at 1550 nm.

The tuning characteristics of this laser were determined by A Burley, a member of the group at the University of Strathclyde. Wavelength dependence with current and temperature were recorded using a BOMEM DA3.002 Fourier transform spectrometer. Details of this work can be found in his thesis *Near-infrared Spectroscopy of Molecules of Metrological Interest* [20]. Summarising: the wavelength tuning rate with temperature was ~ -0.367 $\text{cm}^{-1}/^{\circ}\text{C}$, changing the temperature between 5 and 35 $^{\circ}\text{C}$ resulted in the operating frequency of the laser changing from ~ 6447 to 6435 cm^{-1} . The wavelength dependence with current was non linear with an average tuning rate of ~ -0.033 cm^{-1}/mA . This tuning rate increased at higher currents.

The other characteristics were determined by myself. The power output of the laser was ~ 4.5 mW at 100 mA (25 °C) Threshold current was ~ 30 mA.

Figure 3.7 shows the mode structure of this laser. The side mode suppression ratio was determined to be ~ 33 dB.

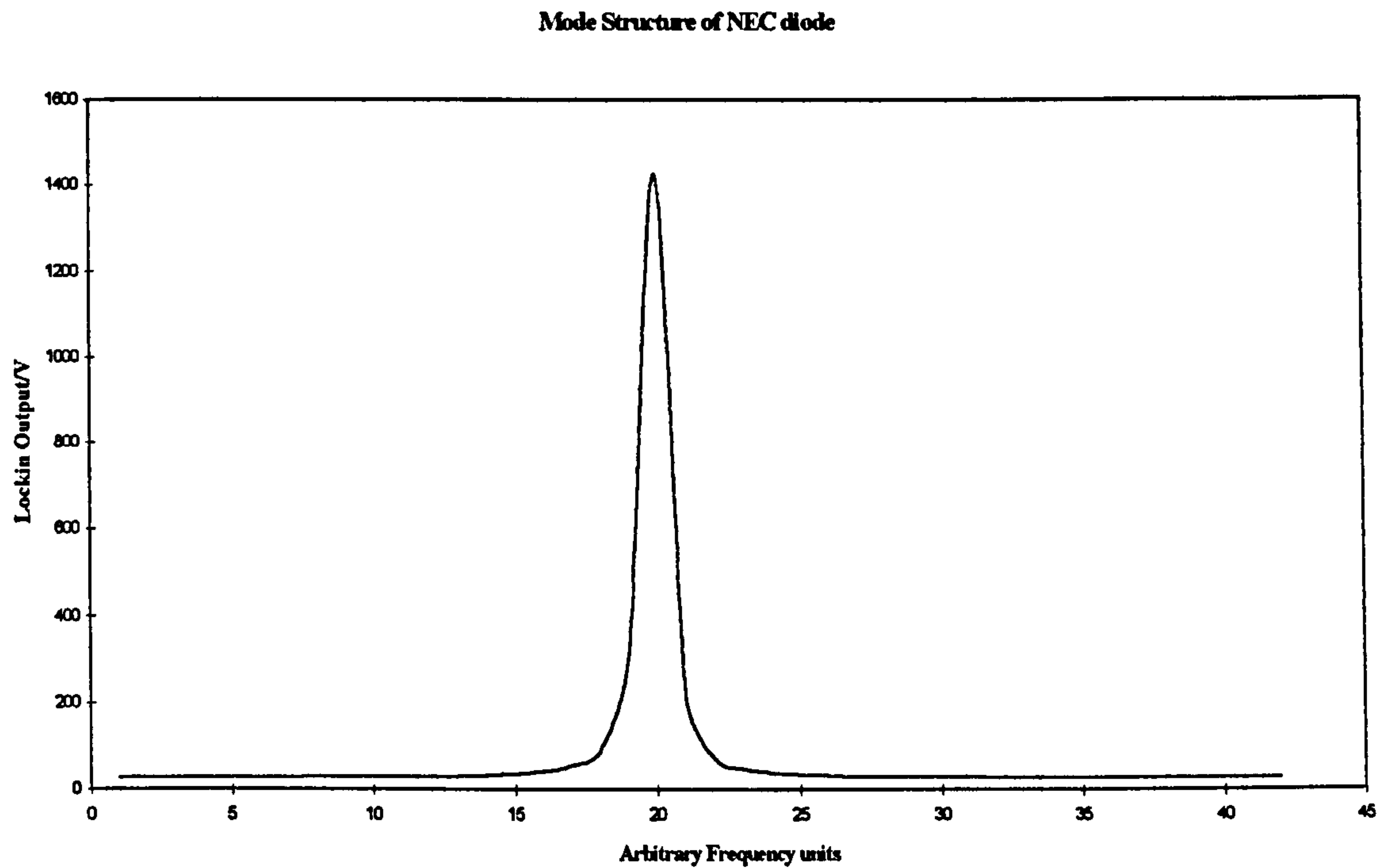


Figure 3.7 - Mode Structure of the NEC Laser

3.3 Spectroscopic Measurements

3.3.1 Introduction

The three lasers described previously, have been used to perform spectroscopic measurements on nitrous oxide (N₂O), methane (CH₄), acetylene (¹²C₂H₂, ¹³C₂H₂) and ammonia (¹⁴NH₃, ¹⁵NH₃). These gases play an important role in atmospheric chemistry and/or are pollutants from large scale industrial processes. The molecular overtone bands in the 1-2 μm region have been much less studied than the fundamental bands in the mid-infrared. In particular, the linestrengths and pressure broadening coefficients of these species are in need for improvement.

Tunable diode laser absorption spectroscopy (TDLAS) offers significant advantages over other spectroscopic techniques, such as Fourier Transform Spectroscopy (FTS). These advantages include better sensitivity (typically a minimum detectable absorption of 1×10^{-4} could be detected. This is a factor of ~ 100 better than what is usually achieved with FTS) and higher resolution (0.0003 cm⁻¹ for the lasers used here). This enables high precision Doppler limited spectroscopic measurements to be taken. The main disadvantage of TDLAS is that absolute frequency calibration is difficult and spectral coverage is very narrow (typically 3-4 nm for each laser).

3.3.2 Experimental Description

The electronic and optical set up used for these measurements is very similar to the one described in Section 5.5.2 and Section 5.5.3. The main differences being: The reference cell channel was replaced with a high Q Fabry-Perot scanning interferometer (FSR 7.5 Ghz), allowing a relative frequency scale to be determined. The laser was amplitude modulated with a mechanical chopper instead of frequency modulated. An alternative temperature controlled laser mount was used, allowing the diode temperature to be varied over an extended range (-20 to +40 °C). This mount was built and designed by Barry M^cDonald a previous member of the group at Strathclyde University. A detailed description of this mount can be found in his thesis [21]. The Herriott multipass cell

used for pathlength enhancement has been extensively described in Chapter 4 of this thesis.

The wavelength of the laser is tuned by scanning the laser current, typically between 40 and 120 mA. As was shown in Section 3.2, this will typically change the laser wavelength by $\sim 2\text{-}3\text{ cm}^{-1}$. In order to achieve continuous coverage over the entire wavelength region accessible to the laser ($20\text{-}30\text{ cm}^{-1}$), a series of such scans are repeated over a wide range of diode operating temperatures (-20 to $+40\text{ }^{\circ}\text{C}$). Typically temperature is incremented in $2.5\text{ }^{\circ}\text{C}$ steps. This ensures that each recorded spectral region overlaps, allowing them to be joined up later to give continuous coverage.

3.3.3 Post Measurement Transformation of Recorded Data

Figure 3.8 shows a typical spectrum recorded with the experimental set up (N_2O , 2.5 Torr, 25.1 m pathlength). It is seen that the spectrum has a large sloping background and that the frequency scale given by the etalon spacing is non linear. If linestrength, broadening coefficient and line position information is to be determined, the background and non linearity must be removed. Once removed, the spectral regions corresponding to each scan must be joined up. Software has been developed in MATLAB to semi automate this transformation process. A program listing can be found in Appendix 2.

The software allows the user to nominate a number of points on the spectrum that he/she thinks are the background signal. The program carries out a polynomial fit to these points and ratios it with the recorded spectrum. The program then automatically finds the etalon peaks and by means of cubic interpolation frequency linearizes the data. An example of the resulting spectrum is shown in Figure 3.9.

Adjoining spectra are loaded up and displayed. The user chooses two coincident points, one from each spectrum. The program will then splice these two spectra together. Linear interpolation is used to ensure that the frequency increment per data point is the same.

An absolute wavelength scale on the recorded spectra is achieved by identifying lines on the spectra that have either been assigned in the literature or have been recorded on the BOMEM FTS available at Strathclyde University.

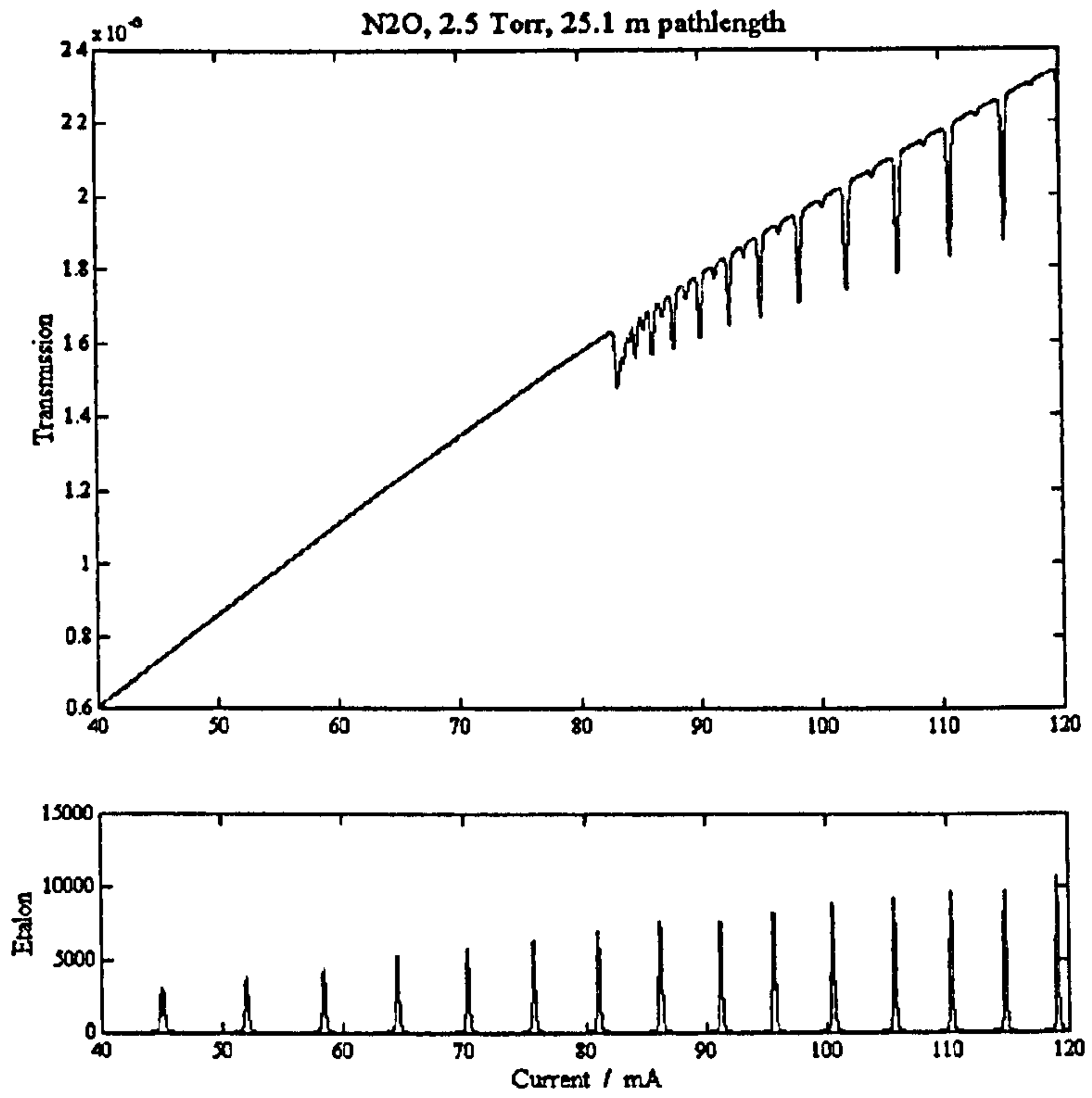


Figure 3.8 - Typical spectrum recorded with TDLAS

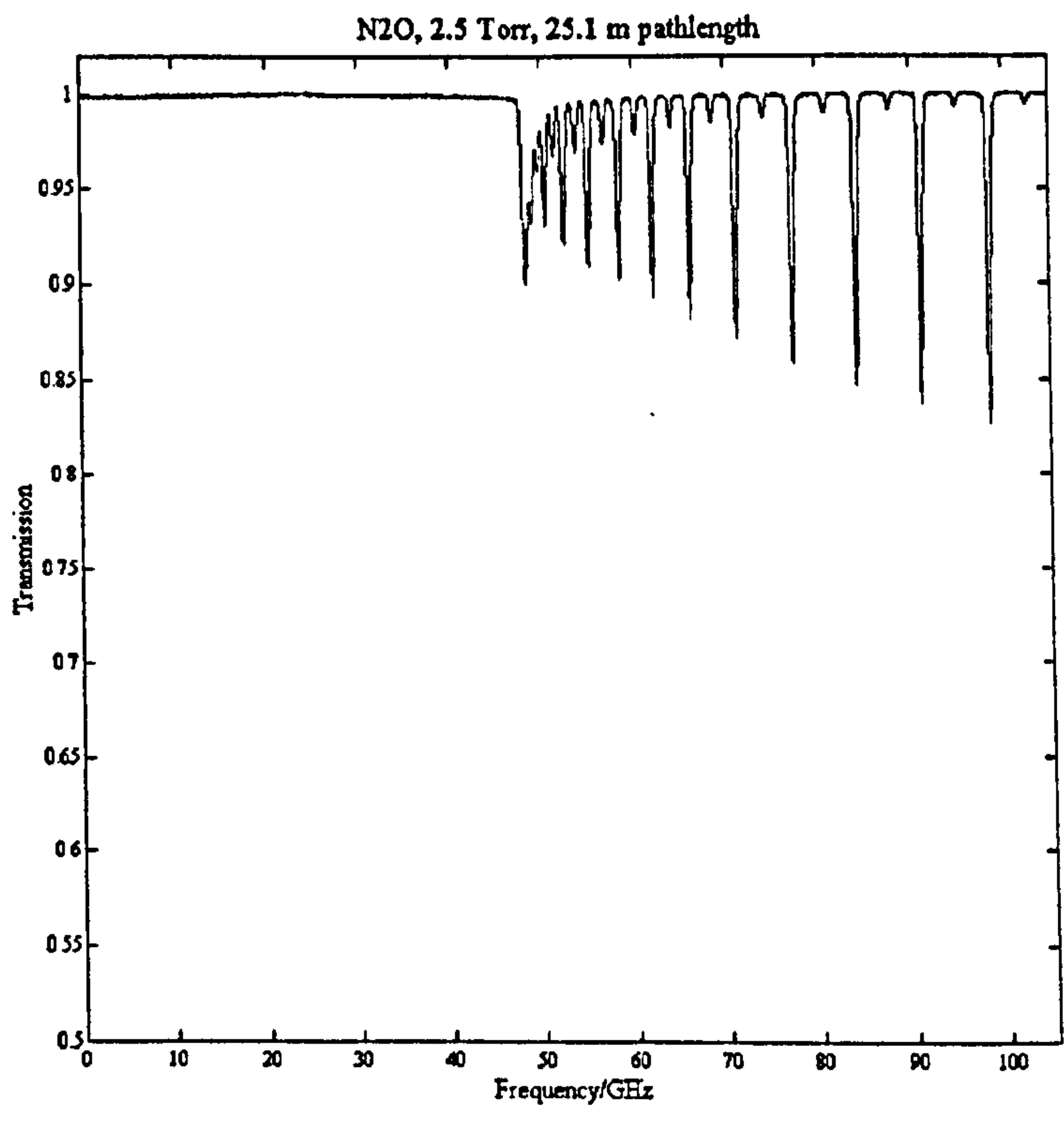


Figure 3.9 - Spectrum after Transformation

3.3.4 Nitrous Oxide

Nitrous oxide was recorded with the Thomson DFB. A 25 m pathlength and 2.5 Torr sample pressure were used. N₂O was transferred to the cell from a 1 litre glass bulb via clean glassware. 12 spectra were recorded at 5 °C intervals between -20 and +40 °C. The cell was refilled every three scans to minimize adsorption and gas leak effects. Each scan took ~ 2 minutes to record, during which the temperature inside the cell was monitored. Temperature was shown to change by less than 0.1 °C during this period. A maximum 2 °C temperature difference was observed over the time taken to record the 12 scans. The average cell temperature was 26 °C.

Rotational Analysis

Figure 3.10 shows the combined spectrum of the 12 separate scans. The observed transitions belong to the 3 ν_3 band. The wavelength calibration was obtained from a FTS spectrum of N₂O in the same region taken on the BOMEM DA3.002. J quantum number assignments were made and a rotational analysis performed. Figure 3.11 shows the relationship between the observed line position and the rotational quantum number J. The smooth transition in the line position with quantum number indicates correct assignment. A least squares polynomial fit of this behaviour has allowed the rotational parameters to be determined. The rotational energy was fit in terms of effective parameters:

$$\begin{aligned} E_R &= BX - DX^2 + HX^3 + LX^4 \\ E_{VR} &= G + E_R \\ X &= J(J+1) \end{aligned} \tag{3.1}$$

where E_R represents the rotational energy and G is the effective vibrational energy. Transition frequencies, $\nu(J' \leftarrow J'')$, are given as:

$$\begin{aligned} \nu(J' \leftarrow J'') &= \nu_0 + E_R' - E_R'' \\ \nu_0 &= G' - G'' \end{aligned} \tag{3.2}$$

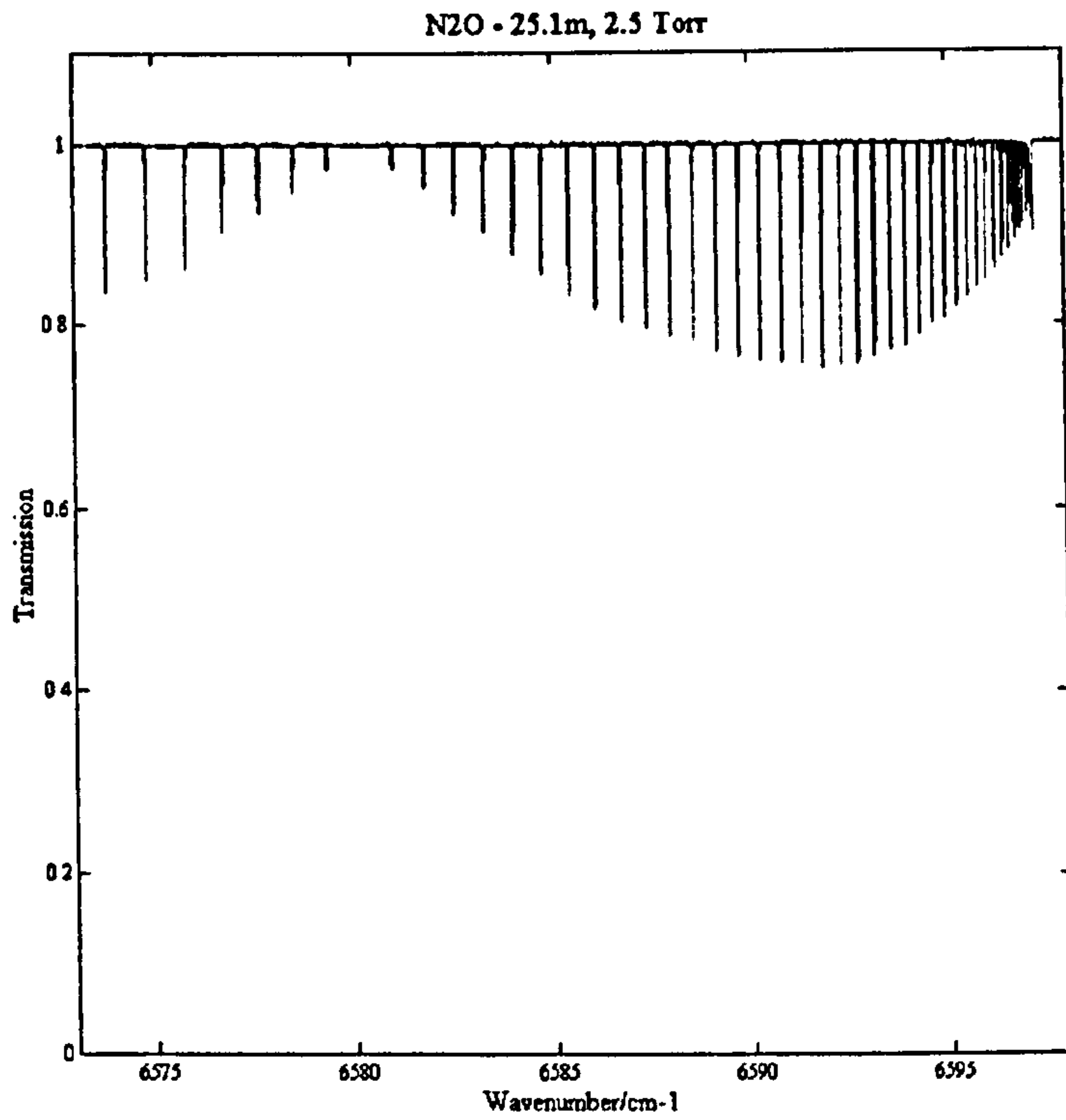


Figure 3.10 - N₂O Spectrum (25.1 m , 2.5 Torr)

N₂O Recorded Line Positions against Rotational Quantum Number, J

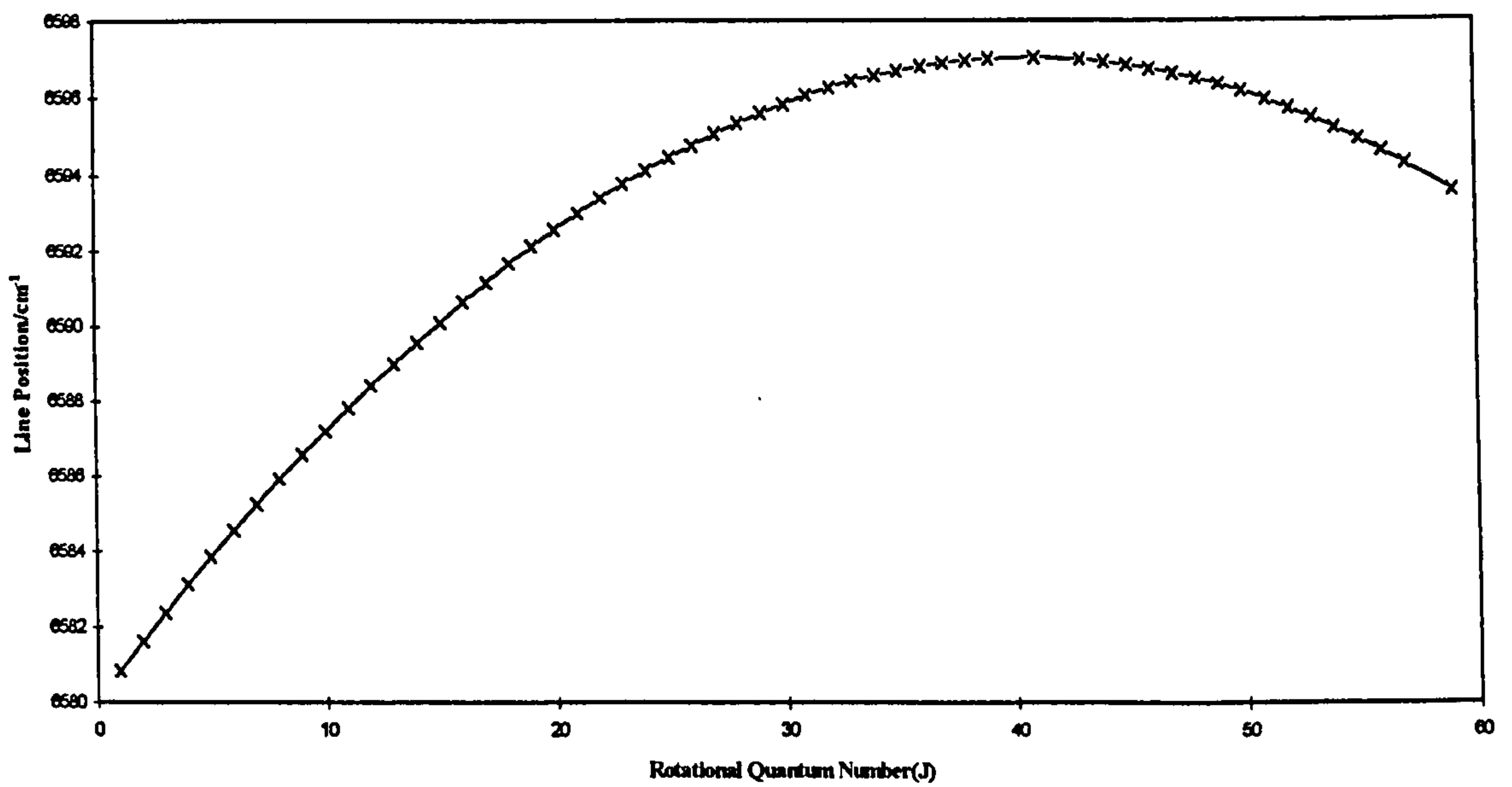


Figure 3.11 - N₂O line positions against rotational quantum number, J

where a prime and double prime denote upper and lower states, respectively, and ν_0 is the band centre. Ground state parameters were taken from work on N₂O carried out by Toth [22]. These parameters and the fitted parameters for the $3\nu_3$ state are listed in Table 3.5. The observed and fitted line positions are listed in Table 3.6. The rms deviation in the fit was 0.028 cm⁻¹. Importantly the observed - calculated residuals did not show any systematic offset.

Ground State [22]	B	0.419011001
	D x 10 ⁷	1.7609193
	H x 10 ¹³	-0.16529
Upper State (3ν₃)	ν ₀	6580.1551238
	B	0.409176616
	D x 10 ⁷	3.045713001
	H x 10 ¹³	176.6870618
	L x 10 ¹⁶	-1.18422164

Table 3.5 - Rotational Parameters for the (3ν₃) band of N₂O

Linestrengths, Linewidths and Broadening Coefficients

The linestrengths for the assigned transitions are listed in Table 3.6. Uncertainties in linestrengths are estimated to be ~ 5% (1σ). This uncertainty was based on an error budget similar to the one described in Section 6.5.4. The main uncertainties arising from the pressure, temperature and absorption pathlength used in the calculation. An additional uncertainty of ~ 1% was associated with the background removal process. The R(39) and R(40) transitions are heavily blended (superimposed). It should be noted that the linestrength given is the combined strength for both transitions.

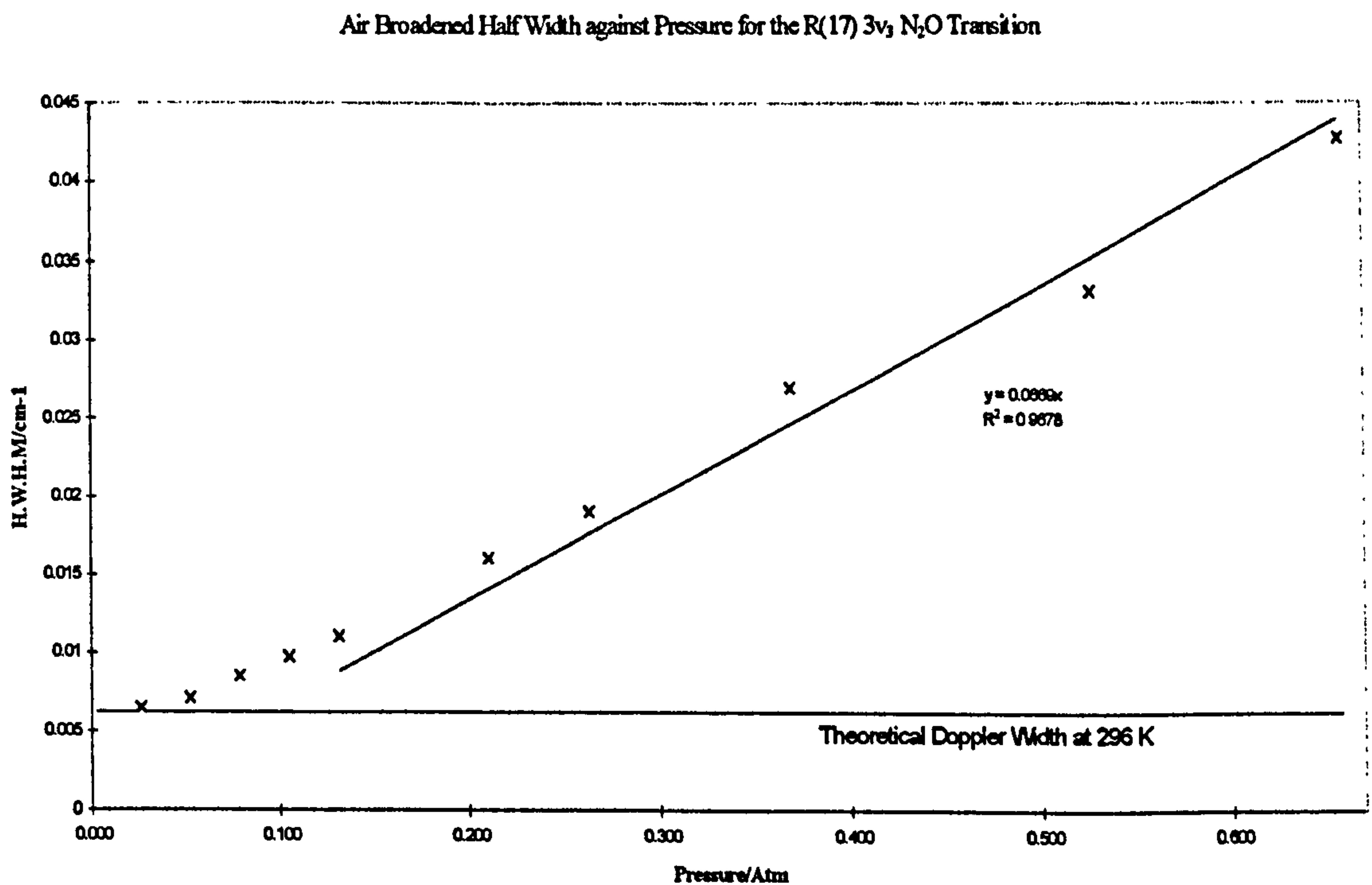
The recorded lineshapes had a HWHM of 0.0061 cm⁻¹. This compares well with the theoretical Doppler width for N₂O (0.00612 cm⁻¹).

The air broadening coefficient for the R(17) transition was also measured. A 1 Torr N₂O sample was mixed with varying sample pressures of dry air and the spectrum

recorded. Figure 3.12 shows a plot of the recorded linewidth against sample pressure. At low sample pressures Doppler broadening dominates. At higher pressures the relationship between the HWHM and sample pressure takes the form:

$$\gamma(P) = \gamma_B P \quad (3.3)$$

where $\gamma(P)$ is the HWHM of the lineshape, P is the sample pressure of the buffer gas and γ_B is the pressure broadening coefficient. A fit of the data to this function has allowed the pressure broadening coefficient to be determined. A value of $0.0669 \text{ cm}^{-1}/\text{atm}$ was calculated. An uncertainty of 3-4% (1σ) is associated with this measurement. The main uncertainty resulting from changes in the FSR of the scanning interferometer with temperature (1-2 %) and the measurements of sample pressure and temperature within the cell. This measured broadening coefficient agrees well with the coefficient for other bands of N_2O listed in HITRAN [23]. A typical value was $\sim 0.0672 \text{ cm}^{-1}/\text{atm}$.



**Figure 3.12 - Air Broadened Half Width against Sample Pressure
R(17) Transition**

Assignment	Linestrength (cm molecule ⁻¹)	Obs (cm ⁻¹)	Calc (cm ⁻¹)	Obs-Calc (cm ⁻¹)
P (7)	1.13E-23	6573.807	6573.877	-0.070
P (6)	1.06E-23	6574.811	6574.833	-0.022
P (5)	9.41E-24	6575.793	6575.769	0.024
P (4)	6.43E-24	6576.731	6576.685	0.046
P (3)	5.03E-24	6577.633	6577.582	0.051
P (2)	3.40E-24	6578.509	6578.538	-0.029
P (1)	1.76E-24	6579.365	6579.337	0.028
R (0)	1.76E-24	6581.017	6580.973	0.044
R (1)	3.12E-24	6581.815	6581.772	0.043
R (2)	5.11E-24	6582.575	6582.551	0.023
R (3)	6.63E-24	6583.326	6583.310	0.016
R (4)	8.25E-24	6584.053	6584.050	0.003
R (5)	9.87E-24	6584.765	6584.770	-0.005
R (6)	1.13E-23	6585.459	6585.470	-0.011
R (7)	1.26E-23	6586.130	6586.150	-0.020
R (8)	1.40E-23	6586.778	6586.811	-0.033
R (9)	1.46E-23	6587.404	6587.451	-0.047
R (10)	1.53E-23	6588.003	6588.072	-0.069
R (11)	1.58E-23	6588.608	6588.673	-0.065
R (12)	1.67E-23	6589.196	6589.254	-0.058
R (13)	1.69E-23	6589.758	6589.815	-0.057
R (14)	1.72E-23	6590.300	6590.356	-0.056
R (15)	1.76E-23	6590.844	6590.876	-0.032
R (16)	1.77E-23	6591.368	6591.377	-0.009
R (17)	1.81E-23	6591.875	6591.858	0.018
R (18)	1.77E-23	6592.331	6592.318	0.013
R (19)	1.76E-23	6592.771	6592.758	0.013
R (20)	1.69E-23	6593.193	6593.178	0.016
R (21)	1.63E-23	6593.595	6593.577	0.018
R (22)	1.62E-23	6593.976	6593.956	0.020
R (23)	1.52E-23	6594.332	6594.315	0.017
R (24)	1.42E-23	6594.663	6594.653	0.010
R (25)	1.37E-23	6594.970	6594.970	0.000
R (26)	1.27E-23	6595.267	6595.267	0.000
R (27)	1.18E-23	6595.541	6595.543	-0.003
R (28)	1.08E-23	6595.795	6595.799	-0.004
R (29)	1.06E-23	6596.018	6596.034	-0.015
R (30)	9.78E-24	6596.244	6596.248	-0.003
R (31)	8.74E-24	6596.449	6596.441	0.008
R (32)	8.05E-24	6596.616	6596.613	0.003
R (33)	7.09E-24	6596.752	6596.765	-0.013

Table 3.6 - Assigned N₂O Transitions of the 3 ν₃ band with linestrengths and linepositions

Assignment	Linestrength (cm molecule ⁻¹)	Obs (cm ⁻¹)	Calc (cm ⁻¹)	Obs-Calc (cm ⁻¹)
R (34)	6.49E-24	6596.876	6596.895	-0.019
R (35)	6.09E-24	6596.988	6597.005	-0.017
R (36)	5.20E-24	6597.076	6597.093	-0.017
R (37)	4.56E-24	6597.145	6597.161	-0.016
R (38)	4.46E-24	6597.194	6597.208	-0.014
R (39),R(40)	6.70E-24	6597.220	6597.238	-0.018
R (41)	2.53E-24	6597.171	6597.185	-0.014
R (43)	2.12E-24	6597.113	6597.127	-0.014
R (44)	1.94E-24	6597.034	6597.048	-0.014
R (45)	1.65E-24	6596.933	6596.948	-0.015
R (46)	1.31E-24	6596.813	6596.828	-0.015
R (47)	1.05E-24	6596.685	6596.687	-0.002
R (48)	9.09E-25	6596.535	6596.525	0.010
R (49)	7.78E-25	6596.347	6596.342	0.005
R (50)	6.55E-25	6596.130	6596.139	-0.009
R (51)	4.62E-25	6595.910	6595.916	-0.005
R (52)	4.54E-25	6595.666	6595.672	-0.006
R (53)	3.35E-25	6595.401	6595.408	-0.008
R (54)	2.58E-25	6595.115	6595.125	-0.010
R (55)	2.11E-25	6594.813	6594.821	-0.009
R (56)	1.99E-25	6594.496	6594.498	-0.003
R (58)	1.16E-25	6593.779	6593.794	-0.014

Table 3.6 - Assigned N₂O Transitions of the 3 ν_3 band with linestrengths and linepositions

3.3.5 Methane

Methane spectra were recorded with the Anritsu laser. A sample pressure of 0.1 Torr was used with a 25.1 m pathlength. The maximum temperature difference during the measurement period was $\sim 1^\circ\text{C}$. Average cell temperature was 24°C . Twenty separate scans were recorded and combined.

Figure 3.13 shows the recorded spectrum of the Q branch of the $2\nu_3$ band. Figure 3.14 shows the Q (10) manifold from this spectrum. It is clear that the lines are heavily blended. This is found to be the case for all the Q branch multiplets except the Q (1) transition which is a single unblended line. Margolis [24] has identified the positions of the individual transitions that make up these manifolds. These line positions have been used in conjunction with a non linear least square fit routine, FITSPEC, written by Kelly Chance [25], to allow the individual strengths of each transition in the recorded spectrum to be determined. Table 3.7 lists the linestrengths determined with this technique and the linestrengths measured by Margolis. In all cases the residuals between the fitted spectrum and the recorded data were less than 2%.

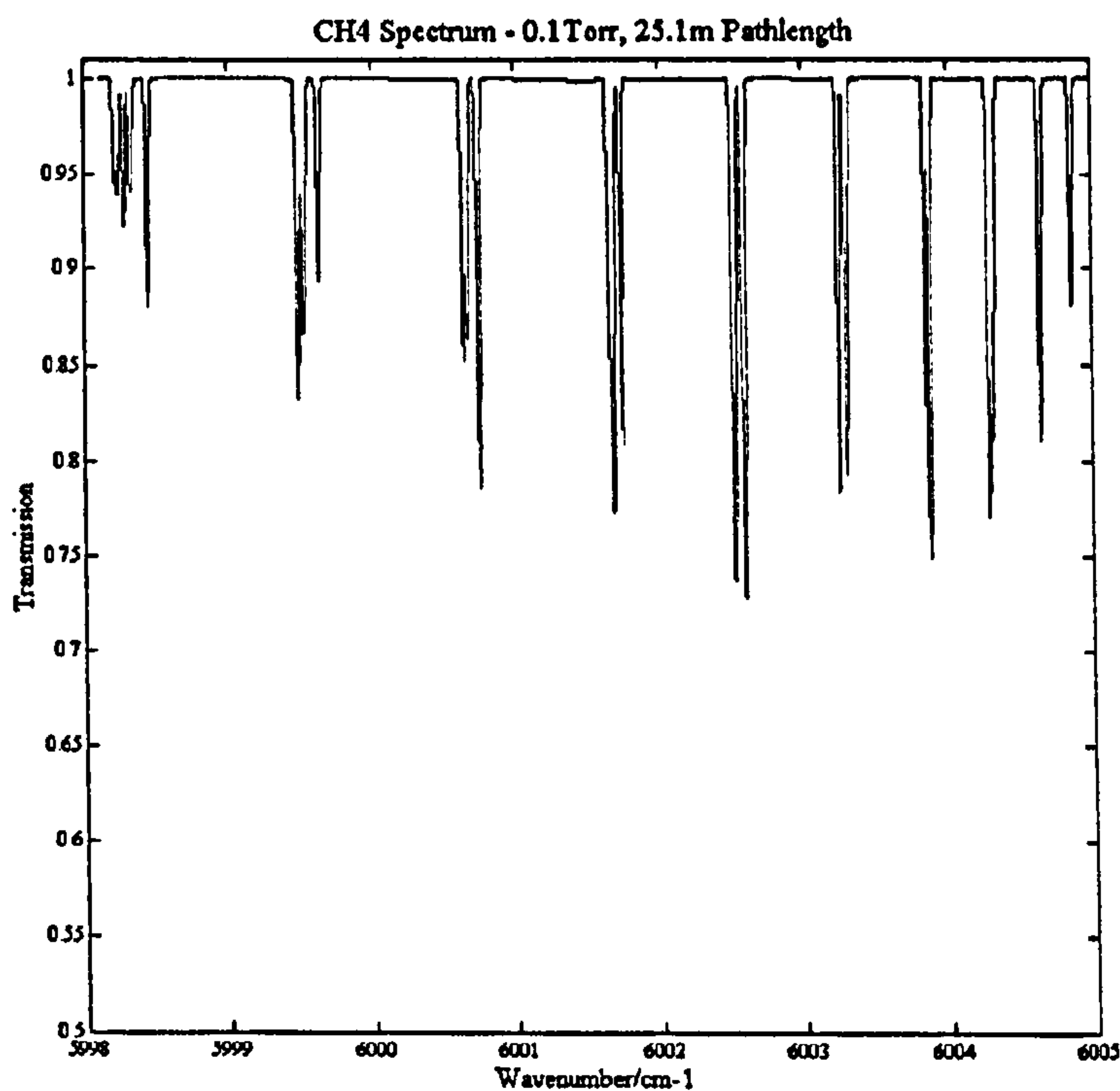


Figure 3.13 - Recorded Spectrum of the $2\nu_3$ Q branch of Methane (0.1 Torr sample pressure, 25.1 m pathlength)

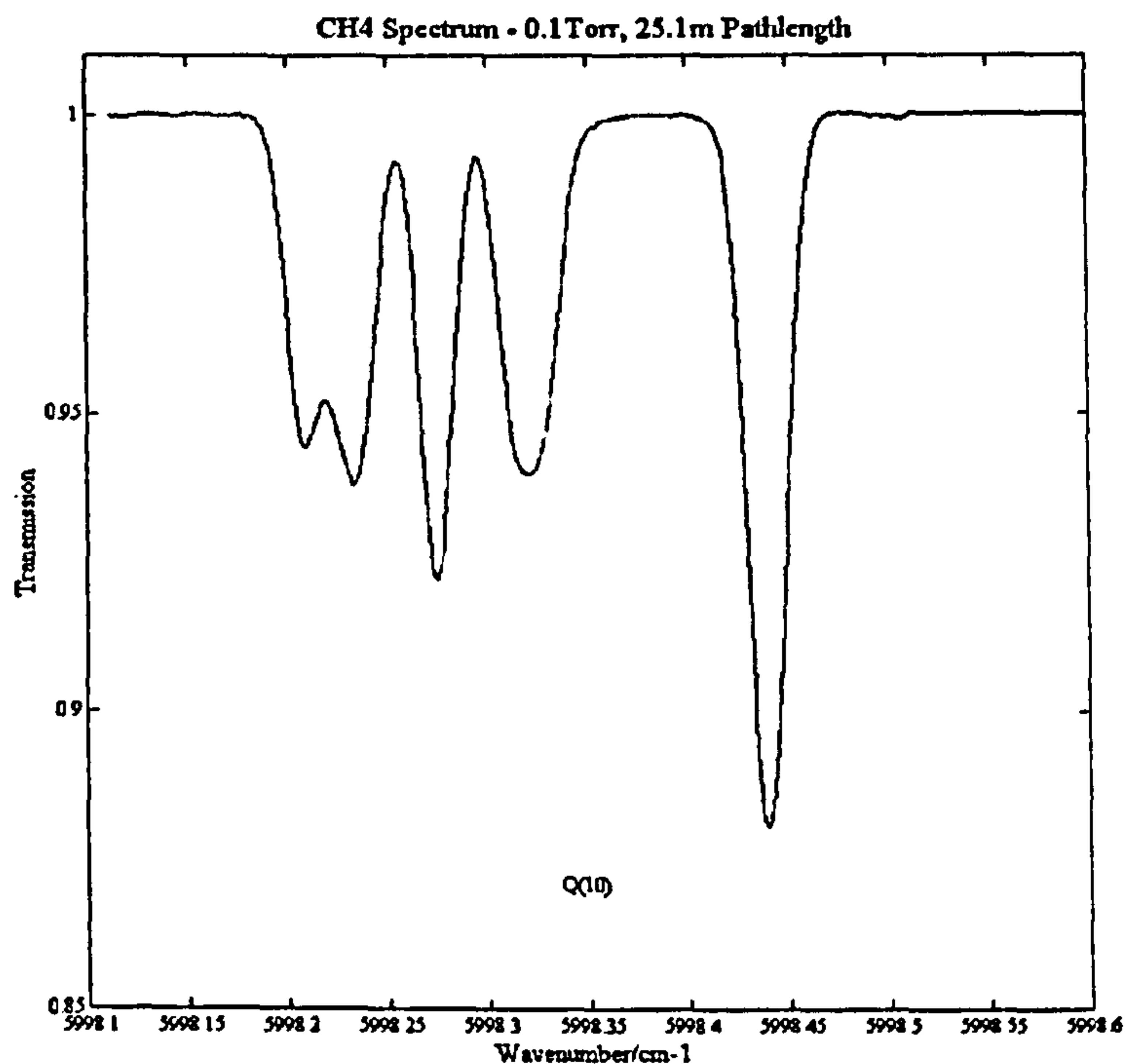


Figure 3.14 - Recorded Spectrum of the $2\nu_3$ Q (10) multiplet of Methane (0.1 Torr sample pressure, 25.1 m pathlength)

Manifold	Frequency (cm ⁻¹)	Strength x 10 ²² (cm molecule ⁻¹) This Work	Strength x 10 ²² (cm molecule ⁻¹) Margolis [24]
Q (10)	5998.21122	1.45	1.439
	.23045	1.15	1.149
	.24261	1.07	1.100
	.28040	2.24	2.220
	.31977	1.33	1.330
	.33479	1.22	1.209
	.43964	1.10	1.070
	.44193	2.13	2.140
	.44275	0.764	0.764
Q (9)	5999.47429	2.22	2.220
	.48102	3.34	3.350
	.49211	2.15	2.120
	.51383	1.90	1.860
	.52704	3.52	3.500
	.54100	1.80	1.820
	.62541	1.88	1.879
	.63430	1.82	1.810

Table 3.7 - Calculated Linestrengths for the Q branch manifolds of the $2\nu_3$ band

Manifold	Frequency (cm ⁻¹)	Strength x 10 ²² (cm molecule ⁻¹) This Work	Strength x 10 ²² (cm molecule ⁻¹) Margolis [24]
Q (8)	6000.63682	2.23	2.250
	.64340	3.00	3.049
	.66346	3.13	3.129
	.67486	2.66	2.660
	.73732	1.05	1.070
	.74837	5.26	5.240
	.75070	2.77	2.779
Q (7)	6001.65242	3.93	3.899
	.66458	3.31	3.310
	.67517	6.18	6.169
	.68410	3.92	3.910
	.73887	2.45	2.500
	.74158	4.80	4.809
Q (6)	6002.51720	6.85	6.839
	.52535	6.85	6.839
	.53672	5.013	5.019
	.58552	5.00	4.959
	.59208	5.87	5.870
	.59654	4.10	4.049
Q (5)	6003.24722	3.74	3.740
	.25405	6.03	6.030
	.29868	5.55	5.540
	.31111	5.69	5.709
Q (4)	6003.83695	6.20	6.190
	.87142	5.95	5.959
	.88305	5.7	5.690
	.89285	8.40	8.339
Q (3)	6004.29301	9.83	9.830
	.31330	5.67	5.690
	.32910	5.78	5.779
Q (2)	6004.64354	4.50	4.490
	.65274	3.61	3.609
Q (1)	6004.86318	3.23	3.229

Table 3.7 - Calculated Linestrengths for the Q branch manifolds of the 2 ν_3 band

The uncertainty associated with the linestrength measurements is $\sim 5\%$ (1σ). The main uncertainty arising from the pressure, temperature, and pathlength used in the linestrength calculation. An additional uncertainty of $\sim 2\%$ is associated with the fitting

procedure. The differences between the Margolis linestrengths and the ones measured in this study are within the measurement uncertainty of both experiments.

The pressure broadening coefficient of the Q(1) transition was measured. The Q(1) transition was chosen because it was a single isolated line. The width of this feature (0.1 Torr, 22 °C) was determined to be $0.0091 \text{ cm}^{-1} \pm 3 \%$. This is in good agreement with the theoretical Doppler width (0.00909 cm^{-1}). Figure 3.15 shows a plot of the recorded linewidth against the dry air sample pressure.

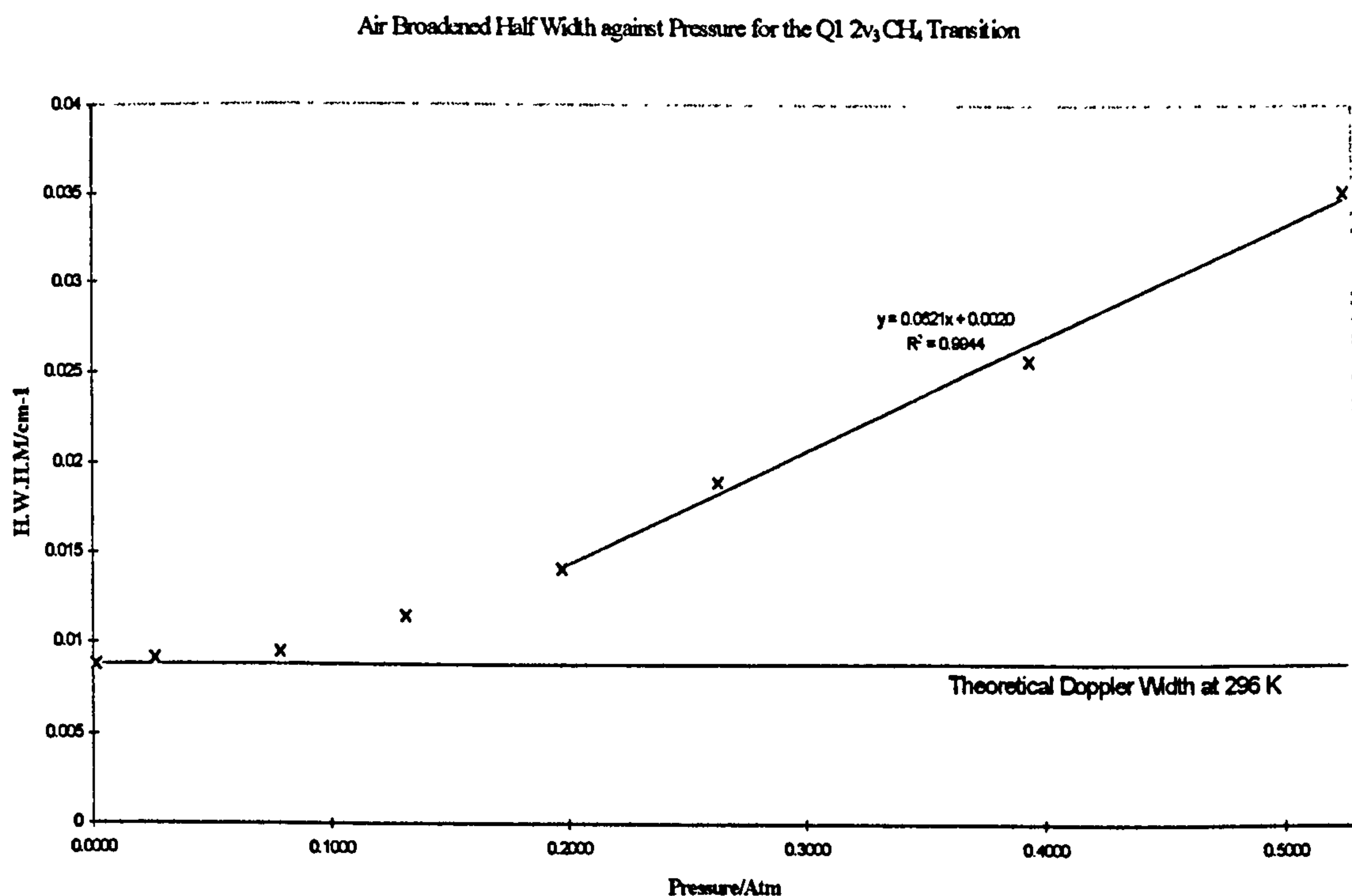


Figure 3.15 - Air Broadened Half Width against Sample Pressure Q (1) Transition

A pressure broadening coefficient of $0.0621 \text{ cm}^{-1}/\text{atm}$ was obtained from this data. The broadening coefficient listed in HITRAN [23] for this particular transition was $0.0641 \text{ cm}^{-1}/\text{atm}$. Again it is seen that good agreement was achieved.

3.3.6 C₁₂ Acetylene

Measurement of Acetylene (¹²C₂H₂) was made with both the NEC and Thomson lasers. Figure 3.16 shows the spectrum recorded with the Thomson laser. A sample pressure of 0.15 Torr and a pathlength of 11.1 m were used. The average cell temperature during measurement was 24 °C. The maximum change in temperature during measurement was 3 °C. Twelve scans were combined.

Karen Keplers work on acetylene [26] has been used to identify many of the transitions in this spectrum. The superior resolution and signal to noise of the spectra recorded with the TDLAS have allowed additional assignments to be made. Table 3.8 lists the identified lines, measured line positions from this study and line positions from Kepler *et al.* Lines marked with an asterisk are new assignments.

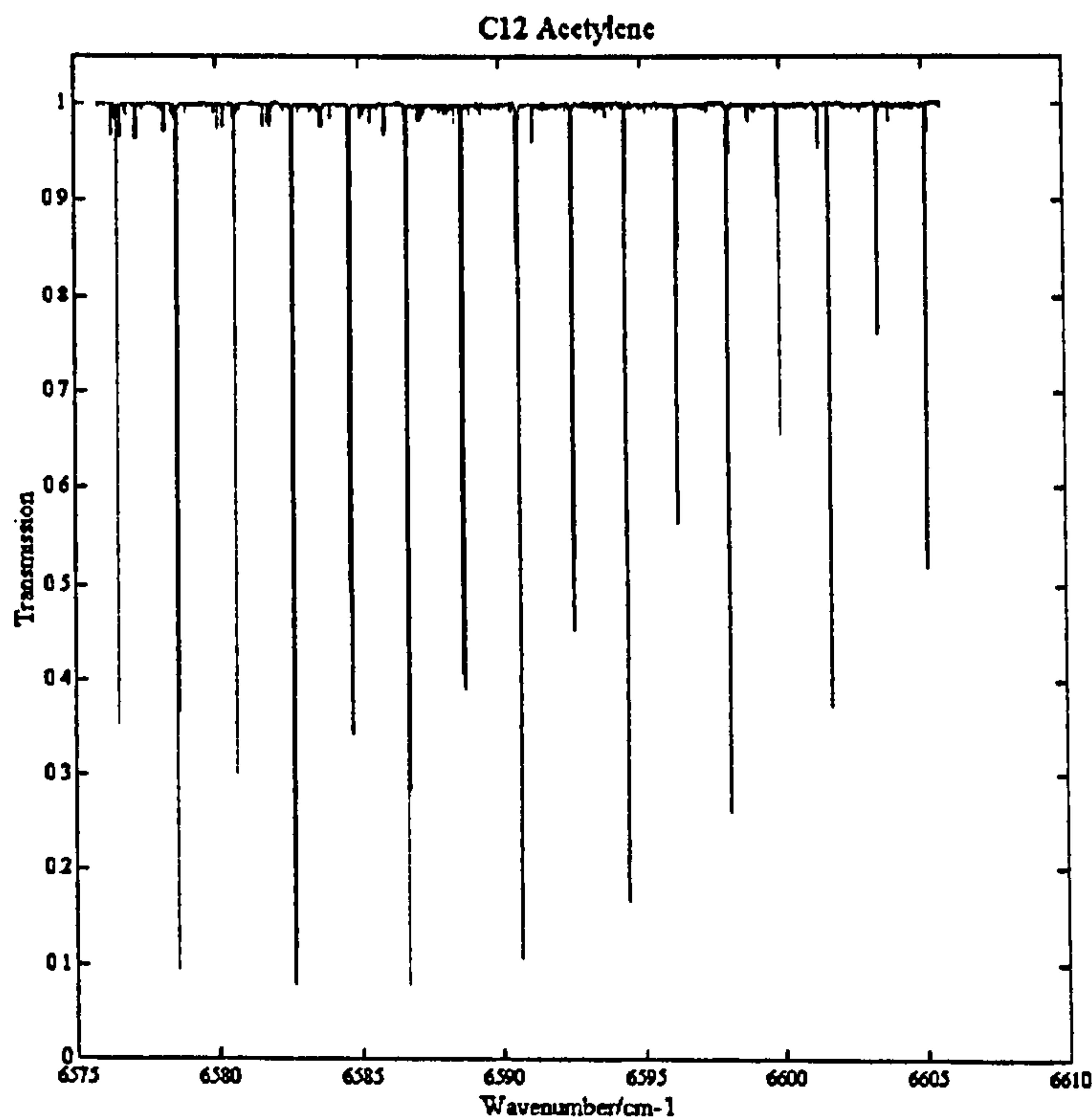


Figure 3.16 - ¹²C₂H₂ (0.15 Torr, 11.1m pathlength)

Rotation	State		Frequency (cm ⁻¹)	Frequency (cm ⁻¹)	Difference
	Upper	Lower	This Work	Keppler [26]	
R8	101 (00)	000 (00)	6576.481	6576.482	-0.00101
R19	101 (01)1f	000 (01)1f	6576.592	6576.594	-0.00239
P18	11011	000 (00)	6577.944		
R20	101 (01)1e	000 (01)1e	6578.123	6578.127	-0.0039
R9	101 (00)	000 (00)	6578.571	6578.575	-0.0045
R21	101 (01)1f	000 (01)1f	6580.199	6580.206	-0.0069
R10	101 (00)	000 (00)	6580.624	6580.644	-0.0197
P17*	11011	000 (00)	6580.72		
R22	101 (01)1e	000 (01)1e	6581.628	6581.652	-0.0238
R25	101 (10)1e	000 (10)1e	6581.908	6581.933	-0.0241
R11	101 (00)	000 (00)	6582.664	6582.685	-0.0208
P16*	11011	000 (00)	6583.272		
R12	101 (00)	000 (00)	6584.688	6584.702	-0.0142
P15	11011	000 (00)	6585.923	6585.936	-0.0133
R13	101 (00)	000 (00)	6586.685	6586.691	-0.0065
P14*	11011	000 (00)	6588.55		
R14	101 (00)	000 (00)	6588.652	6588.654	-0.0016
R15	101 (00)	000 (00)	6590.622	6590.592	0.0303
P13	11011	000 (00)	6591.176	6591.148	0.0284
R16	101 (00)	000 (00)	6592.553	6592.503	0.0497
P12*	11011	000 (00)	6593.767		
R17	101 (00)	000 (00)	6594.433	6594.387	0.0464
R18	101 (00)	000 (00)	6596.277	6596.245	0.0315
P11	11011	000 (00)	6596.306	6596.276	0.0296
R19	101 (00)	000 (00)	6598.104	6598.077	0.0273
P10*	11011	000 (00)	6598.843		
R20	101 (00)	000 (00)	6599.914	6599.882	0.0321
P9	11011	000 (00)	6601.358	6601.324	0.034
R21	101 (00)	000 (00)	6601.697	6601.661	0.0363
R22	101 (00)	000 (00)	6603.432	6603.414	0.018
P8*	11011	000 (00)	6603.84		
R23	101 (00)	000 (00)	6605.152	6605.14	0.0117

Table 3.8 - Assigned ¹²C₂H₂ Transitions (Thomson Laser Spectrum)

A line list (position, transmission) of the all the other lines that have not been identified is given in Appendix 3.

Figure 3.17 shows the spectrum recorded with the NEC laser. A sample pressure of 2.0 Torr and a pathlength of 25.1 m were used. The average cell temperature during measurement was 22 °C. A maximum change in temperature of 2 °C was observed during measurement. Sixteen individual scans were recorded

As before, Karen Kepplers work on acetylene [26] has been used to identify many of the transitions in this spectrum. Table 3.9 lists the identified lines, measured line positions from this study and line positions from Keppler *et al.* Lines marked with an asterix are new assignments. Some of the lines identified come from singly substituted C_{13} isotopic acetylene naturally present at ~ 1% in the acetylene sample used here. These lines are identified in Table 3.9.

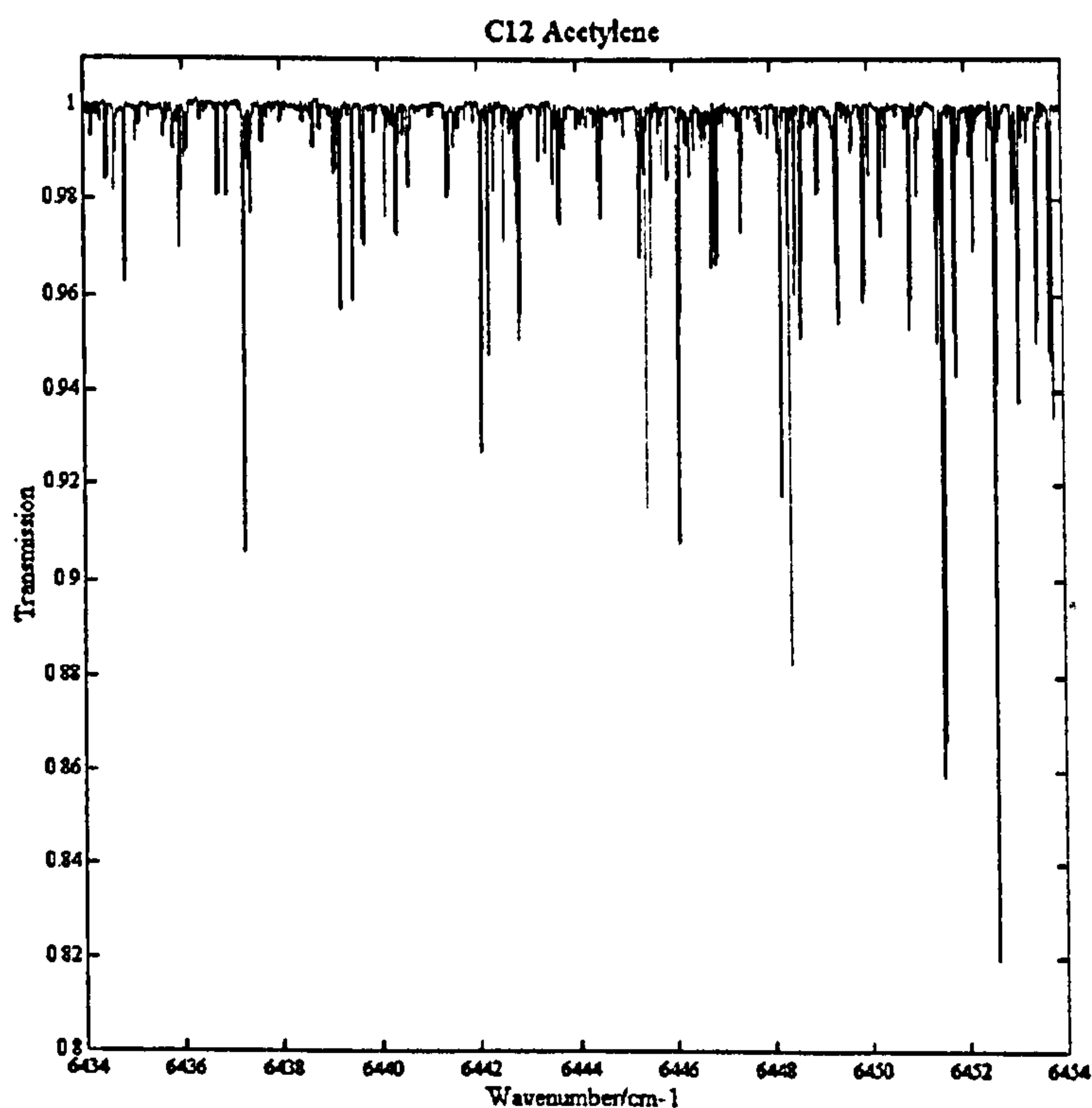


Figure 3.17 - $^{12}C_2H_2$ (2.0 Torr, 25.1m pathlength)

Rotation	State		Frequency (cm ⁻¹)	Frequency (cm ⁻¹)	Difference
	Upper	Lower	This Work	Keppler [26]	
P22 (C ₁₃)*	200 (00)	000 (00)	6434.615		
P42*	101 (00)	000 (00)	6436.043		
P21 (C ₁₃)*	200 (00)	000 (00)	6437.376		
P41	101 (00)	000 (00)	6439.396	6439.38	0.015595
P32	101 (10)1f	000 (10)1f	6442.213	6442.144	0.069107
P40*	101 (00)	000 (00)	6442.789		
P20 (C ₁₃)	200 (00)	000 (00)	6442.845	6442.782	0.063186
P31	101 (10)1f	000 (10)1f	6445.319	6445.257	0.06212
P19 (C ₁₃)	200 (00)	000 (00)	6445.548	6445.486	0.062265
P39	101 (00)	000 (00)	6446.106	6446.047	0.059274
P22	101 (20)2f	000 (20)2f	6446.131	6446.072	0.058974
P32	101 (10)1e	000 (10)1e	6446.908	6446.857	0.051399
P18 (C ₁₃)	200 (00)	000 (00)	6448.214	6448.176	0.038405
P30	101 (10)1f	000 (10)1f	6448.379	6448.343	0.036296
P30	101 (10)1e	000 (10)1e	6448.497	6448.462	0.034788
P38	101 (00)	000 (00)	6449.387	6449.347	0.039984
P31	101 (10)1f	000 (10)1f	6449.92	6449.88	0.039708
P21	101 (11)2f	000 (11)2f	6450.265	6450.227	0.038032
P17 (C ₁₃)	200 (00)	000 (00)	6450.866	6450.838	0.027866
P29	101 (10)1f	000 (10)1f	6451.419	6451.407	0.012285
P29	101 (10)1e	000 (10)1e	6451.544	6451.533	0.011476
P20	101 (20)2f	000 (20)2f	6451.787	6451.777	0.010473
P37	101 (00)	000 (00)	6452.618	6452.618	9.2E-05
P30	101 (10)1e	000 (10)1e	6453.095	6453.097	-0.00226
P16 (C ₁₃)	200 (00)	000 (00)	6453.473	6453.48	-0.00741

Table 3.9 - Assigned C₂H₂ Transitions (NEC Laser Spectrum)

A line list (position, transmission) of the all the other lines that have not been identified is given in Appendix 3.

3.3.7 C¹³ Acetylene

Measurement of Acetylene (¹³C₂H₂) was made with both the NEC and Thomson lasers. An isotopically enriched Acetylene sample was used. Figure 3.18 and Figure 3.19 shows the spectrum recorded with each laser. A sample pressure of 0.3 Torr and a pathlength of 25.1 m were used for the spectrum in Figure 3.18, while a 1.1 Torr sample pressure and 11.1m pathlength were used for the spectrum shown in Figure 3.19.

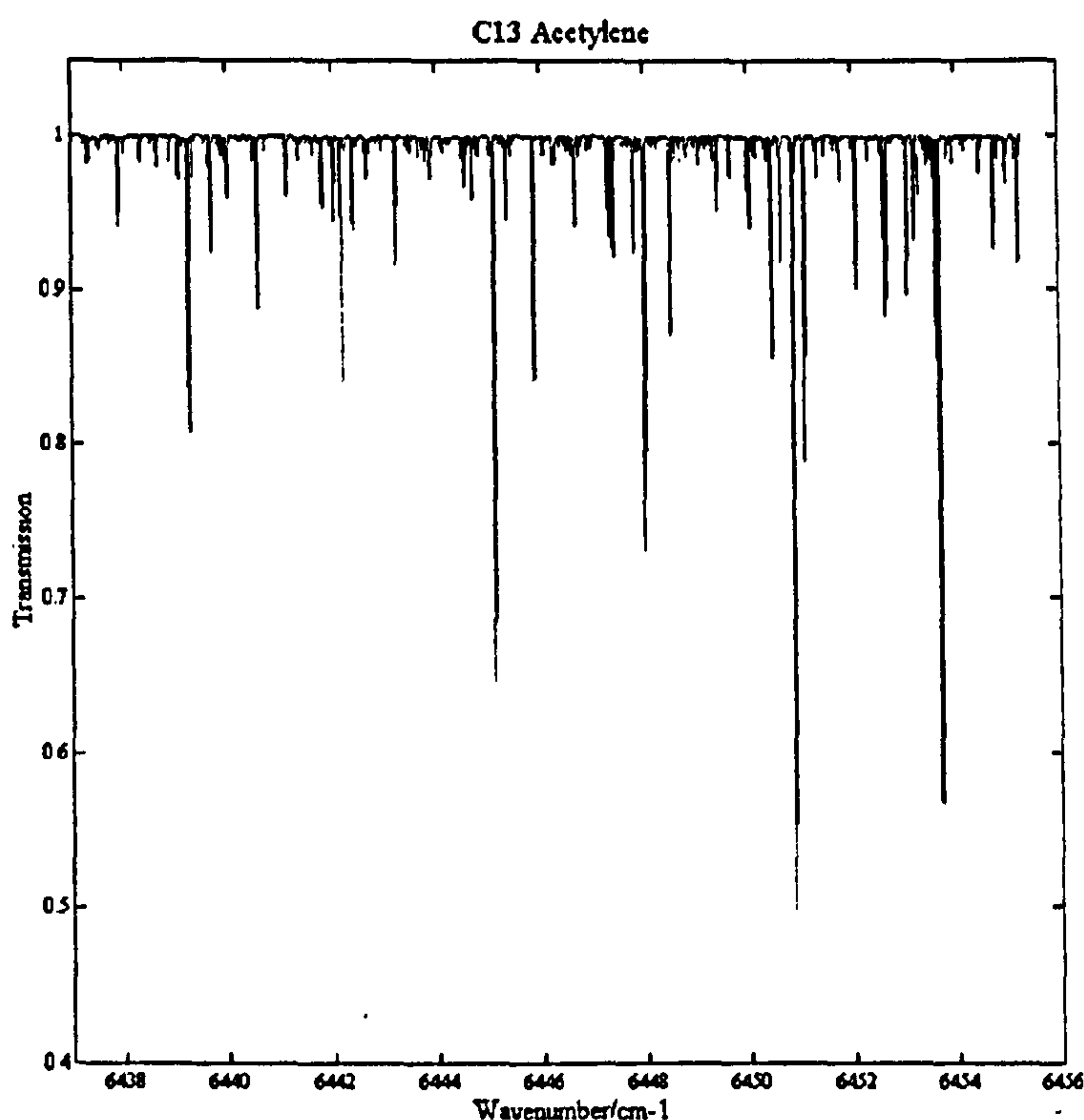


Figure 3.18 - ¹³C₂H₂ (0.3 Torr sample pressure, 25.1 m pathlength) - NEC

Latrasse used a 0.01 cm⁻¹ resolution BOMEM FTS to record ¹³C₂H₂ [27]. This work has been used to help assign and wavelength calibrate the recorded spectra. Table 3.10 lists the identified lines, the recorded line positions from this study and those determined by Latrasse. Lines marked with an asterisk are new assignments.

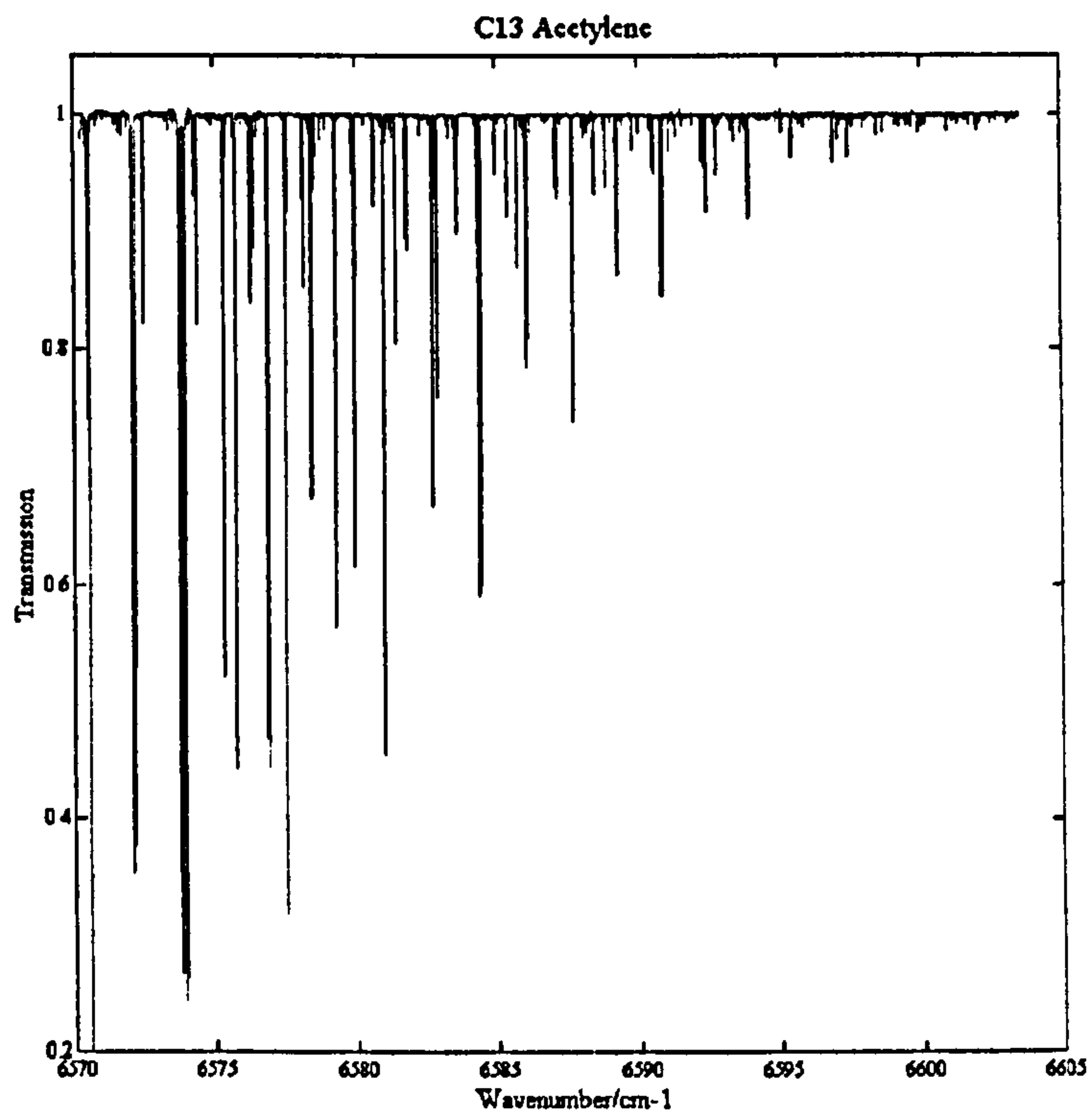


Figure 3.19 - $^{13}\text{C}_2\text{H}_2$ (1.1 Torr sample pressure, 11.1 m pathlength) - Thomson

A line list (position, transmission) of all other transitions not identified in these spectra is given in Appendix 3.

Rotation	State		Frequency (cm ⁻¹)	Frequency (cm ⁻¹)	Difference
	Upper	Lower	This Work	Latrasse [27]	
P32	101(00)	000(00)	6439.270	6439.283	-0.0131
P31	101(00)	000(00)	6442.204	6442.211	-0.0079
P30	101(00)	000(00)	6445.112	6445.118	-0.0059
P29	101(00)	000(00)	6448.004	6448.002	0.0019
P28	101(00)	000(00)	6450.867	6450.868	-0.0009
P27	101(00)	000(00)	6453.701	6453.703	-0.0026
R24	101(00)	000(00)	6570.521	6570.522	-0.0012
R17	110(11)	000(00)	6572.091	6572.099	-0.00775
R25	101(00)	000(00)	6572.153	6572.159	-0.00632
R26	101(00)	000(00)	6573.765	6573.771	-0.00562
R18	110(11)	000(00)	6573.934	6573.939	-0.00476
R27	101(00)	000(00)	6575.338	6575.357	-0.01888
R19	110(11)	000(00)	6575.74	6575.759	-0.01929
R28	101(00)	000(00)	6576.901	6576.922	-0.02167
R20	110(11)	000(00)	6577.53	6577.943	-0.41317
R29	101(00)	000(00)	6578.437	6578.458	-0.02181
R21	110(11)	000(00)	6579.297	6579.324	-0.02675
R30	101(00)	000(00)	6579.936	6579.973	-0.03692
R22	110(11)	000(00)	6581.021	6581.06	-0.03884
R31	101(00)	000(00)	6581.424	6581.467	-0.04308
R23	110(11)	000(00)	6582.739	6582.776	-0.03687
R32*	101(00)	000(00)	6582.894		
R33*	101(00)	000(00)	6584.327		
R24	110(11)	000(00)	6584.417	6584.458	-0.04032
R34*	101(00)	000(00)	6585.742		
R25	110(11)	000(00)	6586.073	6586.118	-0.04514
R35*	101(00)	000(00)	6587.164		
R26*	110(11)	000(00)	6587.71		
R36*	101(00)	000(00)	6588.489		
R27*	110(11)	000(00)	6589.299		
R28*	110(11)	000(00)	6590.875		
R29*	110(11)	000(00)	6592.424		
R30*	110(11)	000(00)	6593.953		
R37*	101(00)	000(00)	6589.826		
R38*	101(00)	000(00)	6591.144		
R39*	101(00)	000(00)	6592.811		

Table 3.10 - Assigned ¹³C₂H₂ Transitions

3.3.8 Ammonia

The Thomson and NEC lasers were used to record the spectra of $^{14}\text{NH}_3$ and $^{15}\text{NH}_3$. Unfortunately, due to time constraints no assignment of the recorded transitions have been made. However, the recorded spectra are shown in **Figure 3.20** through to **Figure 3.23**. A line listing of all the recorded transitions is given in Appendix 3. For $^{14}\text{NH}_3$, only a relative wavenumber calibration has been given, as time constraints have not allowed the spectral features to be identified. The reader is referred to work carried out by Lundsberg-Nielsen on $^{14}\text{NH}_3$ [28] for spectral identification. From the tuning characteristics of the diodes it is known that the spectrum in **Figure 3.20** is in the region $6436\text{-}6456\text{ cm}^{-1}$ and the spectrum in **Figure 3.21** is in the region $6570\text{-}6605\text{ cm}^{-1}$. A partial assignment of the stronger transitions of $^{15}\text{NH}_3$ has been carried out by Duxbury [29].

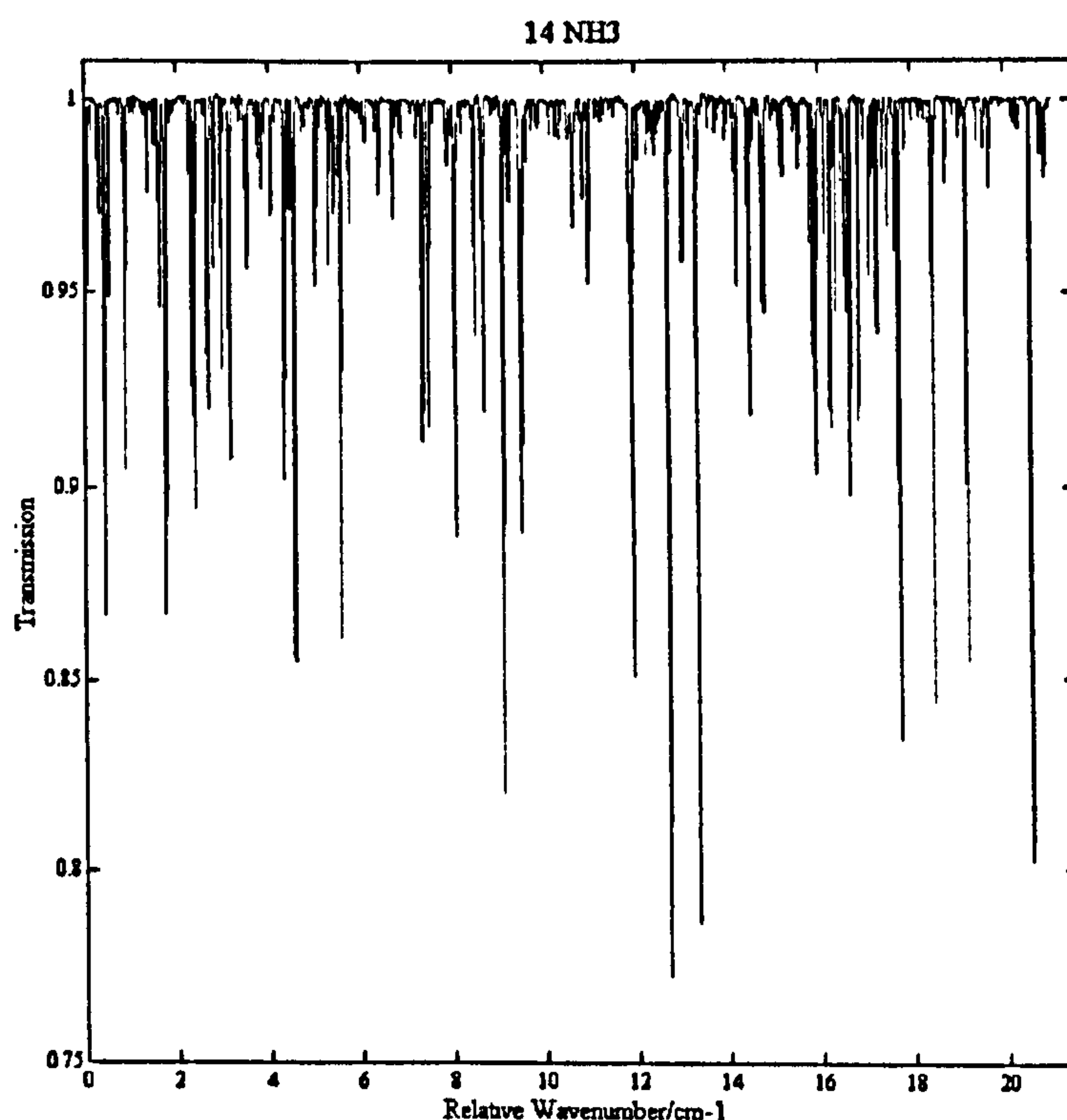


Figure 3.20 - $^{14}\text{NH}_3$ (sample pressure 0.47 Torr, pathlength 25.1 m)

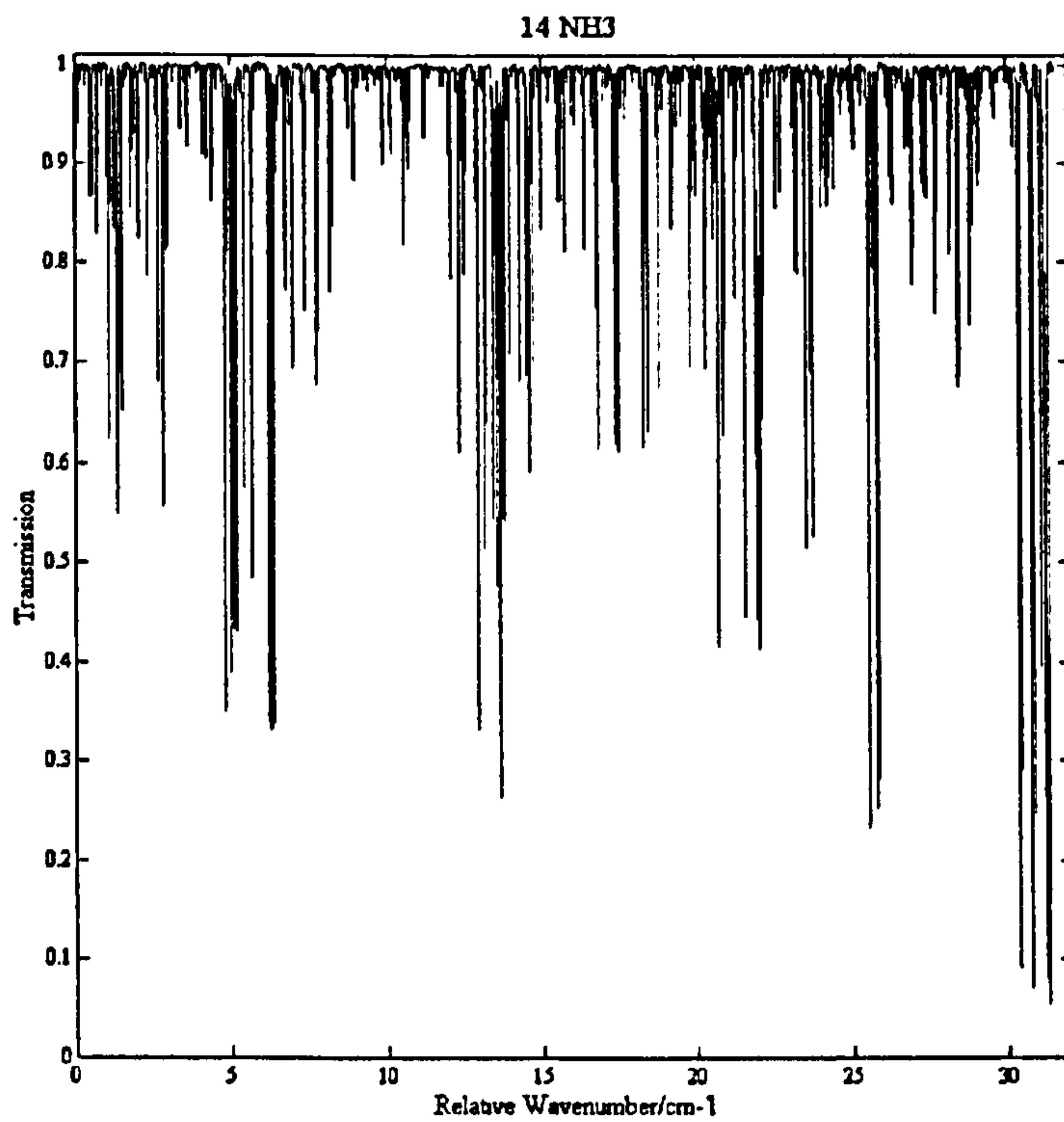


Figure 3.21 - ¹⁴NH₃ (sample pressure 0.47 Torr, pathlength 25.1m)

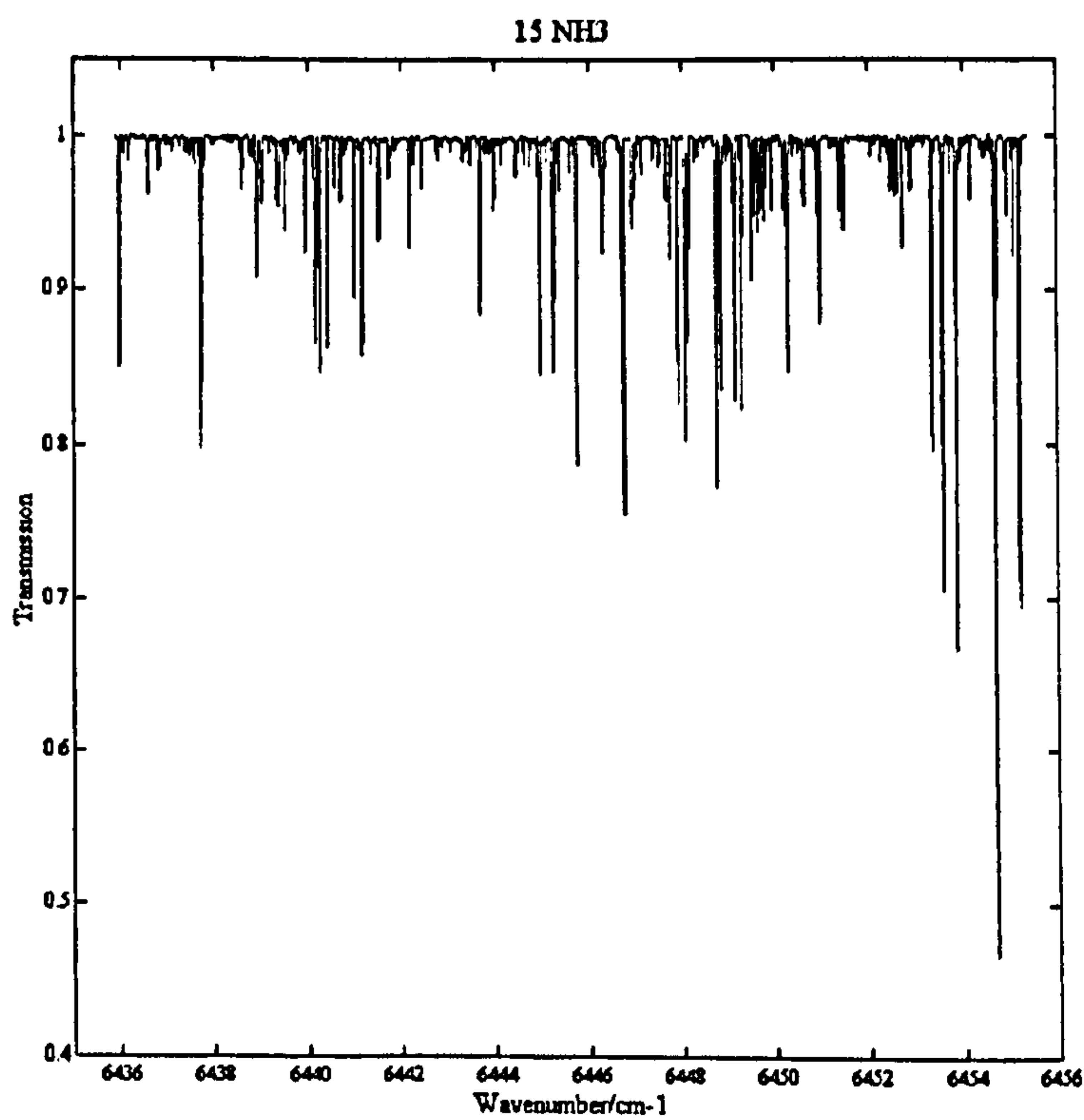


Figure 3.22 - ¹⁵NH₃ (sample pressure 0.47 Torr, pathlength 25.1 m)

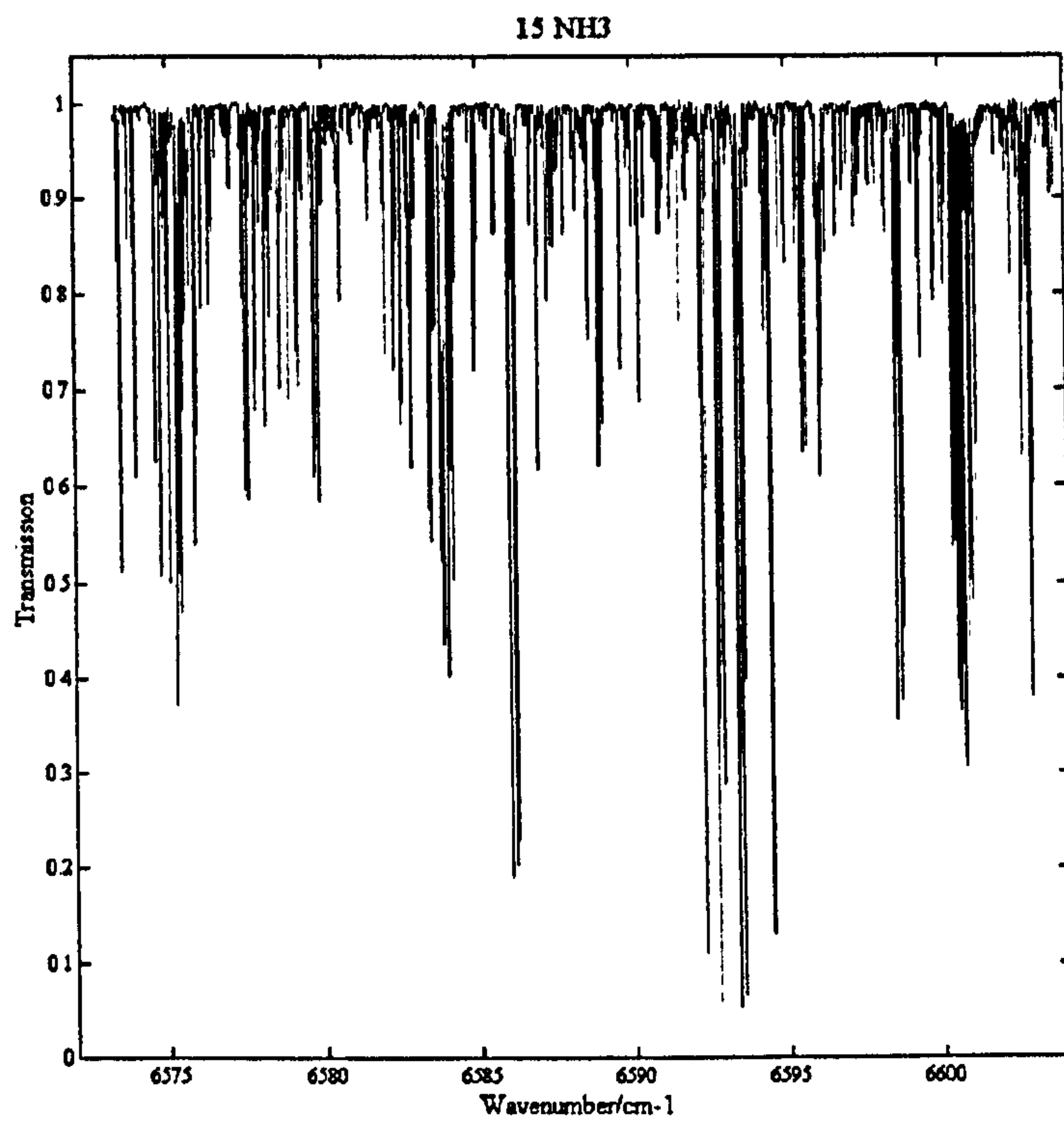


Figure 3.23 - $^{15}\text{NH}_3$ (sample pressure 1.02 Torr, pathlength 11.1 m)

Chapter 4 -

Multipass Cell

4.1 Introduction

From the Beer-Lambert law (section 2.1.7), increasing the pathlength increases the absorbance which will result in improved sensitivity for TDLAS measurements. In the mid-infrared where the fundamental rotational-vibrational transitions of most molecules occur, pathlengths of the order of 1 metre are required to obtain the necessary detection limits. However, the overtone transitions which are found in the near-infrared, are several orders of magnitude weaker and pathlengths up to several hundred metres are required. For a Balloon or aircraft based platform where weight and size restrictions are important design considerations, a folded light path must be used to obtain the necessary pathlength enhancement. For ground based measurements portability is not an overriding design consideration and pathlength can be enhanced using other techniques. The most common of these, involves open path measurements where the detector is placed some way from the laser source or a retroreflector is used to return the beam to the detector near the source. This technique has the advantage that it allows information about gas concentrations over large areas to be retrieved. However, the sensitivity of these instruments is severely hampered by pressure broadening and weather effects such as turbulence and scattering from dust particles. The HAWK [30] instrument uses this technique to determine methane emissions from landfill sites.

Another technique used to obtain high absorption pathlengths is to use optical fibres. Special D-shaped fibres allow light traveling through them to interact with the surrounding gas. The core in these fibres is extremely close to the surface allowing the laser electromagnetic field to extend beyond the fibre [31]. Again pressure broadening limits sensitivity. The fibre transmission is also susceptible to fluctuation if the fibre position is varied. It is therefore concluded that an evacuable folded light path is also the best option for a ground based TDLAS.

Historically, the optical arrangement most commonly used for multipass cells has been the White cell [32]. This arrangement consists of three concave spherical mirrors. A schematic of this is shown in **Figure 4.1**.

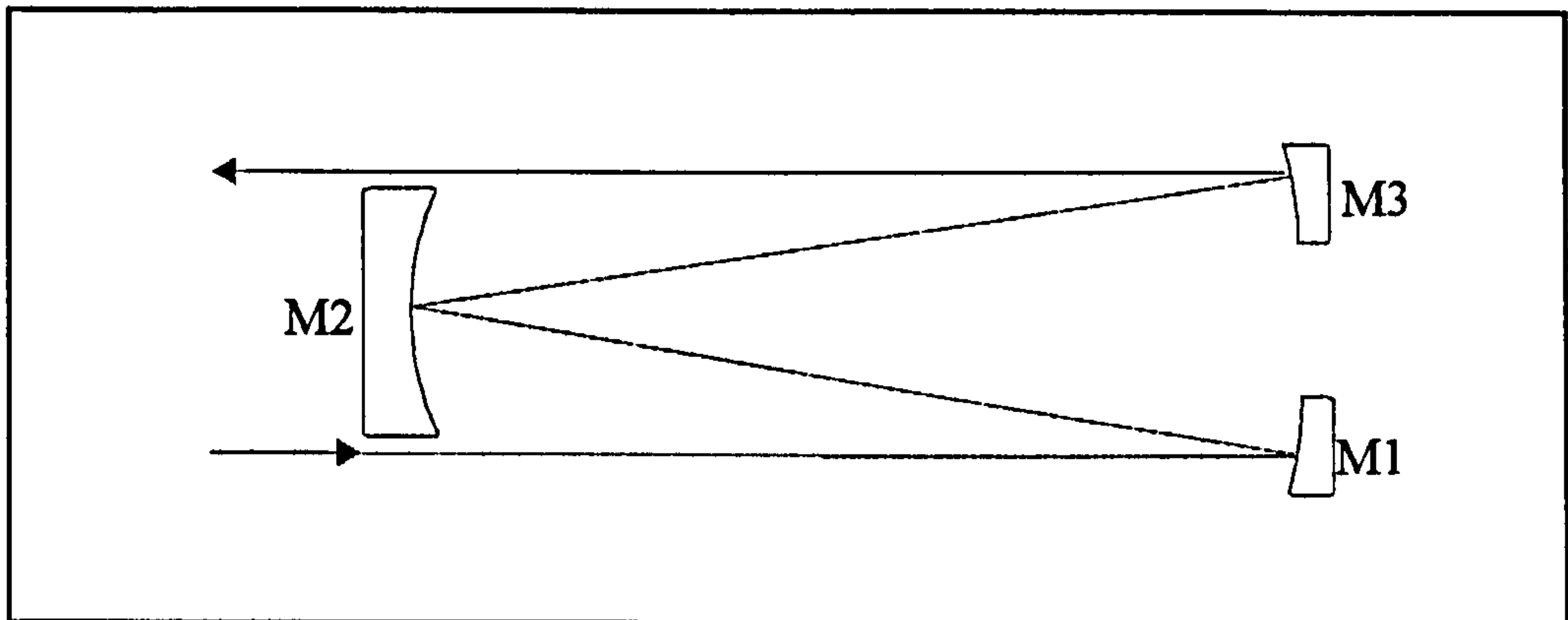


Figure 4.1 - Schematic of White Cell

The beam is focused at the entrance of the cell and is injected so that it hits mirror M1. The beam is then focused onto M2 and is then reflected to mirror M3. The beam can then exit the cell having made four passes as is shown in the schematic. However, mirrors M1 and M3 can be adjusted so that the beam is reimaged onto M2 and is again reflected onto M1. This will facilitate another 4 passes. This process can be repeated a number of times but is limited in practical terms by astigmatism in the mirrors and the mirror dimensions. The typical number of passes that can be supported within this type of cell is usually 32-40.

While the White cell has been successfully used in many instruments, including ATLAS [33], practical experience of the cell has highlighted several significant weaknesses. These include susceptibility to interference fringing, high alignment sensitivity and inefficient use of the mirror surfaces. For these reasons it was decided to investigate other optical arrangements for multipass use.

The literature shows that many other optical arrangements have been used for pathlength enhancement, including the Chernin cell [34], eight mirror multipass cell [6] and the Horn-Pimentel cell [35]. These, however, had not been extensively applied to trace gas monitoring and appeared to suffer many of weaknesses of the White cell. Most promising was the Herriott cell. This cell was successfully used, under hostile conditions, in the ALIAS instrument [11] and was found to be well suited to field applications.

The Herriott cell was first devised by Herriott *et al* [36] in 1964 and was initially intended for use as an optical delay line. It is only in recent years that it has been used as a multipass absorption cell [37].

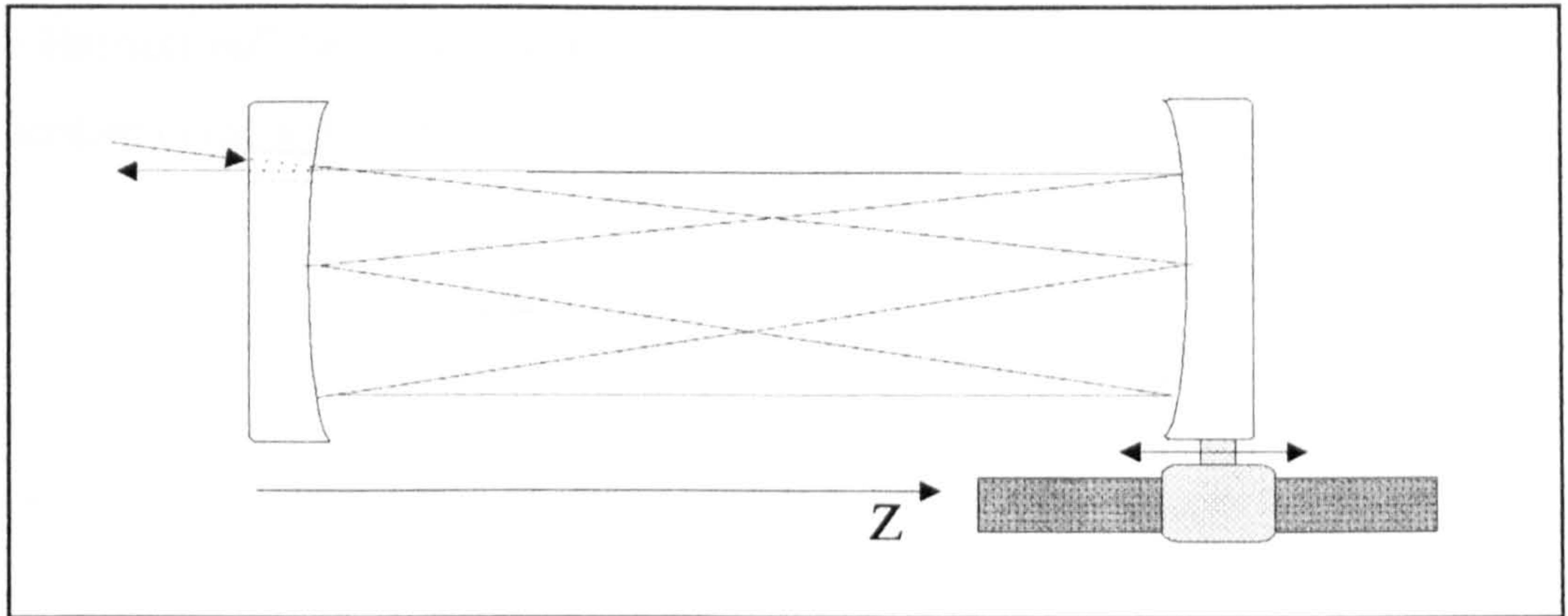


Figure 4.2 - Schematic of Herriott Cell

The cell consists of two identical coaxial spherical mirrors separated by some distance, d . The beam enters through a hole in the front mirror and propagates back and forth between the mirrors tracing out an ellipse on the mirror surface. The beam exits the cell at an angle to the input beam, and the cell can be treated simply as an optical element equivalent to a reflection from a convex mirror in the position of the coupling hole. This property makes it very easy design into optical systems. The number of passes is changed by adjusting the mirror separation. This arrangement is simpler than the White cell and uses the mirror surface more efficiently. A schematic of the cell is shown in **Figure 4.2**.

A set of mirrors were procured (Ealing Cat no. 35-9943) to investigate the suitability of the Herriott cell for use in a portable field instrument. The specification of this set was:

$$R = 60.0 \text{ cm}$$

$$D = 7.5 \text{ cm}$$

$$d_h = 0.4 \text{ cm}$$

$$h = 2.5 \text{ cm}$$

where R is the mirror radii of curvature, D is the mirror diameter, d_h is the diameter of the coupling hole and h is the off axis height of the coupling hole. The mirrors were coated in protected aluminium giving a quoted reflectivity of 95 % at 1550 nm.

These mirrors have also been used to validate theoretical and modelling predictions of the Herriott cell behaviour. The theoretical and modelling descriptions of the cell is described in the following section.

4.2 Herriott Cell

4.2.1 Introduction

As described in Section 4.1, the Herriott cell consists of two identical coaxial spherical mirrors separated by some distance, d . The beam enters off axially through a hole in the front mirror and propagates back and forth between the mirrors tracing out an ellipse on the mirror surface. The following sections describe the relevant theory and the matrix model that will allow the behaviour of a paraxial ray passing through the Herriott cell system to be predicted. These predictions are compared with experimental observations and will be used to assess the suitability of the cell for field applications.

4.2.2 Matrix Description of Optical Elements and Analytical Equations

It is well established that the propagation of a ray through optical media such as homogeneous and isotropic materials, thin lenses and curved reflecting surfaces can be described by simple 2×2 matrices. If beam propagation through a Herriott cell is considered, it is seen that it involves both passage through a homogeneous medium and reflection from a curved mirror. It is evident therefore that the Herriott cell can be described by a combination of these 2×2 matrices.

The matrix description of a spherical mirror of radius R , $\mathbf{R}(R)$, can easily be shown to be [38]:

$$\mathbf{R}(R) = \begin{bmatrix} 1 & 0 \\ -2/R & 1 \end{bmatrix} \quad (4.1)$$

The matrix for propagation through a homogeneous medium, $\mathbf{D}(d)$, is:

$$\mathbf{D}(d) = \begin{bmatrix} 1 & d \\ 0 & 1 \end{bmatrix} \quad (4.2)$$

In terms of ray optics the Herriott cell is equivalent to a series of equally spaced thin lenses as shown in **Figure 4.3**. The lenses are of focal length, f , and they are separated by a distance, d . The thin lens model of the Herriott cell will be used to analyse the behaviour of the paraxial ray .

Beam propagation through a thin lens can be described by the following matrix:

$$\begin{bmatrix} 1 & 0 \\ -1/f & 1 \end{bmatrix} \quad (4.3)$$

The equivalence of the two matrices in equations (4.1) and (4.3) can be shown by substituting the relationship $R = 2f$ into either one.

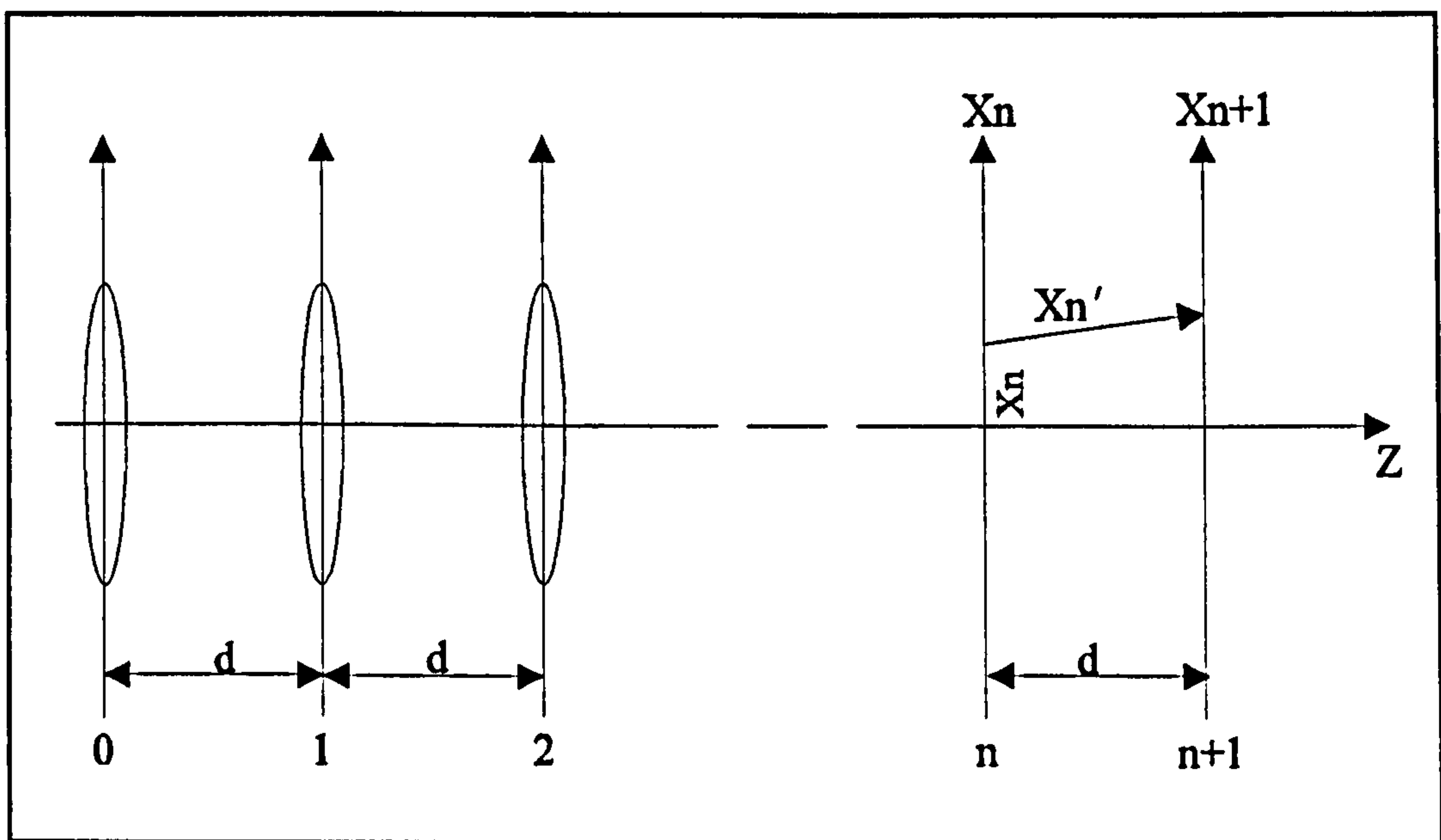


Figure 4.3 - Series of equally spaced thin lenses

Using a Cartesian co-ordinate system, a ray in the section between the n^{th} and $(n+1)^{\text{th}}$ lenses is described by the co-ordinates (x_n, y_n) of the point where it intersects the centre plane of the n^{th} lens, and by the slopes x_n' and y_n' just to the right of this lens. The traversal of the ray in the x and y directions can be treated independently, and as

such, the ray parameters, (x_n, x_n') , will only be considered in the analysis from this point onwards, but a set of identical relationships can be found for (y_n, y_n') .

The matrix relating the ray parameters just after the $(n+1)^{\text{th}}$ lens, (x_{n+1}, x_{n+1}') , to those just after the n^{th} lens, (x_n, x_n') , is the product of the free space and thin lens propagation matrices in equations (4.2) and (4.3), i.e.:

$$\begin{vmatrix} x_{n+1} \\ x_{n+1}' \end{vmatrix} = \begin{bmatrix} 1 & d \\ 0 & 1 \end{bmatrix} \begin{bmatrix} 1 & 0 \\ -1/f & 1 \end{bmatrix} \begin{vmatrix} x_n \\ x_n' \end{vmatrix} \quad (4.4)$$

or, in equation form:

$$\begin{aligned} x_{n+1} &= Ax_n + Bx_n' \\ x_{n+1}' &= Cx_n + Dx_n' \end{aligned} \quad (4.5)$$

where A,B,C and D are the elements of the matrix resulting from the multiplication of the two square matrices.

$$\begin{aligned} A &= 1 - \frac{d}{f} \\ B &= d \\ C &= -\frac{1}{f} \\ D &= 1 \end{aligned} \quad (4.6)$$

The first of (4.5) gives:

$$x_n' = \frac{1}{B} (x_{n+1} - Ax_n) \quad (4.7)$$

and thus:

$$x_{n+1}' = \frac{1}{B} (x_{n+2} - Ax_{n+1}) \quad (4.8)$$

Using the second of (4.5) in equation (4.8) and substituting for x_n' from equation (4.7) gives:

$$x_{n+2} - (A + D)x_{n+1} + (AD - BC)x_n = 0 \quad (4.9)$$

This is the difference equation governing the evolution of the ray through the lens wave guide. Using equation (4.6) it can be shown that $AD - BC = 1$. Consequently, equation (4.9) can be rewritten as:

$$x_{n+2} - 2bx_{n+1} + x_n = 0 \quad (4.10)$$

where:

$$b = \frac{1}{2} (A + D) = \left(1 - \frac{d}{2f}\right) \quad (4.11)$$

Equation (4.10) is the equivalent, in terms of difference equations, of the differential equation $x'' + Gx = 0$, whose solution is $x(z) = x(0)\exp[\pm\sqrt{Gz}]$. A solution of the form $x_n = x_0 e^{inq}$ is tried. Substitution of the solution into equation (4.10) gives:

$$e^{iq} = b \pm i\sqrt{1-b^2} = e^{\pm i\theta} \quad (4.12)$$

where:

$$\cos\theta = b = \left(1 - \frac{d}{2f}\right) \quad (4.13)$$

The general solution can be obtained by taking the linear superposition of $e^{+in\theta}$ and $e^{-in\theta}$. The solution takes the form:

$$x_n = x_{\max} \sin (n\theta + \alpha_x) \quad (4.14)$$

where:

$$(x_{\max})^2 = \frac{4f}{4f - d} (x_o^2 + dx_o x_o' + dfx_o'^2) \quad (4.15)$$

and:

$$\tan \alpha_x = \sqrt{\frac{4f}{d} - 1} / \left(1 + 2f \frac{x_o'}{x_o} \right) \quad (4.16)$$

A set of corresponding solutions can be obtained for the Y co-ordinates. These are:

$$y_n = y_{\max} \sin (n\theta + \alpha_y) \quad (4.17)$$

where:

$$(y_{\max})^2 = \frac{4f}{4f - d} (y_o^2 + dy_o y_o' + dfy_o'^2) \quad (4.18)$$

and:

$$\tan \alpha_y = \sqrt{\frac{4f}{d} - 1} / \left(1 + 2f \frac{y_o'}{y_o} \right) \quad (4.19)$$

It has been shown that the evolution of a paraxial ray through the Herriott cell can be described by sinusoidal equations in x and y. These equations will now be used to help predict the properties of the Herriott cell.

4.2.3 Condition for Re-entrance

A ray is said to be re-entrant if, after a given number of passes, it arrives back at its injection point. If the ray was not coupled out of the cell it would retrace its original path exactly. Re-entrant solutions are used exclusively when using the Herriott cell as a multipass arrangement and allow the beam to be coupled in and out of the cell through one hole. These solutions also offer significant advantages in terms of stability, as will be shown later. The conditions for a re-entrant beam can be obtained from the general solutions for the Herriott cell.

Expansion of equation (4.14) followed by substitution of equations (4.15) and (4.16) into it, gives:

$$x_n = x_o \cos (n\theta) + \sqrt{\frac{d}{4f-d}} (x_o + 2fx_o') \sin (n\theta) \quad (4.20)$$

Similarly for y_n :

$$y_n = y_o \cos (n\theta) + \sqrt{\frac{d}{4f-d}} (y_o + 2fy_o') \sin (n\theta) \quad (4.21)$$

If θ satisfies the condition:

$$n\theta = m\pi \quad (4.22)$$

where n and m are two integers that define the pass number and the pattern of transits through the cell. Substitution of equation (4.22) into equations (4.20) and (4.21) gives:

$$\begin{aligned} x_n &= x_o \\ y_n &= y_o \end{aligned} \quad (4.23)$$

In other words the beam will end up back at its starting point. The condition given in equation (4.22) is known as the re-entrance condition.

The integer, n , will be even as there must be an integral number of round trips if the beam is to enter and exit through a coupling hole in one mirror. Also there must be no common factors (other than two). If such factors exist the pattern will be degenerate and a pattern with lower pass number (reduced by the common factor) will be supported by the cell.

4.2.4 Conditions for Circular Spot Patterns

According to equations (4.14) and (4.17), the locus of the points on the mirror surface will generally lie on an ellipse. For a round mirror the spot pattern circumference will be at its maximum if the spots map out a circular pattern. Maximising the spot pattern circumference has several advantages. These include increasing the maximum number of passes that can be obtained from the mirrors and reducing the possibility of beam overlap, thus minimising possible fringing effects. For the spots to trace out a circular pattern then the following must be true [37]:

$$x_{\max} = y_{\max} \quad (4.24)$$

and:

$$\alpha_x = \alpha_y \pm \frac{\pi}{2} \quad (4.25)$$

Equation (4.24) and (4.25) impose certain conditions on the initial values x_0 , x_0' , y_0 and y_0' . Prescribing x_0 and y_0 , allows the radius of the spot pattern, A , and the slopes x_0' and y_0' to be determined from equation (4.14) and (4.20). Assuming that $x_0 = 0$ and that y_0 has some arbitrary value dictated by the mirror diameter, then the required input slopes for a circular spot pattern are:

$$x_0' = \frac{y_0 \sqrt{\frac{4f-d}{d}}}{2f} \quad (4.26)$$

and:

$$y_o' = \frac{\sqrt{y_o^2 - (x_o'/d)^2} - y_o}{d} \quad (4.27)$$

The pattern radius, A, is defined by the co-ordinate y_o , i.e.:

$$A = x_{\max} = y_{\max} = y_o \quad (4.28)$$

To test the validity of these equations, the Herriott cell was set up over a range of different mirror separations and a HeNe laser was directed into the cell with the required injection slopes. In each case a circular spot pattern was obtained. A beneficial consequence of knowing the injection slopes for circular spot patterns is that they can be used to ensure both mirrors are parallel to each other. An elliptical pattern would result from non parallel mirrors.

4.2.5 Maximum Number of Passes

The maximum number of passes that can be obtained from a given set of mirrors is limited in practical terms by the requirement that neighbouring spots must be separated by the diameter of the coupling hole. If this is not the case then more than one beam will be able to exit through the hole resulting in fringing effects being observed at the detector.

For a circular spot pattern the above criteria will be met when:

$$\frac{2\pi x_o}{N} \geq d_h \quad (4.29)$$

where d_h is the diameter of the hole, x_o is the distance from the centre of the mirror to the coupling hole and N is the number of spots on the mirror. Rearranging (4.29) gives:

$$N \leq \frac{2\pi x_o}{d_h} \quad (4.30)$$

This equation tells us the maximum number of spots that can be obtained on one mirror. The maximum number of passes will just be twice this amount (i.e. N spots on each mirror will give 2N passes). The maximum number of passes, n, is therefore:

$$n \leq \frac{4\pi x_o}{d_h} \quad (4.31)$$

Using the cell parameters of the Ealing mirrors, equation (4.31) predicts that the maximum number of passes that can be supported within this cell will be 64. Experimentally it was possible to get a greater number of passes (up to 15) out the cell, but this could only be achieved by sending the beam through the coupling hole off centre. This effectively reduced the coupling hole diameter. It is therefore concluded that the equation for the maximum number of passes is in good agreement with the experimental observations.

4.2.6 Focusing Properties of the Herriott Cell

In addition to being steered, laser beams are also focused after each bounce. If the beams are to have the same diameter after each pass the input beam must be properly mode matched to the cell.

Considering the ray as a propagating Gaussian beam, Boyd and Kogelnik [39] have shown that if the beam is properly mode matched, the spot size at the mirror surfaces will be:

$$\omega_{(1,2)} = \left(\frac{\lambda d}{2\pi n} \right)^{1/2} \left[\frac{2R^2}{d(R - d/2)} \right]^{1/4} \quad (4.32)$$

The minimum spot size will occur at the centre of the cell and is given by:

$$\omega_o = \left(\frac{\lambda}{\pi n}\right)^{1/2} \left(\frac{d}{2}\right)^{1/4} \left(R - \frac{d}{2}\right)^{1/4} \quad (4.33)$$

If the input beam is not properly mode matched to the cell then the spot size and the focal plane will fluctuate from bounce to bounce. Taking the example of the Ealing mirrors, equations (4.32) and (4.33) predict that the spot diameter on the mirror surfaces, $2\omega_{(1,2)}$, will be 0.98 mm while the beam diameter at the focal plane will be 0.76 mm. It was assumed that the mirrors had a 50 cm separation and a laser source operating at 1550 nm was used.

The value of R (for a given d) for which the mirror spot size is a minimum, is found to be R=d. When this condition is satisfied the system is known as a confocal symmetrical resonator. The spot size at the mirror surfaces becomes:

$$\begin{aligned} \omega_{(1,2) \text{ conf}} &= \sqrt{2} \left(\frac{\lambda d}{2\pi n}\right)^{1/2} \\ \omega_{(1,2) \text{ conf}} &= \sqrt{2} \omega_o \text{ conf} \end{aligned} \quad (4.34)$$

Again assuming a laser wavelength of 1550 nm, the spot diameter at the mirror surfaces is 0.97 mm, while the beam diameter at the focal plane will be 0.76 mm.

These calculations show that the theoretical spot sizes on the mirror surfaces are significantly smaller than the coupling hole diameter used in the Ealing mirrors (4mm). As such this mirror set represents a conservative design, however, the spot sizes will always be larger than the theoretical predictions due to non ideal effects such as scattering and diffraction.

While reducing the coupling hole diameter, would allow more efficient use of the mirrors it would also compromise other more important design criteria such as minimising fringe levels and reducing the spacing sensitivity. Practical experience has shown that a hole diameter which is at least two to three times the spot diameter should be used.

4.2.7 Mode Stability of the Herriott Cell

The ability of an optical resonator to support low (diffraction) loss modes depends on the mirrors separation, d , and their radii of curvature, R . The ratio of the mirror spot size at a given d/R to its minimum confocal ($d/R = 1$) value is given by the ratio of equations (4.32) and (4.34) [38]:

$$\frac{\omega_{(1,2)}}{\omega_{(1,2) \text{ conf}}} = \left[\frac{1}{d/R (2 - d/R)} \right]^{1/2} \quad (4.35)$$

The ratio is plotted out in **Figure 4.4**.

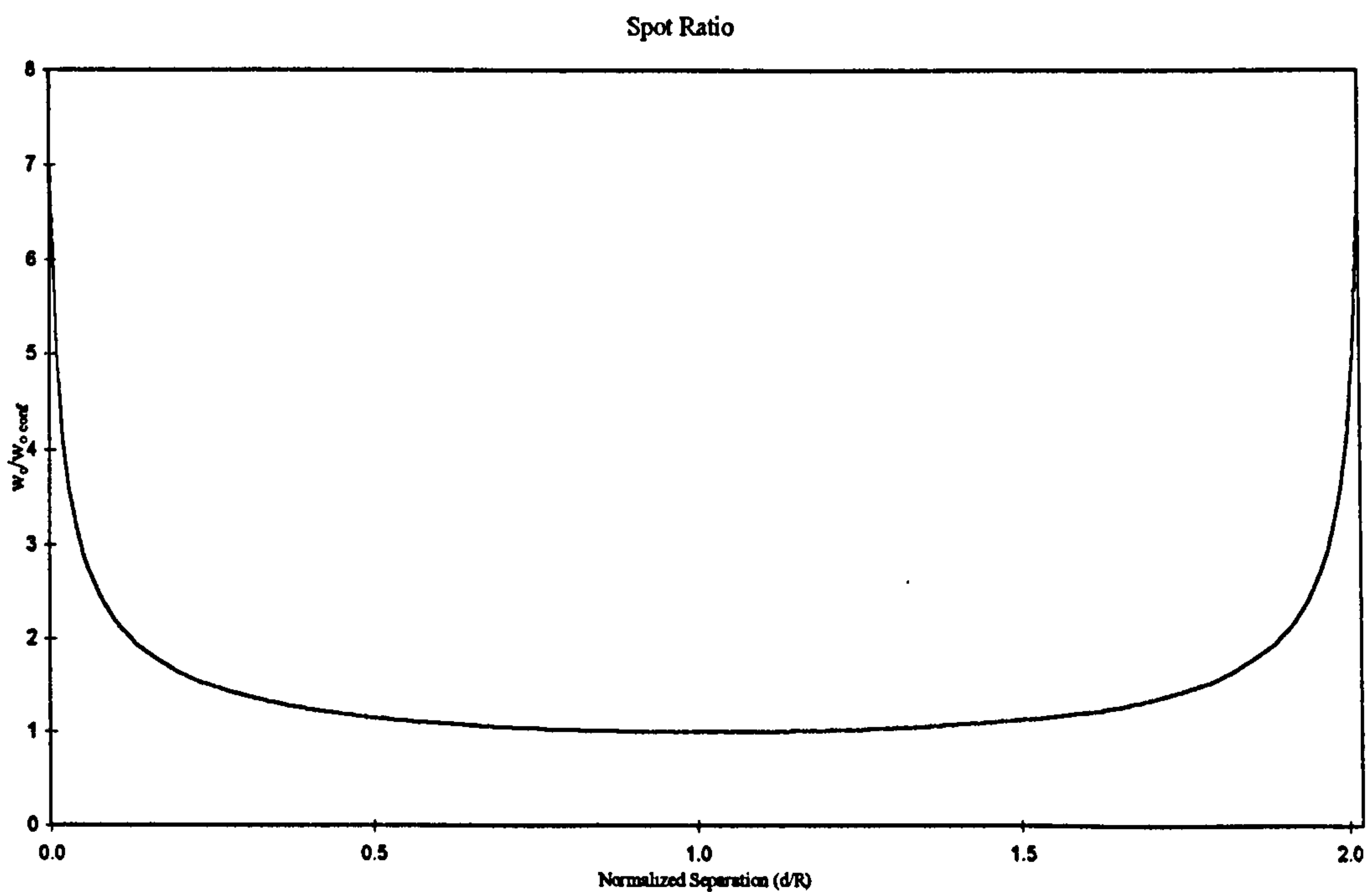


Figure 4.4 - Spot Size Ratio

The ratio becomes infinite at $d = 0$ and $d = 2R$. The diffraction losses under these conditions would be extremely high, as most of the beam energy would miss the cell mirrors. The mirror system will only be stable if the separation is bound as follows:

$$0 < d < 2R \quad (4.36)$$

This is known as the stability condition. However, for practical use the cell spacing is kept within the following range:

$$\frac{R}{2} < d < \frac{3R}{2} \quad (4.37)$$

In this region the maximum spot size is $\sim 15\%$ greater than the minimum confocal value. This ensures that fringing due to beam spot overlap does not become a problem.

4.2.8 Spacing Tolerance

If the mirror spacing is changed from its exact re-entrant position the last spot will be swept across the coupling hole. The tolerance for spacing changes i.e. the range over which the beam still exits through the hole can be determined if we follow McManus [40] and define the transverse spot velocity, $v_s = dS/dd$, with S the lateral position of the last spot on the input mirror. The spacing tolerance will be given by the coupling hole diameter divided by the spot velocity i.e. :

$$\Delta d = d_h/v_s. \quad (4.38)$$

We can use equation (4.14) to determine the (vector) spot velocity:

$$\begin{aligned} v_s = & \quad nx_o \cos(n\theta) \cdot \frac{(1/R)^{1/2}}{(2d - d^2/R)^{1/2}} \hat{x} \\ & + ny_o \cos(n\theta) \cdot \frac{(1/R)^{1/2}}{(2d - d^2/R)^{1/2}} \hat{y} \end{aligned} \quad (4.39)$$

For a nearly re-entrant beam $\cos(n\theta) \approx \pm 1$. Further, if the cell is close to confocal, $d \approx R$ and equation (4.39) simplifies to:

$$v_s \approx \frac{n}{d} (x_o^2 + y_o^2)^{1/2} \quad (4.40)$$

Assuming $x_o = 0$, as in the case of a circular spot pattern, equation (4.40) reduces to:

$$v_s \approx \frac{ny_o}{d} \quad (4.41)$$

In other words the spot velocity increases proportionally with pass number, n , and the input co-ordinate, y_o , but is inversely proportional to the mirror spacing, d .

Taking the example of a 50 pass Herriott cell separated by 56 cm with $y_o = 3$ cm, the spot velocity will be ~ 2.7 . This means that a change in separation of 1 mm will cause the lateral position of the last spot to move by 2.7 mm. Assuming a coupling hole diameter of 4 mm, the spacing tolerance would be 1.5 mm.

To verify the equation for spacing tolerance, the Herriott cell spacing was adjusted with a micrometer and the distance over which the beam still exited through the coupling hole was recorded. The spacing tolerance for the pattern discussed above was 1.60 mm. The discrepancy between the measured and predicted tolerances arose because the cell was not at confocal spacing. If no confocal assumptions are made then the predicted spacing tolerance (equation (4.39)) become 1.49 mm. This is a far better agreement with the observations.

Such tolerances should be easily achievable in a field instrument.

4.2.9 Tilt Sensitivity

Trutna and Byer [37] have carried out a simple geometric analysis to determine how the reflex spot co-ordinates change when either the front or back mirror is tilted. If the front mirror is rotated by an angle Φ_1 about the Y axis, the change in the position of the n^{th} spot on the front mirror is given by:

$$\Delta x_n(\Phi_1) = \frac{R}{2 + d/R} [\text{Cos}(n+1)\theta - \text{Cos}\theta] \Phi_1 \quad (4.42)$$

If instead the back mirror is rotated by an angle Φ_2 about the Y axis, the change in the position of the spots on the front mirror is:

$$\Delta x_n(\Phi_2) = \frac{R}{2 + d/R} [\text{Cos}(n\theta) - 1] \Phi_2 \quad (4.43)$$

If the re-entrant condition ($n\theta = m\pi$) is substituted into both of these equations, the bracketed expressions in each become zero. In other words the position of the exit spot will not change when either mirror is tilted. This is an important result that confirms the inherent stability of re-entrant solutions supported within the Herriott cell. A set of similar relationships can be found for the change in the spot positions on the back mirror, while identical relationships for rotation about the X axis can also be obtained.

This predicted insensitivity to tilt was validated experimentally, however, although the spot position in the plane of the coupling hole does not change, the slopes of the exit beam does. As a consequence, the position of the exit beam outside the cell changes. Using equation (4.42) it is predicted that if the front mirror is tilted through an angle, Φ_1 , the exit beam slope will change by $2\Phi_1$. This was confirmed experimentally and is consistent with the cell being modelled as a single convex mirror at the plane of the coupling hole.

The analytical equations have been used to help understand much of the Herriott cell behaviour. A computer model of the cell based on a matrix description of optical elements has also been developed. This model has allowed further investigation into the Herriott cell behaviour. This model will now be discussed.

4.2.10 Computational Model of The Herriott Cell

The behaviour of the Herriott cell has been explored by direct computation using a program written in the MATLAB language at the University of Strathclyde. A detailed description and listing of this program can be found in Appendix 2.

If we consider one traverse of the Herriott cell, it involves propagation through free space and reflection off one mirror. As described in Section 4.2.2, the matrix for one traverse, $C(R,d)$ is the product of the free space propagation matrix, $D(d)$, and the reflection matrix, $R(R)$.

$$C(R,d) = R(R)D(d) \quad (4.44)$$

The location and slope of a paraxial ray after n traverses, Z_n , is just the product of the matrix for one traverse, $C(R,d)$, raised to the power n , and the initial vector, Z_0 , i.e.:

$$Z_n = C^n Z_0 \quad (4.45)$$

where Z_0 is the vector:

$$Z_0 = \begin{vmatrix} x_0 \\ x_0' \\ y_0 \\ y_0' \end{vmatrix} \quad (4.46)$$

and Z_n is:

$$Z_n = \begin{vmatrix} x_n \\ x_n' \\ y_n \\ y_n' \end{vmatrix} \quad (4.47)$$

This model has been used to confirm the analytical expressions for spacing and tilt sensitivity derived earlier, and to allow spot patterns that are best suited to TDLAS measurements to be chosen.

4.2.11 Maps of Re-entrant Solutions

The model can be used to determine the number of passes in the cell at any given mirror spacing. This is achieved by stepping through values of n for a given separation until a re-entrant solution is found, i.e. the co-ordinates of the n^{th} spot fall within the coupling hole radius. Changes in separation alter the angular advance, θ , on each reflection, allowing a different solution to be supported. If this process is repeated for a range of different separations, a map of re-entrant solutions is generated. The map is independent of choices of specific cell parameters and proves to be useful tool in the design and set up of the Herriott cell.

Due to the finite sizes of the beam, coupling hole and mirror, the number of passes, n , that can be supported within the cell has been limited to a maximum value of 110.

Figure 4.5 shows how the pass number changes with the normalised separation (R/d). To make these calculations, it was assumed that the mirror diameter was 7.5 cm with a 4 mm diameter coupling hole placed 3.5 cm above the centre of the mirror.

If the region marked #1 on **Figure 4.5** is examined, it is seen that at a normalised separation of 0.937, 50 passes are supported within the cell. If a 50 pass Herriott cell with 50 cm spacing needs to be designed, then the required radii of curvature would be, $R = 50/0.937 = 53.67$ cm. If on the other hand the mirrors were required to be separated by 78cm, the required radii would be 83.24 cm. In other words, a specific solution can be obtained at any given separation by changing the radii of curvature. This is an important result, as the converse is also true, i.e. a specific solution can be found for a given radii of curvature by changing the mirror separation. This means that any error in the radii of curvature can be compensated for by changing the mirror spacing, allowing the tolerances in the precision of manufacture to be kept relaxed and the cost of manufacture to be reduced.

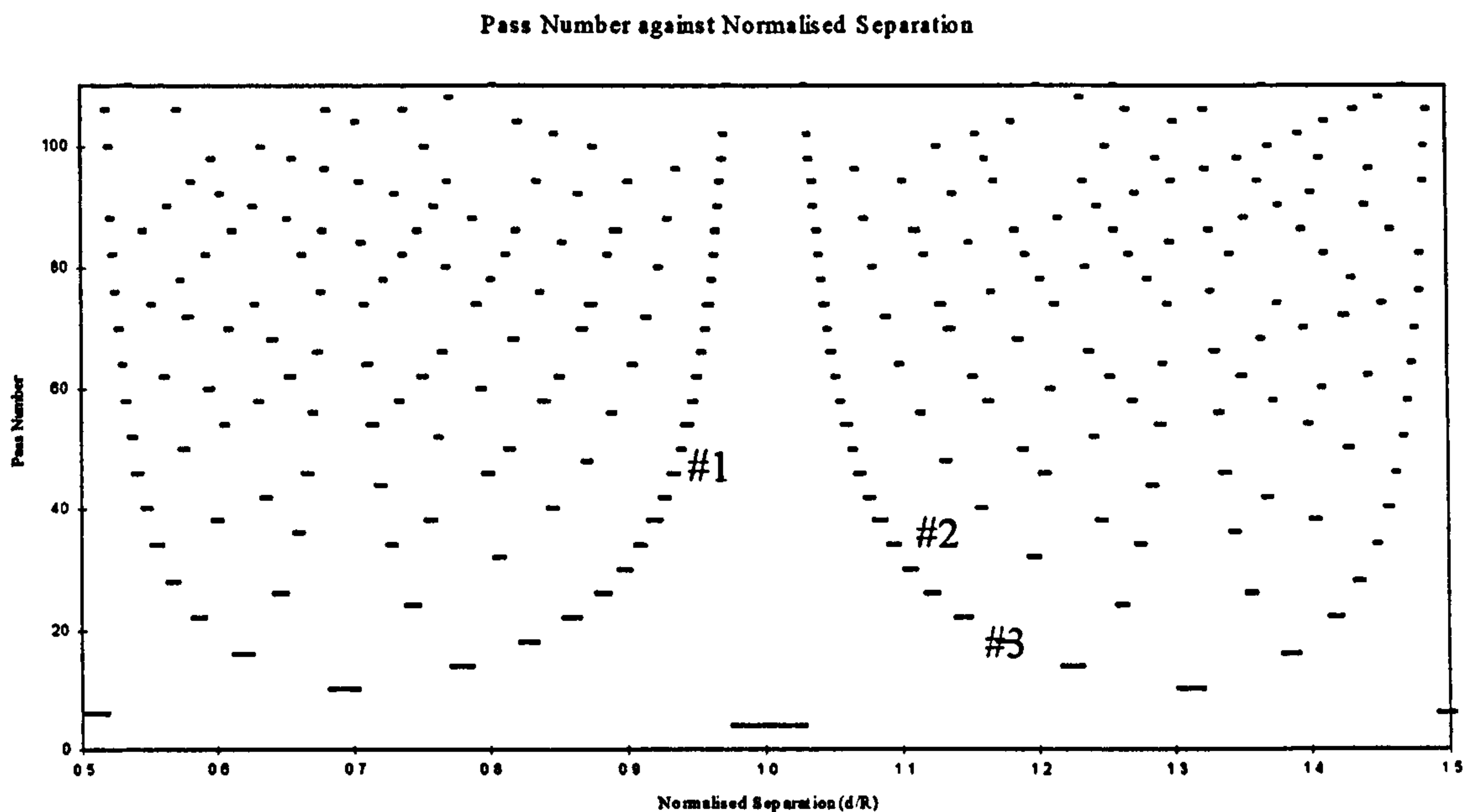


Figure 4.5 - Map of Re-entrant Solutions

This result also has another important consequence. If both the mirrors and the material separating them are made of the same material, any changes in ambient temperature will cause the cell to expand/contract isotropically. Consequently, any change in the separation will be matched by a corresponding change in the mirror radii of curvature, allowing the same re-entrant solution to be supported within the cell. This has been validated using the model and is of great benefit for field applications where ambient temperature variations up to $\pm 30^{\circ}\text{C}$ can be expected. In a White cell such temperature variations would significantly affect the alignment, resulting in ambiguity in the number of passes supported within the cell. In the Herriott cell no such ambiguity will exist, allowing the effect of temperature variations on the pathlength to be determined precisely. This behaviour was confirmed in Chapter 7, where successful operation of a Herriott cell at temperatures as low as 77K was achieved.

Figure 4.6 shows the spot pattern obtained for a Herriott cell giving 50 passes at 50 cm separation. The small circle represents the coupling hole. The pass numbers for the front mirrors are shown in Figure 4.7. All the even pass numbers are located on the front (coupling hole) mirror with the odd numbered passes on the back mirror.

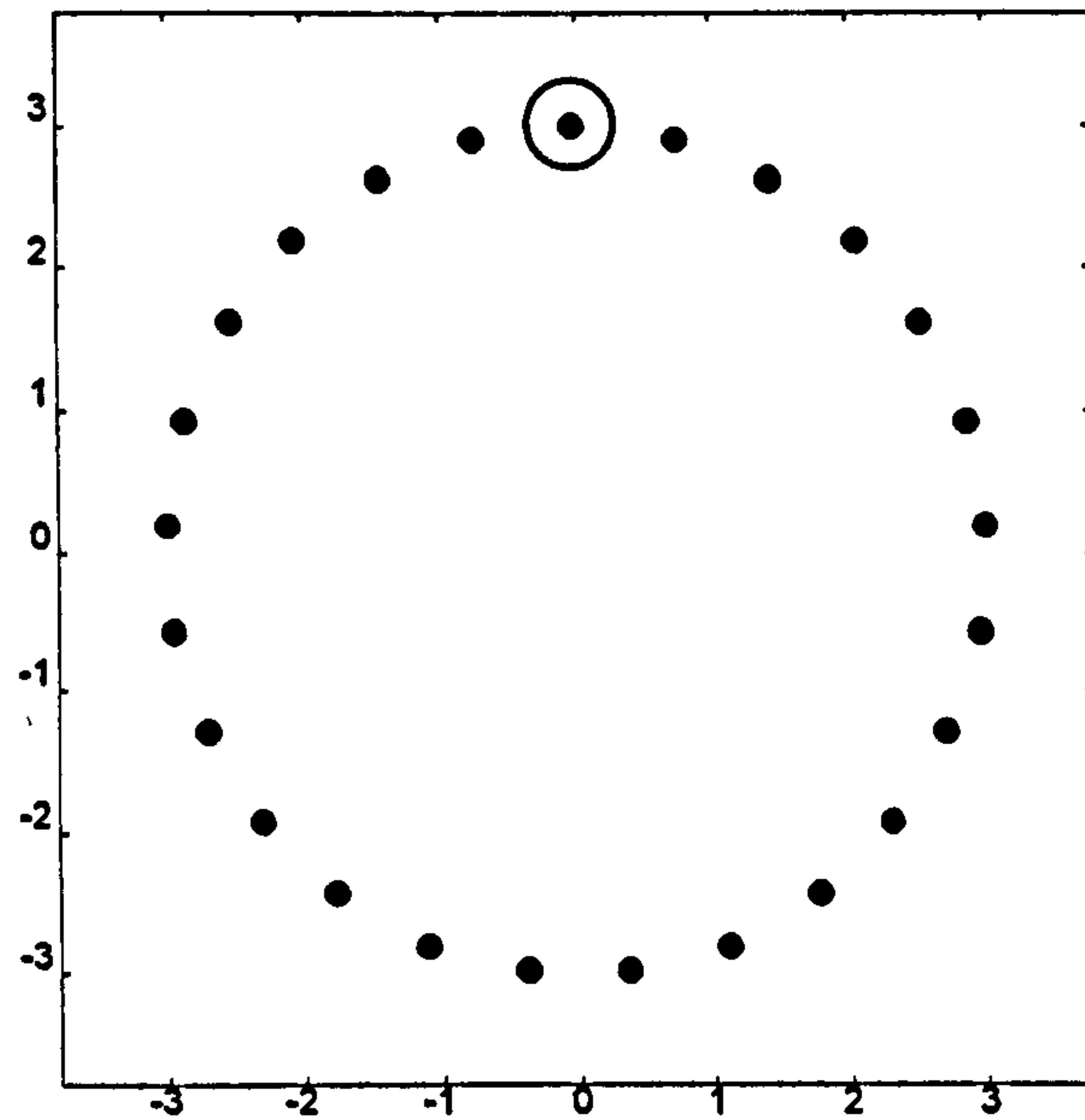


Figure 4.6 - 50 Pass Spot Pattern , $d = 50\text{cm}$

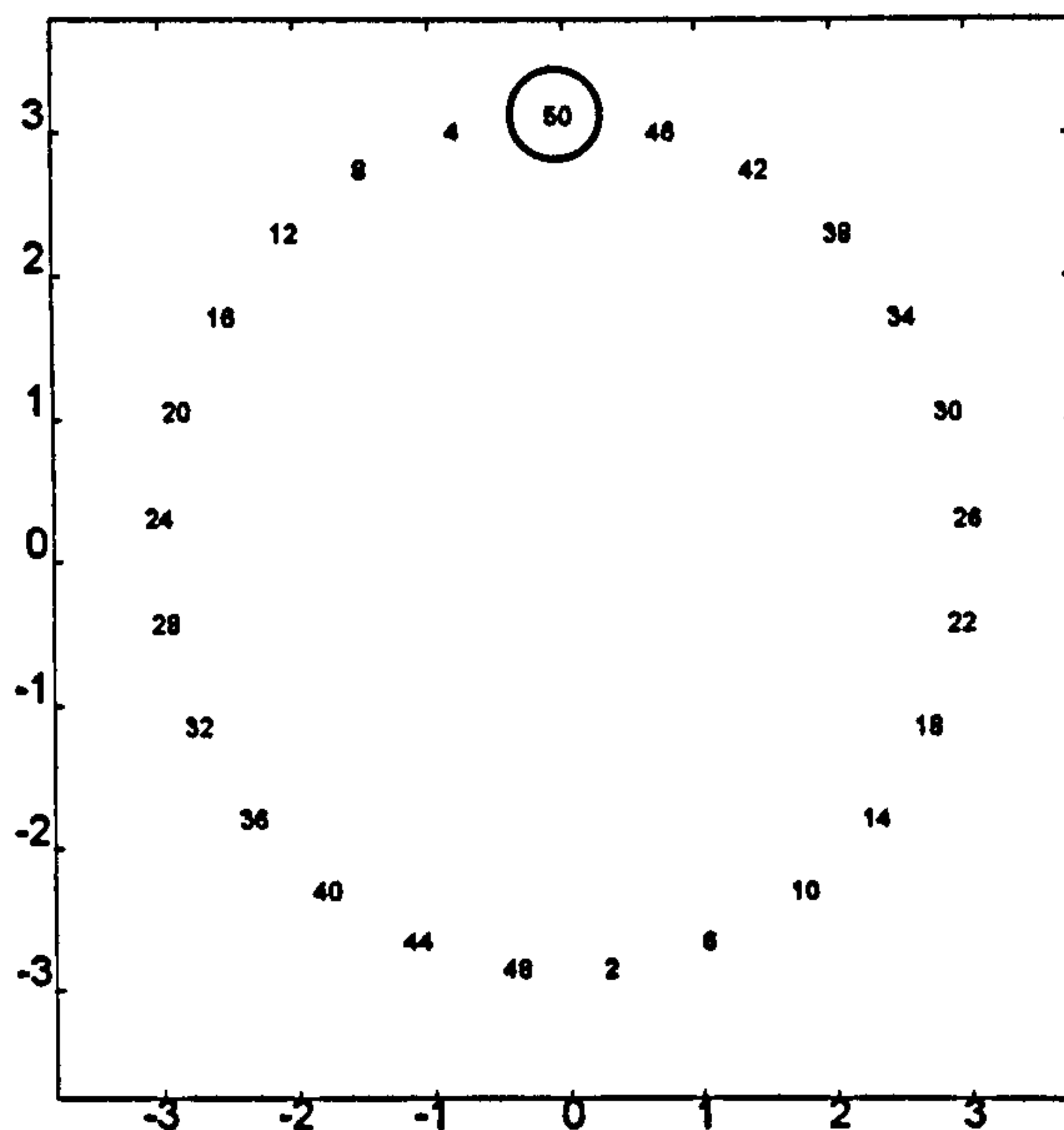


Figure 4.7 - Even Pass Numbers on Front Mirror

The modelled spot patterns highlight that the reflex spots only cover a very small area of the mirror. Thus, although more efficient than the White cell in terms of the number of passes that can be supported within a cell, a large percentage of the mode volume is still not used.

4.2.12 Modelled Spacing Tolerance

If Figure 4.5 is examined more closely, it is evident that as the pass number increases, the distance over which the beam will still exit the coupling hole gets correspondingly smaller. This distance was defined in Section 4.2.8 as the spacing tolerance. The regions #2 and #3 in Figure 4.5 occur at similar normalised separations, but #3 has twice as many passes as #2. A close up of this region is shown in Figure 4.8.

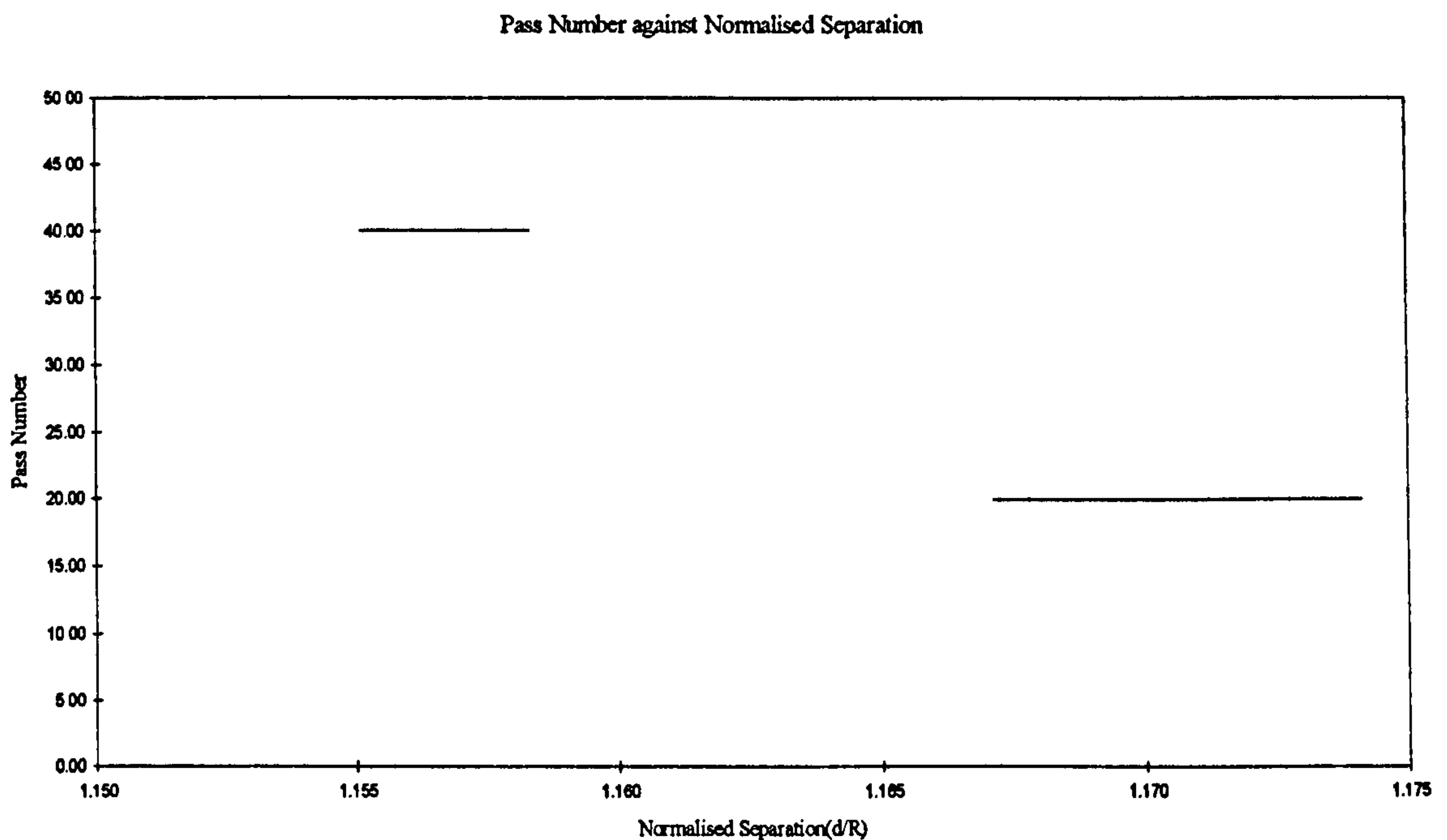


Figure 4.8 - Expanded Region of Figure 4.5

The spacing tolerance for the two regions, as determined from Figure 4.8, are 1 mm and 2 mm respectively. Thus the spacing tolerance, to a first approximation is proportional to the pass number. The analytical equation for spacing tolerance (equation (4.41)) given in Section 4.2.8, predicts tolerance values of 1.02 mm and 2.01 mm. It is clear that there is good agreement between the modelled results, the derived analytical equations and the experimental observations

4.2.13 Modelled Tilt Tolerance

Tilt can be accounted for in the model by adding a constant to the slopes, (α_x', α_y') , at each reflection off the tilted mirror. If for example the back mirror was tilted by the angle Φ_1 in the x direction, the beam would undergo an additional angular deflection of $2\Phi_1$ on each reflection off that mirror. **Figure 4.9** and **Figure 4.10** show the spot pattern obtained when there is no tilt and when there is a 0.5° tilt in the x direction.

It is evident that while the shape and orientation of the spot pattern as a whole has changed, the order of appearance of the spots has not. More significantly the coordinates of the exit spot have remained unchanged. In other words the position of the exit spot on the front mirror is independent of tilt. The model is therefore in good agreement with the analytical expressions and experimental observations (Section 4.2.9). The model was also used to determine how the exit beam slopes changed when either mirror was tilted. As expected, when the front mirror is tilted through an angle Φ_1 , the exit beam slope will change by $2\Phi_1$.

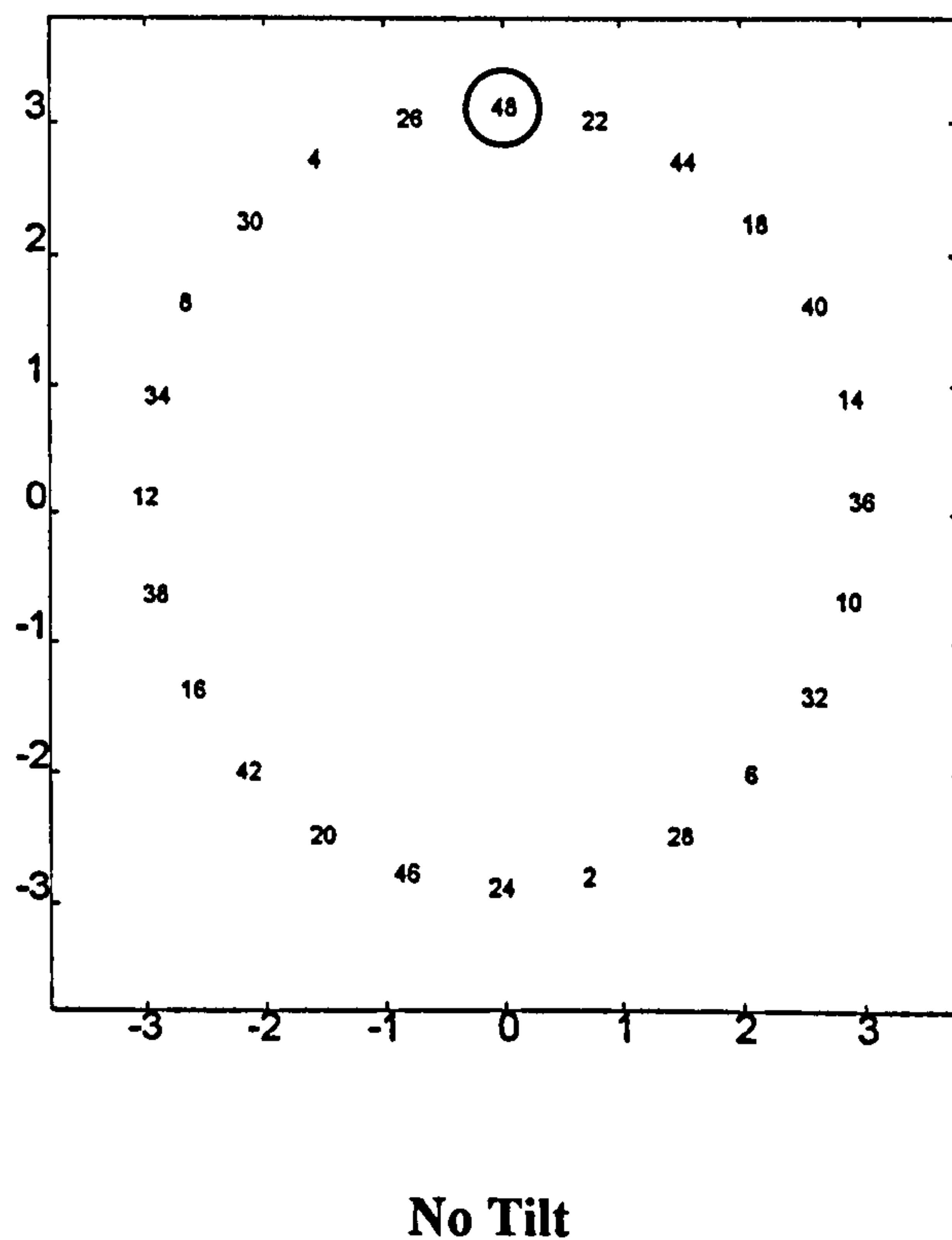
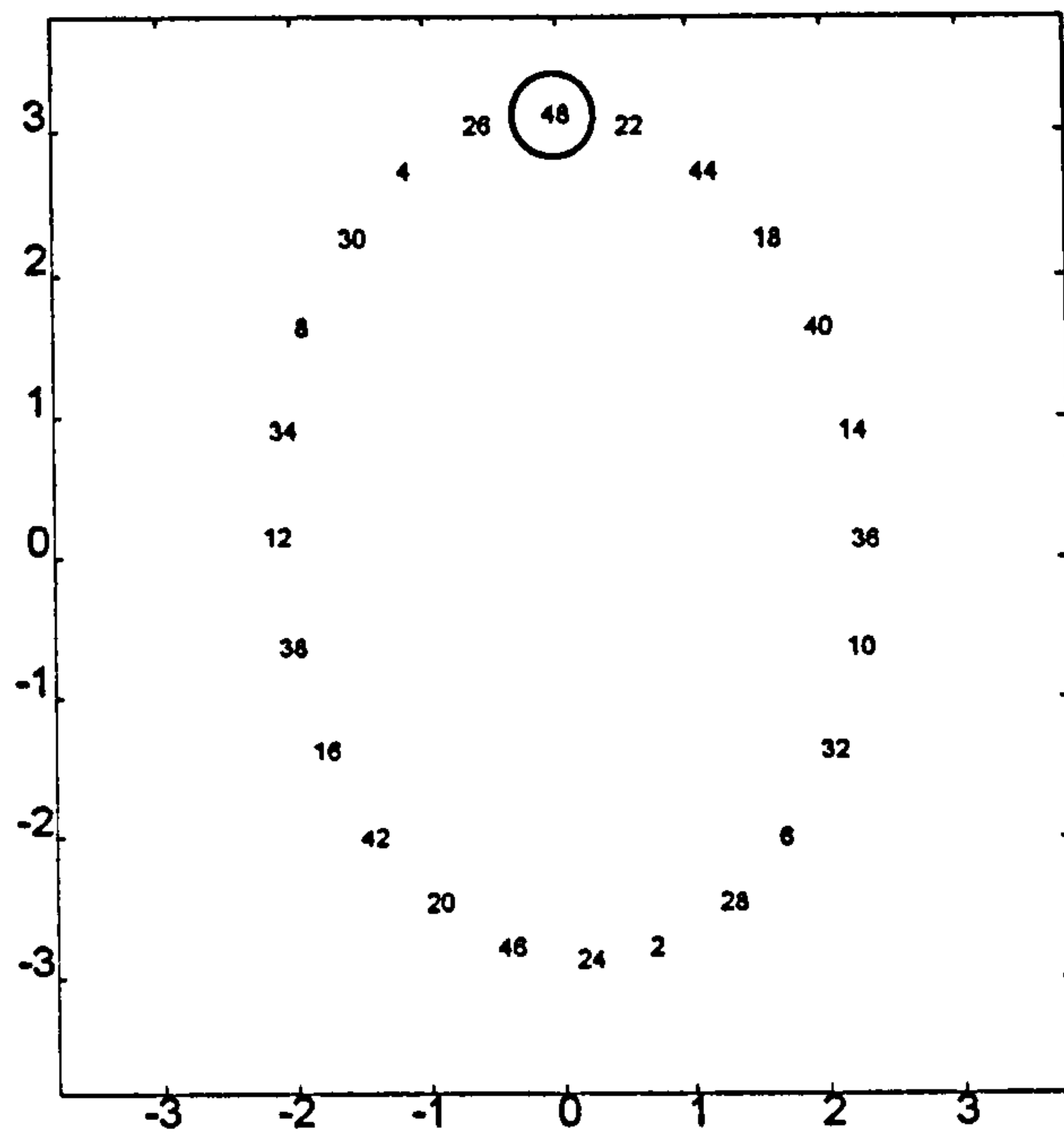


Figure 4.9 - Spot Pattern without a 0.5° tilt in the x direction



0.5° Tilt

Figure 4.10 - Spot Pattern with a 0.5° tilt in the x direction

4.2.14 Best Choice Spot Patterns

In TDLAS measurements one of the main factors limiting sensitivity is optical interference fringes. These are caused by interference at the detector of coherent beams that have travelled different pathlengths. One of the main sources of such fringing will be the multipass cell. It will be discussed how, through a suitable choice of spot pattern, the elimination of such fringing can be made significantly easier.

In a Herriott cell, the most important source of fringing will be the spillover of light from the beam spots that neighbour the coupling hole on the input mirror. Ideally it should be possible to make the neighbouring spot spacing large enough so that no spillover will occur, however, imperfections on the mirror surface will produce a cone of near forward scattered light surrounding the main beam, making the beam diameter larger than the theoretical calculations (Section 4.2.6). Also, poor coupling of the beam

into the cell will cause diffraction, again leading to a larger than expected beam diameter. Practically, the non ideal effects mentioned above and the requirement for low volume, high pass number cells make it impossible to totally eliminate fringing. Therefore, other techniques to *remove* the fringes must be used. These techniques, including piezo cell mirror dithering, digital filtering or the use of a Brewster plate spoiler are discussed in detail in Section 5.4, but common to all is the requirement that the Free Spectral Range (FSR) of the fringes must be considerably different from the width of the absorption feature that is being recorded.

For a Herriott cell the FSR is given by [40]:

$$\text{FSR} = \frac{c}{\Delta d} \quad (4.48)$$

where c is the speed of light and Δd is the path difference between the interfering beams. Using equation (4.48) it can be seen that in order to obtain spot patterns that will give narrow FSR fringes, the path difference between the exit spot and either of the two neighbouring spots (S1 and S2) should be as large as possible. This will be the case when both S1 and S2 correspond to spots nearly midway through the beam orbit. Kebabian and McManus [41] have shown that re-entrant solutions can be grouped into families that have a fixed relationship between the pass number n and the integer m . **Figure 4.11** shows the re-entrant solutions against the normalised separation and the groups of families to which they belong. It is found that each family has a distinct relationship between the exit spot number and the two nearest neighbour spot numbers, S1 and S2. **Table 4.1** lists the families and the spot number relationships. Of all the families listed, only the $2m \pm 4$ family has both its neighbouring spots close to midway through the beam orbit. This family will therefore have narrow FSR fringing and will be best suited to TDLAS measurements. **Figure 4.12** shows the spot patterns for the $2m \pm 4$ and $2m \pm 2$ families.

Experimentally the variation of the fringe FSR with different spot patterns has been observed using an experimental set up identical to the one described in Section 5.5.

Pass Number against Normalised Separation

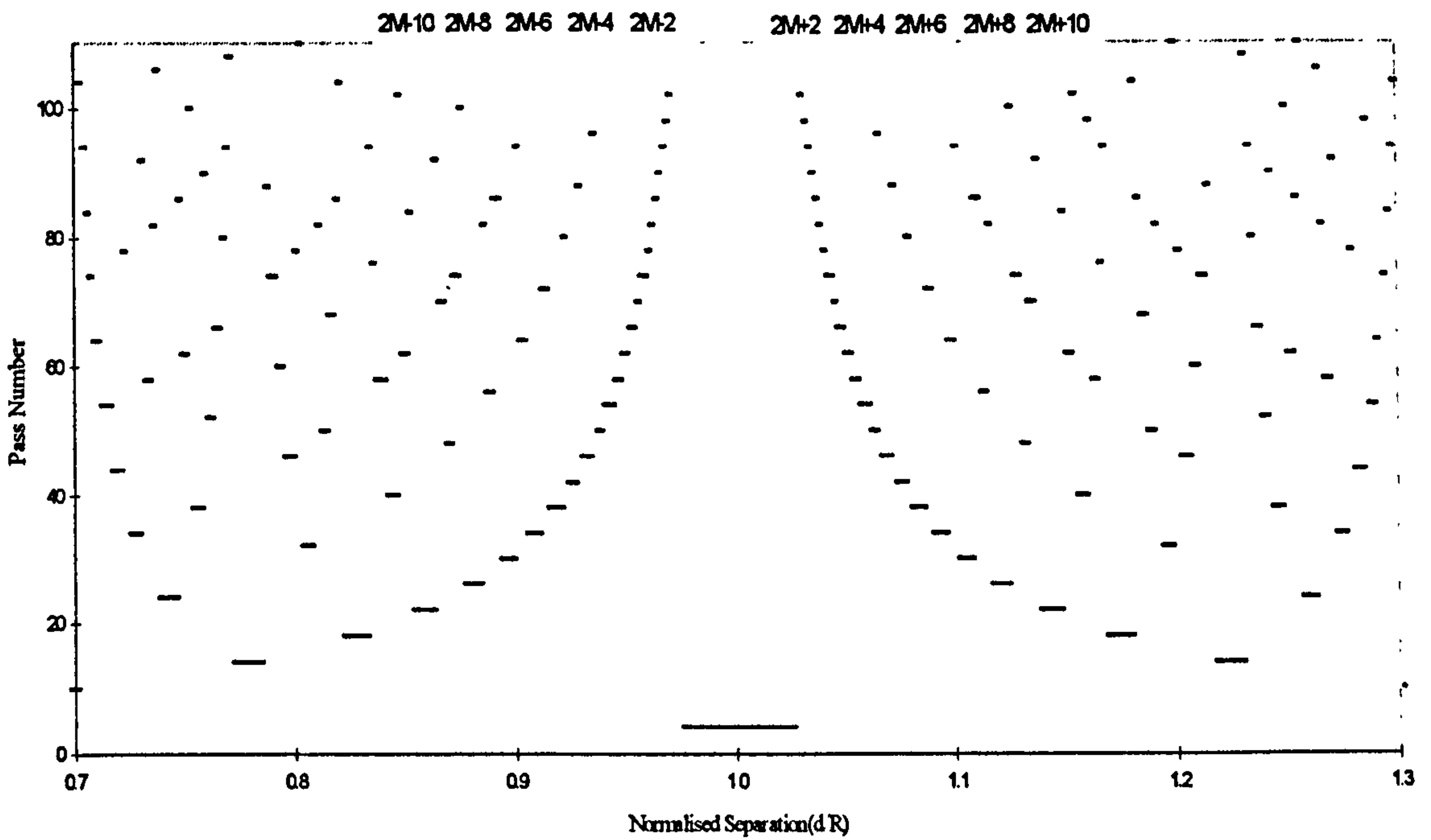
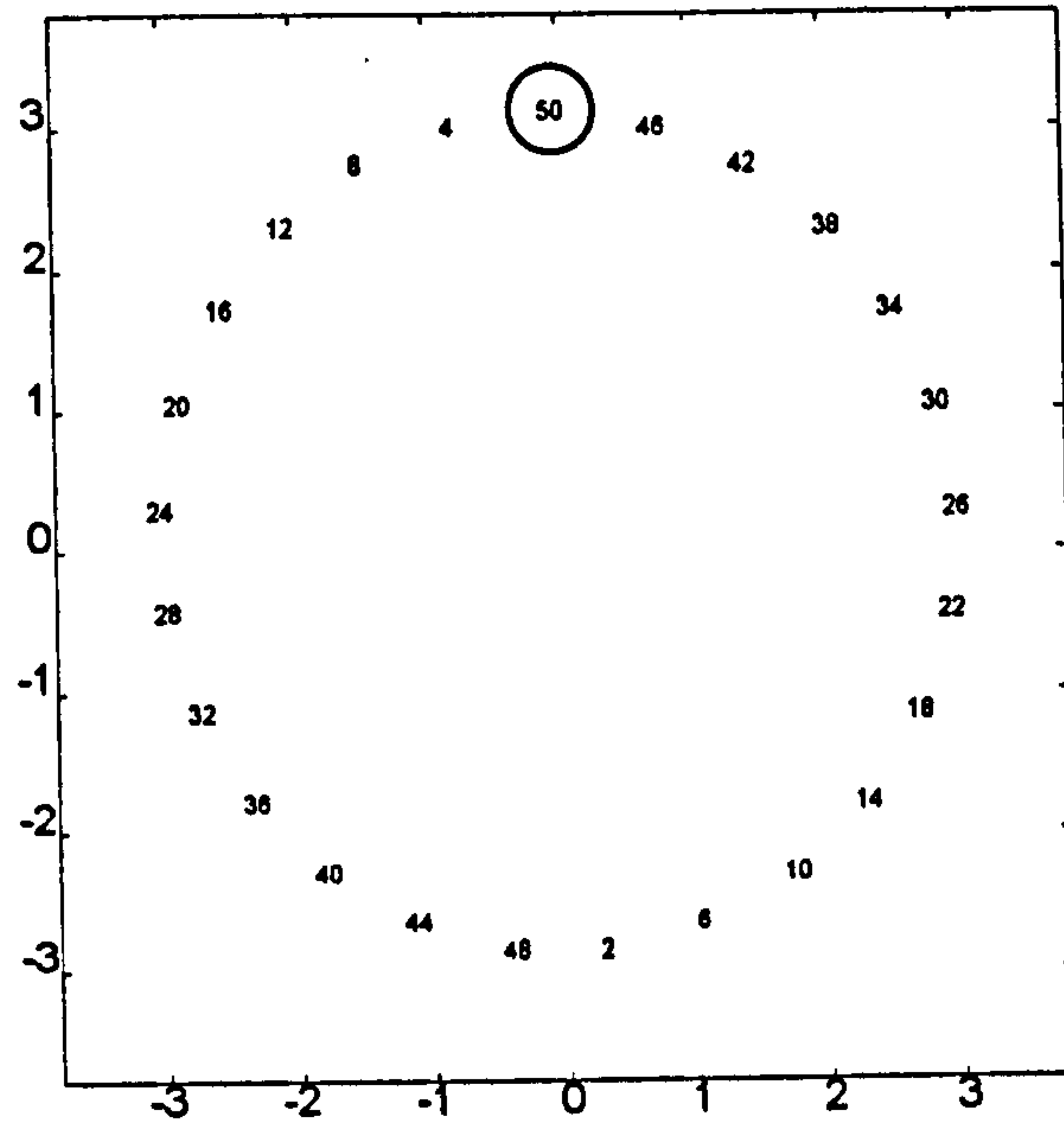


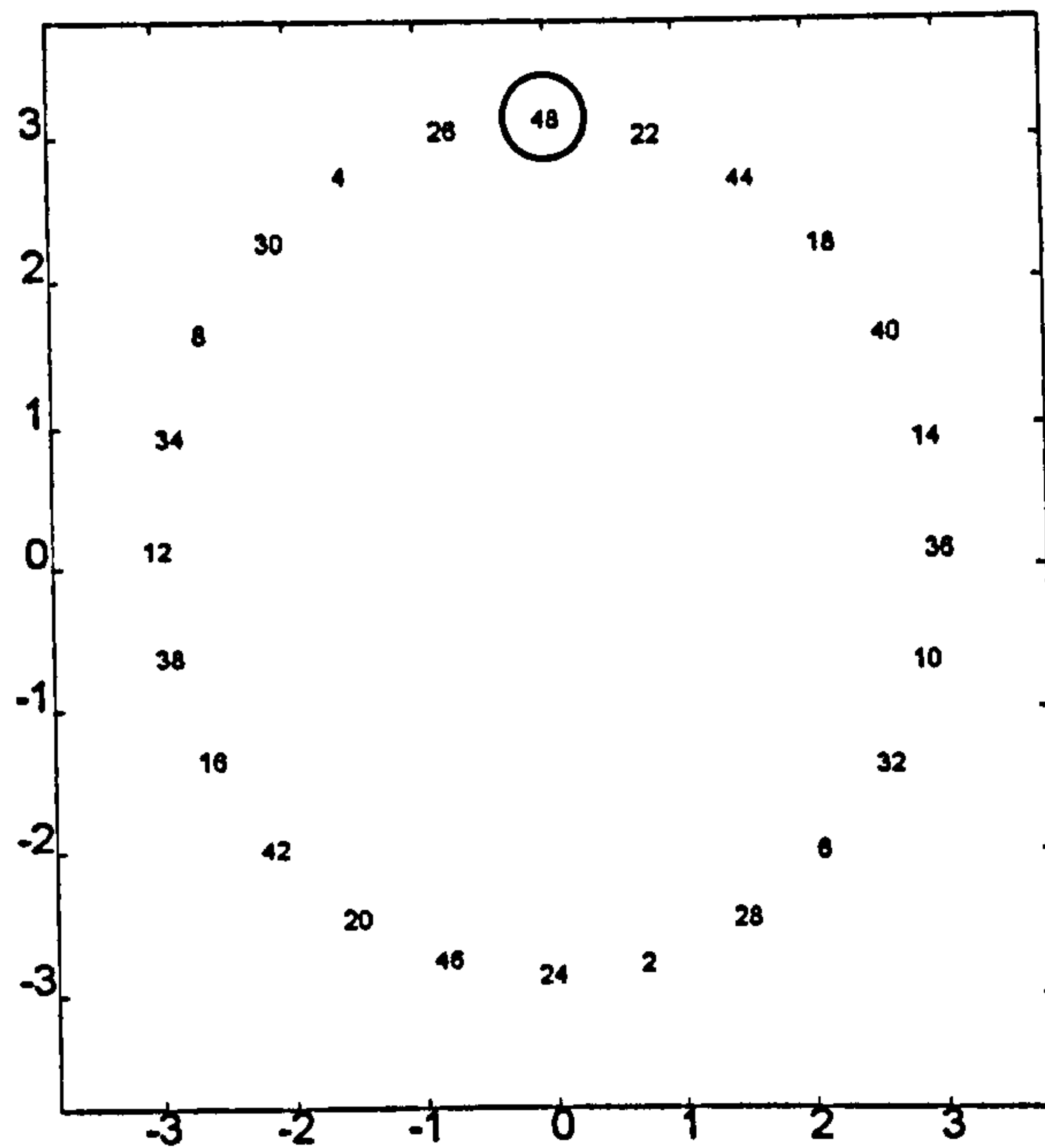
Figure 4.11 - Re-entrant Solutions and their Family Groupings

Family	Neighbouring Spot Numbers (S1,S2)
$n = 2m \pm 2$	$(4), (n - 4)$
$n = 2m \pm 4$	$(n/2 - 2), (n/2 + 2)$
$n = 2m \pm 6$	$(n/3 \pm 4/3), (2n/3 \pm 4/3)$
$n = 2m \pm 8$	$(n/4 \pm 1), (3n/4 \pm 1)$
$n = 2m \pm 10$	$(n/5 \pm 4/5), (4n/5 \pm 4/5)$ or $(2n/5 \pm 4/5), (3n/5 \pm 4/5)$

Table 4.1 - Family Groupings and Spot number relationships



$2m \pm 2$



$2m \pm 4$

Figure 4.12 - Spot Patterns for the $2m \pm 2$ and the $2m \pm 4$ families

4.2.15 Cell Solution chosen for ground based TDLAS.

For the ground based instrument described in this thesis (Chapter 5), the 48 pass solution shown in Figure 4.12 will be used. This solution is found at a mirror separation of 52.1 cm. The computer model predicts an absorption pathlength of 25.1m. A spacing tolerance of 1.5 mm ensures that this solution will have the required stability for field applications. In addition, this choice of solution will ensure that the FSR of fringing generated by neighbouring spots to the coupling hole will be significantly smaller than the width of the feature being measured. The performance of this Herriott cell in the ground based spectrometer is assessed in Chapter 6.

4.2.16 Conclusions

A model of the Herriott cell, based on a matrix description of the optical elements, has been written in the MATLAB language. This model coupled with analytical equations has been used to understand and predict the behaviour of the cell. All predictions were validated experimentally using mirrors bought from Ealing Electro-Optics. Modelling predictions have allowed a suitable solution for the ground based TDLAS to be determined.

It was found that the Herriott cell was insensitive to tilt and spacing changes and had several advantages over the traditionally used White cell. These included the ability to choose patterns that produced fringes with a small FSR and the ability of the cell to maintain a given re-entrant solution over a wide temperature range. Consequently it is concluded that the Herriott cell is well suited for field applications.

The main drawback of this multipass arrangement is that inefficient use of the mirror surfaces means that the cells can only support a moderate number of passes. It is felt that for a ground based instrument this inefficiency would not pose a problem, but in the case of a piggy back balloon deployment where size and weight must be kept to a minimum such inefficiencies could not be tolerated. Thus another more *efficient* solution must be found. Such a solution is discussed in the following section.

4.3 Astigmatic Herriott Cell

4.3.1 Introduction

So far, only the conventional Herriott Cell has been considered for use as a multipass arrangement. A different variant of this cell, known as the astigmatic Herriott cell [36], uses mirrors that have two different radii of curvature. This variant offers several advantages, the main one being that the circulating beam more fully fills the whole volume of the cell, giving a more even distribution of spots on the mirror surface. Thus for a given number of passes, the beam spots will be more widely spaced than in the conventional case, allowing smaller mode volumes and smaller mirrors. Figure 4.13 shows a schematic of an astigmatic mirror.

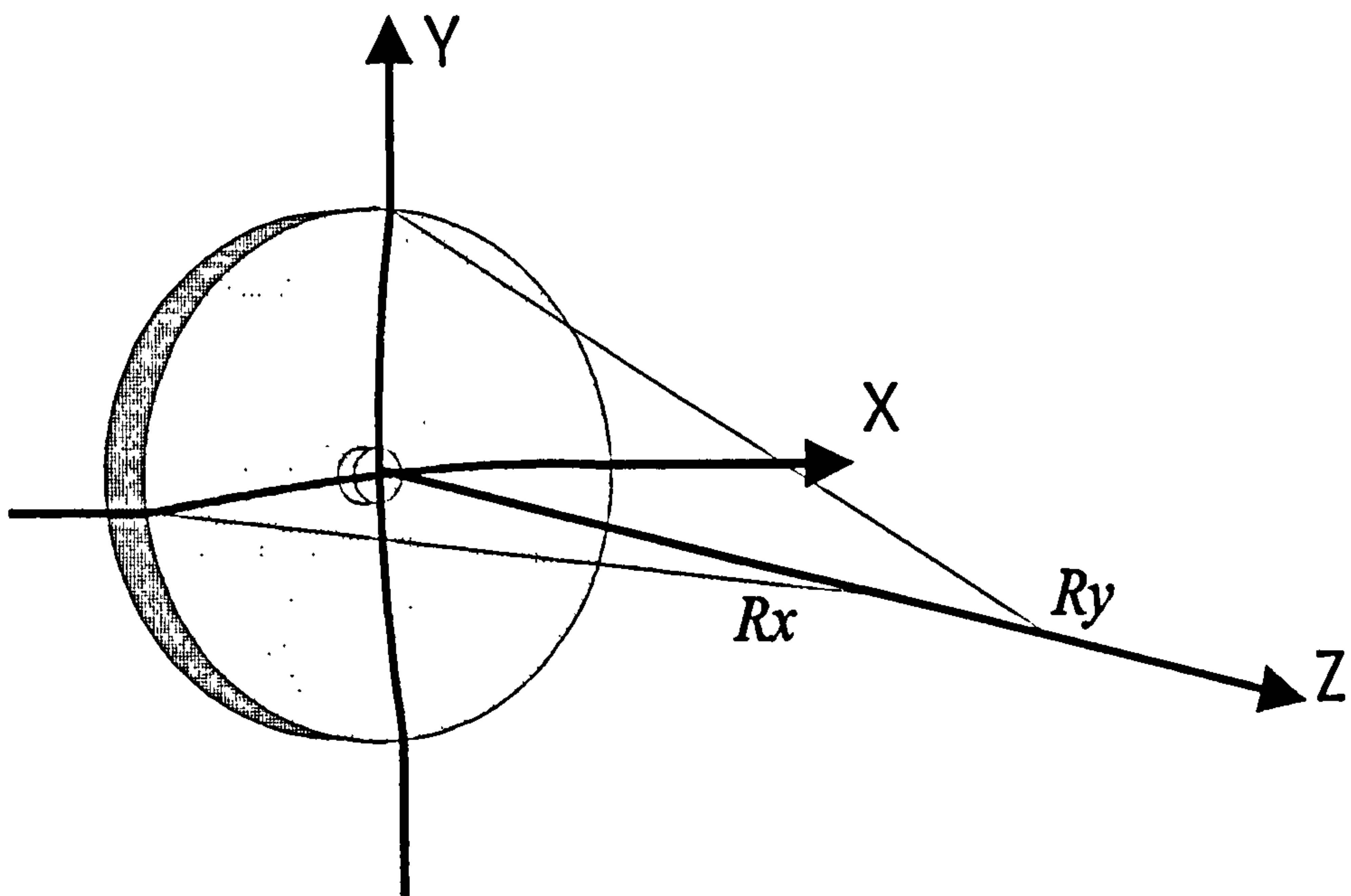


Figure 4.13 - Schematic of Astigmatic Herriott Cell Mirror

One of the main factors that has inhibited the use of astigmatic cells has been the need for extreme precision in manufacturing the mirrors. Unlike in the conventional Herriott cell, where errors in the radii of curvature can be compensated for by changing the mirror spacing, the astigmatic case requires that the re-entrant condition be achieved

for both axes simultaneously. This is not possible through changes in the mirror separation only.

McManus *et al* [40] have demonstrated that changes in the mirror spacing coupled with the rotation of one mirror about its optical axis, can provide the required compensation to both radii of curvature. Using this technique allows the mirrors to be made with relaxed tolerances and reduced cost.

The properties of the astigmatic Herriott cell will now be investigated.

4.3.2 Properties of the Astigmatic Herriott Cell

McManus *et al* [40] have derived analytical expressions to describe the propagation of a paraxial beam through the astigmatic Herriott cell. A 4x4 matrix description of the cell can be reduced to a pair of coupled difference equations that naturally yield analytical expressions for circulation frequencies within the cell. This approach is analogous to the 2x2 matrix description of the conventional Herriott cell, and the reduction of such a matrix to a single difference equation, as was described in Section 4.2.2. Using this technique they have shown that the co-ordinate of the reflex spot after n passes is:

$$\begin{aligned} x_n &= (A^2 - 2 - e^2)x_{n-1} - x_{n-2} + \varepsilon y_{n-1} \\ y_n &= (B^2 - 2 - e^2)y_{n-1} - y_{n-2} - \varepsilon x_{n-1} \end{aligned} \quad (4.49)$$

where:

$$\begin{aligned} A &= 2 - [(\alpha_x d) \cos^2 \tau + (\alpha_y d) \sin^2 \tau] \\ B &= 2 - [(\alpha_x d) \sin^2 \tau + (\alpha_y d) \cos^2 \tau] \\ e &= 1/2(\alpha_x - \alpha_y)d \sin(2\tau) \\ \varepsilon &= -1/4[(\alpha_x - \alpha_y)d]^2 \sin(4\tau) \end{aligned} \quad (4.50)$$

and where:

$$\begin{aligned}
\alpha_x &= 2/R_x \\
\alpha_y &= 2/R_y \\
\tau &= \theta_t/2
\end{aligned}
\tag{4.51}$$

θ_t is the rotation angle between the mirrors. The coupling term, ε , will usually be small. If the rotation angle between the two mirrors was zero, the coupling term would become zero, and the resultant motions would be sinusoidal in x and y, as in the case of the conventional Herriott cell.

McManus *et al* have also derived equations that give the cosines of the angular advances in two passes:

$$\begin{aligned}
\cos\theta_{x2} &= 1/2[A^2 - 2 - e^2 - \xi] \\
\cos\theta_{y2} &= 1/2[B^2 - 2 - e^2 + \xi]
\end{aligned}
\tag{4.52}$$

with:

$$\xi = 1/2(B^2 - A^2)\{-1 + [1 - 4\varepsilon^2 / (B^2 - A^2)]^2\}^{1/2}
\tag{4.53}$$

For a simplified analysis, in which the two mirrors are assumed to be identical, with no rotation or tilt, equations originally presented by Herriott *et al* [42] can be used. In this simplified analysis, the equations for the spot co-ordinates after n passes become:

$$\begin{aligned}
x_n &= X_o \sin(n\theta_x) \\
y_n &= Y_o \sin(n\theta_y)
\end{aligned}
\tag{4.54}$$

where:

$$\begin{aligned}
\theta_x &= \cos^{-1}(1 - d/R_x) \\
\theta_y &= \cos^{-1}(1 - d/R_y)
\end{aligned}
\tag{4.55}$$

X_0 and Y_0 are the co-ordinates of the point where the injection beam first hits the back mirror and they define the size of the overall spot pattern. R_x and R_y are the mirror radii of curvature and d , as in earlier sections, is the mirror separation. It should be noted that the expressions in (4.54) assume that the entrance/exit point is at the centre of the mirror (i.e. at the co-ordinate $(0,0)$). It is seen that the spots trace out a path on the mirror surfaces that is sinusoidal in x and y , but with different frequencies, i.e. a Lissajous pattern.

4.3.3 Conditions for Re-entrance

As a result of having two independent re-entrant equations describing the circulation of the beams within the cavity, two independent conditions for re-entrance are required. These are:

$$\begin{aligned} n\theta_x &= m_x \pi \\ n\theta_y &= m_y \pi \end{aligned} \tag{4.56}$$

Substitution of these conditions into the equation (4.53) gives $x_n = 0$ and $y_n = 0$, i.e. the beam returns to the entrance point. As in the case of the conventional Herriott cell, n must be an even integer and there should be no common factors (other than two) within the set of integers $\{n, m_x, m_y\}$.

These integers define the pattern of transits through the cell and the choice of n , m_x and m_y can have a significant effect on the cell behaviour. When m_x and m_y are both chosen to be even, the beam undergoes a whole number of complete transverse oscillations. This means that the output beam will exit from the diagonally opposite corner from the one it was originally aimed at. Under these conditions, the beam acts as if it is reflecting from a convex mirror, equivalent to the back of the cell input mirror, at the coupling hole. This is identical to the behaviour of the conventional Herriott cell and is especially useful when incorporating the cell into an optical design. Solutions with even m_x and m_y tend to have symmetrical patterns, allowing a more even distribution of spots. It is for these reasons that patterns with even m_x and m_y are exclusively used when searching for suitable solutions.

4.3.4 Maximum Number of Passes

In the conventional Herriott cell, the beam maps out a single ellipse on the mirror surface. In the case of the astigmatic cell, with the coupling hole at the centre of the front mirror, the beam maps out a rectangular pattern defined by the initial injection co-ordinates, X_0 and Y_0 . Assuming that a circular mirror is used, the pattern area will be maximised when both injection co-ordinates are the same, i.e. the pattern will be square. It is assumed that the spots must at least be separated by the diameter of the hole, d_h , and that they are evenly spaced over the mirror surface. The maximum number of passes, n , using these assumptions, can be shown to be:

$$n \leq \frac{8X_0^2}{d_h^2} \quad (4.57)$$

If $X_0 = 3.5$ cm and $d_h = 0.4$ cm, the maximum number of passes would be 612. This compares with a maximum of 110 passes for the conventional Herriott cell under identical conditions. It is clear from this comparison that the astigmatic Herriott cell gives a significantly greater number of passes over the standard cell.

4.3.5 Other Properties

Even when using the simplified analytical expressions for the astigmatic Herriott cell, the expressions for the focusing properties (Section 4.2.6) and the stability condition (Section 4.2.7) become complicated. If rotation of one mirror about the optical axis is included, the expressions become extremely complicated. If it is assumed that the two radii of curvature are similar, i.e. $R_x \approx R_y$, then the expressions for the conventional Herriott cell are a good approximation [42]. This is also true for the spacing tolerance (Section 4.2.8) and tilt sensitivity (Section 4.2.9). The exact behaviour of the cell will therefore only be investigated numerically using the 4x4 matrix model. This 4x4 matrix description of the astigmatic Herriott cell will now be described.

4.3.6 4x4 Matrix Description of Astigmatic Herriott Cell

The 4x4 matrix description for the astigmatic cell is similar to the 2x2 matrix description of the conventional Herriott cell (Section 4.4.1). The matrix for one traverse, $C(R,d)$, is the product of the free space propagation matrix, $D(d)$, and the reflection matrix, $R(R)$, where:

$$D(d) = \begin{bmatrix} 1 & d & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & d \\ 0 & 0 & 0 & 1 \end{bmatrix} \quad (4.58)$$

and:

$$R(R_x, R_y) = \begin{bmatrix} 1 & 0 & 0 & 0 \\ -2/R_x & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \\ 0 & 0 & -2/R_y & 1 \end{bmatrix} \quad (4.59)$$

When the mirror axes are rotated by an angle θ_t to the co-ordinate axes, the reflection matrix becomes [40]:

$$R'(R_x, R_y) = T(-\theta_t)R(R_x, R_y)T(\theta_t) \quad (4.60)$$

where $T(\theta_t)$ is the rotation matrix [40]:

$$T(\theta_t) = \begin{bmatrix} \cos\theta_t & 0 & \sin\theta_t & 0 \\ 0 & \cos\theta_t & 0 & \sin\theta_t \\ -\sin\theta_t & 0 & \cos\theta_t & 0 \\ 0 & -\sin\theta_t & 0 & \cos\theta_t \end{bmatrix} \quad (4.61)$$

The rotation matrices mix x and y components of the beam co-ordinates. The behaviour of the cell, however, is similar to the unrotated case, with essentially sinusoidal x and y spot motions.

As in the case of the *standard* Herriott cell, the co-ordinate of the beam spot after n passes, Z_n , is given by $C^n Z_0$, with:

$$Z_0 = \begin{pmatrix} x_0 \\ x_0' \\ y_0 \\ y_0' \end{pmatrix} \quad (4.62)$$

and:

$$Z_n = \begin{pmatrix} x_n \\ x_n' \\ y_n \\ y_n' \end{pmatrix} \quad (4.63)$$

This model has been incorporated into a MATLAB program which has been used to design and investigate the properties of the astigmatic Herriott cell. A program listing can be found in Appendix 2. The results of this work will now be discussed.

4.3.7 Maps of Re-entrant Solutions

As in the case of the conventional Herriott cell, creating a map of re-entrant solutions can be a useful tool in helping to design and set up a cell configuration. Due to the astigmatism, the pass number is mapped against the two dimensional space of (θ_x, θ_y) . Once again the map of solutions is completely general and is independent of specific choices of cell parameters. It is generated in an identical fashion to the *standard* cell, with the model being used to step through values of n at an arbitrary point (θ_x, θ_y) , until a re-entrant solution is found. Figure 4.14 shows a very small region of the map

In this case the solutions are mapped against (ϕ_x, ϕ_y) . ϕ_x and ϕ_y are known as the reduced angular variables [40], and are defined as:

$$\begin{aligned}\phi_x &= \theta_x - \pi/2 \\ \phi_y &= \theta_y - \pi/2\end{aligned}\tag{4.64}$$

The reduced angular variables are used simply as a matter of convenience.

It is seen that the map is made up of overlapping circular zones. Each zone on the map represents a re-entrant solution, with the number of cavity passes indicated by the colour. Zones with larger diameters correspond to lower pass solutions. The maximum pass number was limited to 1000.

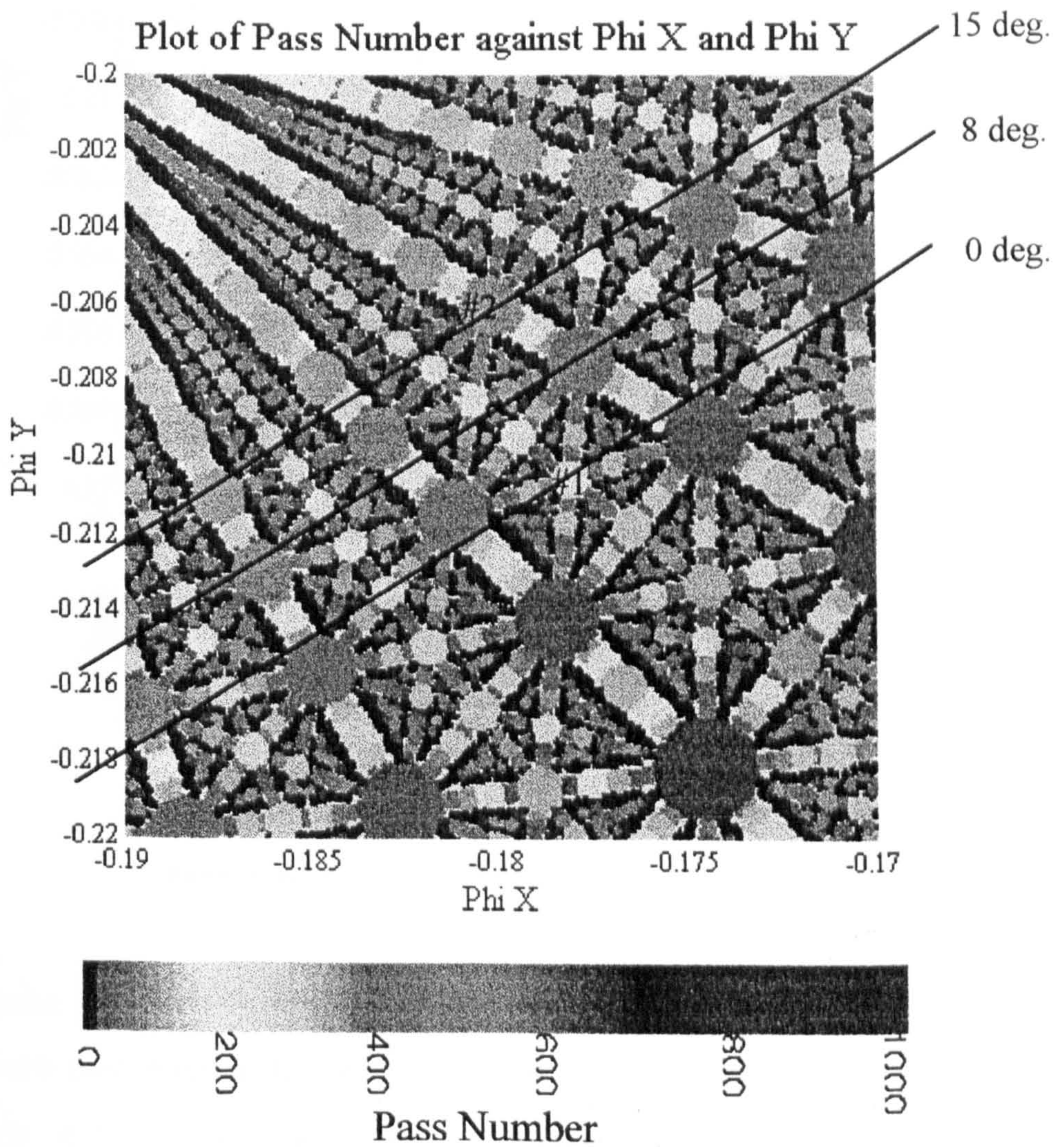


Figure 4.14 - Map of Re-entrant Solutions

Figure 4.15 shows the sub-set of the solutions in which the beam acts as if it were reflecting from a single mirror located at the cell input, i.e. m_x and m_y both even. As stated previously, only members of this sub-set are considered as possible configurations. It is evident from **Figure 4.15** that considerably fewer solutions belong to this subset.

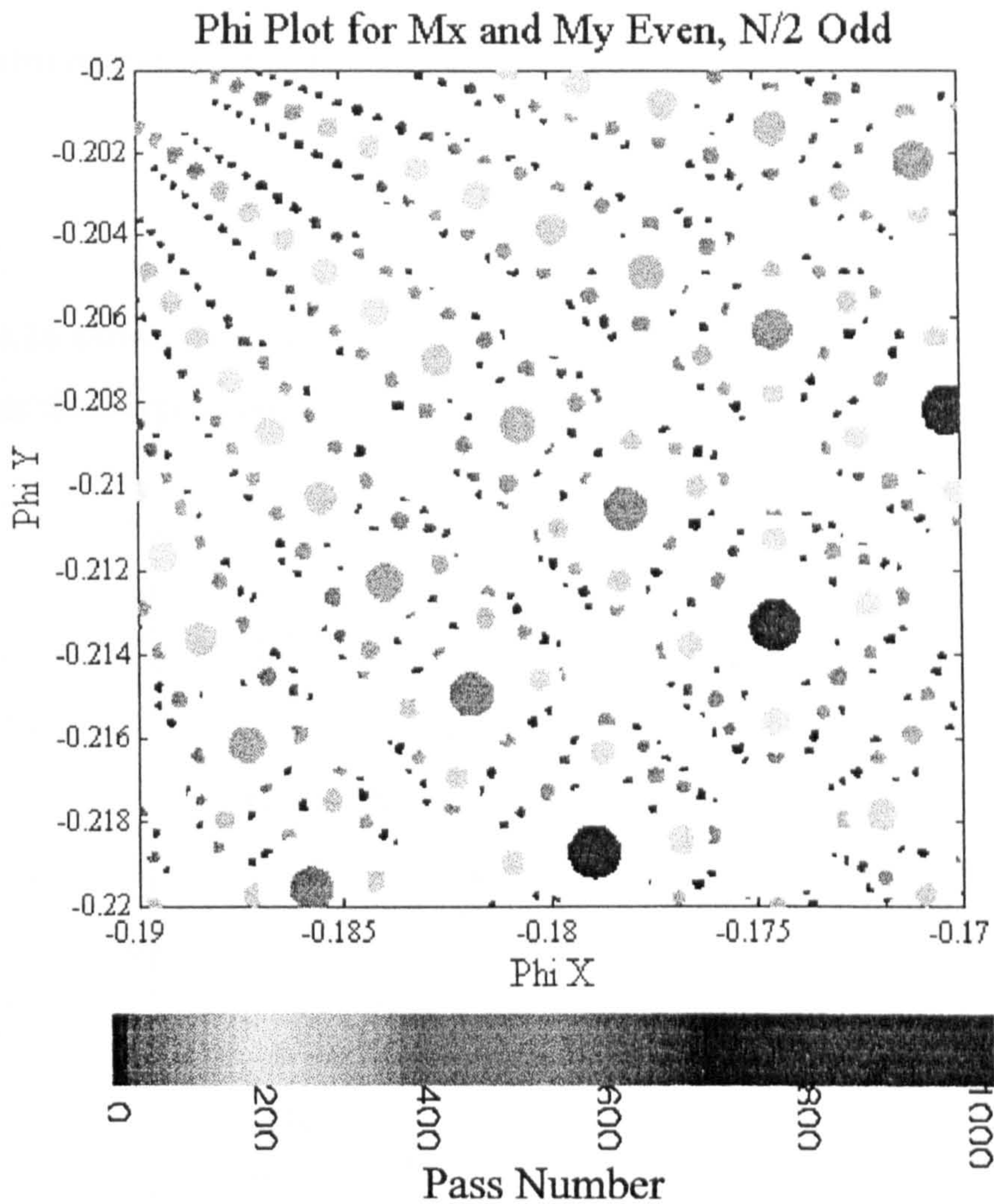


Figure 4.15 - Subset of Re-entrant Solutions

To determine specific cell parameters from these maps, a zone corresponding to a solution with pass number, n , should be chosen and the centre co-ordinates of the zone, i.e. (θ_x, θ_y) , should be incorporated into equations (4.54). This will recover the normalised separations, which can then be used to determine the required radii of curvature for a given mirror spacing. As an example, if the region marked #1 on

Figure 4.14 is considered, it corresponds to a re-entrant solution with 194 passes. The centre co-ordinates of this zone are $(-0.178, -0.211)$. If these co-ordinates are converted from reduced angular variables into (θ_x, θ_y) , and incorporated into equation (4.54), the normalised separations are:

$$R_x/d = 0.822$$

$$R_y/d = 0.791$$

For a mirror spacing of 50 cm, the required radii of curvature would therefore be:

$$R_x = 60.83 \text{ cm}$$

$$R_y = 63.23 \text{ cm}$$

Figure 4.16 shows the spot pattern obtained for this solution. The circle in the centre represents the coupling hole.

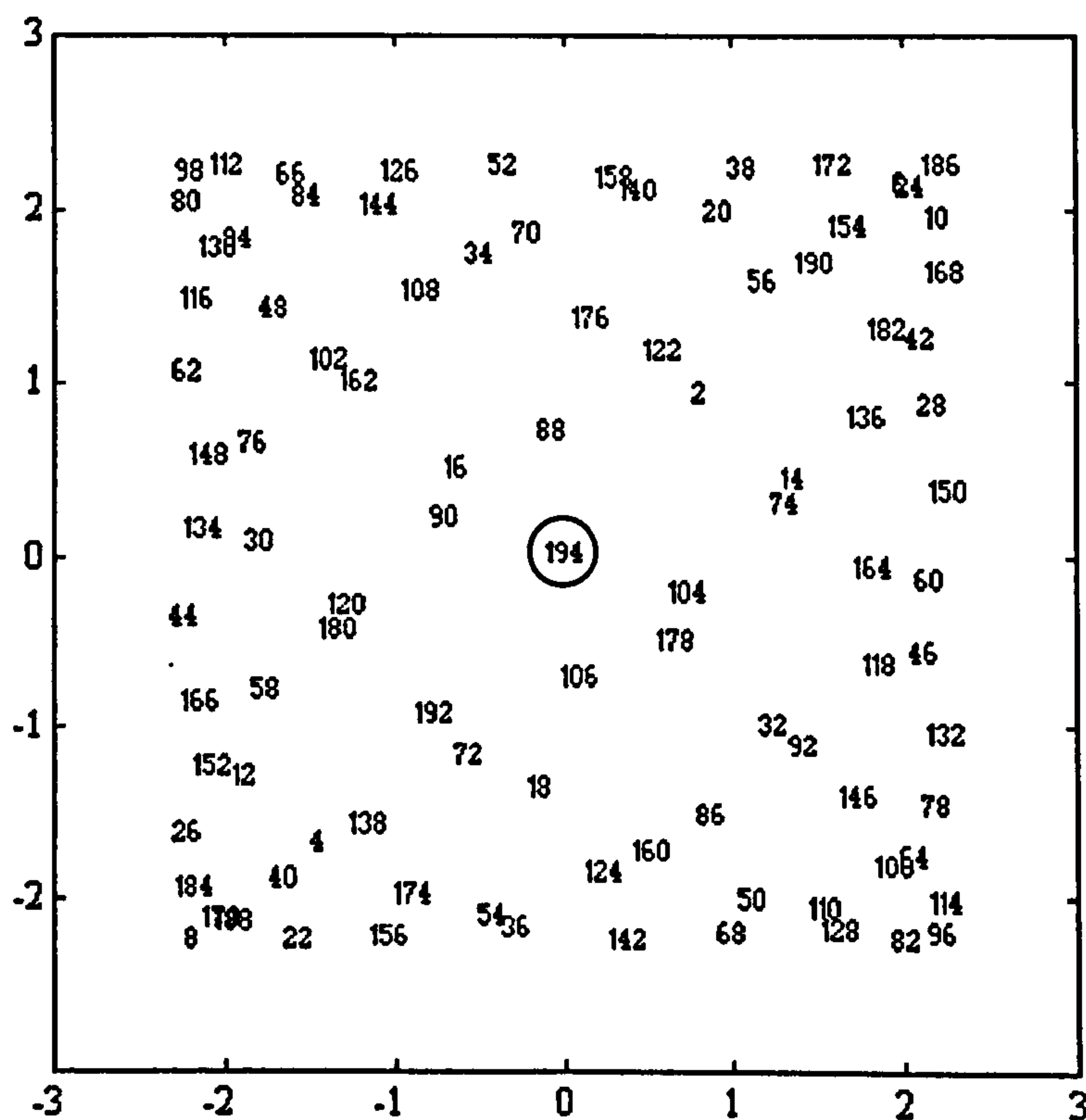


Figure 4.16 -194 Pass Solution at $(1.392, 1.360)$

Adjustments in the mirror rotation and spacing, map out trajectories through the (θ_x, θ_y) space on the map of re-entrant solutions. A set of these trajectories are shown

in **Figure 4.14**. The trajectories can be determined using the equations in (4.52) and (4.53) and can be used to ascertain the required spacing and rotation changes to move from one solution to another. For example movement from the solution marked #1 on **Figure 4.14** to the solution marked #2 can be achieved through a rotation of 15° and a change in separation of 2.0 mm. These trajectories are approximately linear, if the rotation angle remains fixed.

4.3.8 Design Considerations and Design Criteria

The MATLAB model has been used to design an astigmatic Herriott cell, which will provide a long pathlength in a small volume. A pathlength of ~ 100 m is required to obtain the necessary detection limits for the weaker overtone transitions of the molecules that are of interest. In order to keep the instrument small, a mirror separation of 50 cm was decided upon, dictating that the required number of passes from the astigmatic cell would be of the order of 200.

It was decided that it would be beneficial if the mirrors could also support a 100 pass solution at a separation of one metre. Such a solution would be useful for work in the mid-infrared where low laser powers would limit the number of passes that could be obtained. This solution would also be used as a backup if the higher pass solution proved to be too sensitive to spacing and tilt changes in a field application.

With the basic criteria set, it is important to decide what factors make a specific spot pattern a suitable candidate for absorption measurements. The design considerations involved in choosing a suitable pattern are listed below.

Low Fringe Levels

Fringing from within the cell can be a major contribution to noise levels. It is therefore important that any pattern has its innermost spots as widely spaced from the centre as possible, reducing the possibility of beam spillover.

Easy Removal of Fringes

Any fringing that does occur should have a Free Spectral Range significantly smaller than the linewidth of the feature that is being measured. This is achieved by making

sure that the spots nearest the coupling hole are close to midway through their orbit.

Minimum Mode Size

Another important criteria for a suitable spot pattern is that the mirror spacing should be close to confocal. This will minimise the mode size of the propagating beam, allowing the cell to be made smaller. This is advantageous where rapid cell response times are required. Patterns close to confocal will have by definition, values of m_x and m_y which are approximately half the value of the pass number, n .

Tilt Sensitivity

Certain patterns will be less sensitive to tilt than others. Patterns must therefore be checked to ensure that intermediate spots are not susceptible to being driven out the coupling hole through misalignments.

Recognisable

The spot pattern should be easy to recognise as it will be difficult to visually count the reflex spots on the mirror surface.

Suitable Neighbour

The spot pattern should have a low pass solution nearby. This low pass solution will have a large spacing tolerance and will therefore be easy to find. The low pass pattern will act as a marker for the high pass solution. The computer model can be used to determine the required adjustments to both spacing and twist which will allow movement from the marker pattern to the actual solution.

4.3.9 Candidate Patterns

Using the model a systematic search for candidate patterns was carried out. The initial search was aimed at finding solutions with even m_x and even m_y whose patterns were close to confocal and had wide spacing around the coupling hole. Shown below in **Figure 4.17** is an example of a *typical* solution generated with the model. It does not fulfil these initial design criteria. The solution does not have even m_x and m_y and as a

result a characteristic asymmetrical pattern is produced. It is also noted that neighbouring spots are close to the coupling hole.

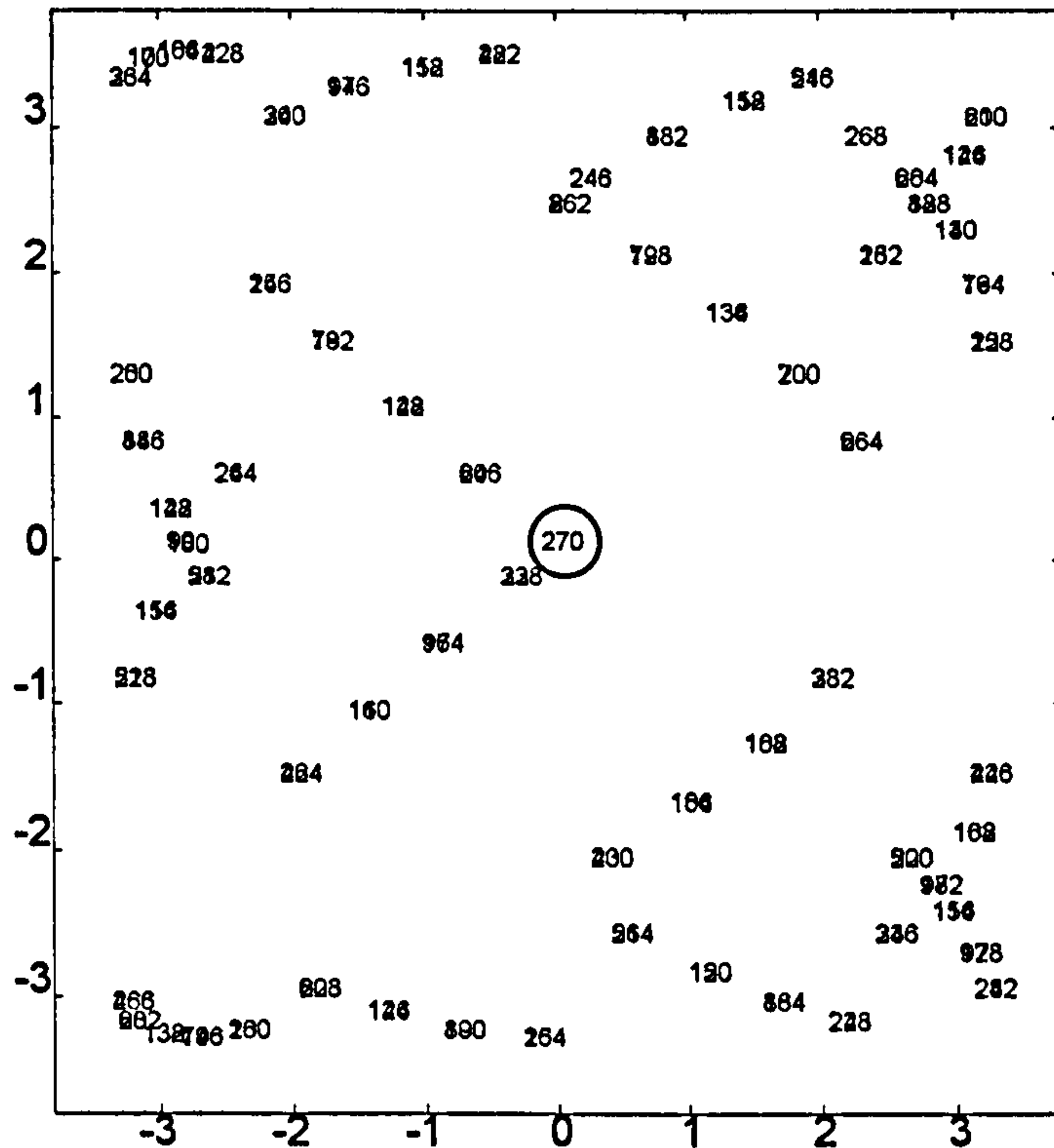


Figure 4.17 - Pattern with odd m_x , odd m_y {270,101,93}

After a comprehensive search, the number of candidate solutions was narrowed down to two. These were:

<u>Solution 1</u>	<u>Solution 2</u>
N = 202	N = 202
d = 50cm	d = 50cm
$M_x = 84$	$M_x = 90$
$M_y = 78$	$M_y = 92$

Each one will give a nominal pathlength of 101 m with a 50 cm mirror spacing. Figure 4.18 and Figure 4.19 show the spot patterns corresponding to these solutions.

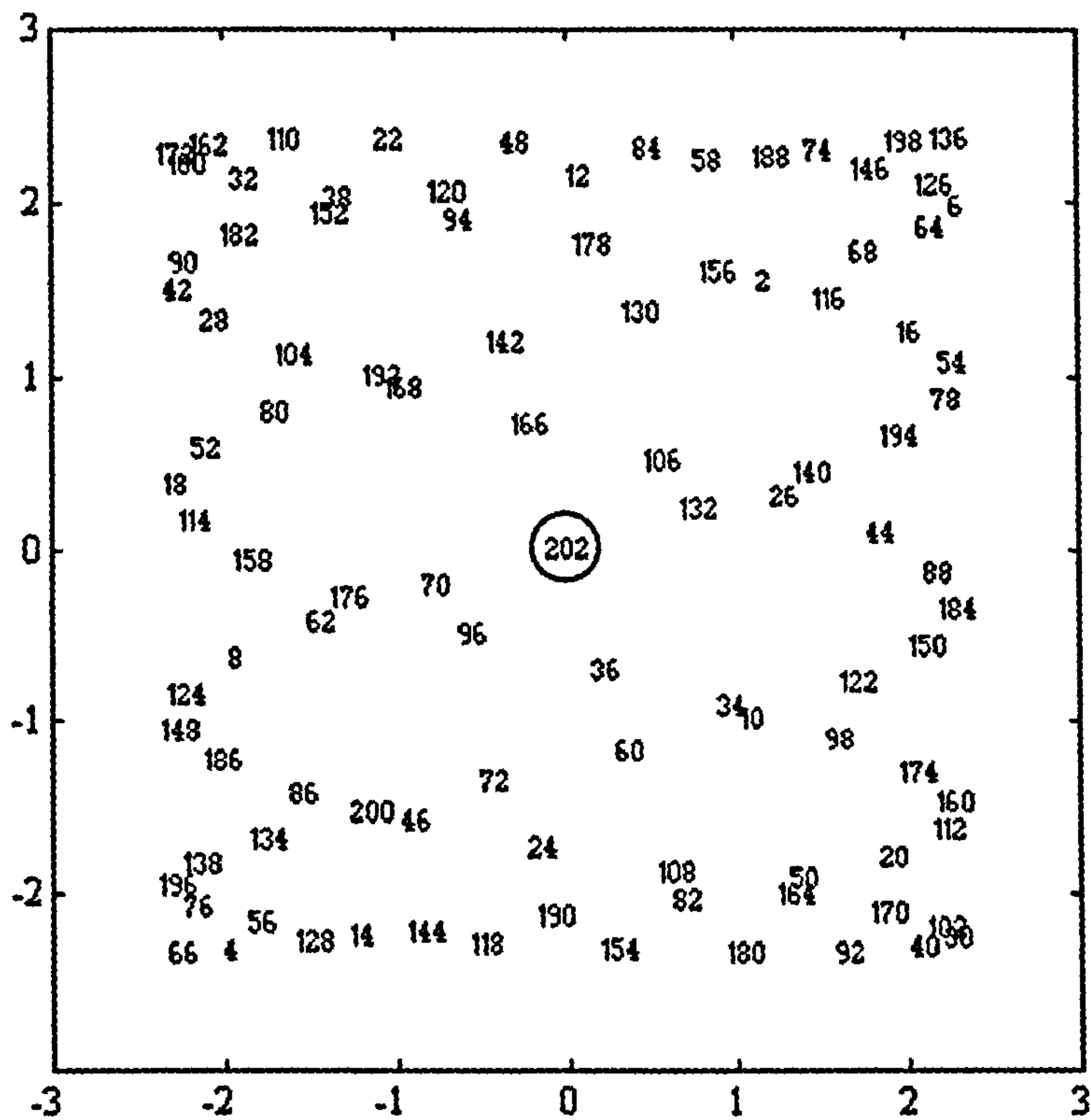


Figure 4.18 - Solution 1 , $m_x = 84$, $m_y = 78$

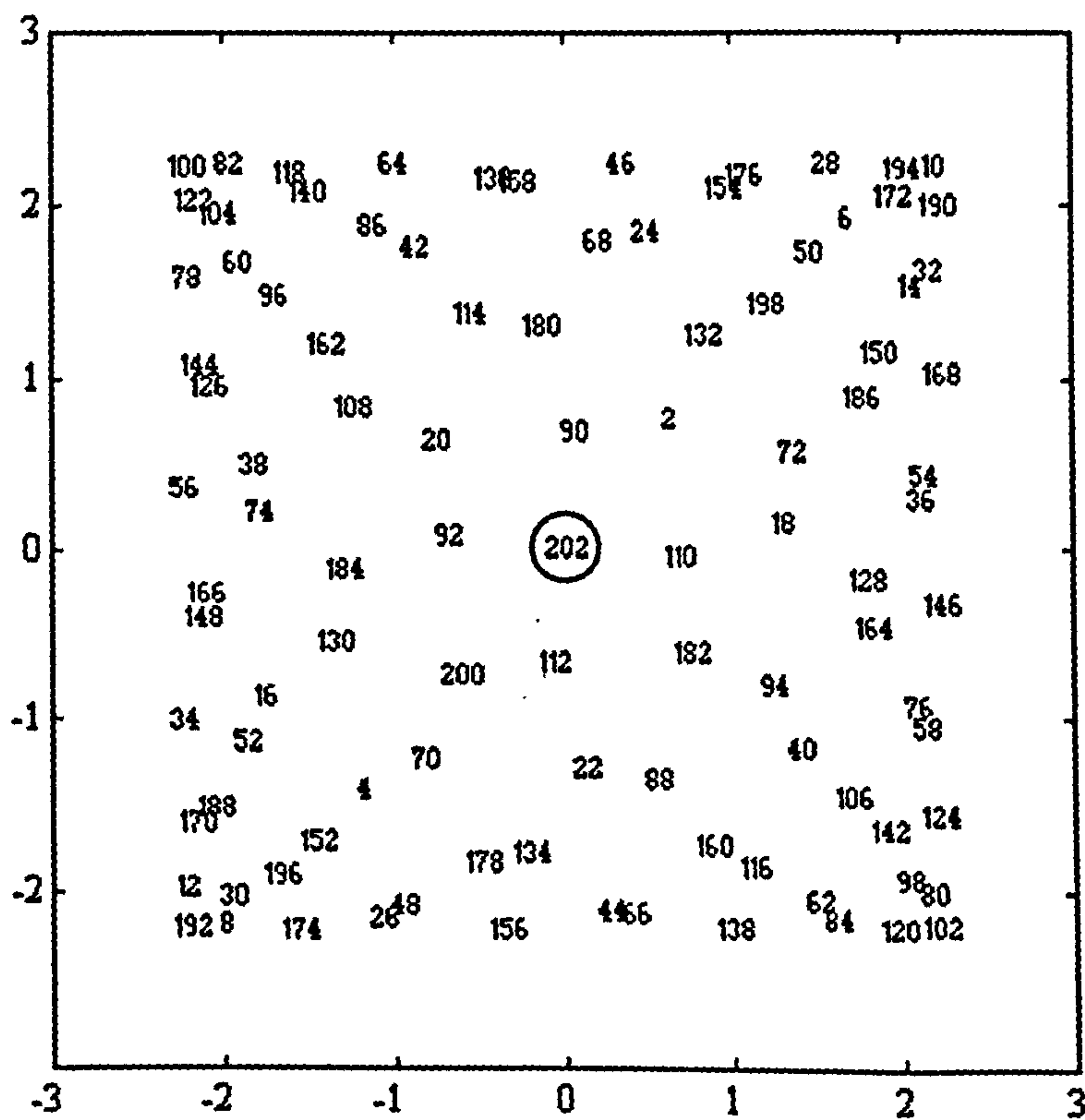


Figure 4.19 - Solution 2 , $m_x = 92$, $m_y = 90$

Both patterns fulfil the principle criteria, described earlier. Additionally, each solution was further tested against the secondary criteria listed in Section 4.3.8. Solution 1 was found to be better suited to absorption measurements. The reasons are outlined below. Although both patterns proved to be equally insensitive to tilt and were easily recognisable, solution 2 had spots neighbouring the coupling hole with pass numbers that were not close to midway through the beam orbit. i.e. one of the closest spots to the entrance/exit hole was pass number 200. This would produce fringing with a FSR of the order of 300 MHz, which is equivalent to the typical linewidth of a light molecule (~ 200 MHz). Consequently, the removal of these fringes, without distortion to the absorption feature, would be extremely difficult.

The next criteria which had to be met was that the pattern should have a low pass number solution nearby. To ascertain this, a map of re-entrant solutions was generated in the regions where each pattern was found. **Figure 4.20** shows the map of re-entrant solutions about solution 1.

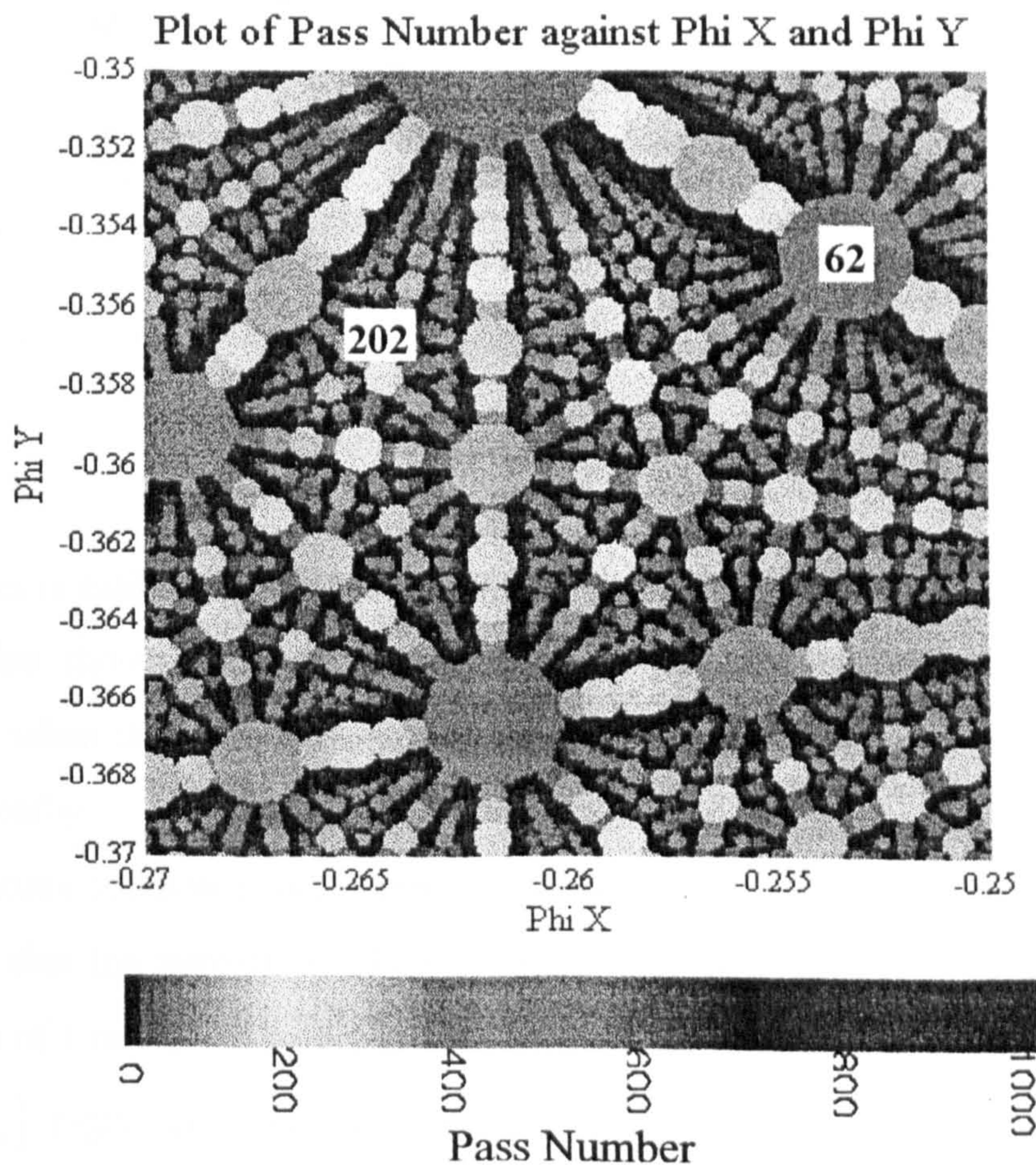


Figure 4.20 - Plot of Pass Number against Phi X and Phi Y, Solution 1

Solution 1, corresponding to 202 passes, has been labelled on Figure 4.20. Also marked is a nearby solution with 62 passes. Movement between these two patterns can be achieved through a rotation of 20° followed by a change in spacing of 5 mm. The spot pattern corresponding to the 62 pass solution is shown below.

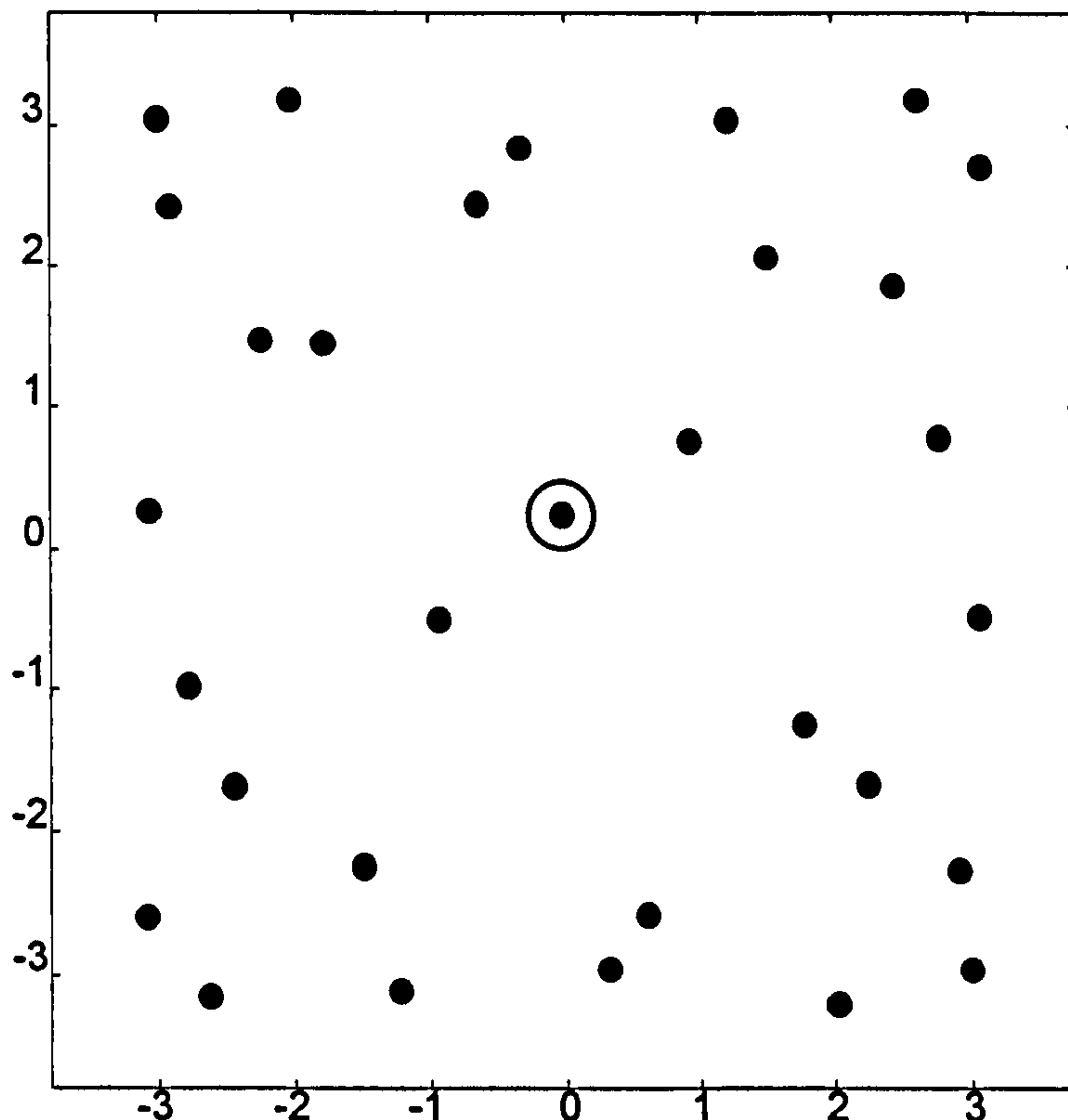


Figure 4.21 - 62 Pass Marker Solution

This pattern is easily recognisable and has most of its spots near the outside edge of the mirror. This should make identification easy. The unsuitability of solution 2 was confirmed when the map of re-entrant solutions showed that there were no low pass patterns nearby.

Having chosen solution 1 as the best suited pattern for TDLAS, all that remained was to ensure that the mirrors would also be able to support a 100 pass solution at a separation of 1 metre. To ascertain this, a map of re-entrant solutions was generated in the (θ_x, θ_y) region corresponding to a 1 metre spacing. Four candidate patterns were found in this region. The best of these patterns had 98 passes with a mirror spacing of

99 cm. Figure 4.22 shows the 98 pass pattern. This pattern is recognisable and the spots neighbouring the coupling hole are well spaced and are nearly midway through the beam orbit.

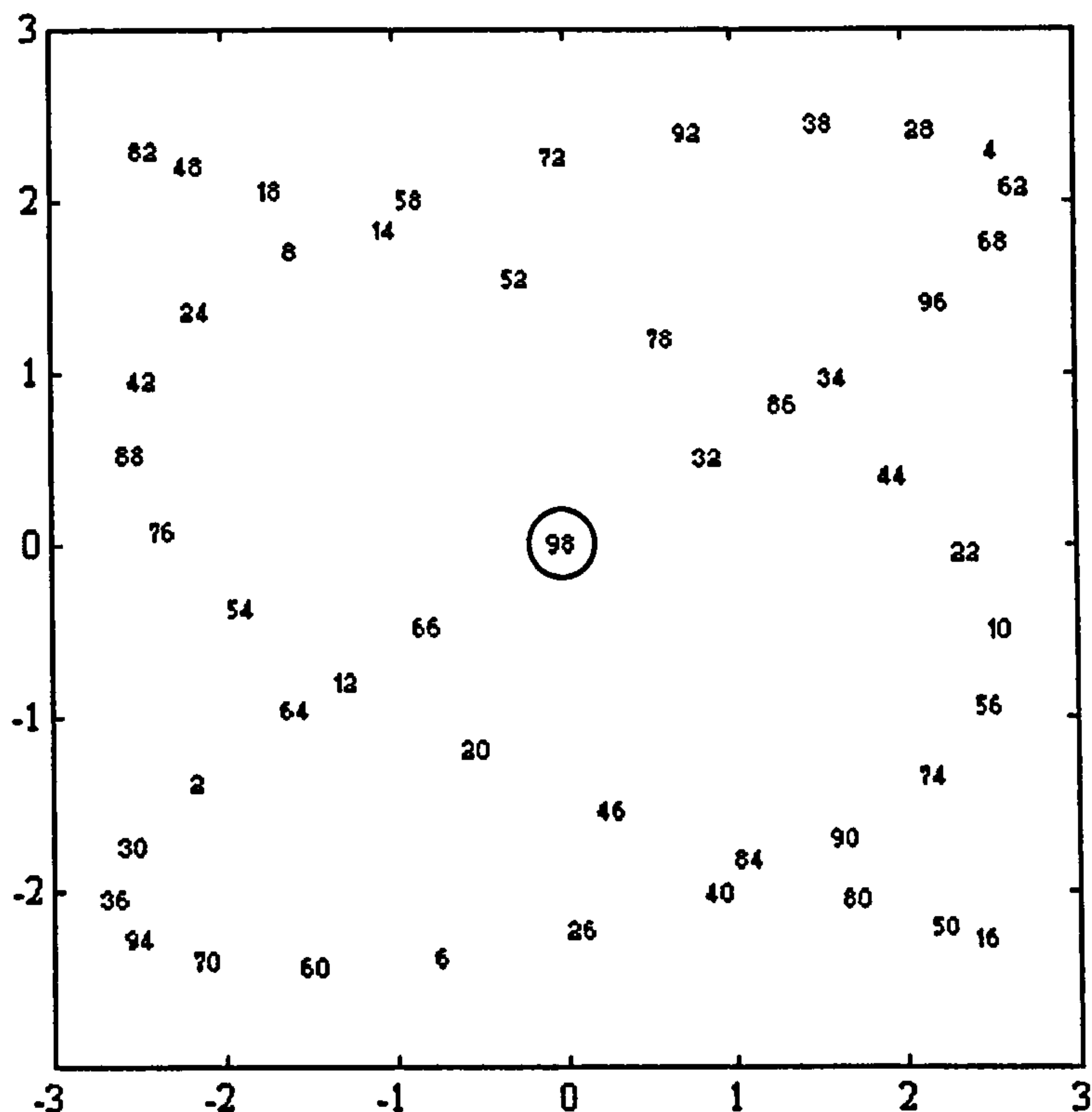


Figure 4.22 - 98 Pass solution at 99 cm separation

It is concluded that the use of the 4x4 matrix model of the astigmatic Herriott cell has allowed a systematic search for a candidate pattern, suitable to TDLAS measurements, to be carried out. This search has provided a pattern that fulfils the design criteria described in Section 4.3.8. The chosen solution, {202,84,78}, will provide a pathlength of 101 metres at a separation of 50 cm and will also support a 98 pass solution with a 99 cm separation.

Having chosen a suitable pattern, the specification of other cell parameters must be decided. These include the manufacturing material, the coupling hole diameter and the precision of manufacture. Again the model was used to determine many of these parameters.

4.3.10 Mirror Specification

Radii of Curvature - Built in Deviations

The solution, as it stands, will appear at a mirror axis angle of 0 degrees. This is not desirable as the adjustability goes to zero at zero rotation angle. Consequently, it would be difficult to compensate for any errors in the radii of curvature. To overcome this, deviations must be built into the mirror radii so that the solution appears at a mirror axis relative angle of ~ 15-20 degrees. The required deviations can be easily determined by using an approximate expression for the *effective* radii against the rotation angle [43]:

$$\begin{aligned} R_{x\text{-eff}} &\sim R_x / [\cos^2 \tau + \gamma \sin^2 \tau] \\ R_{y\text{-eff}} &\sim R_y / [\cos^2 \tau + \gamma^{-1} \sin^2 \tau] \end{aligned} \quad (4.65)$$

where τ is the symmetric half angle between the mirror axes, and $\gamma = R_x/R_y$. The centre co-ordinates of the solution zone, (θ_x, θ_y) , are used to determine $R_{x\text{-eff}}$ and $R_{y\text{-eff}}$. The simultaneous equations in equation (4.65) can then be solved to give the required radii of curvature, R_x and R_y . The answer comes out fairly close to what you would get from the exact expressions in equation (4.52). Equation (4.65) also shows how the effective astigmatism is at a maximum when the axes are aligned, and zero when the axes are at 90° ($\tau = \pm 45^\circ$).

The required radii of curvature to allow the 202 pass solution to appear at an axis angle of ~ 20 degrees are:

$$R_x = 77.274 \text{ cm}$$

$$R_y = 67.425 \text{ cm}$$

The spot pattern for the rotated case, as shown in **Figure 4.23**, is a slightly distorted version of the pattern in **Figure 4.18**. The model predicts that the solution will appear

at a rotation angle of 19.9 degrees and a separation of 49.967 cm. The 98 pass solution will appear at a rotation angle of 18 degrees.

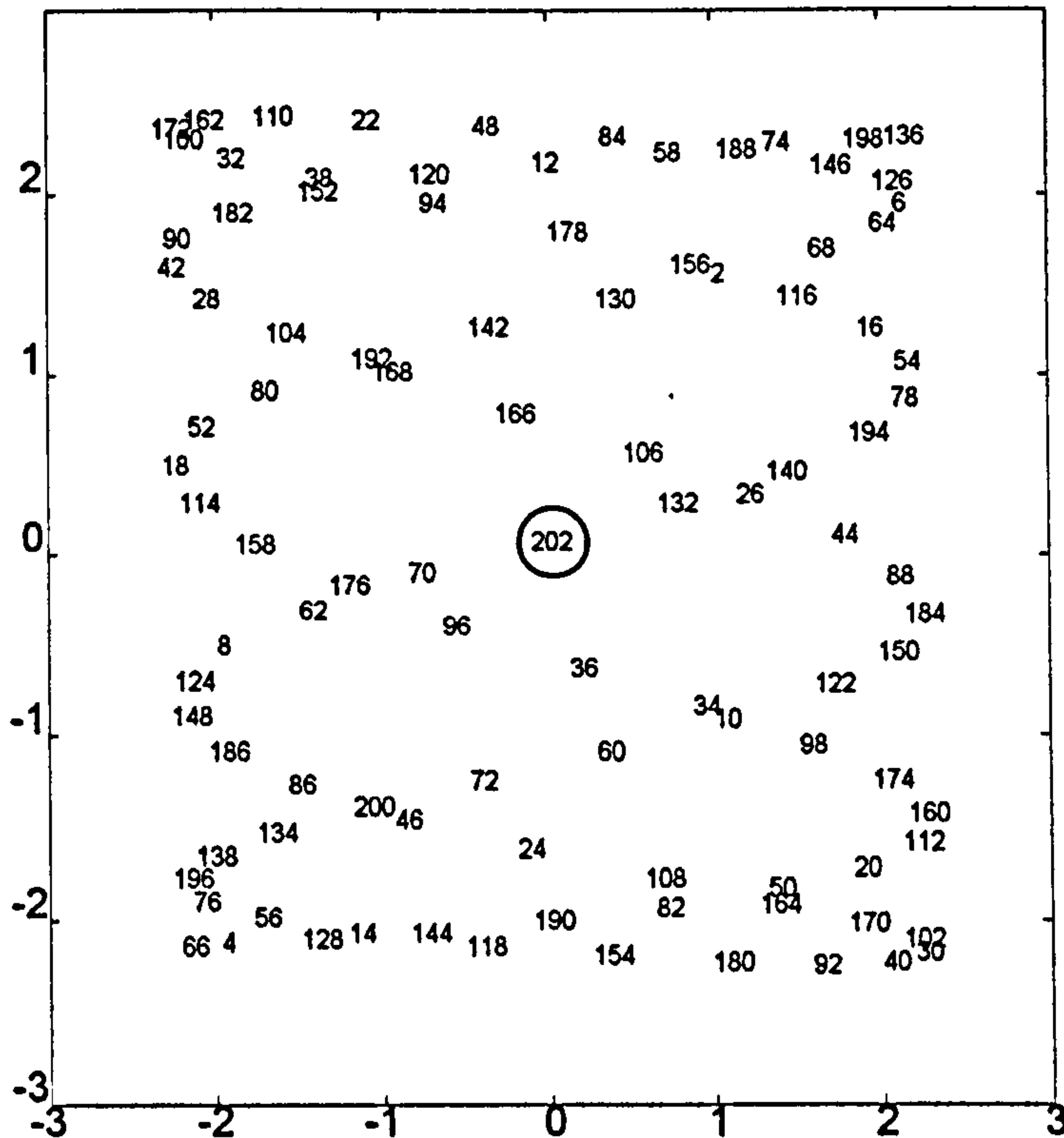


Figure 4.23 - Rotated Spot Pattern

Mirror Precision

As described in Section 4.3.1, errors in the mirror radii of curvature for an astigmatic cell can be compensated for by a combination of spacing and twist adjustments. The model has been used to ascertain the maximum error in the radii of curvature that can be accommodated by such changes. To determine this, the spacing and twist settings for the chosen re-entrant pattern are used, and a small error in the radii of curvature is incorporated into the model. The spacing and twist are then changed so that the beam once again exits the cell in the same pattern. This process is repeated until spacing and twist adjustments can no longer be used to compensate for the errors. The maximum error will dictate the required precision of manufacture.

Figure 4.24 shows the required twist and spacing adjustments against the percentage error for the chosen pattern {202,84,76}. For a 1% error in the radii, i.e. $R_x = R_x/1.01$, $R_y = R_y/0.99$, the required spacing and twist adjustments are 0.11 cm and 14.8

degrees. The maximum error that could be accommodated was $\sim 10\%$. This would represent a very relaxed tolerance on the radii. For the purpose of manufacture, a 1% tolerance was specified.

If spacing and twist adjustments were not possible, the model predicts a radii of curvature tolerance better than 0.001 % would be necessary. Such tolerances would practically be difficult to achieve and would certainly be prohibitively expensive to manufacture.

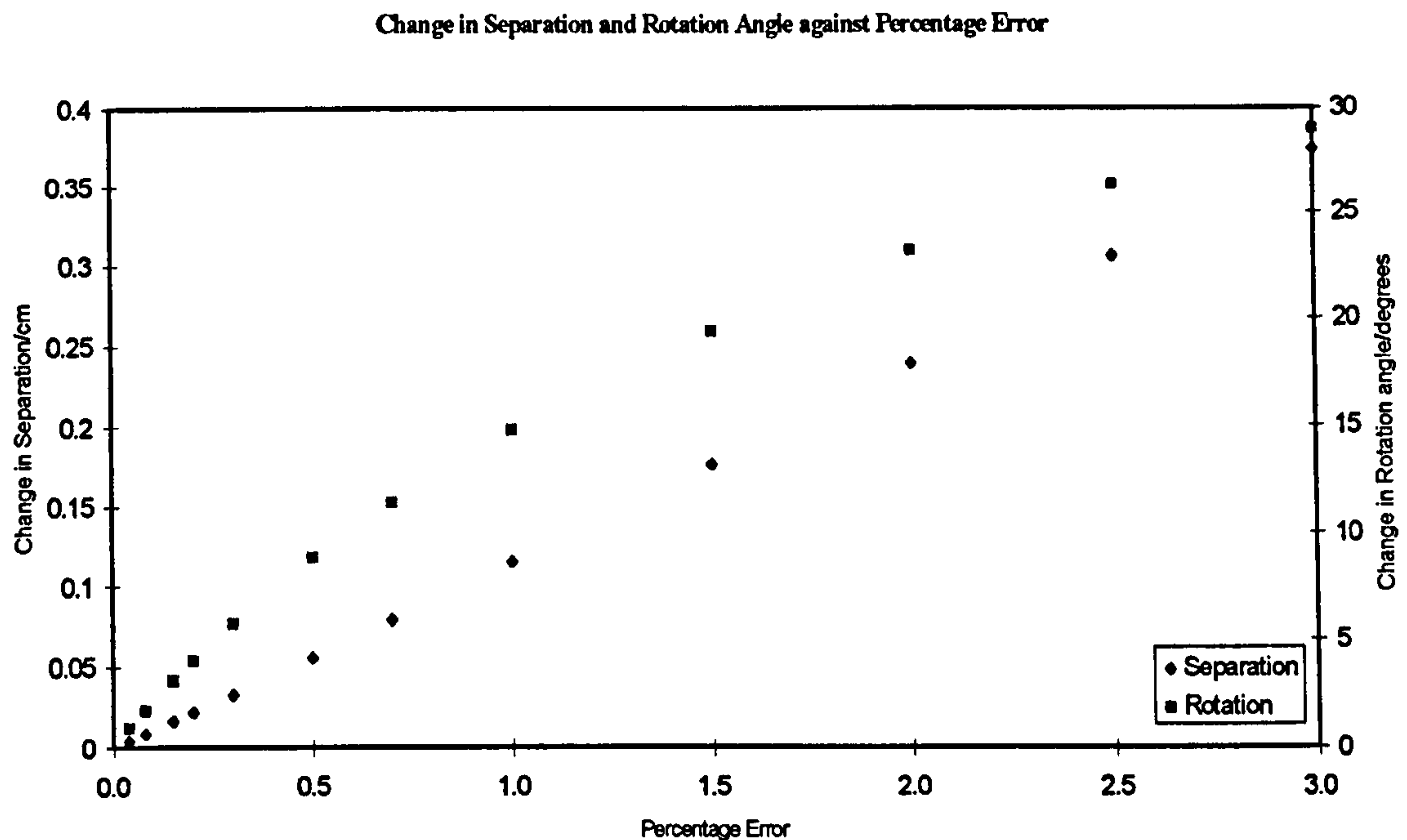


Figure 4.24 - Spacing and Rotation Changes against Percentage Error

Reflectivity

The coating chosen for the multipass cell, is extremely important. With over 200 reflections, a large percentage of the beam energy will be lost at the mirror surfaces. Examining the noise characteristics of multipass cells, shows that when the laser power falls to less than $1/e$ of its initial value, there will be no further gain in sensitivity. For the high power near-infrared lasers, this is not actually strictly true [44], however, minimising reflection losses is an important factor in providing good instrument performance. A reflectivity of 99.2 % would allow $\sim 20\%$ of the beam power to be transmitted after 202 passes. It was felt that this would certainly be adequate.

The cell will be used with many different laser sources in the near and mid-infrared (0.8 - 10 μm), therefore the coating must provide the specified reflectivity over this range. Particularly important is that it should also have a high enough reflectivity ($\sim 98\%$) in the visible (0.6 μm), so that the pattern and exit beam of the trace laser (HeNe) can be seen. The coating must also be able to withstand the hostile environment it would experience in field applications.

Discussions with coating vendors revealed that coating LH 41 was ideally suited. This was a broadband dielectric protected silver coating with a specified reflectivity of $\sim 98\%$ at 0.6 μm and $> 99.2\%$ from 1.0-10 μm .

Mirror and Coupling Hole Diameter

A mirror diameter of 7.5 cm was chosen. This would easily allow up to 200 passes to be supported within the cell, as shown by the pass number calculations in Section 4.3.4. Models of the spot pattern on a mirror of that size, showed that the distance of approach between the exit spot and its nearest neighbour was ~ 7 mm. The coupling hole radius was chosen to be half this distance, i.e. 3.5 mm. In order to allow easier beam injection, the hole was tapered and opened out to 9mm diameter at the back of the mirror.

These parameters represent a conservative design, and while it would be possible to support a similar number of passes on a mirror half this size, the chosen values represents a compromise between a small cell mode volume and the reduction of fringing effects and good spacing tolerance.

Material of Manufacture

The chosen material of manufacture was aluminium. This was chosen because it is easily machinable, has a low density, has high strength and is relatively cheap. The main drawback of this material is that it has a relatively large expansion coefficient compared to other materials such as INVAR ($\sim 1 \times 10^{-6}$) [45]. However, as discussed in Section 4.2.10, if the mirrors and the material separating them are made of the same material the cell will expand/contract isotropically, allowing the same re-entrant solution to be supported within the cell. Consequently, the expansion coefficient was not an influential factor in the choice of material.

Other Parameters

The specification of the other cell parameters are listed below.

Surface Quality - scratch dig 40/20

Surface Accuracy - $\lambda/2$ at 600 nm

Mounting Arrangements - 4 M4 Tapped Holes (see machine drawings-Appendix 4)

The mirrors were made by an American company, Opticon [46], at a cost of \$7000.

This included a one of tooling charge of \$2500 and a coating charge of \$700.

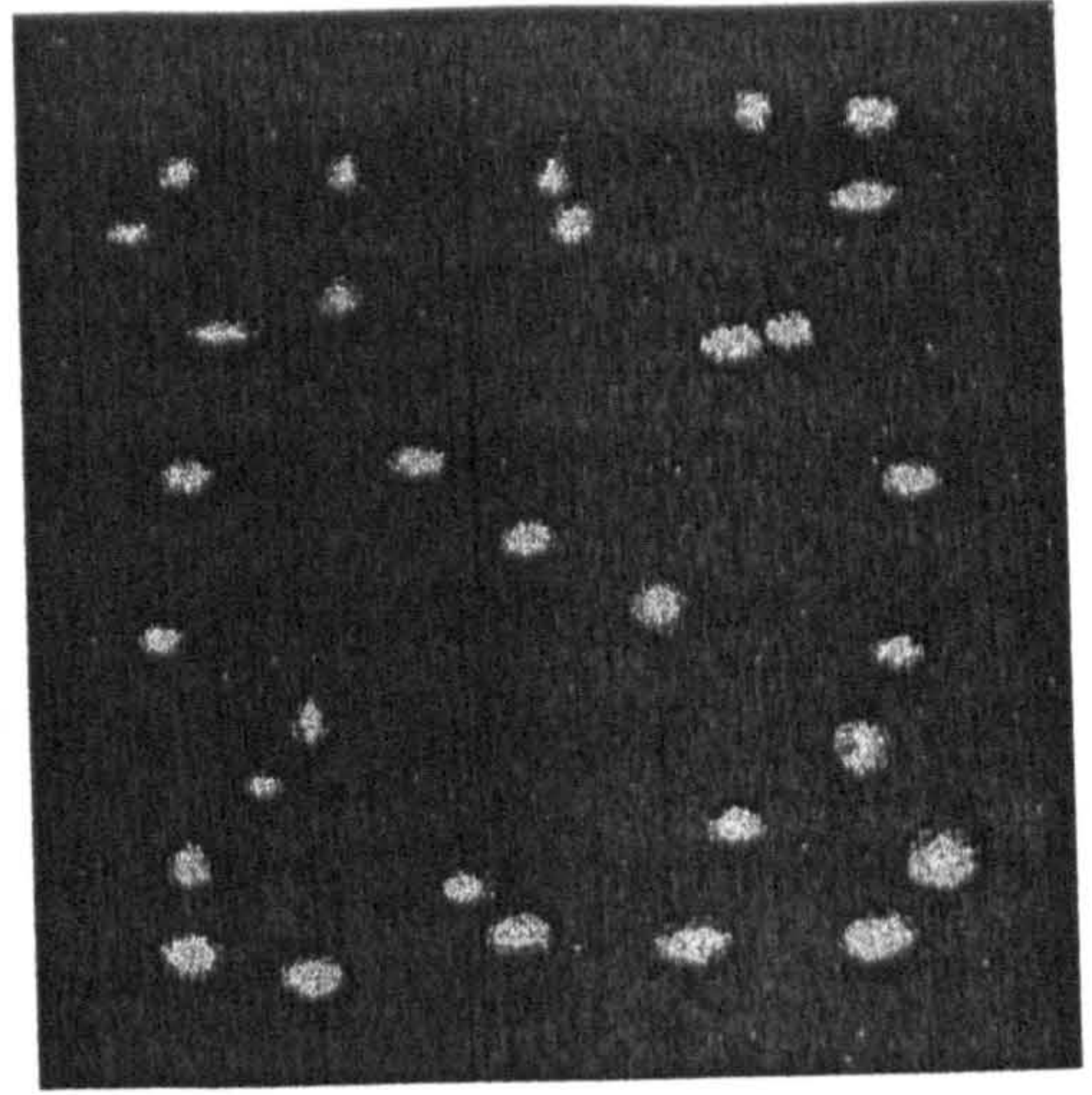
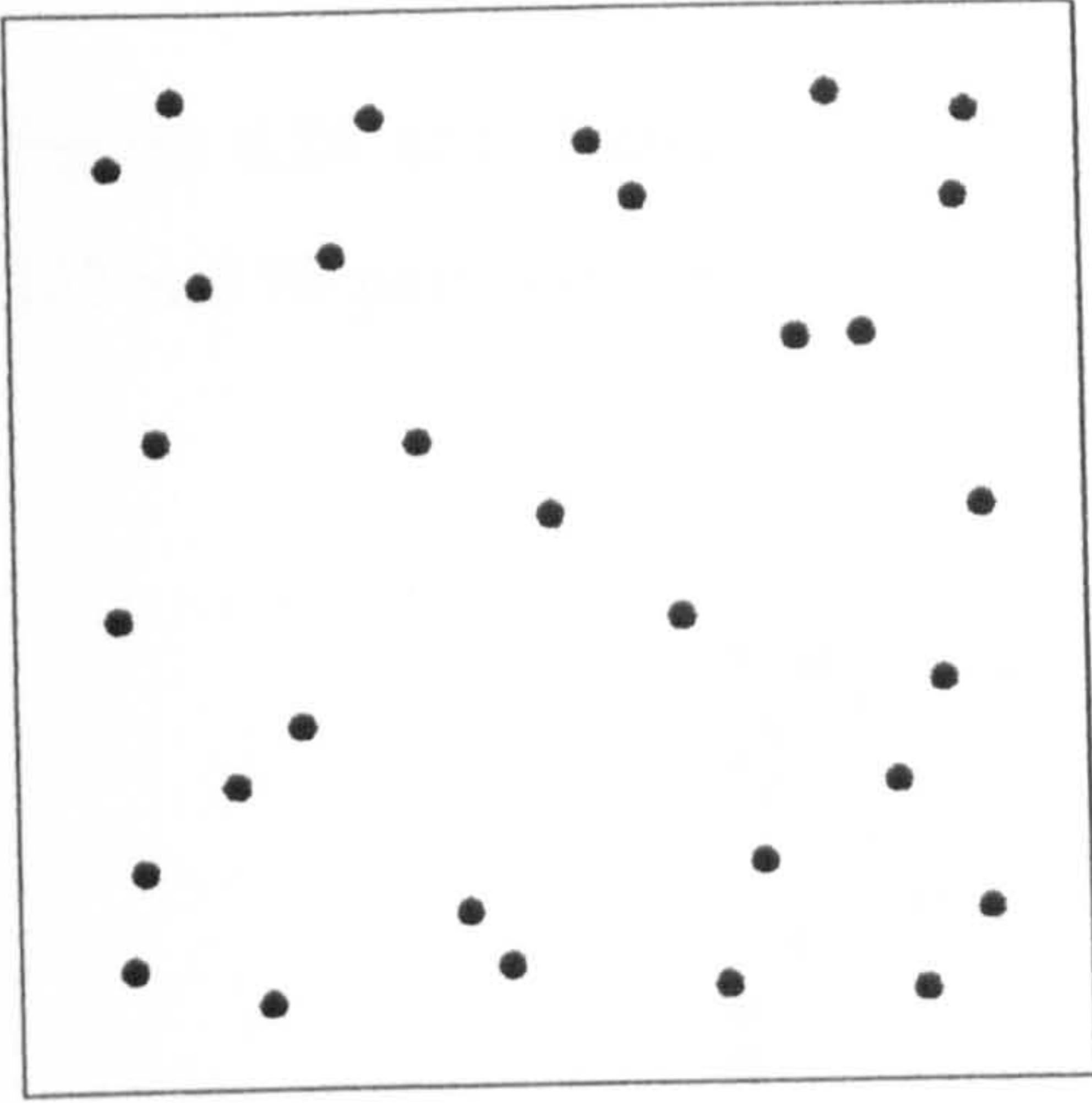
The experimental observations obtained with these mirrors were compared with the modelling predictions. The findings are discussed in the next section.

4.3.11 Modelling Predictions and Experimental Observations

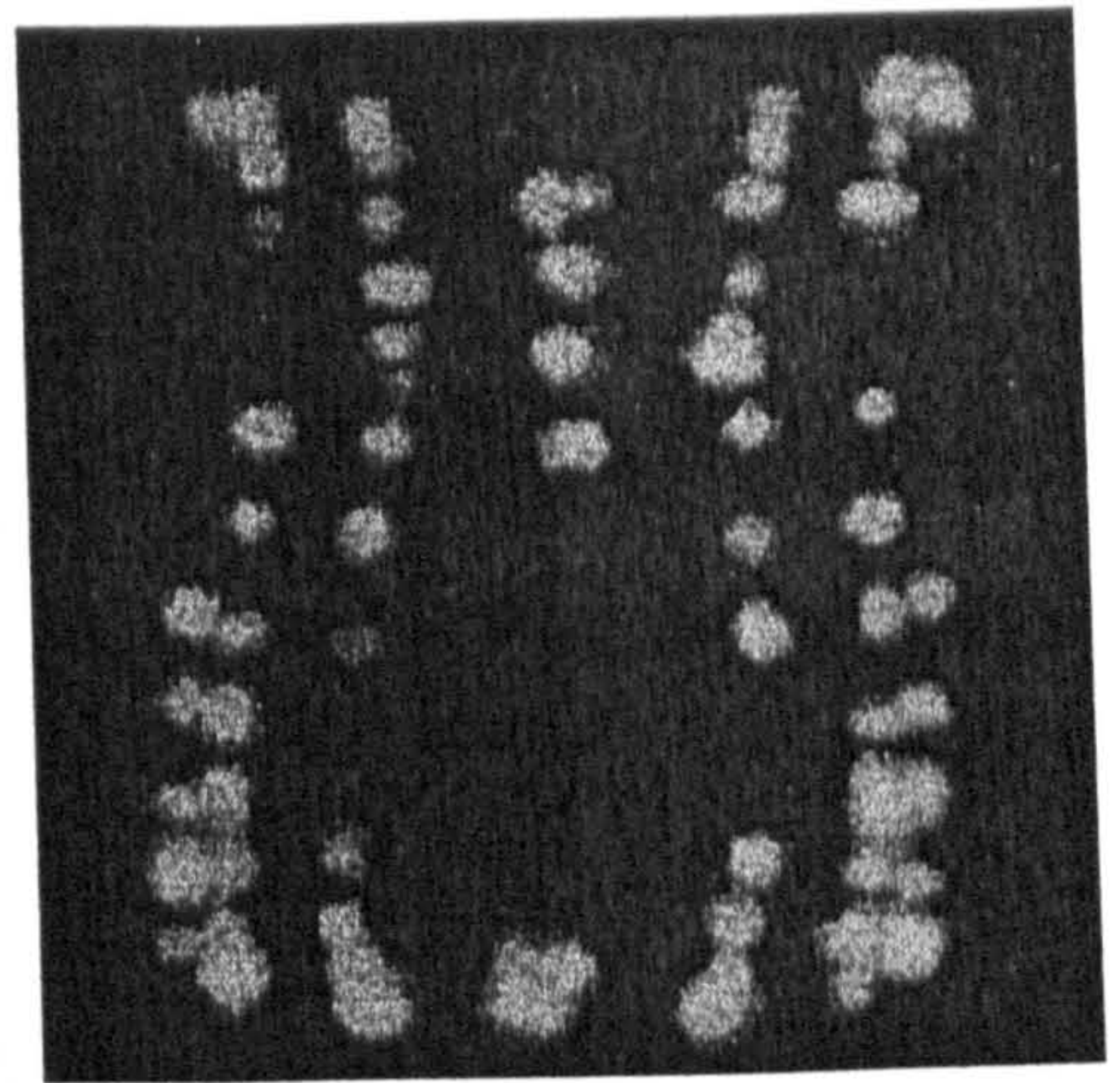
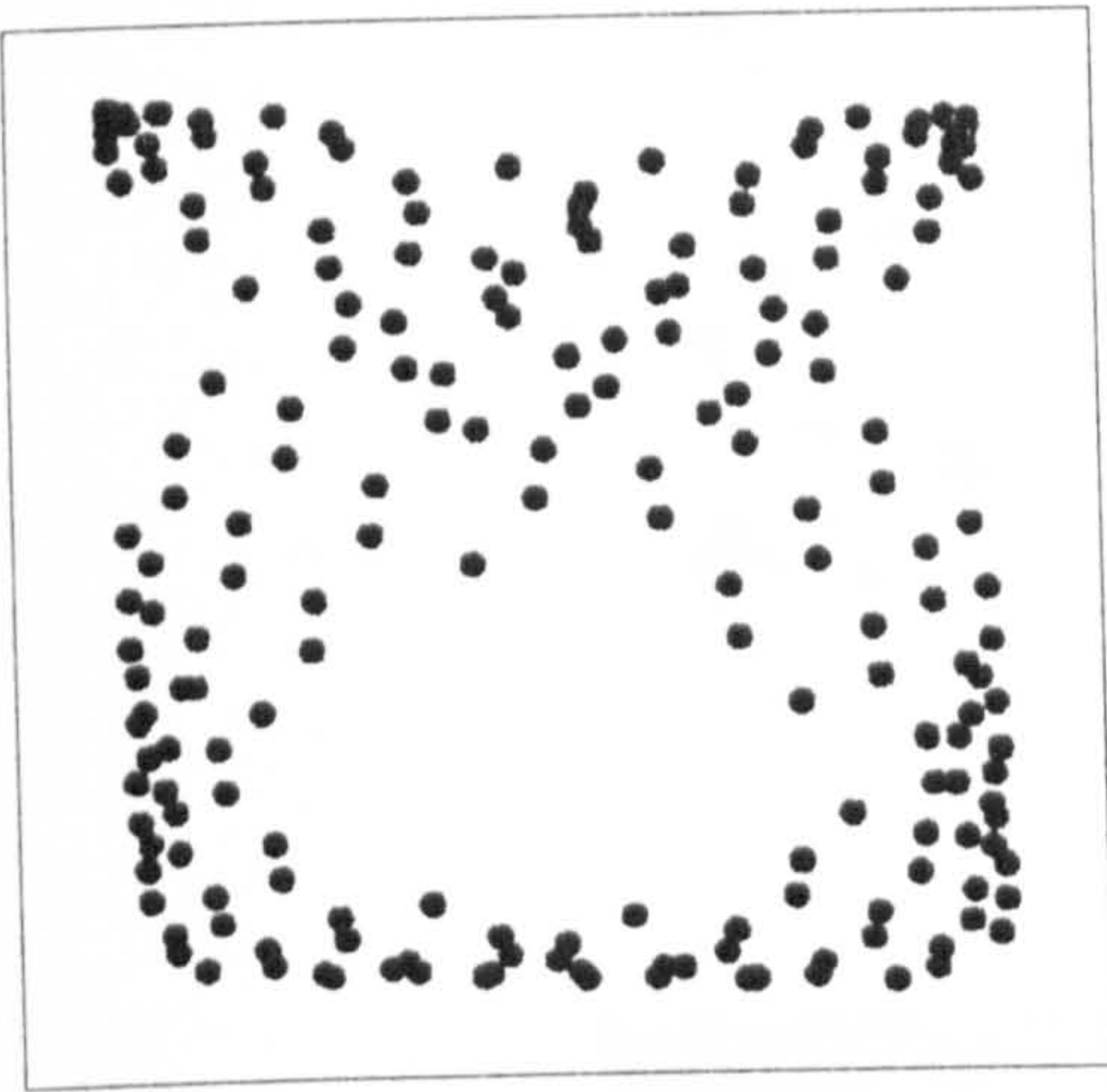
The astigmatic mirrors were set up on an aluminium breadboard. The front coupling hole mirror was mounted in a Newport Gimbal mount (model 605-4-DM). This allowed the mirror to be tilted about the X and Y axes with a resolution of 0.01 degrees. The back mirror was mounted on a Newport rotation stage (model M-UBG80CC, resolution 0.001 degrees), which in turn was attached to a micrometer stage with a resolution of 5 μm . This arrangement provided all the degrees of movement and the necessary resolution to allow a detailed comparison with the modelling predictions to be carried out.

202 Pass Solution Set Up and Observed Spot Patterns

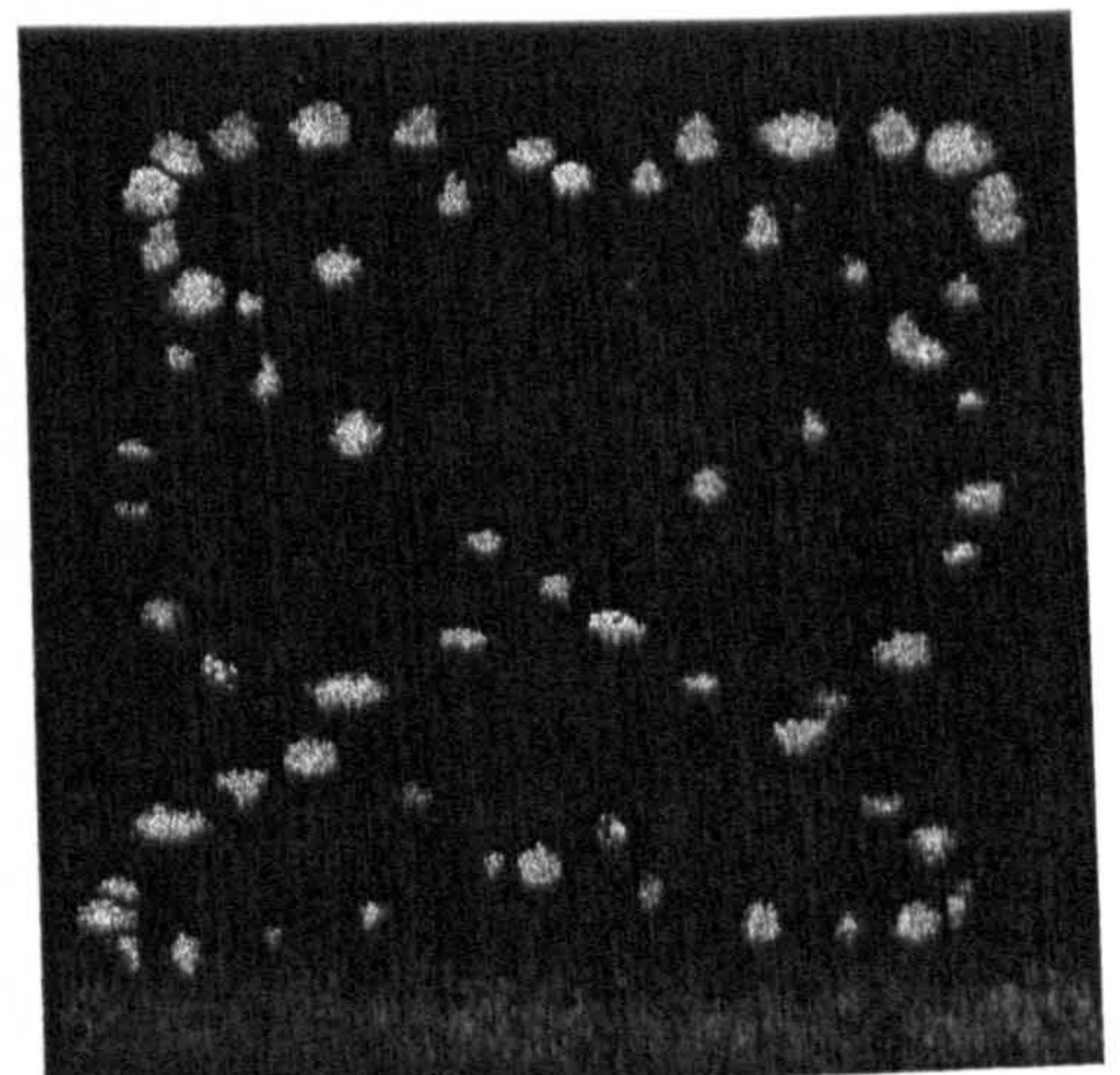
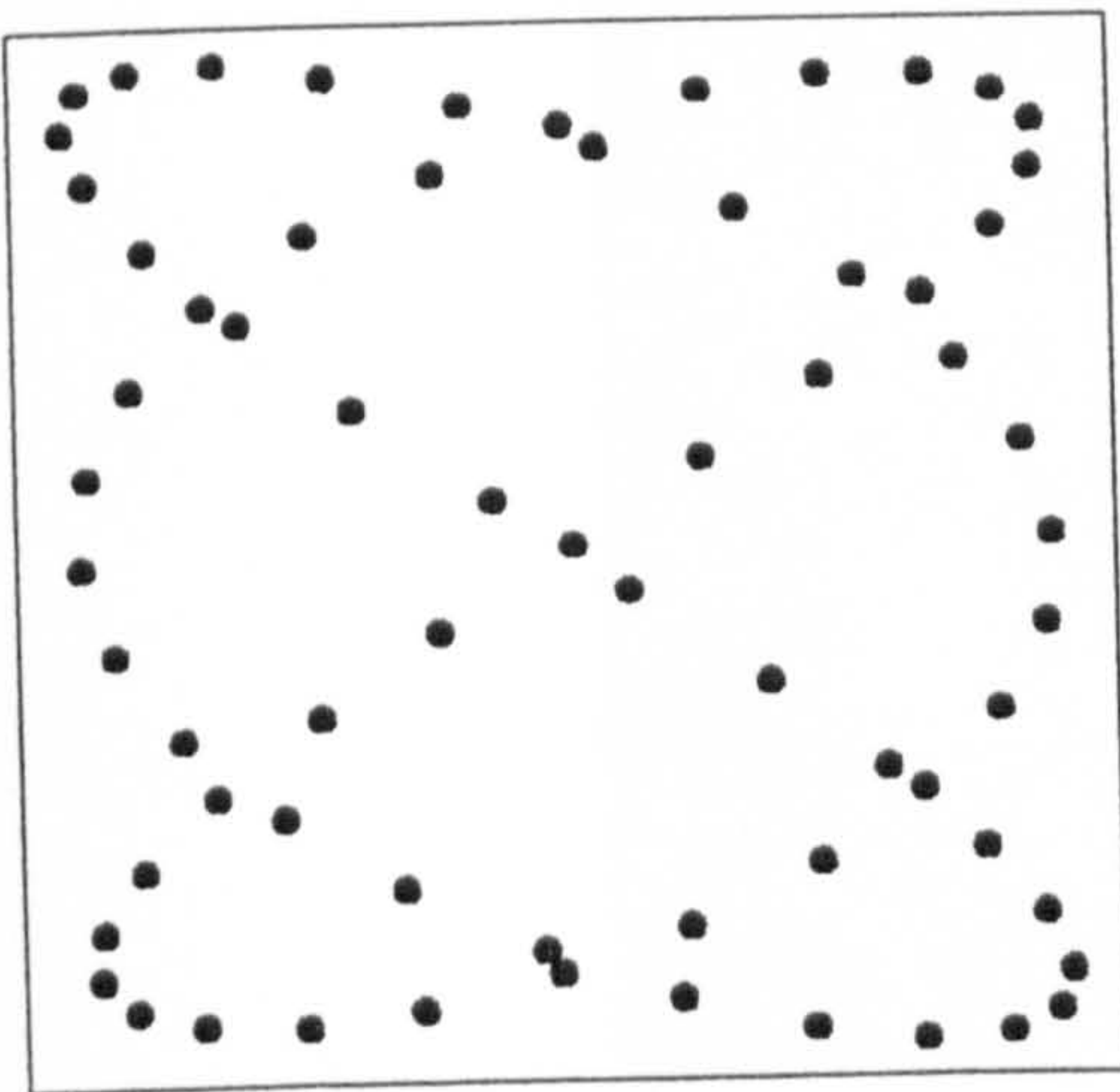
As described in Section 4.3.8, the 202 pass solution was chosen with a low pass marker solution nearby (see Figure 4.20). Although the marker solution was easily found, it proved difficult to move from this solution to the 202 pass pattern in one movement. The model was therefore used to find intermediate patterns that could be used as *stepping stones* between the two solutions. This technique proved to be far more successful, and once it had been completed a couple of times, the 202 pass solution could be found in a matter of minutes. Figure 4.25 shows the modelled marker patterns. Beside each one is a picture of the spot pattern that was actually observed with the mirrors.



$d = 50.50 \text{ cm}$, $\tau = 0^\circ$, $N = 62$



$d = 50.50 \text{ cm}$, $\tau = 19.9^\circ$, $N = 400$



$d = 50.4 \text{ cm}$, $\tau = 19.9^\circ$, $N = 134$

Figure 4.25 - Marker Patterns

Figure 4.26 and **Figure 4.27** show the modelled and observed spot patterns for the 202 and 98 pass solutions.

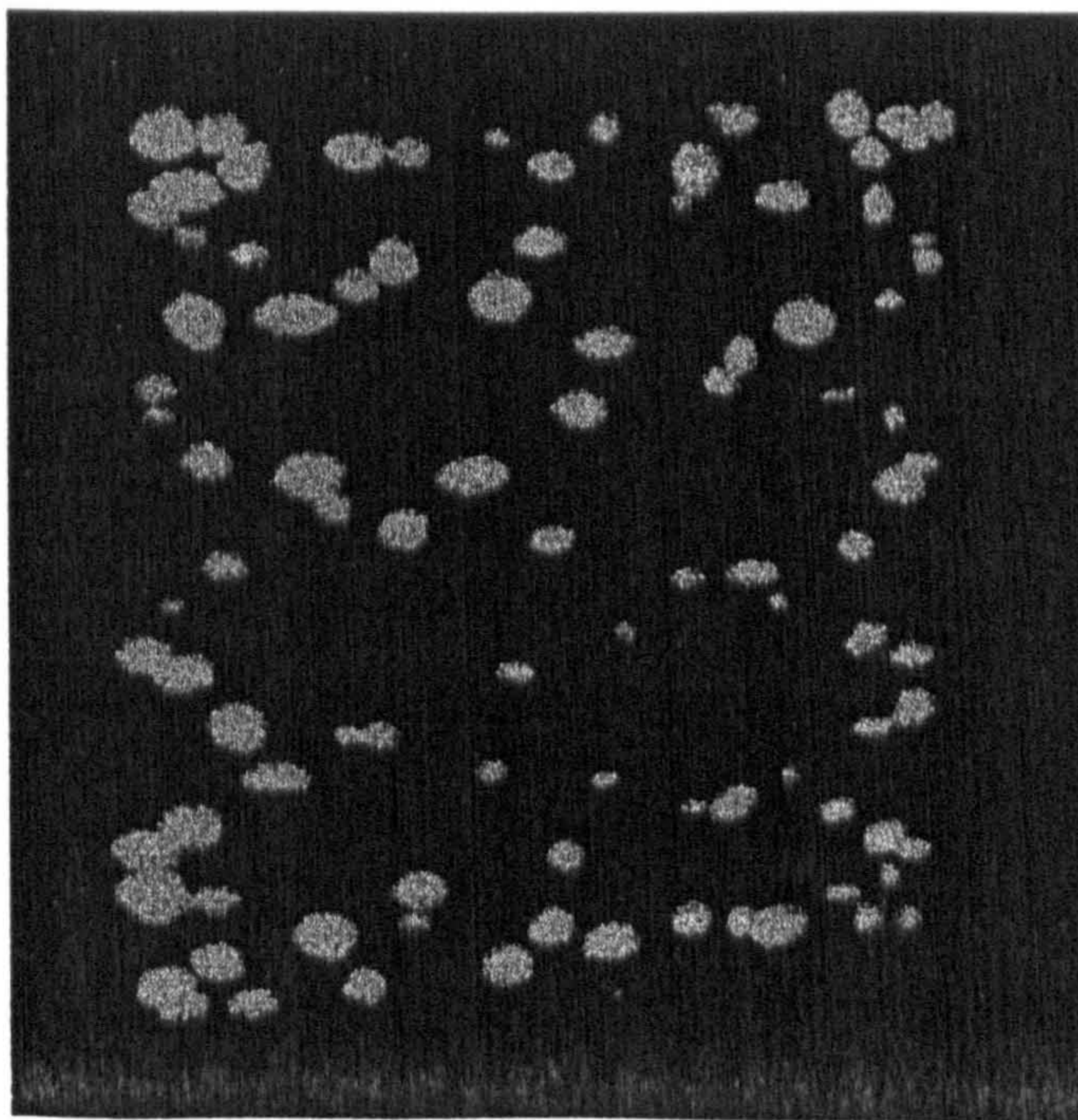
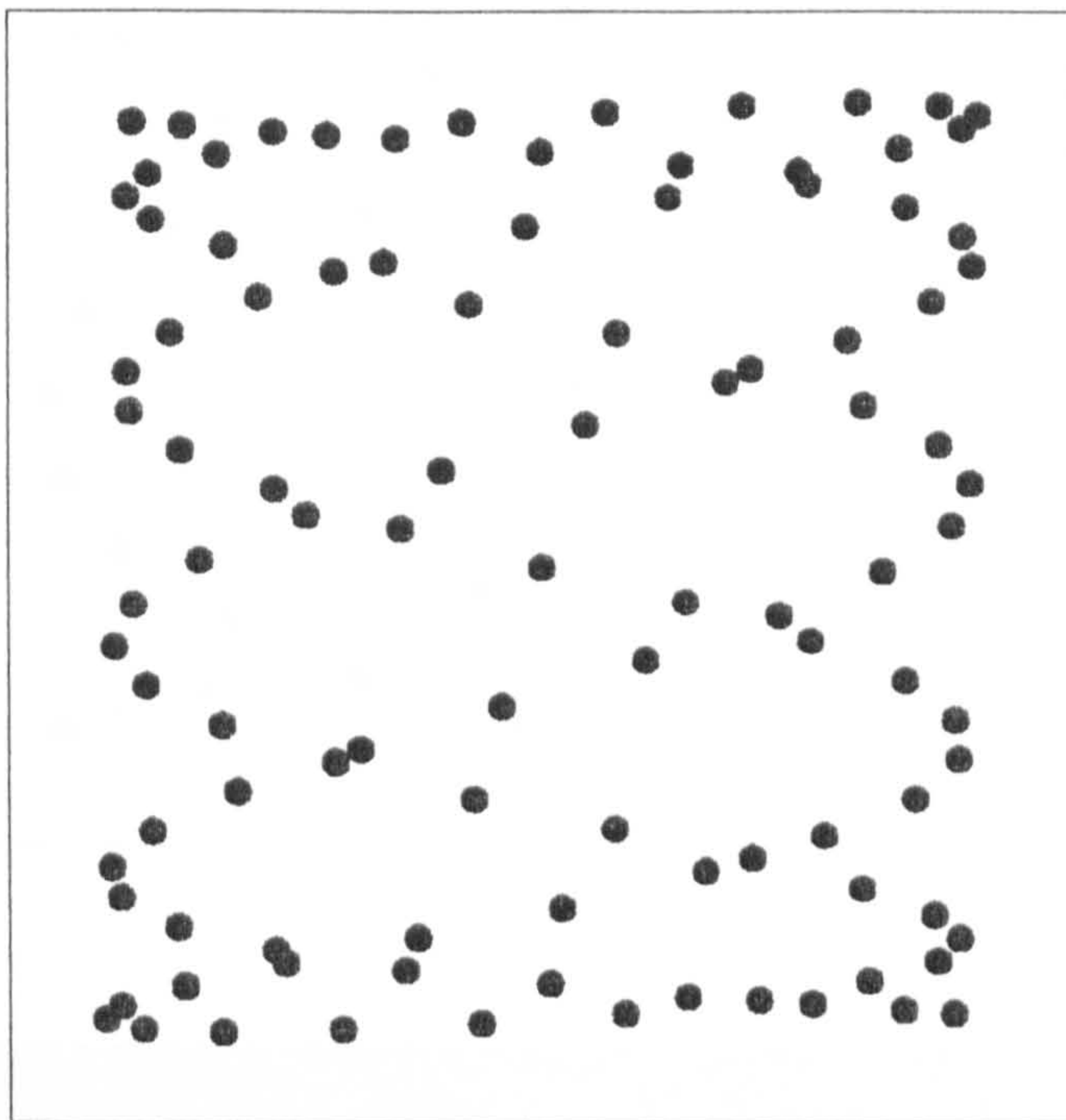


Figure 4.26 - 202 Pass Solution

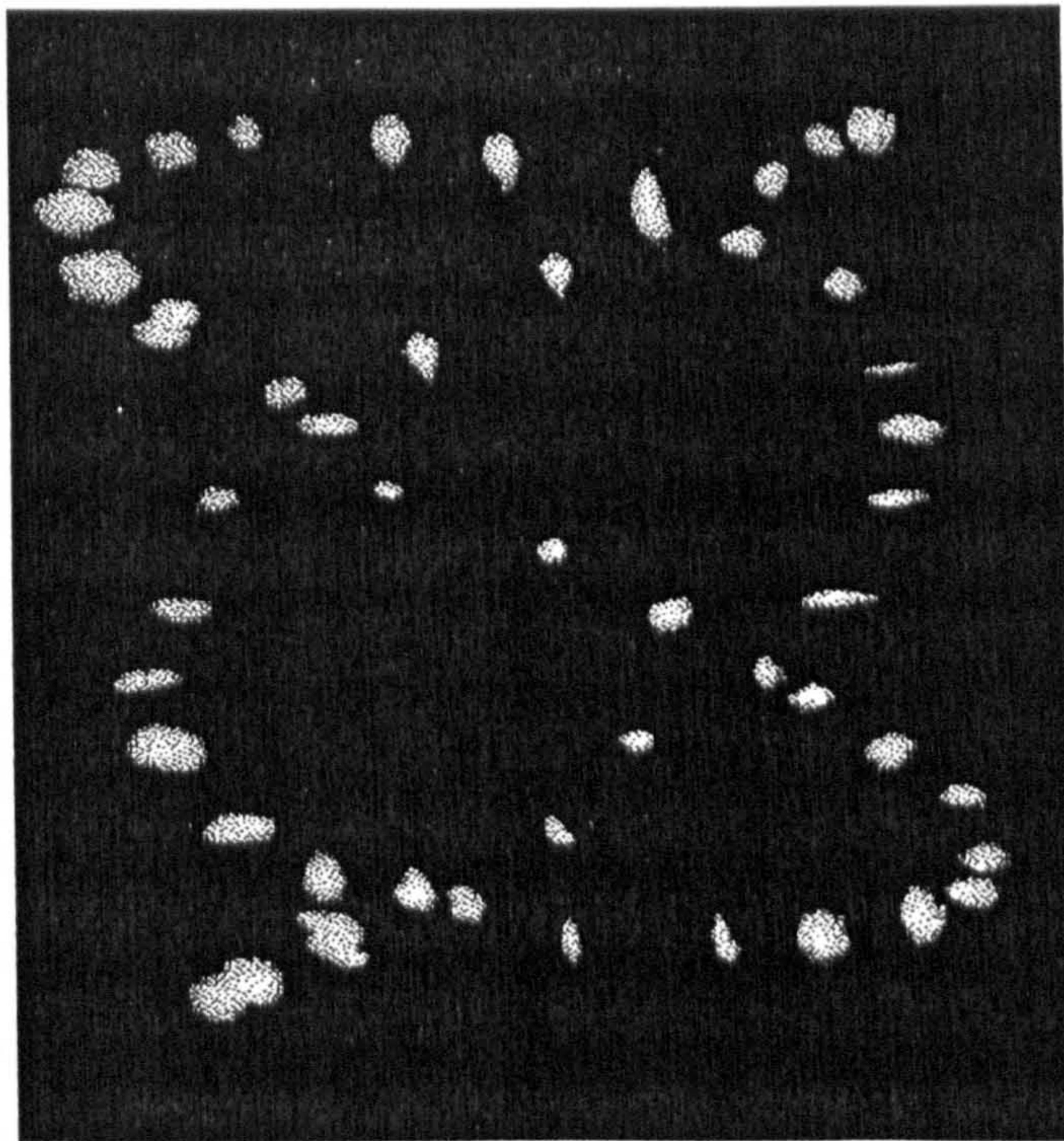
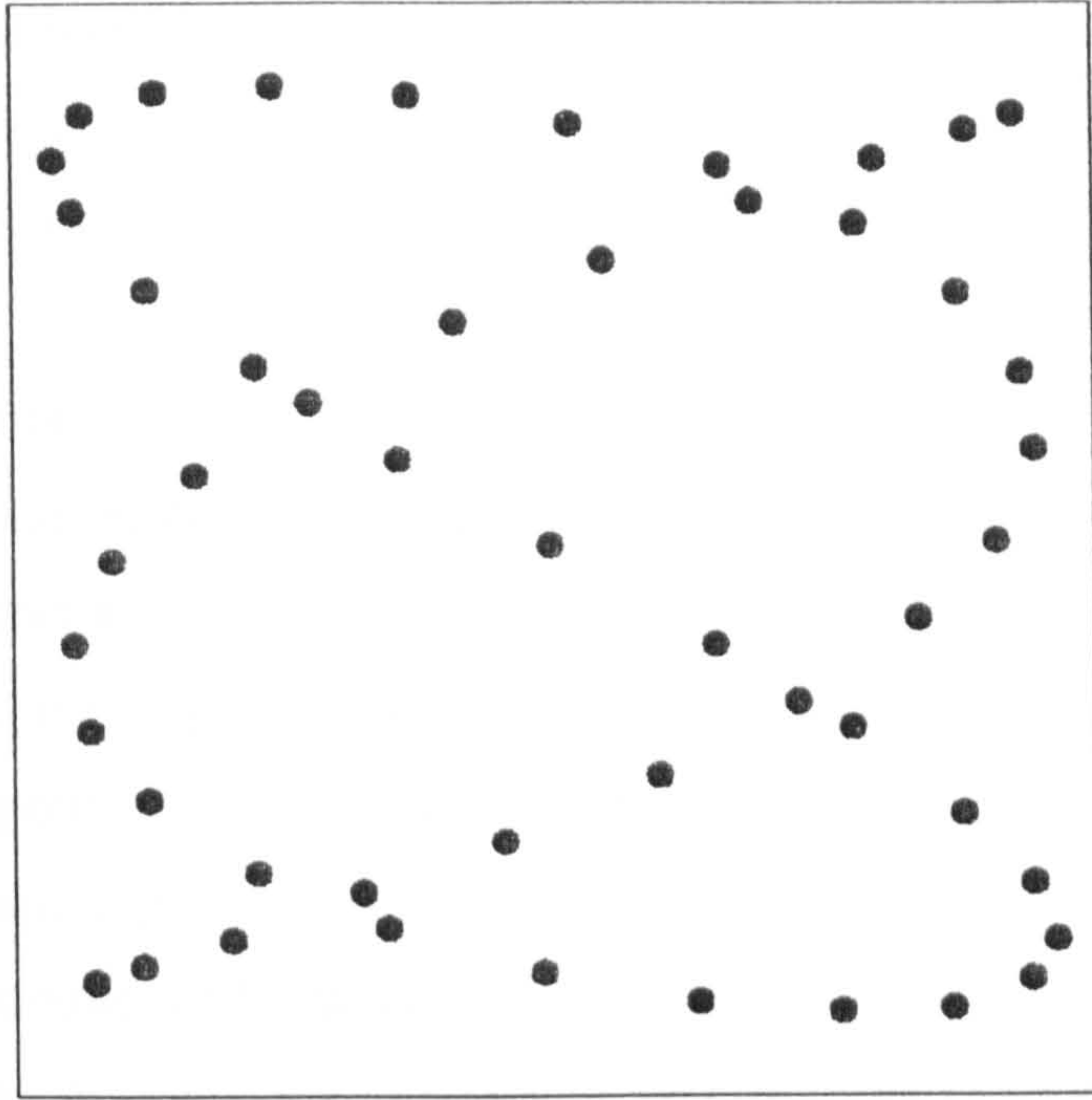


Figure 4.27 - 98 Pass Solution

It is seen that a good match between the modelled and observed patterns was achieved. The variation in spot size results from intensity variations. The 202 pass solution

appeared at a rotation angle of ~ 20 degrees, and a separation of 49.9 cm while the 98 pass solution was found at 98.9cm and ~ 17.8 degrees. Using the model it has been calculated that the error in the radii of curvature is less than 0.1 %. This is well within the specified accuracy.

Spacing Tolerance

The spacing tolerance, defined in Section 4.2.8, is the separation change over which the cell can support a given re-entrant solution. This was measured experimentally for both the 202 and the 98 pass solutions. The tolerances were measured to be 0.75 mm and 2.1 mm respectively. The model predicted tolerances of 0.76 mm and 2.2 mm, while the analytical expression derived for the non astigmatic cell predicted 0.69 mm and 2.8 mm. It is concluded that the modelled and observed spacing tolerances were in good agreement. A reasonable agreement was also obtained using the analytical expression for the conventional Herriott cell.

If the spacing tolerance for the 98 pass solution is compared with the measured tolerance for the 48 pass solution, in the conventional cell (Section 4.2.15), it is seen that the higher pass solution can accommodate greater changes in the mirror spacing. This is possible because the astigmatic cell uses the mirror surfaces far more efficiently, and can consequently accommodate larger coupling holes. This property means that the spacing tolerance for the 202 pass solution should be practically achievable in a field instrument.

Rotation Tolerance

Changing the relative angle between the two mirror axes, will change the effective radii of curvature and result in the movement of the reflex spots on both mirrors. The rotation tolerance is defined as the maximum rotation that can be accommodated before another pattern will be supported within the cell. The model was used to predict the rotation tolerance for both the 202 and 98 pass solutions. The predicted values were ± 1.23 degrees and ± 1.52 degrees. The measured tolerances were ± 1.25 degrees and ± 1.5 degrees. Again, the modelling predictions and experimental observations were in excellent agreement. The positions of the reflex spots were

significantly more sensitive to changes in spacing than in rotation. It is therefore expected that such tolerances could be easily met in a field application.

Tilt Tolerance

The model has been used to investigate the effects of tilt on the astigmatic Herriott cell. The behaviour was found to be identical to that of the conventional cell with the position of the exit beam on the front mirror being independent of tilt. However, tilt will cause movement of intermediate spots on the mirror surfaces and can drive them out the coupling hole. The maximum tilt that can be tolerated before such an event occurs is defined as the tilt tolerance. The measured tolerance for the 202 and 98 pass solutions was 0.6 degrees and 0.66 degrees respectively. The modelled results were in good agreement.

Reflectivity

The mirror reflectivity was measured in the visible and at $1.65\ \mu\text{m}$ by recording the laser intensity before and after the beam had circulated within the cell. The measurements were taken at 62, 98 and 202 passes. The reflectivity in the visible was $\sim 97.8\%$, while at $1.65\ \mu\text{m}$ it was $\sim 99.25\%$. This agrees well with the specified performance of the coating. The good agreement in the measured reflectivity at three different pass numbers is a good indication that the actual pass number and modelled pass number were also in agreement.

Recorded Spectra

Spectra of ambient methane at atmospheric pressure were recorded with an open path astigmatic cell arrangement. **Figure 4.28** shows a typical spectrum obtained. This is the Q (6) manifold, consisting of six individual transitions that are heavily blended at atmospheric pressure, hence the slightly unusual lineshape.

The inability to evacuate the cell at the time this spectrum was recorded meant that it was not possible to check for slowly varying etalon fringes. Noise levels on this spectrum were equivalent to a minimum detectable absorption of 6×10^{-7} .

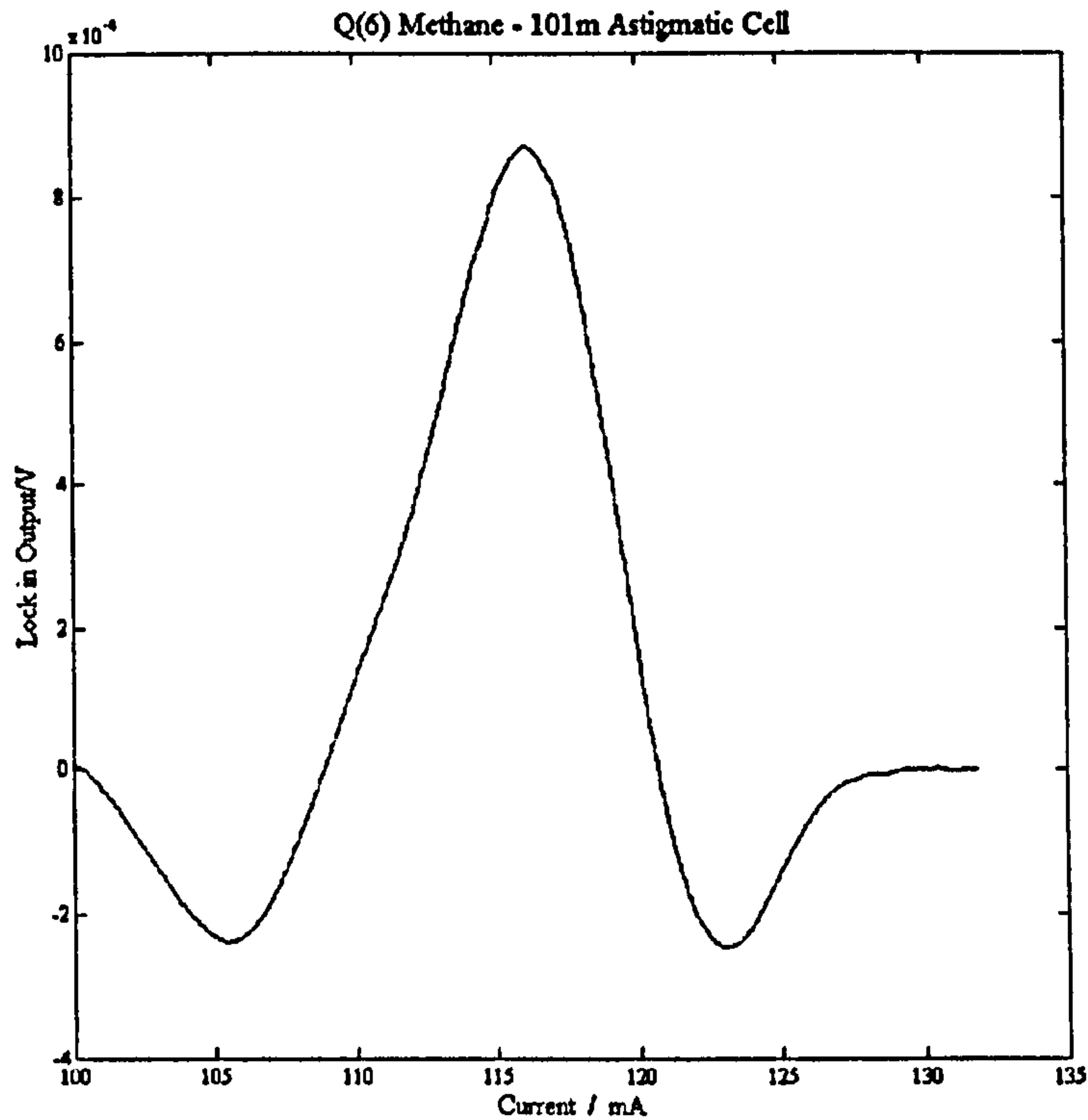


Figure 4.28 - Ambient Methane, Q (6) transitions (101m Astigmatic Cell)

4.3.12 Conclusions

A model of the astigmatic Herriott cell, based on a 4x4 matrix description of the optical elements, has been written in the MATLAB language. This model has been used extensively to design an astigmatic cell for TDLAS measurements.

The astigmatic cell behaviour was found to be substantially similar to the conventional Herriott cell, however, the astigmatic cell uses the mirror surfaces more efficiently, allowing higher pass numbered solutions to be supported. Having a more even distribution of spots also allowed for an enlarged coupling hole diameter giving better spacing tolerances and a reduced possibility of fringing. The chosen design provided 202 passes at a separation of 50 cm and 98 passes at a separation of 99 cm. The modelling predictions and experimental observations were found to be in excellent agreement

4.4 Other Work

This section details other work carried out on multipass cells, including investigation into multiple beam optical arrangements where more than one beam can be supported within the cell. Additional ways of increasing the number of passes that could be supported within the Herriott cell were also investigated.

4.4.1 Multiple Beam Cells

Another advantage of the Herriott cell, is its ability to support more than one independent optical path. The ALIAS instrument [11] supported four independent beams, each going through 80 passes, in a conventional Herriott cell, allowing different gas species to be measured simultaneously. Four coupling holes were placed in the front mirror with four beams entering and exiting through one coupling hole each. Investigation into the possibility of supporting four independent paths in the astigmatic cell was investigated with the model and the findings confirmed experimentally.

The spot pattern for four beams being injected into the cell is shown in **Figure 4.29**. Each is injected at a slightly different angle and goes through 82 passes before exit. An advantage of the astigmatic cell is that all the beams can enter and exit through one coupling hole in the centre of the mirror. This facilitates a simpler input/output optical system than was used in ALIAS. In addition, four beams can easily be supported on 7.5 cm diameter mirrors, whereas the ALIAS instrument required 15 cm diameter mirrors. Martin [10] has successfully validated the modelling predictions for multiple beam cell operation with the astigmatic mirrors described previously.

4.4.2 Additional Techniques to increase the pass number of a Herriott Cell

Investigation into ways of increasing the pass number of the conventional Herriott cell without the expense of designing astigmatism into them was undertaken. The first method investigated, involved placing a perturber mirror into the system. This

secondary mirror picked off the last spot before exit, directing the beam into a new elliptical pattern. This re-direction could be repeated several times. In this case several ellipses are seen and a corresponding increase in the pathlength is obtained. Figure 4.30 shows a schematic of the observed spot pattern. Experimentally, it was shown that this method could be used to increase the pathlength, however some of the inherent stability properties of the cell were lost. It was also difficult to get the beam to exit out of the coupling hole. Consequently, it was concluded that the perturber mirror system was not suited to field applications.

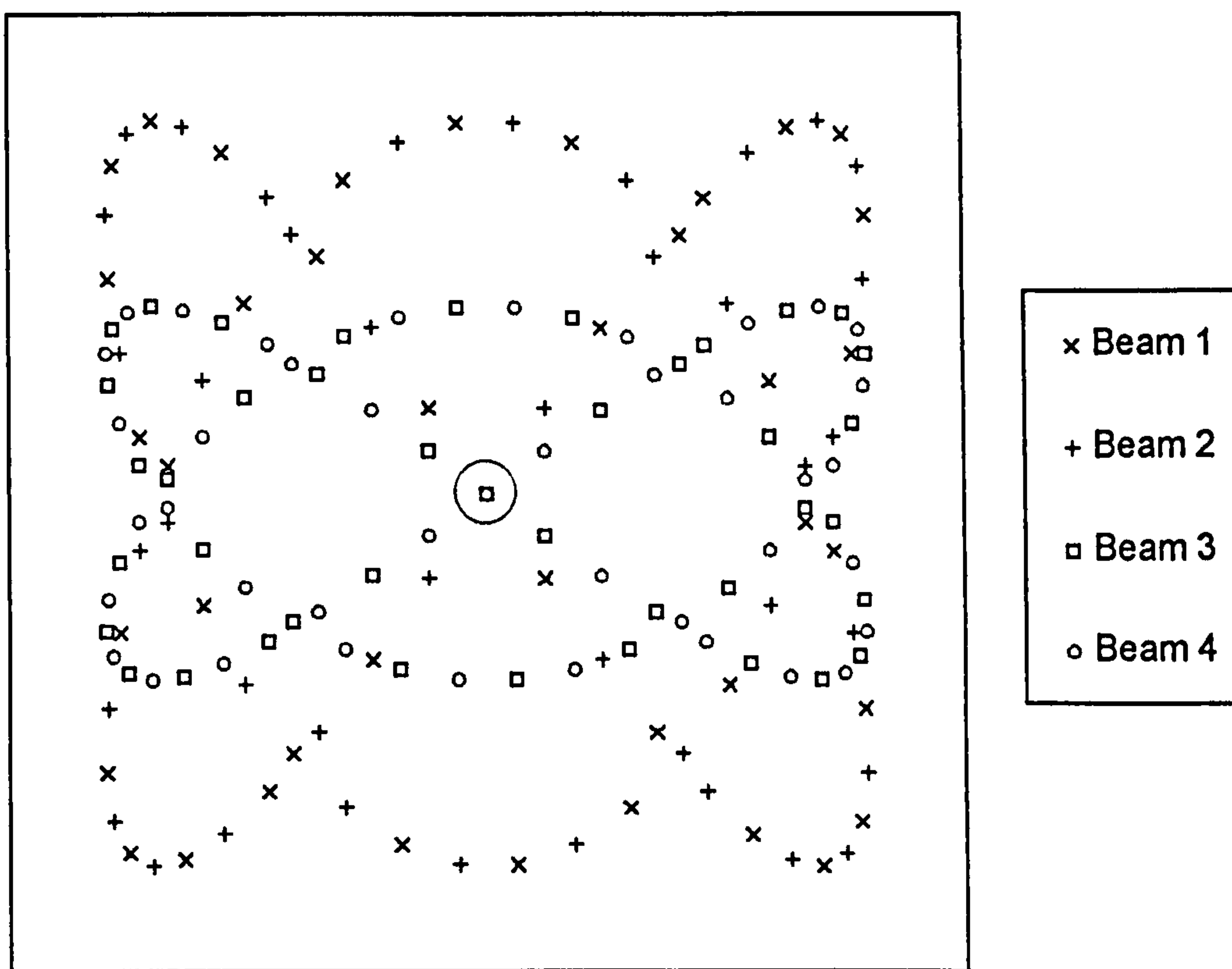


Figure 4.29 - Modelling prediction for Multiple Beam Herriott cell

Another method of increasing the cell pathlength is to stress the mirror surface so that it becomes astigmatic. Experimentally this was achieved by over tightening two of the four locating bolts which held the mirror in place This stressed the surface along one

axis, thus providing the necessary astigmatism. This technique was shown to increase the pathlength by a factor of three to four.

Not knowing the exact astigmatism in the mirrors, however, meant modelling was impossible. Consequently, it was difficult to achieve re-entrance. It was concluded that this method would also not be suited to field applications. A particular concern was that temperature induced creep would change the level of astigmatism in the mirrors and hence the pass number.

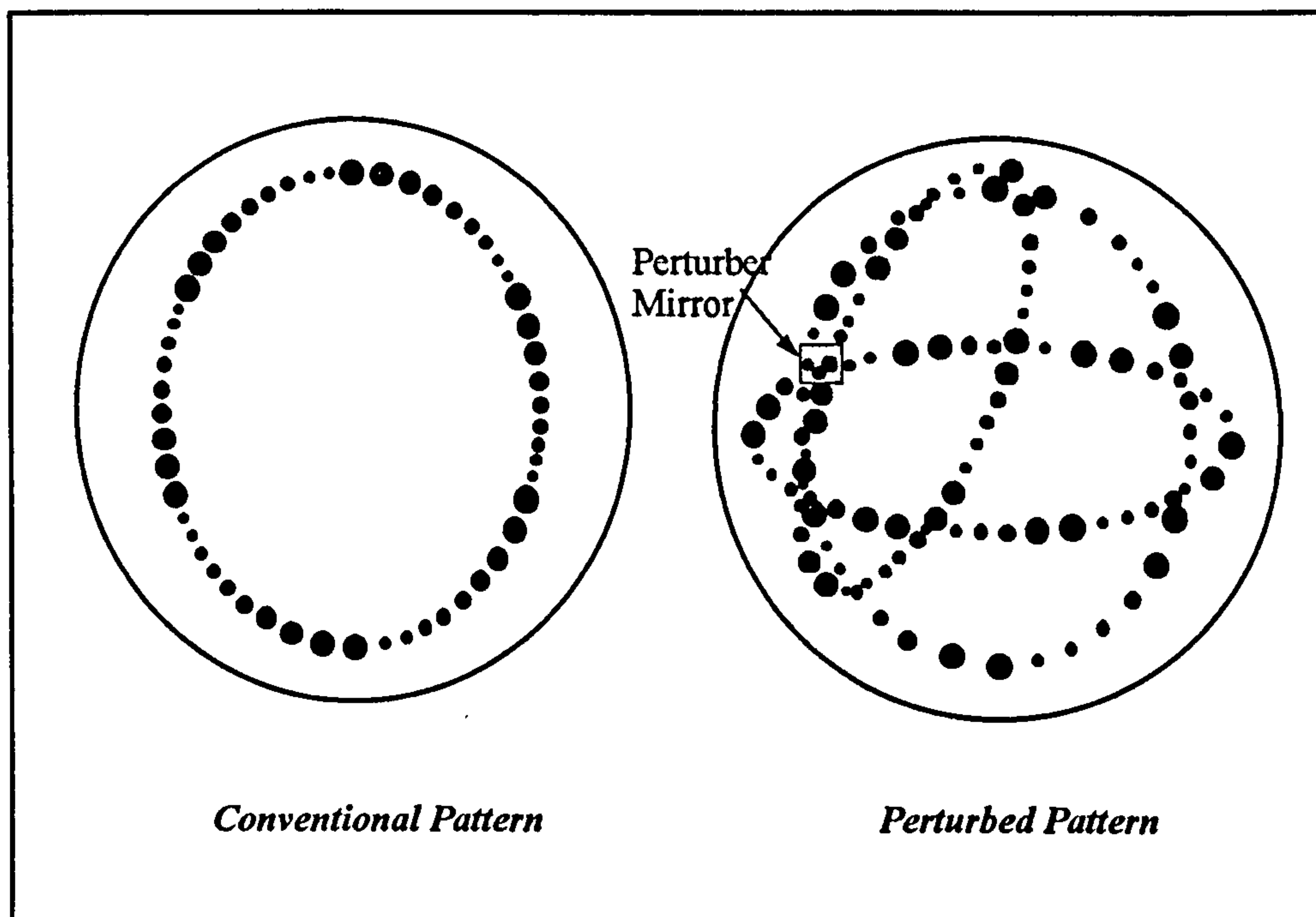


Figure 4.30 - Spot Patterns for Conventional and Perturbed Herriot Cell

Chapter 5 -

Ground Based Tunable Diode Laser

Absorption Spectrometer (TDLAS)

Applications and Design

5.1 Introduction

This chapter describes the design and development of a high resolution (0.00033 cm^{-1}) ground based near-infrared tunable diode laser absorption spectrometer (TDLAS).

As discussed previously (Chapter 1), accurate and precise monitoring of trace gases and industrial pollutants has become increasingly important over recent years, due to increased concern about global warming, ozone depletion and degradation of air quality. While a wide variety of techniques exist for measuring these gases, the very low concentrations typically encountered, (parts per trillion (ppt) up to parts per million (ppm)) have resulted in tunable diode laser absorption spectroscopy becoming one of the leading techniques for high sensitivity atmospheric analysis of trace gas constituents.

One disadvantage of TDLAS is that each laser has an extremely small wavelength coverage (typically 3nm) . This usually precludes measuring more than one gas species with each device. For the development and testing of this instrument two DFB lasers were procured. The first, a device from Anritsu operated at 1650 nm and covered the Q branch of the $2 \nu_3$ band of methane. The second laser was manufactured by Thomson semiconductors and covered the $3 \nu_3$ nitrous oxide band at 1520 nm. A detailed description of these devices and their operating characteristics can be found in Chapter Three.

Methane and nitrous oxide both play an important role the chemistry of the atmosphere and are pollution products from large scale industry. More information about the potential applications of this instrumentation is given in Section 5.2.

The instrument described here has been designed to:

- Take advantage of near-infrared tunable diode lasers and detectors now available, which can be mounted in compact, lightweight packages requiring no cryogenic cooling.
- Use the available high power of near-infrared sources to achieve long atmospheric pathlengths and therefore high sensitivity.

- Use the high spectral resolution and high mode purity of near-infrared distributed feedback (DFB) lasers to obtain equivalent gas detection levels to mid-infrared instruments.
- Use the high detection bandwidth of near-infrared detectors to enable high frequency detection, reducing the effect of low frequency laser excess noise.
- Make use of the high quality beam profile of near-infrared lasers to minimise stray optical reflections and etalon fringes to obtain maximum sensitivity.
- Use the latest design of multipass cell to minimise etalon fringing and allow large optical pathlengths in small volumes.
- Employ rapid current tuning of lasers to obtain fast response measurements.

It was hoped that these design features would provide sensitivities close to those achieved with mid-infrared TDLAS.

Also included in this chapter is a study of the different detection schemes commonly used with TDLAS. This study was aimed at assessing which detection scheme was best suited to this particular application, with an emphasis on high sensitivity, practicality and cost effectiveness.

Finally, a detailed investigation into fringe removal techniques is described. Optical fringing is found to be one of the limiting factors in the obtainable sensitivity of TDLAS.

5.2 Near-Infrared TDLAS Applications

5.2.1 Introduction

This section describes potential applications for a ground based TDLAS. In particular, special attention has been given to applications involving the measurement of methane and nitrous oxide. These gases play an important role in tropospheric/stratospheric chemistry. A greater knowledge about the size and identity of sources and sinks of these species will help quantify and improve our understanding of the role these gases play in global warming and ozone depletion.

The instrument also has many potential applications as a monitor for atmospheric pollutants.

5.2.2 Methane and Nitrous Oxide Measurements

As detailed above, methane and nitrous oxide play an important role in the greenhouse effect and ozone depletion. Details of the role these gases play will now be discussed.

Methane

The effect of methane on global warming

Methane concentrations have increased from a pre industrial value of 700 ppb to 1780 ppb today [3]. The recent rate of concentration change per year (during the 1980's) has been 0.8%. This increase in concentration is a direct consequence of human activities, the main sources being rice paddies, animal husbandry, landfills, biomass burning and fossil fuel production and use [6].

The direct radiative forcing due to the increase in methane concentration since pre-industrial times is about 0.5 Wm^{-2} [3].

The effect of methane on ozone concentrations

A small amount (< 15%) of the CH₄ released at the Earth's surface diffuses into the stratosphere where it can act as a terminator for the Cl atom, by forming HCl:



Although HCl can recycle to Cl via reactions with OH, some diffuses down to the tropopause and is rained out, thereby acting as a permanent Cl sink. The observed increase in CH₄ may therefore result in lowered Cl atom concentrations and hence decreased Cl atom destruction of O₃. A doubling of CH₄ concentrations would cause an estimated 3% increase in the total observed ozone column [6].

Nitrous Oxide

The effect of nitrous oxide on global warming

Nitrous oxide concentrations have increased from a pre industrial value of 275 ppb to a current value of 330 ppb [3]. The recent rate of concentration change per year (over the past four decades) is 0.25% [3]. There are many small sources of N₂O both natural and anthropogenic. The main anthropogenic sources are from agriculture, biomass burning and a number of industrial processes (adipic acid and nitric acid production). Natural sources are expected to be twice as large as anthropogenic sources [6].

The radiative forcing due to the change in N₂O since pre industrial times is about 0.1 Wm⁻² [3].

The effect of nitrous oxide on ozone concentrations

Ground level emissions of N₂O are responsible for the bulk of oxides of nitrogen in the stratosphere. In the stratosphere, N₂O is mainly broken down by photolysis but is also broken down by its reaction with energetic oxygen atoms produced by solar ultraviolet radiation:



This process leads to NO_x production that allows the catalytic chain of reaction given in equation (1.1) to be initiated. It is estimated that if N_2O concentrations increase by 20% over the next 60 years a 3% reduction in the total ozone column will result [6].

5.2.3 Measurement Techniques

It has been shown that methane and nitrous oxide play an important role in both global warming and ozone depletion. Ground based measurements can help our understanding of these processes by allowing us to identify and quantify the different sources and sinks of these species. Some of the techniques used to measure and identify these sources and sinks will now be discussed.

Source and Sink Quantification - Flux Measurements

The quantification of sources and sinks of gaseous species is achieved by measuring the gas flux normal to the surface. The most popular technique used for measuring gas fluxes is that of eddy correlation. In this, the three components of the wind velocity are correlated with fast (<1s) concentration measurements to yield the net flux of the gas normal to the surface. The wind velocity can be measured with three-axis sonic anemometers and the gas concentration with near-infrared TDLAS. Up to date, concentration measurements have been made with mid-infrared TDLAS. An example of such measurements are those taken by Fowler at peat bog sites in northern Scotland [47]. Measurement of both methane and nitrous oxide fluxes were taken using a mid-infrared TDLAS developed by Aerodyne Research Inc. [48]. Fowler estimated that concentration retrievals had to be made with a precision better than 1%.

An extension of this technique is to spatially map gaseous emissions using open path measurements. In this case the multipass cell is discarded and the beam is directed at a

retroreflector some distance away. The beam is returned by the retroreflector to the detector. This allows the average gas concentrations over large areas to be determined. Measurement of the gas flux across the boundary can be made downwind on a vertical plane set up, by using mirrors on two masts and passing the beam between them at different heights. Again mid-infrared TDLAS have conventionally been used. An example of such an instrument is HAWK developed by Partridge at the National Physical Laboratory [30]. This has been used to spatially map methane concentrations at a land fill site.

The final technique uses flux boxes to determine flux concentrations. The flux box is a metal container with one of its ends open in order to be placed against the ground. In most flux box designs the box is fitted with a bleed pipe, whose purpose is to release gas from inside the box (if the rate of gas ingress is high). Assuming a gas tight seal is formed between the box and ground, the horizontal distribution of gas at the measurement site can be eliminated from the measurement, allowing the vertical flux only to be measured. The gas concentration is recorded by pumping gas from the flux box through the multipass cell. The gas from the cell is recirculated back into the flux box, so that gas pressure inside the box remains fairly constant throughout the measurement period. Measurements of methane gas fluxes from a land fill site using flux boxes have been carried out by the National Physical Laboratory [49]. They, however, monitored concentrations with flame ionisation detectors (FID's). TDLAS should provide better sensitivity and faster response times over FID's.

Source and Sink Identification - Isotopic Signature

Isotope ratio measurements of gaseous species can provide important information about the species global budget, as well as about the gas production and consumption processes occurring within the various sources. Taking methane as an example, $^{14}\text{CH}_4$ measurements have been used to estimate the contribution of fossil sources such as natural gas and methane from coal mining or from the oil industry. In contrast the stable isotope ratios, $^{13}\text{CH}_4/^{12}\text{CH}_4$ ($\delta^{13}\text{C}$) and $^{12}\text{CH}_3\text{D}/^{12}\text{CH}_4$ (δD), are determined by the different methane production processes, i.e., a) biogenic methane production by

acetate fermentation, b) thermogenic production (at high temperatures) and c) incomplete combustion of biomass or fossil fuels. The isotopic signatures for the three different processes are clearly distinguishable. For many biogenic CH₄ sources, the CH₄ flux into the atmosphere is reduced by partial bacterial oxidation of the CH₄, which causes an enrichment of both the $\delta^{13}\text{C}$ and the δD values. Further effects modifying the isotopic signature include the gradual isotopic enrichment of residual organic material in the course of degradation and molecular diffusion, which may alter isotope signatures on geological timescales. Thus it is seen that parallel $\delta^{13}\text{C}$ and δD measurements allow conclusions to be drawn about the production, consumption and transport of methane.

Conventionally, isotopic measurements have been performed exclusively with mass spectrometry, however, a mid-infrared TDLAS was recently used by Bergamaschi *et al* [50] to measure both the $\delta^{13}\text{C}$ and the δD values for methane. Such measurements could in future be performed using a near-infrared TDLAS.

Nitrous oxide production and consumption processes also exhibits characteristic N₂¹⁸O / N₂¹⁶O isotopic signatures. These signature have been measured by Wahlen and Yoshinari [51].

5.2.4 Atmospheric Pollutants

There has been an increase in concern over recent years about the release of pollutants into the atmosphere. This has led to an increase in legislation and guidelines aimed at controlling the release of these pollutants. Near-infrared TDLAS should fulfil an important role in assessing compliance with new legislation, as well as direct safety monitoring of pollutants. Pollutants of concern include HCl, HF, H₂S and NH₃. They are given off at low concentrations (10ppb - 10ppm) by the petrochemical, metal processing and incineration industries [52]. There is a requirement for a high sensitivity monitor for these species capable of long term unattended operation. Near-infrared TDLAS should be capable of such measurements.

There has also been widespread concern about exhaust emissions from motor vehicles. In particular there is a need to better specify vehicle CO, volatile organic compound

(VOC) and NO_x emissions under actual operating conditions. A mid-infrared TDLAS has been developed by Aerodyne Research Inc. to carry out measurements of NO_x on moving vehicles [48]. The instrument measures concentrations in an open path between the TDLAS on one side of the road and the retroreflector on the other. The instrument could measure a minimum detectable concentration of 5ppm of NO over a measurement time of 40 ms. Recently a near-infrared TDLAS operating at 1579nm has also been developed by Unisearch Associates Inc. to measure CO/CO₂ ratios [53]. This instrument also measures gas concentrations in an open path across the road.

5.2.5 Summary

It is clear that there are a wide variety of applications for a ground based near-infrared TDLAS, including fundamental studies of atmospheric chemistry as in the case of methane and nitrous oxide measurements and industrial applications such as the monitoring of pollutants like HCL and NH₃. While many techniques already exist for making these measurements, none combine the ability to perform in situ measurements with high sensitivity and be capable of long term unattended operation. As such, near-infrared TDLAS should provide significant advantages over existing technology.

5.3 Detection Technique

5.3.1 Introduction

The chosen method of detection is extremely important as it can have a significant effect on the sensitivity of the instrument and the complexity and cost of the detection electronics. Investigation of the literature, followed up by experimentation in the laboratory has been carried out to determine which detection scheme would be best suited to TDLAS measurements. The different detection schemes considered are described below.

5.3.2 Direct Measurement/Amplitude Modulation

The simplest of all the detection schemes involves recording the direct signal from the detector whilst tuning the laser through the absorption feature. Direct measurement, however, is not generally used as it suffers from all the attendant difficulties associated with detecting small differences in large background signals. The principal sensitivity limitation for direct absorption measurements will be laser excess noise. This noise has a $1/f$ form typical of any semiconductor device and is caused by the random spontaneous emission of photons from the diode. The detector is also a semiconductor based device which will exhibit noise with $1/f$ characteristics, however, the laser noise is generally much larger than the detector noise unless the laser power output is particularly low ($< 10 \mu\text{W}$).

Figure 5.1 shows the noise spectrum of the Anritsu 1650nm DFB laser recorded with a Spectrum Analyser (MI TF2371). This spectrum clearly shows the typical $1/f$ characteristics of laser excess noise.

Amplitude modulation involves modulating the laser amplitude, usually with a mechanical chopper, whilst tuning the laser over the absorption feature. The signal is detected at the modulation frequency using a lock in amplifier. The main advantage of this scheme is that the lock in amplifier effectively acts as a band pass filter about the modulation frequency, reducing the bandwidth of the detection electronics and hence

reducing the noise. Figure 5.2 shows a typical spectrum obtained using amplitude modulation.

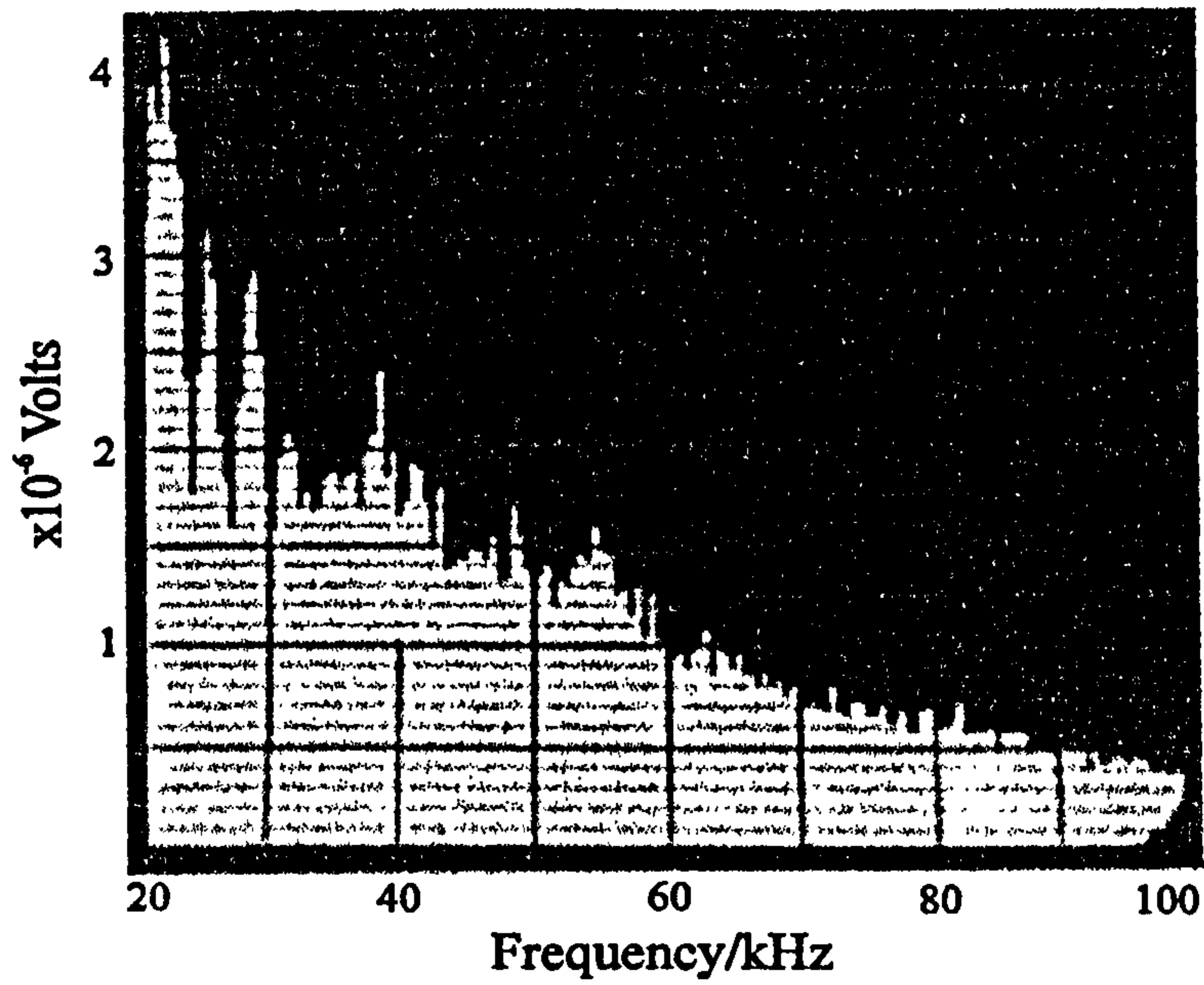


Figure 5.1 - Noise Spectrum of Anritsu diode

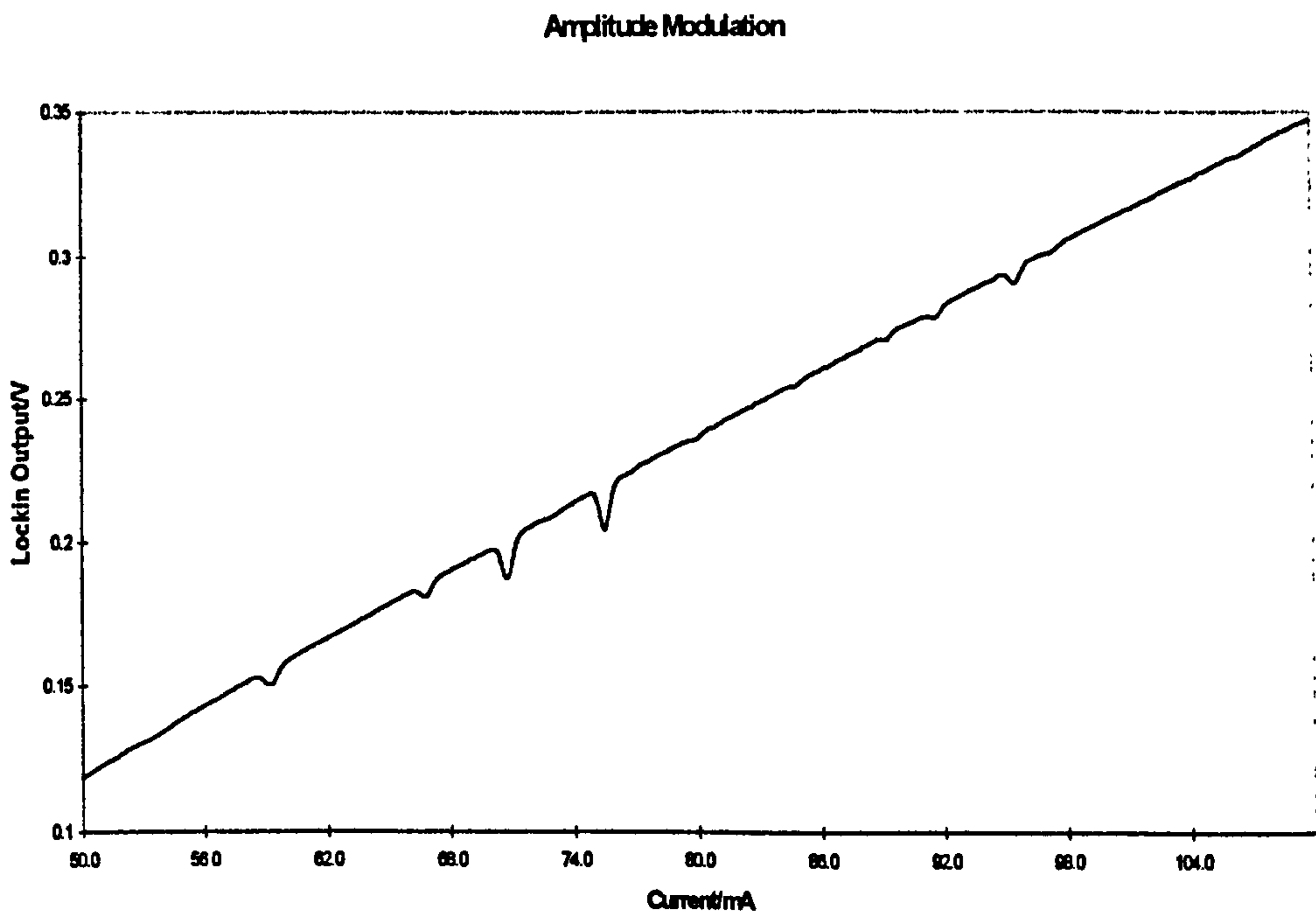


Figure 5.2 - Amplitude Modulation Spectrum of the $\nu_1 + \nu_3$ C_2H_2 band

Again, it is clear that this scheme will also suffer from the difficulties associated with measuring small changes on a large background signal. The mechanical nature of the modulation also means that the detection band can only be shifted to frequencies of the order 1-2 kHz. Laser excess noise is still dominant in this region.

Amplitude modulation has been extensively used in the laboratory at Strathclyde. The best sensitivity that could be achieved in practice was equivalent to a minimum detectable absorption of 1×10^{-4} in a 1 Hz bandwidth. This was about an order of magnitude better than was achieved with direct measurement, but is still well short of the required sensitivity for trace gas detection of N_2O or CH_4 .

5.3.3 Sweep Integration

An alternative method to amplitude modulation is sweep integration, whereby the absorption line is scanned rapidly many times and the signal averaged to produce a direct absorption signal. Scan rates can vary between 200 Hz and 300 kHz. The sensitivity should in theory improve with the square root of the scan rate. The use of a high pass filter to cut out low frequency noise and a pre-subtraction technique to reduce the sloping background have been shown to produce sensitivities close to the best obtained with other detection techniques [54]. The main disadvantage of sweep integration is that high bandwidth current controllers and detection electronics are required, making the system costly. In addition it has been shown that optical fringing, which cannot be averaged out, ultimately limits the achievable sensitivity of all detection techniques (Section 6.3). Consequently, it was decided high frequency sweep integration was not a cost effective option to obtain the required sensitivity.

5.3.4 Frequency Modulation

A variety of frequency modulation schemes exist. In all approaches, the diode laser frequency is modulated by adding an ac component to the laser injection current. The absorption signal is then detected with a lock in amplifier or RF mixing schemes, depending on the modulation frequency employed.

Frequency Modulation Spectroscopy (FMS) uses modulation frequencies comparable to or greater than the absorption linewidth (100 MHz to several giga-hertz) [55]. One tone FMS detects the signal at the modulation frequency while two tone FMS uses a pair of closely spaced frequencies and detects the signal at the difference frequency (1-10 MHz). At these frequencies laser noise will be negligible and the system in theory will give detector limited performance. This is equivalent to absorption's smaller than 10^{-7} [56].

Wavelength modulation spectroscopy (WMS), on the other hand, uses modulation frequencies that are much lower than the absorption linewidth that is being measured. Typical modulation frequencies of 1-100 kHz are used. The signal can be detected at the modulation frequency (1f) or a multiple of the modulation frequency (2f, 3f, 4f etc.). For this reason, WMS is commonly referred to as Harmonic detection. At these lower frequencies laser noise can still be significant and sensitivities equivalent to an absorption of $\sim 10^{-6}$ have been practically achieved [32, 44, 57]. Theoretical descriptions of both approaches have been covered extensively in the literature. These descriptions will now be reviewed.

Wavelength Modulation Spectroscopy

Theoretical Description

A laser whose injection current is modulated at a frequency, ω_m , will have an instantaneous frequency that can be expressed as:

$$\nu(t) = \bar{\nu} + a \cos(\omega_m t) \quad (5.3)$$

where $\bar{\nu}$ is the mean frequency which is slowly tuned by ramping the diode current. If $G(\nu)$ is the absorption line shape function, then the detector output, $S(\nu)$, will be:

$$S(\nu) = G[\bar{\nu} + a \cos(\omega_m t)] \quad (5.4)$$

If a Lorentzian line shape is considered, the output from the detector would take the form:

$$S(x, m) = \frac{1}{1 + [x + m \cos(\omega_m t)]^2} \quad (5.5)$$

where x , the normalised frequency, is defined as:

$$x = \frac{\bar{\nu} - \nu_0}{\Delta\nu} \quad (5.6)$$

and m , the modulation index, is:

$$m = \frac{a}{\Delta\nu} \quad (5.7)$$

m represents the maximum deviation in terms of frequency half widths, $\Delta\nu$, from the carrier frequency ν_0 .

The detector signal is usually fed into a lock in amplifier which selects one of the signal harmonics. In the case of very small modulation depths, Wilson [58] has shown that the n^{th} harmonic will be proportional to the n^{th} derivative of the absorption line. However, it has been shown that the harmonic signals are maximised at larger modulation amplitudes [59]. At these large modulation depths, the lineshape is no longer proportional to the derivative of the absorption feature and exact expressions for the Fourier components must be used. Wilson has shown these to be:

$$\begin{aligned} H_n(\nu) &= \frac{2}{\pi} \int_0^\pi S(\nu) \cos n\theta d\theta \\ &= \frac{2}{\pi} \int_0^\pi G(\bar{\nu} + a \cos\theta) \cos n\theta d\theta \end{aligned} \quad (5.8)$$

$H_n(\nu)$ is the normalised n^{th} Fourier component/harmonic of the detector output. Again considering a Lorentzian pressure broadened line, the second harmonic component of the detector output would be:

$$H_2(x, m) = \frac{2}{\pi} \int_0^{\pi} \frac{1}{1 + [x + m \cos \theta]^2} \cos 2\theta d\theta \quad (5.9)$$

This integral can easily be evaluated computationally over any normalised frequency and modulation depth. Evaluating such integrals allows the theoretical output from a Lock in amplifier to be determined. Similar integrals can also be derived for both Gaussian Doppler broadened lines and intermediate Voigt lines.

Software was developed in MATLAB at Strathclyde to evaluate the necessary integrals. A listing of this code can be found in Appendix 2.

Peak Heights

As stated previously, the harmonic signals are maximised when the modulation index is increased such that the lineshapes are no longer proportional to the derivatives. Reid and Labrie [59] have determined the modulation index required to maximise the peak height for a second harmonic Doppler and Lorentz line. Their work was extended by Silver [60] to include peak heights for $n = 1, 2, 4, 6$. Using the software developed at Strathclyde, this work has been further extended to include $n = 1, 2, 3, 4, 5, 6$. Table 5.1 shows the modulation index and normalised peak heights, $H(n)$, at the different detection harmonics for both Doppler and Lorentzian lineshapes.

It is seen that at higher detection harmonics, the required strength of modulation increases while the peak signal decreases. This would indicate that the optimum harmonic would be $n = 1$, however, it will be shown later (section 5.3.5) that when signal to noise considerations are taken into account this is not the case. It is also evident that the peak heights of the Doppler broadened lines are greater than those for the Lorentz lineshape. This indicates that harmonic detection is best suited to the Doppler lineshape. Voigt lineshapes, naturally, will have intermediate peak heights.

<u>Harmonic</u>	<u>Doppler</u>		<u>Lorentz</u>	
n =	m	peak- H(n)	m	peak - H(n)
1	1.6	0.574	2.0	0.500
2	2.1	0.438	2.2	0.343
3	3.3	0.361	3.6	0.234
4	3.6	0.236	5.0	0.177
5	4.7	0.190	5.6	0.145
6	5.2	0.160	6.5	0.121

Table 5.1 - Computed Peak Heights and Optimal Modulation Indices

Comparison of Theory with Experimental Results

A detailed comparison between the theoretical lineshapes for second harmonic detection and those obtained experimentally was carried out by Reid and Labrie [59]. In general, excellent agreement was found for both Doppler and Lorentzian lineshapes, however, this work was carried out using lead salt lasers operating in the mid-infrared. It was felt that it would be worthwhile extending this comparison to include the $n = 1, 2, 4$ harmonics using the near-infrared DFB lasers available at Strathclyde.

Figure 5.3 and **Figure 5.4** show the odd and even harmonic lineshapes of a Doppler broadened line as predicted by the computer model. For each harmonic the optimum modulation index was used. The lineshapes are broadly similar to those you would obtain at lower modulation indices where the lineshapes are proportional to the derivatives. However, it is evident that at higher harmonic detection and greater modulation depths the lineshapes become broadened.

Comparison between the modelled and experimental lineshapes provided good agreement. **Figure 5.5** shows a Doppler broadened (1.5 Torr) water line recorded using second harmonic detection at $\sim 1.52 \mu\text{m}$. This line was chosen as there were no nearby lines that would cause interference. The points marked with crosses show the modelled lineshape. Experimentally, the optimum modulation level was determined by observing the lock in output on an oscilloscope and increasing the modulation until the second harmonic peak height reached a maximum. For second harmonic detection this

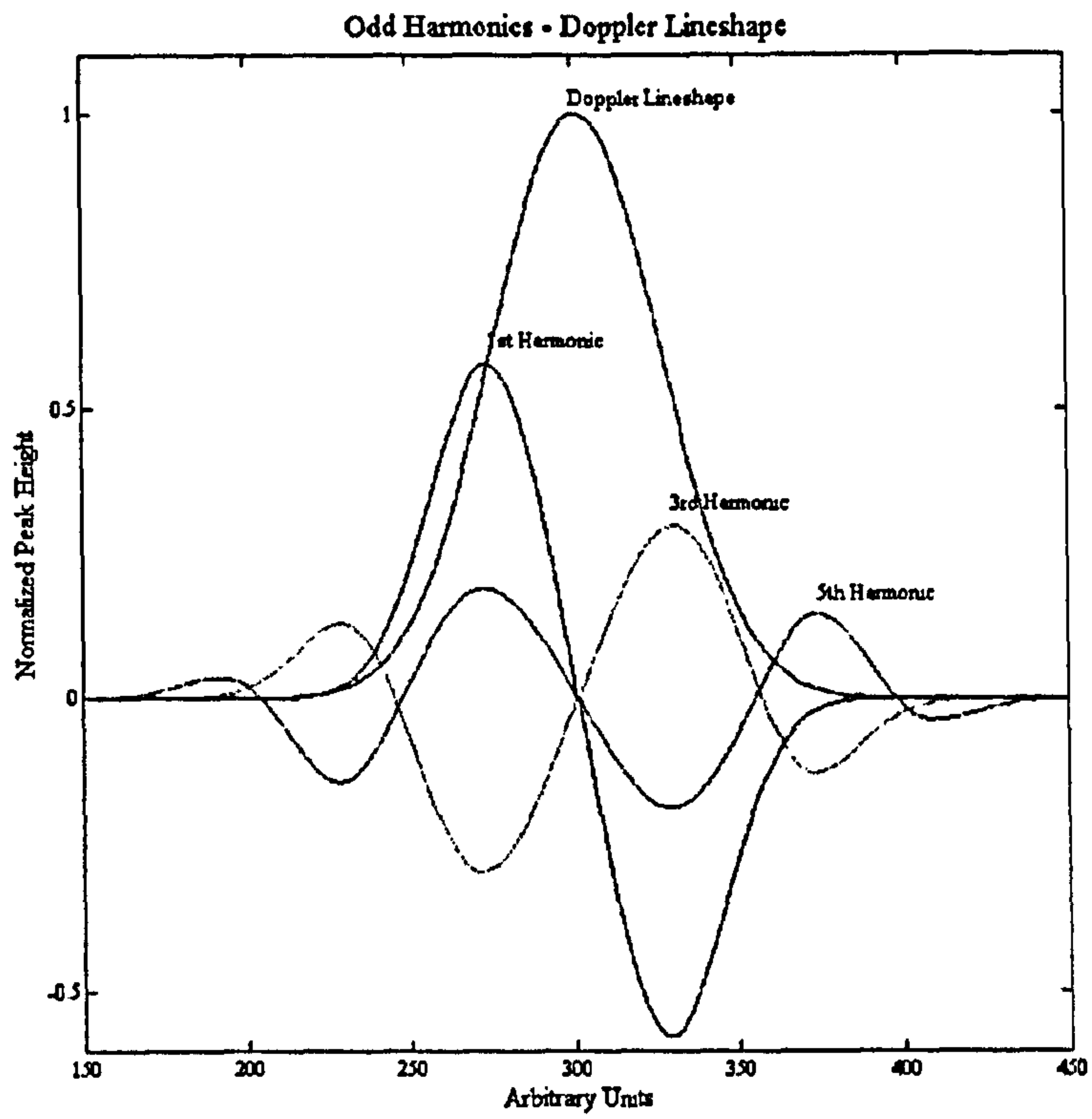


Figure 5.3 - Odd Harmonics

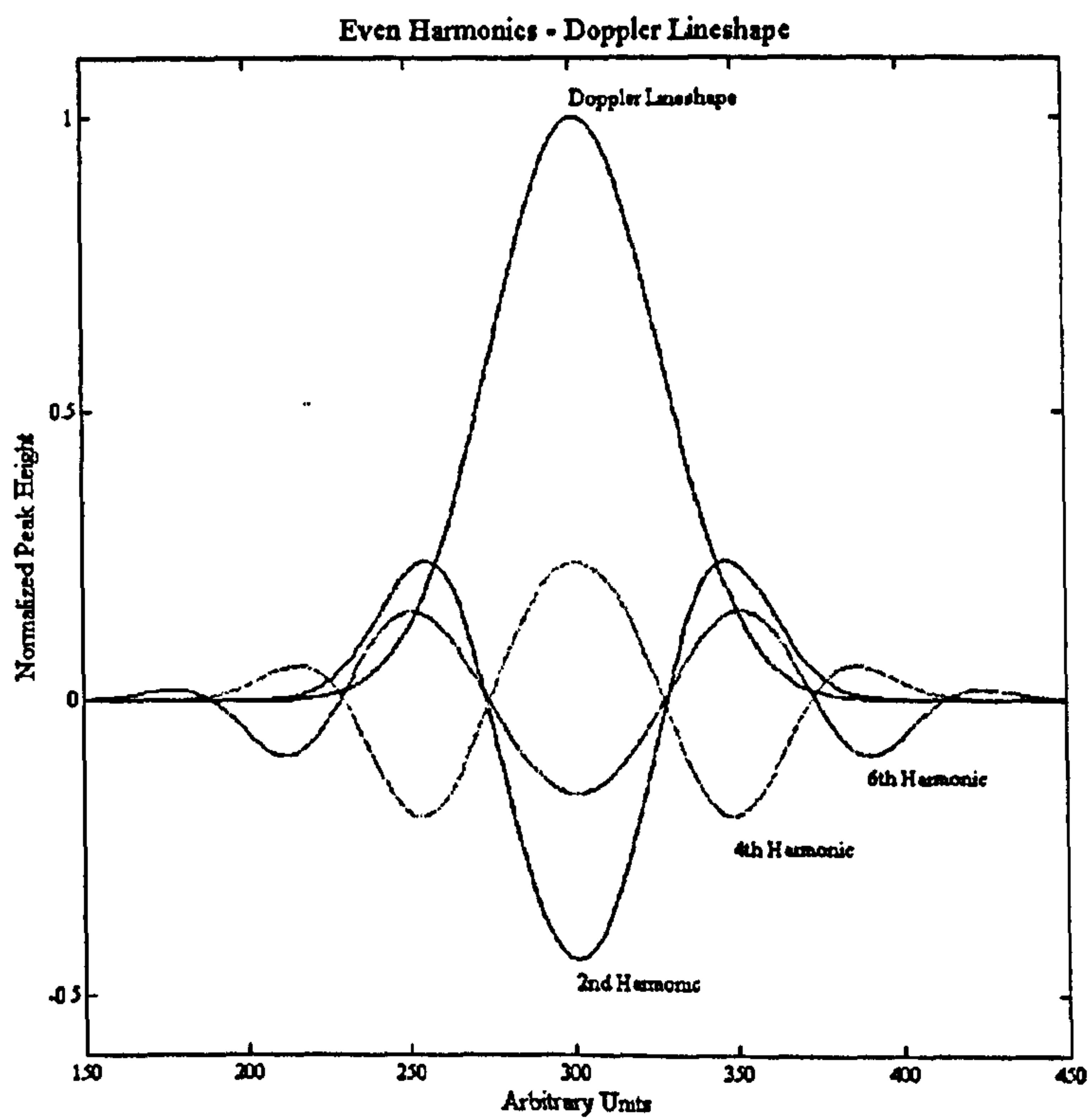


Figure 5.4 - Even Harmonics

should occur at a modulation index of 2.2. The experimentally determined modulation index that provided the best fit with the modelled output was 2.212. This is an excellent agreement.

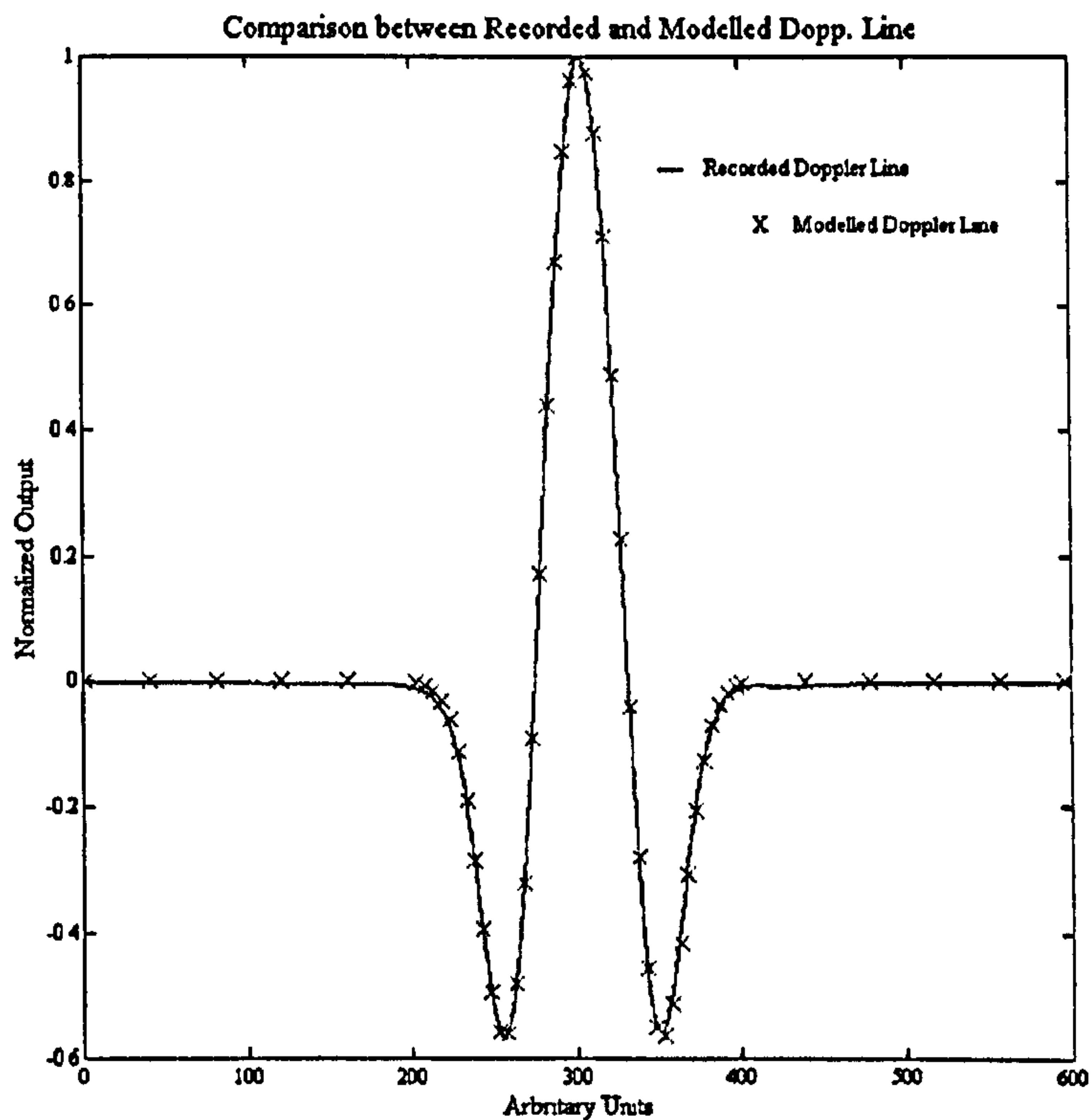


Figure 5.5 - Comparison between experimental and modelled 2nd Harmonic Doppler Line

The residuals between the two lineshapes were better than 0.1% in the central region of the line. The residuals, however, increase to $\sim 1\%$ at the wings. This was felt to be caused by a slight Lorentzian component to the recorded line shape. This would seem to be confirmed by the observation that the wings of the recorded line extend out further than the modelled output.

The fit of the same line in the pressure broadened regime (1.5 Torr H₂O + 300 Torr N₂) gave residuals of approximately 1%. While still reasonable, the fit was not so good as the Doppler broadened line. This was felt to be caused by the increased

modulation depths required for the Lorentzian lineshape. At these larger modulation depths, the linear frequency tuning assumption used in the theoretical description is no longer valid.

The 1st harmonic Doppler lineshape exhibited residuals of $\sim 0.1\%$ while the 1st harmonic pressure broadened line had residuals of $\sim 1\%$. The 4th harmonic residuals for both lineshapes were significantly worse ($>4\%$). Again, it was felt that the departure from the modelled lineshape resulted from the breakdown of the linear frequency tuning assumption at higher modulation depths. The good fit, for both the first and second harmonic Doppler lines, where smaller modulation depths are used, would seem to confirm this.

The theoretical expressions for the harmonic components of different lineshapes can only be used to generate the harmonic lineshape of single unblended lines. In practice recorded lines are usually either blended or have some interference from nearby lines. Software has been developed in MATLAB to allow the harmonic components of more complicated lineshapes to be determined.

The program, which is listed in Appendix 2, calculates the Fourier component of a synthetic or experimentally recorded direct absorption spectrum on a point by point basis. For each point on the recorded spectrum, $S(j)$, the integral given in equation (5.10) must be calculated.

$$H_n(j) = \frac{2}{\pi} \int_{i=0}^{i=z} S(j) \cos n\theta_i d\theta \quad (5.10)$$

The upper limit of the integral, $i = z$, corresponds to the number of data points that are found within the modulation limits, $m\Delta\nu$, where m is the modulation index and $\Delta\nu$ is the HWHM of the recorded absorption features. z can be determined using equation (5.7):

$$z = \frac{m\Delta\nu}{\nu'} \quad (5.11)$$

where ν' is the frequency tuning rate per data point. The program automatically carries out cubic interpolation of the data set to ensure an integral number of point fall within the modulation limits.

At each integration step, i , θ_i must be calculated. θ_i is given by:

$$\theta_i = \text{Cos}^{-1}\left(\frac{i\nu'}{m\Delta\nu}\right) \quad (5.12)$$

Calculating θ_i for the limits of the integration in equation (5.10), i.e. $i = 0$ and $i = z$, gives us $\theta_{i=0} = 0$ and $\theta_{i=z} = \pi$. Thus it is seen that the integration limits are the same as those given in the expression for the theoretical Harmonic components in equation (5.8). Indeed it is seen that the expressions in equations (5.10) and (5.8) are identical. The program was used to generate the second harmonic component of a single Doppler line first. This allowed a comparison with the second harmonic Doppler line generated using the theoretical expression given by Wilson [58]. It was found that the residuals between the two spectrum could be reduced to less than 0.2 %. To achieve this level of agreement it was necessary to refine the integration by increasing the number of points (through interpolation) within the integration limits.

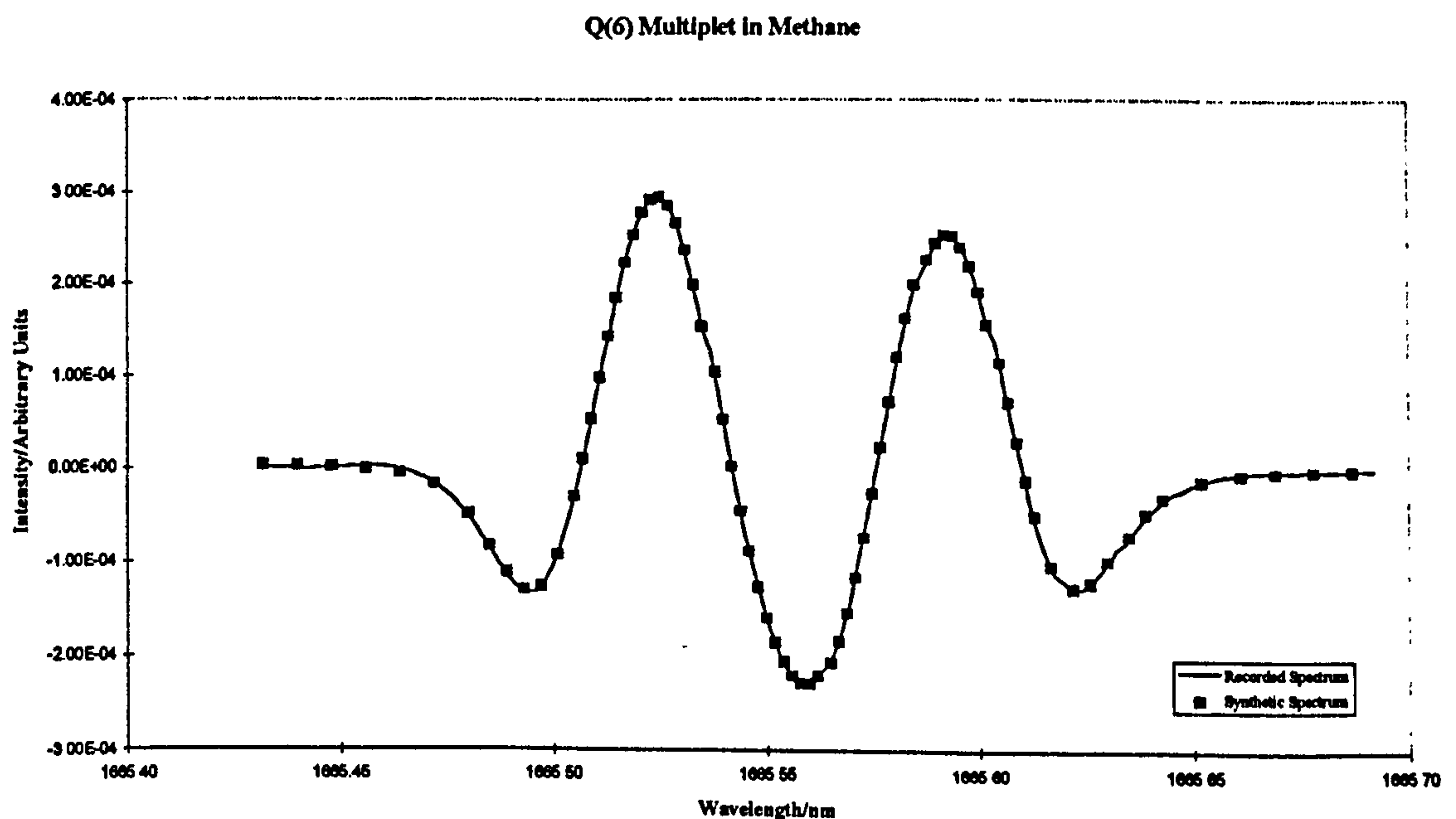


Figure 5.6 - Comparison between experimental and theoretical second harmonic component of the Q (6) multiplet in methane

Figure 5.6 shows a recorded spectrum of the Q6 multiplet of the $2\nu_3$ band of methane. This feature consists of six lines which are heavily blended. The second spectrum on this figure is the second harmonic spectrum generated by the program. The rms residuals between the two are better than 0.3%.

Frequency Modulation Spectroscopy

As discussed earlier, Frequency Modulation Spectroscopy (FMS) is essentially an extension of conventional Wavelength Modulation Spectroscopy (WMS). WMS has already been described theoretically using the laser intensity, however, FMS theory is cast in terms of the laser electric field. The electric field representation retains the phase information, giving a fuller picture of the results. Since both approaches are essentially the same, it is evident that the electric field representation can equally be applied to WMS. J A Silver [60] has performed mathematical derivations for frequency modulation as applied to a laser system. Here only a brief overview of the derivations are given.

Theoretical Description

If a time dependent phase shift, $\phi(t)$, is imposed on the laser carrier frequency, the resulting electric field is:

$$E(t) = E_0 \exp[i\omega_0 t + i\phi(t)]$$

$$\phi(t) = \beta \sin(\omega_m t) \tag{5.13}$$

The maximum excursion from the centre frequency ω_0 will be $\beta\omega_m$ where ω_m is the modulation frequency. The FM index β can be related to the wavelength modulation index by:

$$m = \beta x_m \tag{5.14}$$

However, the laser intensity cannot be assumed to be uniform over the range of frequency modulation. Consequently, simultaneous frequency and amplitude modulation of the electric field occurs:

$$E(t) = E_0 [1 + M \sin(\omega_m t + \psi)] \exp[i\omega_0 t + i\beta \sin(\omega_m t)] \quad (5.15)$$

where M is the amplitude modulation index and ψ is the AM-FM phase difference. M is defined as:

$$M = \frac{|I_0 - I_{\max}|}{2I_0} \quad (5.16)$$

where I_0 is the laser intensity at line centre and I_{\max} is the maximum laser intensity within the modulation limits.

The modulation harmonics are determined using Fourier analysis of the detector current that results from the interaction of the modulated laser field with the absorption feature. In the absence of absorption the photocurrent may be non zero because of the simultaneous amplitude modulation. The portion of the signal resulting from this is known as Residual Amplitude Modulation (RAM) or $R(M)$, and can significantly reduce sensitivity by two effects: introducing a strongly sloping baseline to the signal and adding noise due to the amplitude modulation of the power fluctuation of the laser. Silver [60] has shown that the RAM components of the laser power are given by:

$$I_{\text{RAM}}^{\text{one tone}} = 2I_0 R(M) = \begin{cases} 2MI_0 \sin(\theta + \psi) & n = 1 \\ 1/2 M^2 I_0 \cos(\theta + 2\psi + \pi) & n = 2 \\ 0 & n > 2 \end{cases} \quad (5.17)$$

where θ is the detector phase angle. The in phase (0 or π) component corresponds to pure absorption.

The electric field representation can be used to determine theoretical sensitivity limits for both WMS and FMS. Comparison of these theoretical limits will allow the best

detection technique for near-infrared TDLAS to be determined. This sensitivity analysis is now discussed.

5.3.5 Theoretical Sensitivity Analysis

The main sources of noise that will limit sensitivity in TDLAS measurements are shot noise, detector/thermal noise from the preamplifier, laser excess noise and RAM. Ancillary electronics such as the lock in amplifier, signal generator and the temperature and current controller will also act as noise sources but they can normally be specified to ensure they do not limit sensitivity. Consequently, they are not included in the sensitivity analysis at this stage, but are quantified in Section 6.5. The four main sources of noise are now described in more detail.

Shot Noise

Electric current has noise due to the finite nature of the charge carriers. There is always some non-uniformity in the electron flow which generates noise in the current. The shot noise is given by:

$$i_{\text{shot}}(\text{rms}) = (2qI\Delta f)^{1/2} \quad (5.18)$$

where q is the charge on an electron, Δf is the detection bandwidth and I is the RMS AC current. If frequency modulation is employed, Silver [60] has shown that the shot noise is given by:

$$i_{\text{shot}}(\text{rms}) = \left[2q\Delta f r_i \langle P_o \rangle \left(1 + \frac{M^2}{2} \right)^n \right]^{1/2} \quad (5.19)$$

where P_o is mean power at the detector, n is the detection harmonic and r_i is the detector responsivity. The responsivity is given by:

$$r_i = \frac{q\eta}{hv_o} \quad (5.20)$$

where η is the detector quantum efficiency, h is Plancks constant and v_o is the laser frequency.

Detector/Thermal Noise

Detector/Thermal noise is generated in every resistor. It occurs because of thermal fluctuations in the electron density within the resistor itself. The noise is given by:

$$i_{\text{thermal}}(\text{rms}) = \left(\frac{4kT_{\text{eff}}\Delta f}{R_1} \right)^{1/2} \quad (5.21)$$

where k is Boltzmann's constant, T_{eff} is the preamplifier effective temperature and R_1 is the detection electronic input resistance. Careful design of the detector preamplifier should ensure that is not a dominant noise source.

Laser Excess Noise

Laser excess noise has a characteristic $1/f$ form. While the reasons for this form are not generally well understood it is clear that excess noise will make measurements at low frequencies difficult. The excess noise component is given as:

$$i_{\text{excess}} = \frac{r_i \Delta f^{1/2} \sigma_{\text{ex}}}{f^b} \quad (5.22)$$

The parameter σ_{ex} is a system dependent constant defined as the excess noise at 1 Hz bandwidth and 1 Hz frequency. The frequency exponent, b , will typically have a value of 1, however, it can vary between the range 0.8 to 1.5.

Residual Amplitude Modulation (RAM)

Silver [60] has shown that the RAM induced noise depends on the product of the standard deviation of the laser power, σ_p , with the AM function, $R(M)$, as defined in Equation (5.17), i.e.:

$$i_{\text{RAM}} = \sqrt{2} r_i R(M) \sigma_p \quad (5.23)$$

Combined Noise

All of the four main noise sources are incoherent and as such the total random noise will be the square root of the sum of the squares of each noise contribution, i.e.:

$$i_{\text{noise}} = \left(i_{\text{shot}}^2 + i_{\text{thermal}}^2 + i_{\text{excess}}^2 + i_{\text{RAM}}^2 \right)^{1/2} \\ = \left(2q\Delta f \left[r_i \langle P_o \rangle \left(1 + \frac{M^2}{2} \right)^N + \frac{2kT_{\text{eff}}}{qR_1} \right] + r_i^2 \left(\frac{\Delta f \sigma_{\text{ex}}^2}{f^b} + 2R^2(M) \sigma_p^2 \right) \right)^{1/2} \quad (5.24)$$

Detector Signal

The detector signal is given by the product of the laser power arriving at the detector and the detector responsivity. However, as was shown in the WMS description in Section 5.3.4, the normalised peak height of the detector output varies with detection harmonic and lineshape. The normalised height, $H(n)$, must also be incorporated, i.e.:

$$i_{\text{signal}} = \sqrt{2} r_i \langle P_o \rangle H(n) \quad (5.25)$$

The normalised peak heights have been shown to be fairly independent of the modulation frequency and can thus be used in the FMS regime also [60].

Minimum Detectable Absorption

The minimum detectable absorption (MDA) is defined as the absorbance corresponding to the maximum peak height at unit signal to noise ratio. The MDA is given by:

$$\text{MDA} = \frac{i_{\text{noise}}}{i_{\text{signal}}} \quad (5.26)$$

The expressions presented above were programmed into MATLAB at Strathclyde University (see Appendix 2), allowing the MDA for different harmonics under both the WMS and FMS regimes to be determined. The findings are now discussed.

System Dependent Parameters

As detailed in the previous section, several system dependent parameters need to be evaluated before the theoretical sensitivity limits can be determined. A brief overview of how this was done is given below. The evaluated parameters for the two diode systems are tabulated in **Table 5.2**.

σ_p

σ_p was defined in the previous section as the standard deviation of the laser power. It was measured by recording the preamplified detector output using a digitising oscilloscope (Le Croy - 600 Mhz B.W). This scope has statistical facilities allowing the rms deviation of the signal to be determined. While the detector and preamplifier will also contribute noise to this signal, their contribution should be small if the full laser intensity is focused onto the detector. If the detector responsivity and preamplifier gain are known, the standard deviation can be given as a fraction of the total laser power.

σ_{ex}

σ_{ex} is the excess noise at 1Hz frequency and 1Hz bandwidth. The output from the preamplifier and detector, with full laser power incident, was fed into a MI TF2371 Spectrum analyser. Figure 5.1 shows the noise frequency spectrum of the Anritsu 1650nm DFB. Again, assuming that the detector responsivity and preamplifier gain are known it is quite simple to define the excess noise as a fraction of the laser power. The spectrum analyser output also allows the frequency exponent, b , to be determined.

M

M is the amplitude modulation index. It can be evaluated by measuring the slope efficiency of the laser and the frequency tuning rate. M will be given by:

$$M = m\omega_{1/2} \frac{dP}{dI} / \frac{d\omega}{dI} \quad (5.27)$$

Where the slope efficiency, dP/dI , is defined as the change in laser intensity brought about by a change in the diode operating current. The tuning rate, $d\omega/dI$, is defined as the change in laser frequency brought about by a change in operating current. For near-infrared lasers these parameters can be assumed to be constant over typical operating currents and temperatures, however, the tuning rate will be dependent on the modulation frequency employed.

Figure 5.7 shows how the tuning rate of the Thomson diode varies within the frequency range 500 Hz to 50 kHz. The tuning rate was measured by optimising the modulation depth for a second harmonic Doppler broadened line over this frequency range. Since it has already been shown that optimum modulation occurs at a modulation index of 2.2 and given that the Doppler width of any gas can be theoretically determined using equation (2.48), the tuning rate at each modulation frequency can be easily calculated. It is seen that the d.c tuning rate ($0.025 \text{ cm}^{-1}/\text{mA}$), as determined in section 3.2.2, is significantly greater than the tuning rate at higher frequencies. This decrease in tuning rate with increasing frequency is consistent with the thermal time constant involved in the diode tuning mechanism. Bandwidth limitations in the current controller, however, prevented measuring the tuning rate at

higher frequencies. For the purpose of the WMS and FMS comparison, the fit to the measured rates below 50kHz (WMS), which is given by the equation in Table 5.2, has been extrapolated to the high frequency (>50 MHz) region associated with FMS detection. Similar behaviour was found for the Anritsu diode.

Parameter	Thomson Diode InGaAs Detector with 150 k Ω Transimpedance Amplifier	Anritsu Diode Ge Detector with 100 k Ω Transimpedance Amplifier
P_o	5 mW	1 mW
ν_o	6580 cm $^{-1}$	6060 cm $^{-1}$
η	0.54	0.52
$\frac{dP}{dI}$	0.083 mW/mA	0.013 mW/mA
$\frac{d\omega}{dI} (f)/ (cm^{-1}/mA)$	0.0124 [f (kHz) $^{-0.2049}$]	0.0054 [f (kHz) $^{-0.2140}$]
R_i	150 k Ω	100 k Ω
T_{eff}	296 K	296 K
Δf	100 kHz	100 kHz
σ_p	8x10 $^{-4}$ P $_o$	5x10 $^{-4}$ P $_o$
σ_{ex}	5x10 $^{-5}$ P $_o$	5x10 $^{-5}$ P $_o$
b	1	1
Ψ	$\pi/2$	$\pi/2$

Table 5.2 - Evaluated Laser Parameters

The parameters used for the two laser systems are listed in Table 5.2. These parameters have been used to determine the theoretical noise performance of the instrument under both WMS and FMS regimes. The findings are now discussed.

Tuning Rate against Modulation Frequency

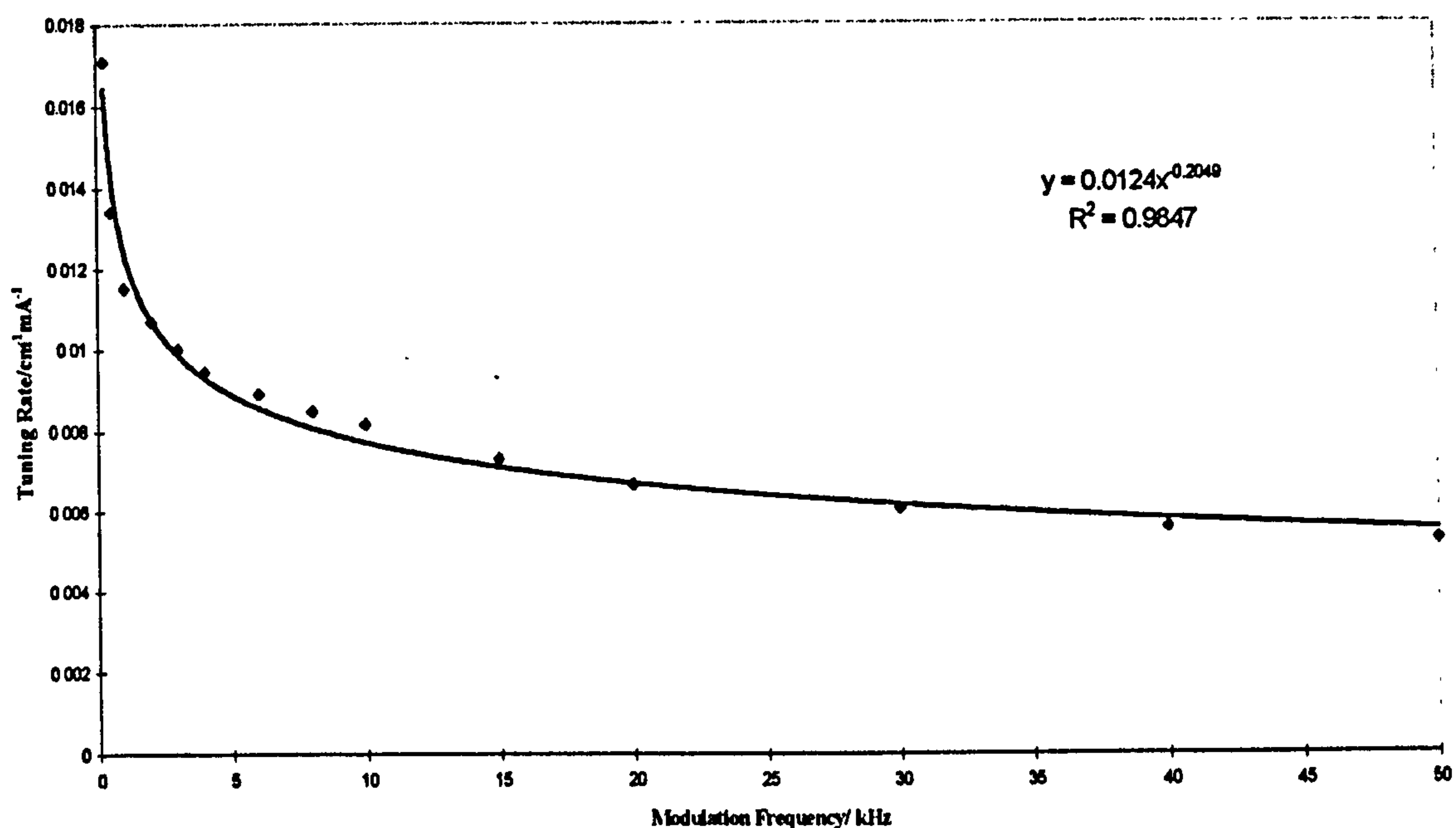


Figure 5.7 - Tuning Rate against Modulation Frequency for Thomson Diode

Theoretical Sensitivity Limits

Figure 5.8 shows the theoretical sensitivity limits for WMS using 1st, 2nd, 4th and 6th harmonic detection for the Thomson and Anritsu laser diode systems. The performance of both lasers was extremely similar. Table 5.3 generalises the findings for both lasers. Laser excess noise dominates all detection methods below frequencies of ~ 10 kHz. At very low frequencies (< 50 Hz) 1st harmonic detection provides the best sensitivity (4×10^{-5}). This is due to the larger signal obtained with 1st harmonic detection. At higher harmonics, the decrease in the peak intensity results in poorer sensitivity in this region.

As the frequency is increased up to ~ 10 kHz, the sensitivity of all methods improve as a result of the decrease in the excess noise. The improvement in the sensitivity for 1st harmonic detection is markedly less rapid in this region when compared with the

others. This is due to greater RAM noise. In fact, RAM noise becomes dominant above 10kHz for 1st harmonic detection, resulting in the sensitivity getting progressively worse at higher frequencies. This is a consequence of increasing RAM induced noise brought about by the reduction in frequency tuning rate at higher modulation frequencies. Thus the best sensitivity for 1st harmonic WMS occurs at 10 kHz and is equivalent to a MDA of 1×10^{-5} .

One tone FMS would have a sensitivity limit of 5×10^{-4} . Again it is dominated by RAM noise. The predicted sensitivity is about 2 orders of magnitude worse than what is claimed for this technique by Cooper *et al* [56]. The difference is attributed to a complete lack of RAM noise in the diode that they used. This lack of RAM noise is peculiar to their diode and similar predicted sensitivity limits would be expected with other near I-R diodes that exhibit more consistent behaviour .

The best sensitivity in the region 1-500 kHz is obtained by using 2nd harmonic detection. Higher harmonics have similar noise levels but smaller signals. At 100kHz the predicted sensitivity for 2nd harmonic WMS is 1×10^{-7} . Beyond 500 kHz RAM noise begins to dominate 2nd Harmonic detection and sensitivity limits get worse. Higher harmonic WMS which does not suffer from RAM induced noise exhibits better sensitivity limits in this region. Excess noise remains dominant up to 100 MHz. Above this region shot noise is dominant and the best sensitivity, equivalent to a MDA of 4×10^{-8} , is achieved using 4th harmonic WMS.

The behaviour of the Anritsu diode was remarkably similar to the Thomson device. The main difference being that RAM noise became dominant at slightly lower frequencies. This is a consequence of the smaller current tuning coefficient exhibited by this laser which meant that the laser current had to be modulated more to provide similar modulation depths to the Thomson diode. The lower power output of this laser resulted in increased Detector/Thermal noise levels. Overall, however, sensitivity limits for both devices were very similar over the full range of detection frequencies.

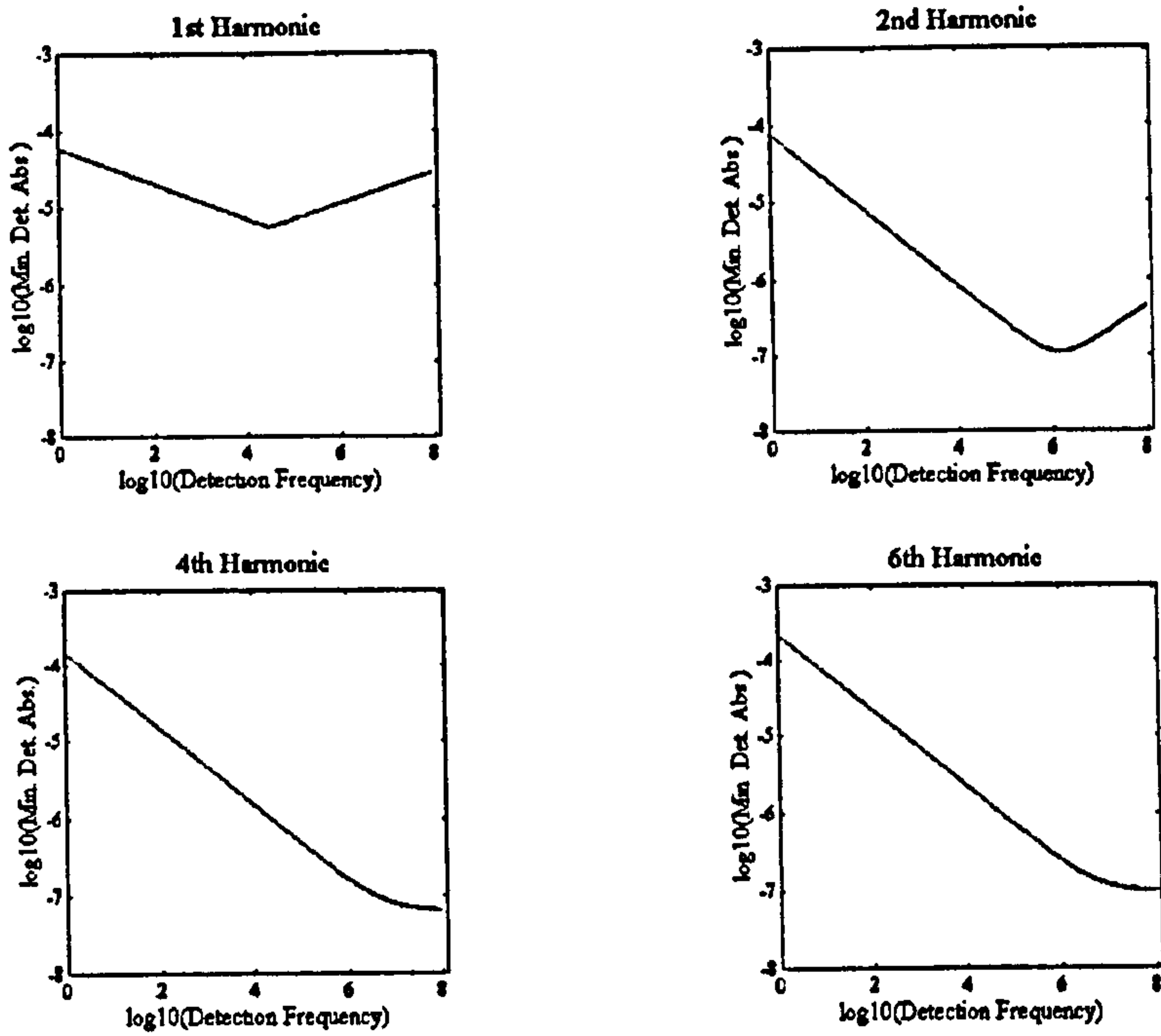
Considering the Thomson diode, it is seen that the best sensitivity is achieved using 4th harmonic WMS at detection frequencies in the MHz range. However, 2nd harmonic detection provides sensitivity limits that are just over a factor of two worse at kHz detection frequencies. The ability to use cheaper conventional commercial electronic components and low bandwidth photodiodes at kHz frequencies instead of discrete RF

components in the MHz region with all the attendant problems associated with this was felt to far outweigh the slight improvement in sensitivity. Consequently 2nd Harmonic detection in the 50-100 kHz region was felt to be the best detection scheme. The Anritsu device also showed very little improvement in sensitivity by going to high detection frequencies. Thus again it is concluded that 50-100 kHz 2nd harmonic WMS is the most suitable detection scheme.

Detection Scheme	Detection Frequency	Typical Min. Det. Abs. in 1Hz Band Width	Dominant Noise Source
1f	0 to 10 kHz	1×10^{-5}	Excess
	10 to 100 kHz	3×10^{-5}	RAM
	0.1 to 1 MHz	6×10^{-5}	RAM
	1 to 500 MHz	9×10^{-5}	RAM
2f	0 to 10 kHz	2×10^{-6}	Excess
	10 to 100 kHz	1×10^{-7}	Excess
	0.1 to 1 MHz	2×10^{-7}	Excess and RAM
	1 to 500 MHz	1×10^{-6}	RAM
4f	0 to 10 kHz	3×10^{-6}	Excess
	10 to 100 kHz	4×10^{-7}	Excess
	0.1 to 100 MHz	6×10^{-8}	Excess
	100 to 500 MHz	4×10^{-8}	Shot
6f	0 to 10 kHz	4×10^{-6}	Excess
	10 to 100 kHz	5×10^{-7}	Excess
	0.1 to 100 MHz	8×10^{-8}	Excess
	100 to 500 MHz	4×10^{-8}	Shot

Table 5.3 - Summary of Sensitivity Analysis

Theoretical Sensitivity Calculation for Thomson 1.52 μm
Diode with 150 k Ω Preamplifier



Theoretical Sensitivity Calculation for Anritsu 1.65 μm
Diode with 100 k Ω Preamplifier

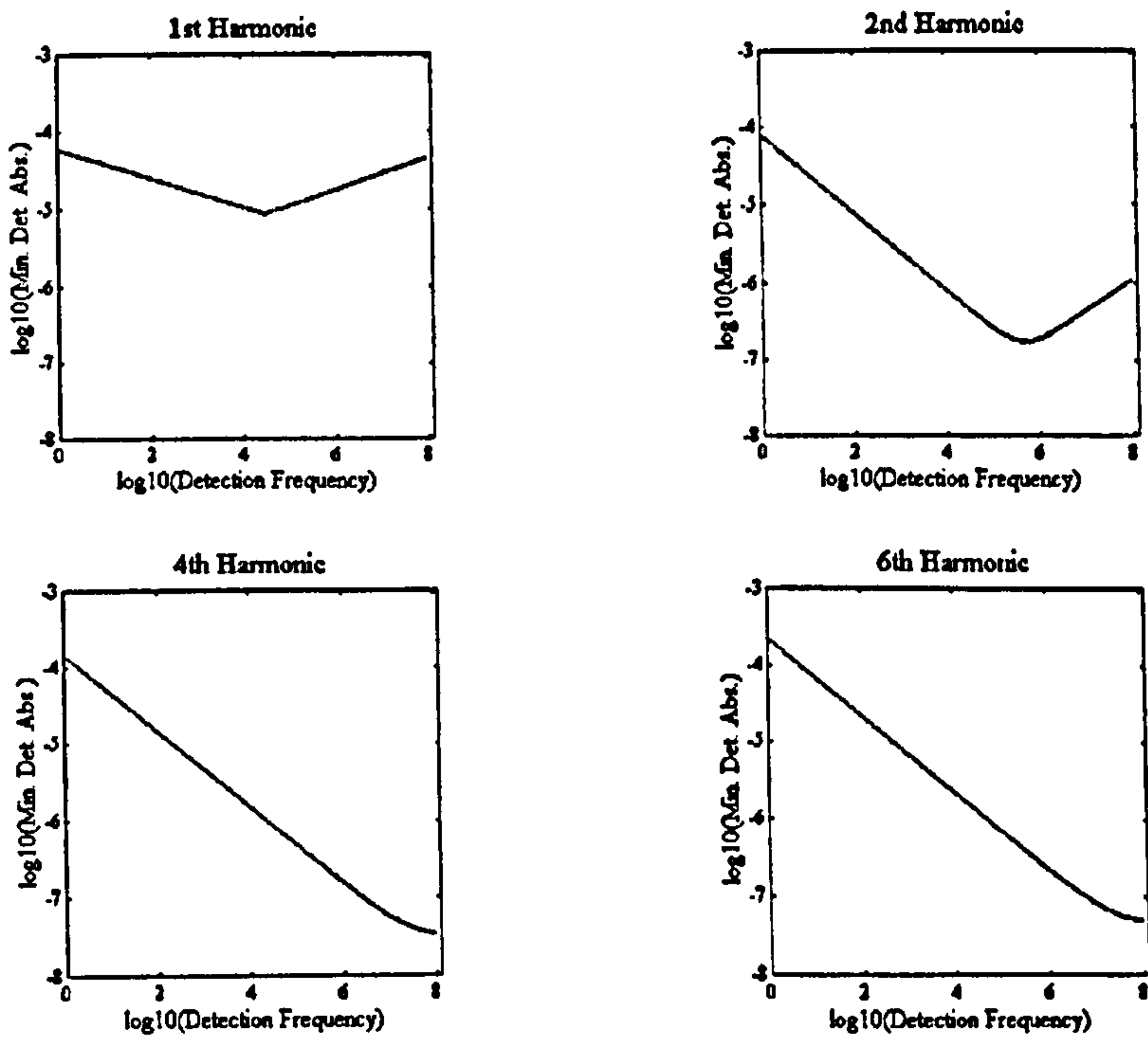


Figure 5.8 - Theoretical noise levels for different detection schemes for the Thomson and Anritsu lasers

Additional Techniques to improve Sensitivity

RAM Removal

The theoretical description of FMS showed that a phase difference, ψ , exists between AM and FM components of the detected signal. This difference in phase can be used to remove the noise and sloping baseline due to RAM. This is achieved by selecting a suitable detector phase angle, θ , so that the RAM component defined in (5.17) is reduced to zero. Taking the case of 2nd harmonic WMS, the RAM term will become zero if the following is true:

$$\theta + \pi + 2\psi = \frac{\pi}{2} \text{ or } \frac{3\pi}{2} \quad (5.28)$$

Experimentally the RAM baseline could be removed by changing the phase by $\sim 25^\circ$ from the optimum phase condition ($\theta = 0$ or π). This indicates that the phase difference, ψ , between AM and FM is $\sim 30^\circ$. This is in good agreement with the findings of Feher and Martin [31].

The removal of RAM induced noise would improve noise levels by between 10% and 50 % depending on the modulation frequency chosen. Changing the detector phase angle by 25° , however, will cause a reduction in signal intensity by $\sim 15\%$.

Signal Averaging

The sensitivity can be improved by averaging the signal. Sensitivity will improve with the square root of the number of points averaged. This will only improve sensitivity to a level determined by optical fringing which is statistically dependent.

5.3.6 Conclusions

Theoretical calculations show that near optimum sensitivities can be achieved using second harmonic WMS in the 50-100 kHz region. Shot noise limited performance can be obtained for higher harmonic WMS if frequencies above 100 MHz are used, but the increase in the sensitivity (typically a factor of two for both lasers tested here) was not felt to be worthwhile when the additional complexity and cost of the detection electronics was taken into consideration. In addition, it has been shown that poorer fits are obtained between theoretical and experimentally recorded lineshapes for higher harmonic WMS. This will adversely affect the accuracy of concentration retrievals (Section 6.2). Finally, the increased linewidth associated with higher harmonic detection will increase the possibility of unwanted interference from nearby lines.

These calculations have extrapolated the tuning efficiency behaviour as measured at low frequencies to the high frequency region. While there is no explicit evidence that such an extrapolation is valid, Cooper *et al*'s work [56] measuring the sensitivity limits for a similar experimental set up, showed similar behaviour to that predicted for the two lasers used here. In particular, the reduction in the sensitivity at the very highest frequencies, brought about by the reduction in tuning efficiency, is well mimicked.

It should also be noted that while theory predicts quantum limited performance for both devices at high detection frequencies, practical experience gained at Strathclyde University has shown that the noise levels will always be dominated by mechanical vibrations and optical fringing (Section 6.3). This is in agreement with the findings of Bomse *et al* [55] and Nakagawa *et al* [62]. Generally, the measurements taken at Strathclyde have indicated that if no active fringe reduction is employed, optical fringes would limit sensitivity to a MDA of 1×10^{-6} . Different methods of fringe reduction have therefore been investigated to determine which would be best suited to the proposed application. The findings are discussed in the following section.

5.4 Fringe Removal

5.4.1 Introduction

As was discussed in Section 5.3.6, optical fringing brought about by the interference of two coherent beams at the detector is one of the main factors that stop the instrument achieving the sensitivity predicted by theory. This section looks at the different methods that can be used to eliminate fringing and determines their effectiveness and suitability to the proposed system.

Common to all methods investigated is the necessity that the fringe FSR be significantly removed from the period of the absorption feature.

5.4.2 Brewster Plate Spoiler

The Brewster plate spoiler works by continuously changing the effective path difference between the two interfering beams that are causing fringing. The change in pathlength is achieved by oscillating an opaque plate ($n > 1$) about the Brewster angle. The Brewster angle is chosen so as to minimise insertion losses when polarised lasers are used.

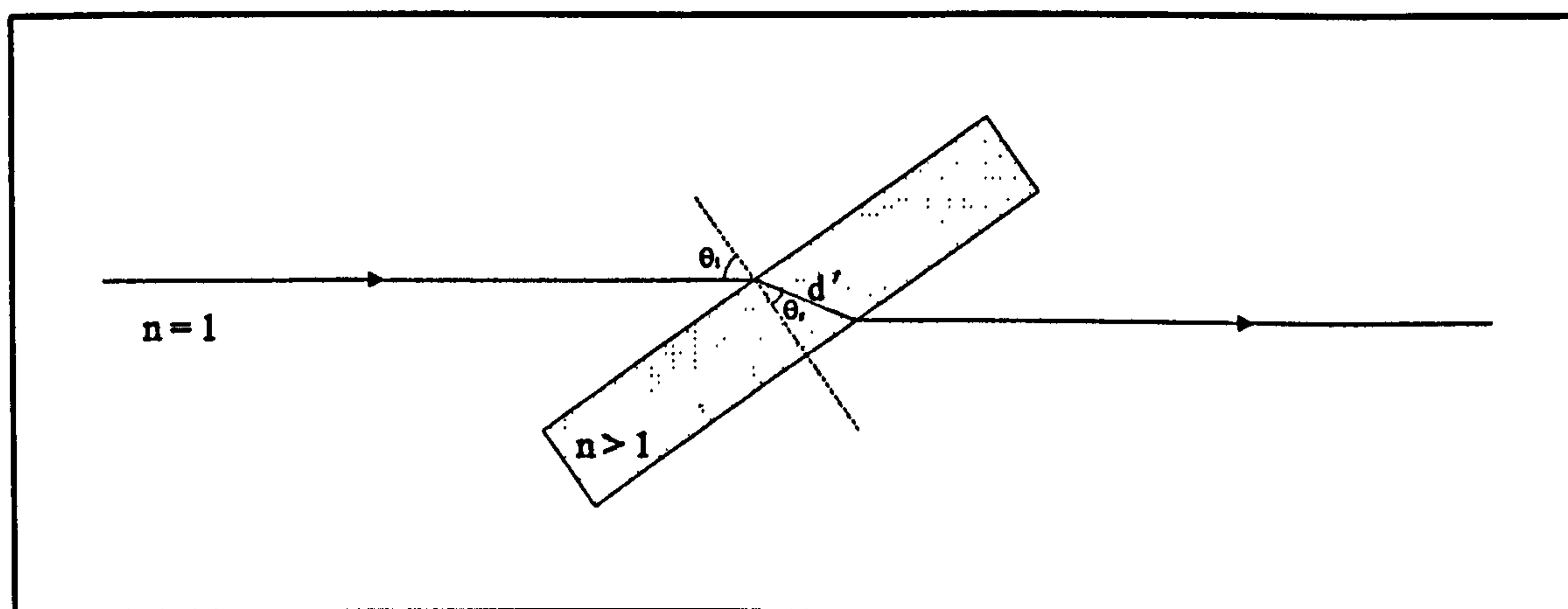


Figure 5.9 - Schematic of a Brewster plate spoiler

Figure 5.9 shows a schematic of a Brewster plate spoiler. Oscillation of the plate about the Brewster angle will cause the distance d' to change. This change will cause the pathlength between the cavity surfaces on either side of the plate to oscillate, which will in turn cause an oscillation of the fringe FSR. Webster [62] has shown that the change in pathlength between the cavity surfaces, brought about by a change in the angle of incidence of the Brewster plate from θ_{i1} to θ_{i2} , will be (assuming the plate is in free space):

$$\Delta L = d_1' [\cos(\theta_{i1} - \theta_{r1}) + n] - d_2' [\cos(\theta_{i2} - \theta_{r2}) + n] \quad (5.29)$$

where n is the refractive index of the Brewster plate.

If the oscillation frequency is greater than the laser scan rate, the varying fringe output will effectively be time averaged. In order for this averaging to be successful the minimum change in pathlength must be equivalent to half the fringe FSR. It can easily be shown that this is equivalent to changing the pathlength by one quarter of the laser operating wavelength, i.e.:

$$\Delta L_{\min} = \frac{\lambda}{4} \quad (5.30)$$

This is an important result as it shows that the required change in pathlength depends only on the operating wavelength of the laser and is independent of the cavity length. Thus the required pathlength change is independent of the fringe FSR.

In practise, Webster found that fringe reduction was optimised by changing the pathlength by three or four times more than the minimum requirement. At $1.5 \mu\text{m}$ this would correspond to $\sim 1-1.5 \mu\text{m}$. If a 0.5 mm thick fused silica plate ($n=1.458$) is used at the Brewster angle (56.555°), a 0.25° oscillation would change the pathlength by $\sim 1.5 \mu\text{m}$. Thus it is seen that very little movement of the plate is required.

Practical Set Up and Results

To obtain the fine angular oscillations required, a scanning galvanometer was used. A scanner was procured from General Scanning Inc. (Model G300PD). This had an angular resolution of 0.01° and was identical to the scanner used successfully by Webster in his experiments [62]. General Scanning Inc. also provided a control electronics board which offsets and/or amplifies waveforms supplied by a function generator. A 0.5 mm thick fused silica plate was attached to the scanner with epoxy resin.

Initial investigation was aimed at trying to reproduce the results obtained by Webster in the mid-infrared ($7.65\ \mu\text{m}$) with a $1.52\ \mu\text{m}$ DFB laser. The schematic of the optical set up used is shown below.

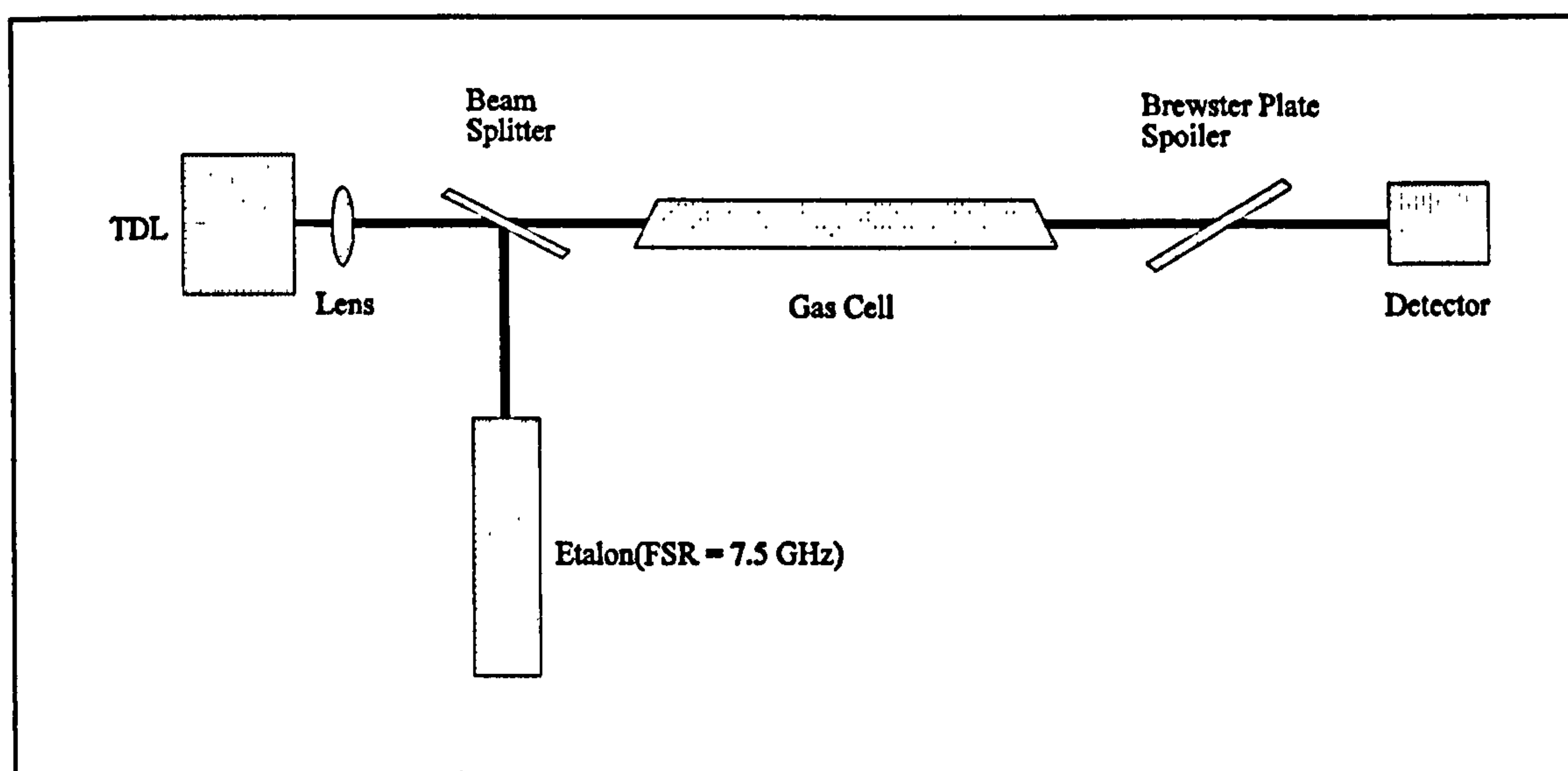


Figure 5.10 - Schematic of the Optical Set up

2nd Harmonic WMS detection was used with a modulating frequency of 10 kHz. The system was intentionally aligned to promote fringing between the detector and the laser source which were separated by 1m. The cell was 15 cm long and was filled with a 5 Torr N_2O and 100 Torr N_2 mixture. Figure 5.11 shows the scan recorded. The observed lines are part of the $3\nu_3$ band of N_2O . They correspond to absorptions of the

order of two to three percent. It is seen that the fringing is equivalent to a MDA of $\sim 0.1\%$. The etalon channel was used to determine the FSR of the fringes. The FSR is ~ 160 MHz. This is in good agreement with the theoretical FSR (150 MHz) associated with the cavity surface spacing.

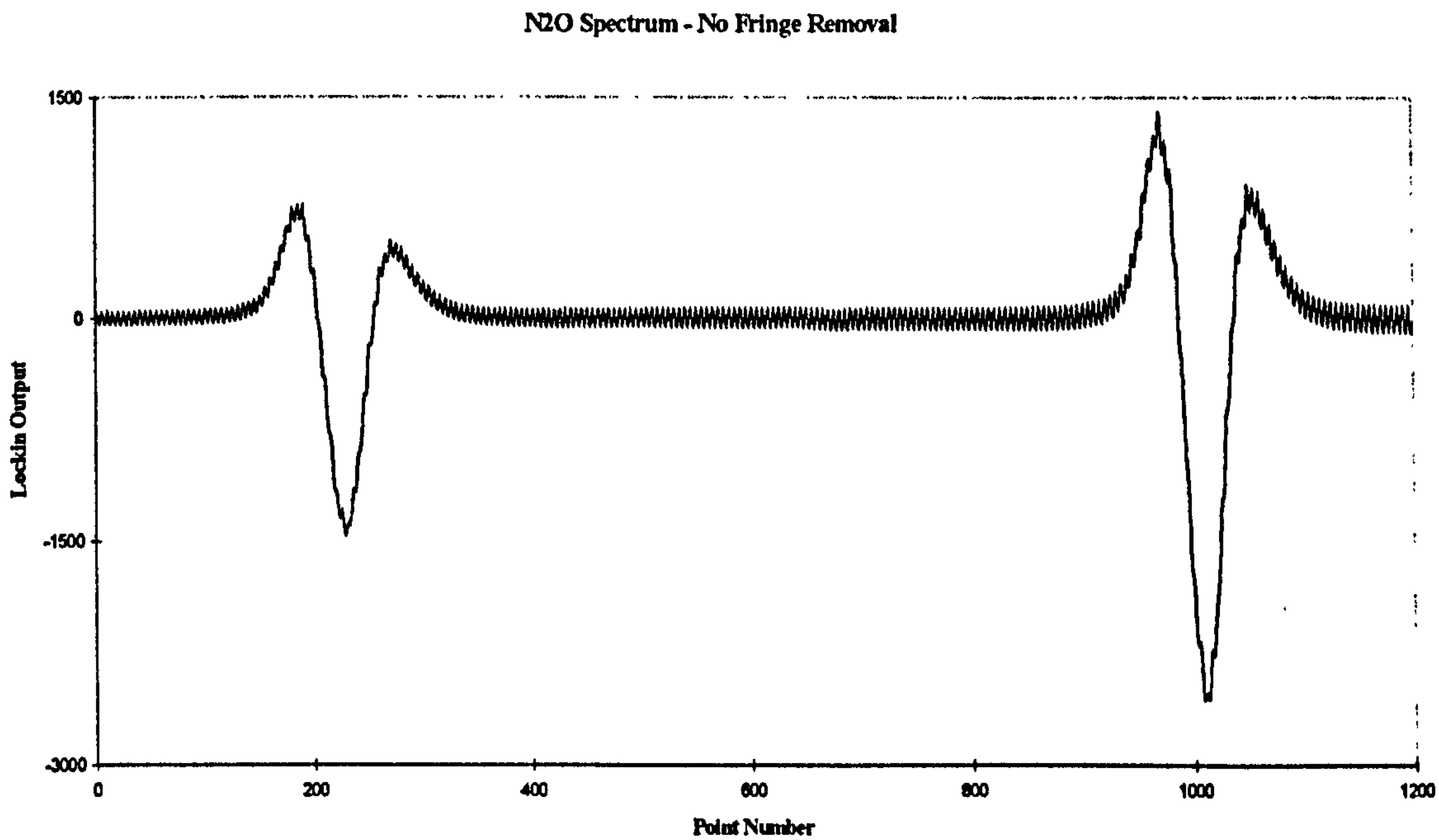


Figure 5.11 - N₂O Spectrum without Fringe Spoiling

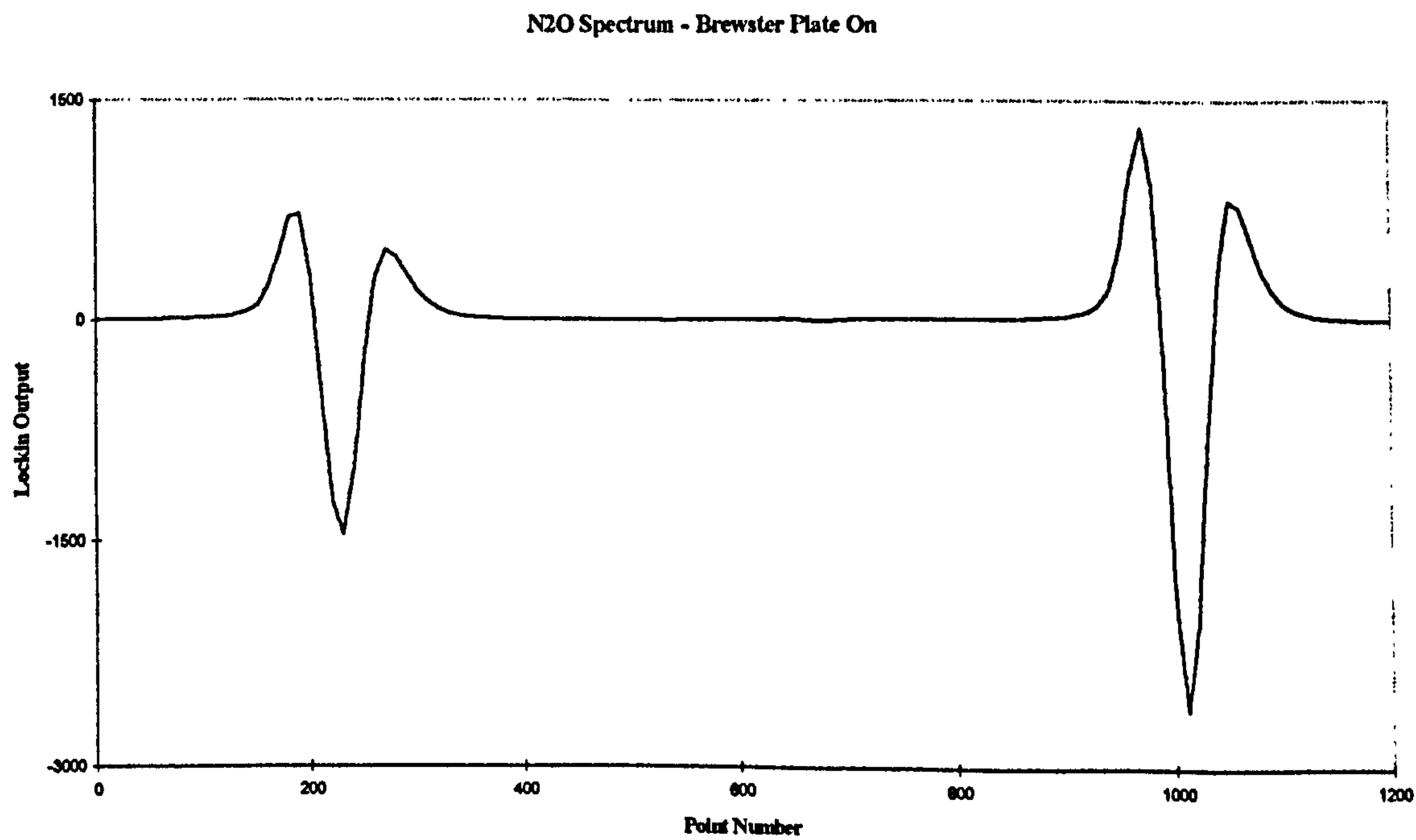


Figure 5.12 - N₂O Spectrum with Fringe Spoiling

The Brewster plate spoiler was turned on and the scan repeated. **Figure 5.12** shows the spectrum that was obtained. It is seen that the fringe amplitude has reduced significantly (by two orders of magnitude). Experimentation with different oscillation frequencies and amplitudes was carried out to determine which gave the best reduction in fringe levels. It was found that the optimum oscillation frequency depended on the scan rate of the laser, with higher frequencies being required for faster scan rates. For a typical scan rate of ~ 1 mA/sec the optimum oscillation frequency was ~ 60 Hz. This frequency, however, depends on the fringe FSR, as the oscillation must cause an averaging of the fringe amplitude in less than the time taken for the TDL to scan over half of one fringe period.

The optimum oscillation amplitude was found to be four to five times the minimum requirement given in equation (5.30). This was in good agreement with Webster's results. At higher amplitudes, signal amplitude modulation caused by beam displacement at the detector and variations in the plate transmission with angle of incidence began to dominate. Choosing the oscillation frequency so that it and its harmonics lie outwith the effective bandwidth of the lock in amplifier should eliminate this problem. The maximum reduction in the fringe level was typically two orders of magnitude.

Webster also suggested that the Brewster plate could be used to spoil fringes generated within a multipass cell. This claim was investigated by removing the reference cell and incorporating a Herriott cell into the optical system shown in **Figure 5.10**. For the Brewster plate to be used successfully it must be incorporated at some point between the two surfaces that are generating the fringing. In the case of the Herriott cell this will be between the two spherical mirrors. Since it is difficult to determine which beams within the cell are producing fringing, the plate should be mounted so that all beams pass through it.

Inserting the plate in the middle of the cell significantly distorted the 48 pass solution that was set up (see solution described in Section 4.2.15). Both the cell separation and beam input slope had to be altered to bring back the original solution. It was also noted that the beam throughput was significantly reduced (between 50-80 % depending on the plate orientation). The various beam trajectories within the cell made it impossible to have the plate at the Brewster angle for the majority of passes. Several plate

oscillations and amplitudes were tried, but the end result in all cases was a reduction in the signal to noise obtained without the plate in place. This was felt to be caused by the reduction in laser throughput caused by the plate itself. Anti reflection coating the plate would significantly reduce this problem, however, financial and time constraints prevented further investigation into this.

One of the main advantages of the Brewster plate spoiler is that it can easily be placed at any point in the optical path. As such, it proved to be a useful diagnostic tool when trying to find which surfaces in the optical set up were producing fringing. It is concluded that this technique would not be suitable for fringe reduction within the multipass cell. Apart from the reduction in throughput discussed earlier, the beam displacement brought about by the plate is not ideal for field applications. Small changes in the plate position caused by external vibration etc. would be amplified each time the beam passed through it, possibly resulting in large displacements at the detector. Throughput would also be affected by small changes in the plate orientation as a direct consequence of the variation in plate transmittance with the angle of incidence. Again this would be amplified in a multipass set up.

5.4.3 Piezo Mirror Dithering

Piezo mirror dithering works on the same principle as a Brewster Plate Spoiler. That is it changes the separation between the two optical surfaces responsible for generating the fringing. The change in separation is achieved by mounting one of the optical surfaces to a piezoelectric element. Applying a sinusoidal voltage to this element will cause the separation between the two surfaces to change continuously, which will in turn modulate the fringe FSR. As in the case of the Brewster plate spoiler, this FSR modulation allows the fringes to be averaged or *washed* out. This technique has been successfully used by Stanton and Silver [63] to reduce fringe amplitudes by up to two orders of magnitude.

Experimental Set up and Results

One of the main disadvantages of the Brewster plate spoiler was that it was not suitable for removing fringes generated within the Herriott multipass cell. Investigation into the suitability of piezoelectric dithering for this application was therefore carried out. A schematic of the Herriott cell used is shown in Figure 5.20. A piezoelectric element from Strathclyde University (unknown origin) was attached between the back mirror of the Herriott cell and the Kinematic mount (Section 5.5). The element was electrically insulated from both the mirror and mount with small pieces of Mylar. A Burleigh piezo controller was used to apply a sinusoidal waveform to the piezo. Figure 5.13 shows a spectrum of air. Piezo fringe dithering was *turned on* approximately half way through recording the spectrum. It is seen that the fringe amplitude has dramatically reduced (by a factor of 30). The small absorption features are water lines and correspond to $\sim 0.0005\%$ absorptions.

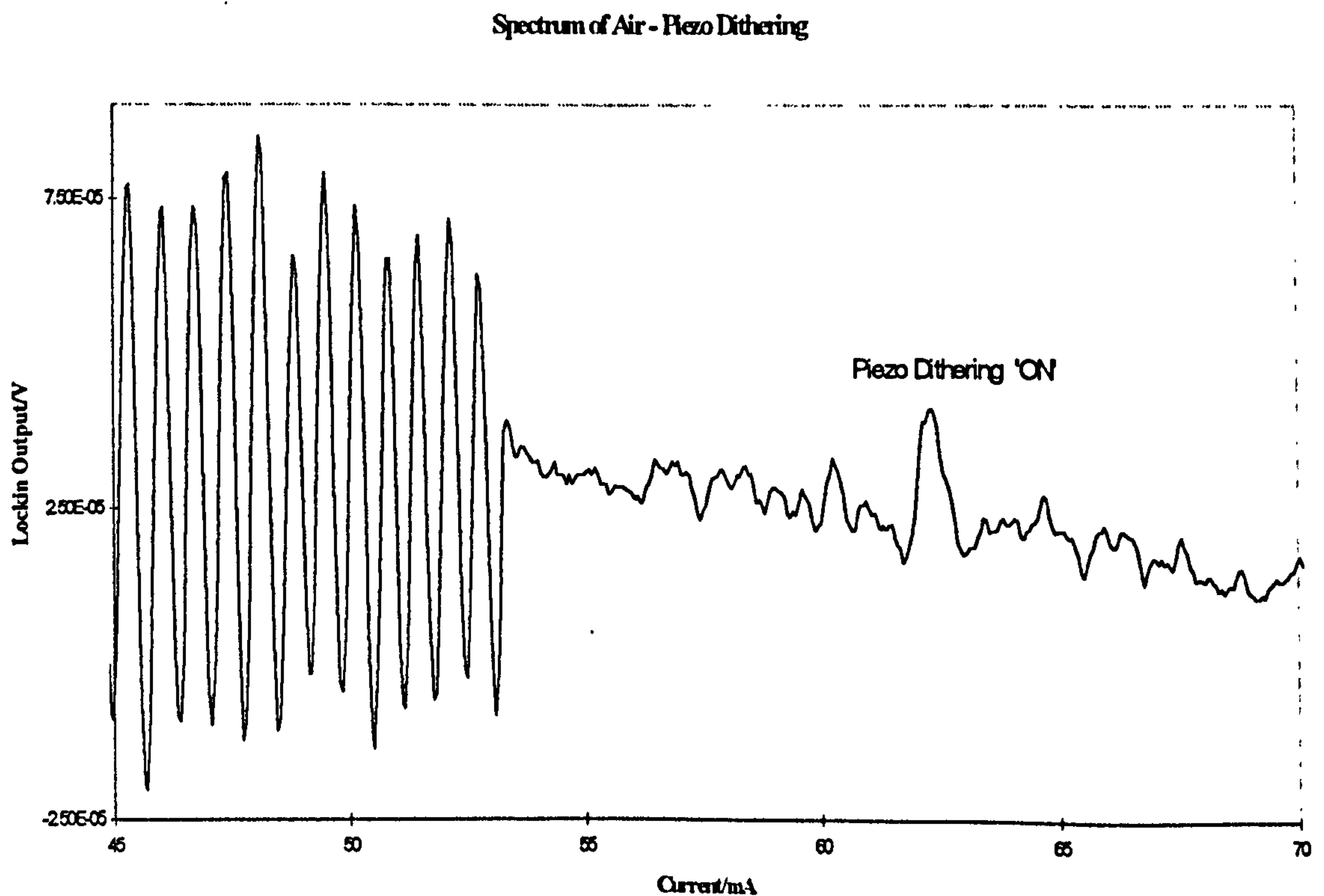


Figure 5.13 - Spectrum of Air with Piezo Dithering

Thus it is seen that piezo dithering can significantly reduce fringing generated within a Herriott multipass cell. Long term use of this fringe reduction technique has shown that fringe amplitude can typically be reduced by a factor of twenty. This technique, combined with careful optical design and alignment can give sensitivity limits equivalent to a minimum detectable absorption of $\sim 2 \times 10^{-7}$ (see section 6.3). The optimum dithering frequency, as with the Brewster plate spoiler, depended on the scan rate of the laser. It was typically in the region 50 - 150 Hz.

5.4.4 Fourier Transform Fringe Removal

Post measurement removal of fringing can be performed by using Fourier transform techniques. MATLAB has powerful Fourier transform tools built into it which can be used to remove fringing whose period is either substantially greater or smaller than the absorption feature that is being measured. Details of how this is done are given below. MATLAB can calculate the discrete Fourier transform of any data set. This allows a spectral analysis of the recorded data to be performed. Fringing can be removed by setting the frequencies corresponding to the unwanted fringe period to zero and back-transforming the spectrum. The software used to perform this process is listed in Appendix 2.

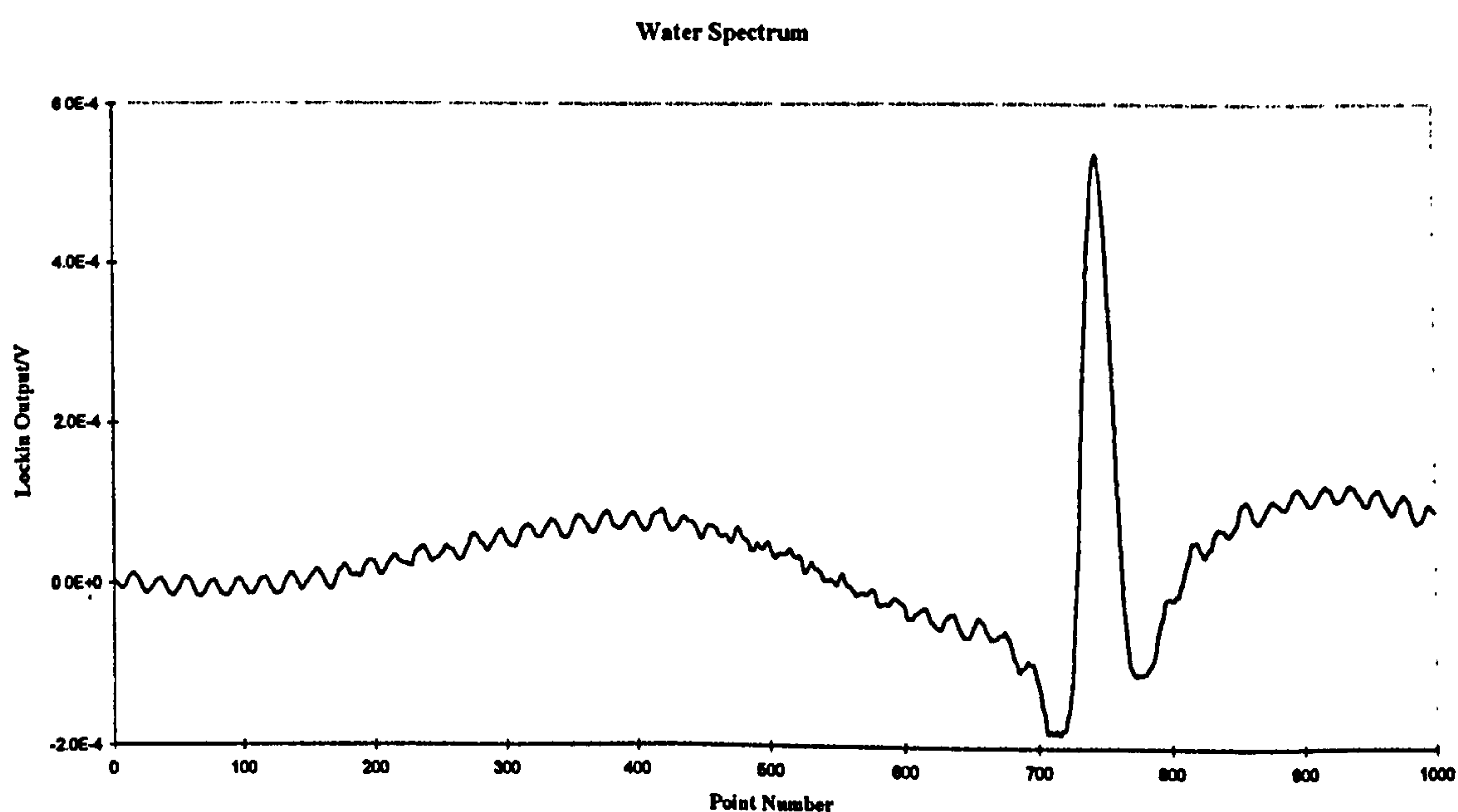


Figure 5.14 - Water Spectrum exhibiting both high and low frequency fringing

Figure 5.14 shows a spectrum of water recorded at 1520nm. It is evident that the spectrum exhibits both low and high frequency fringing. This is generated by etaloning from a non wedged window used at the cell entrance and etaloning from within the multipass cell respectively.

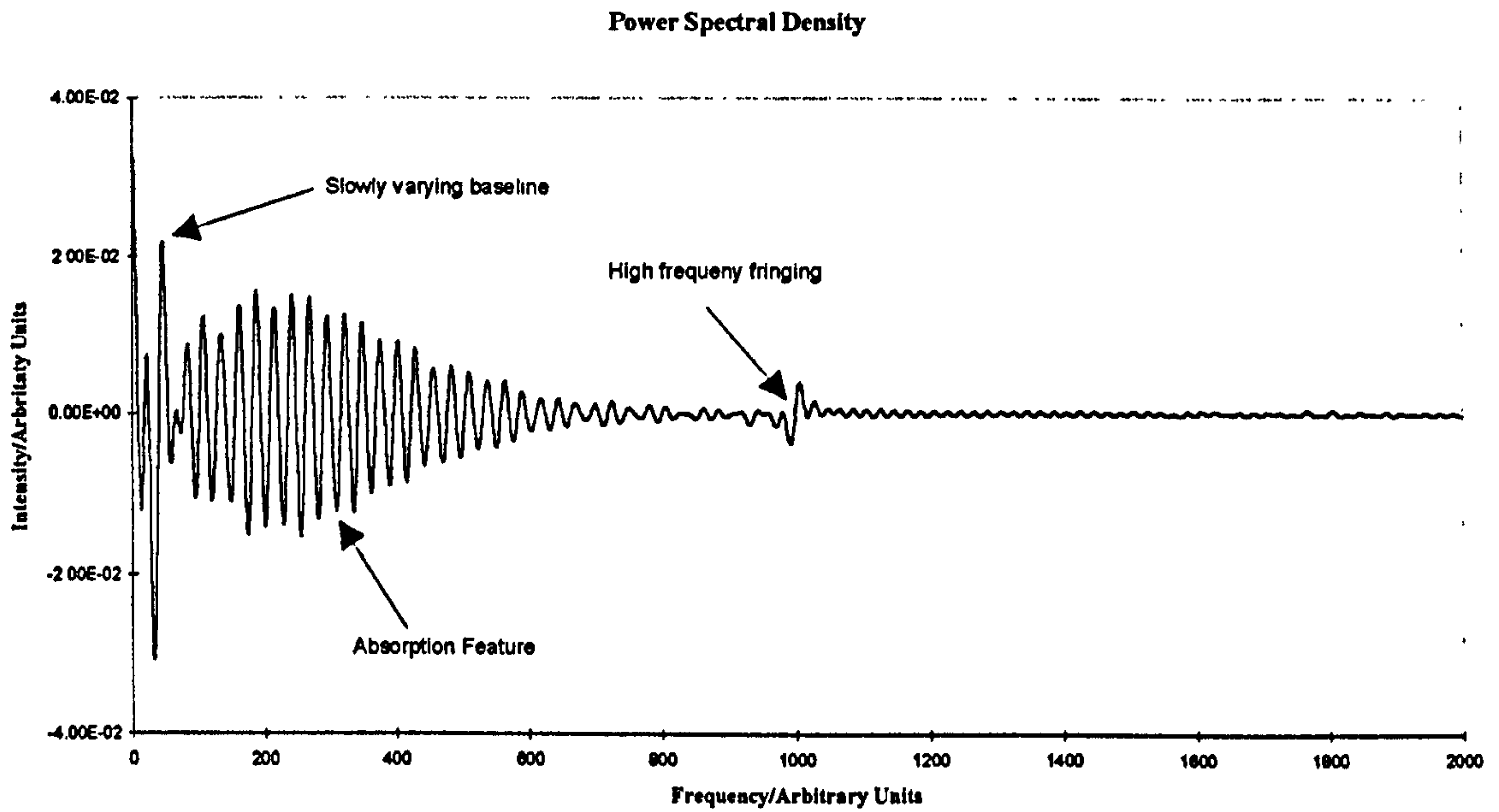


Figure 5.15 - Power Spectral Density of Water Spectrum in Figure 5.14

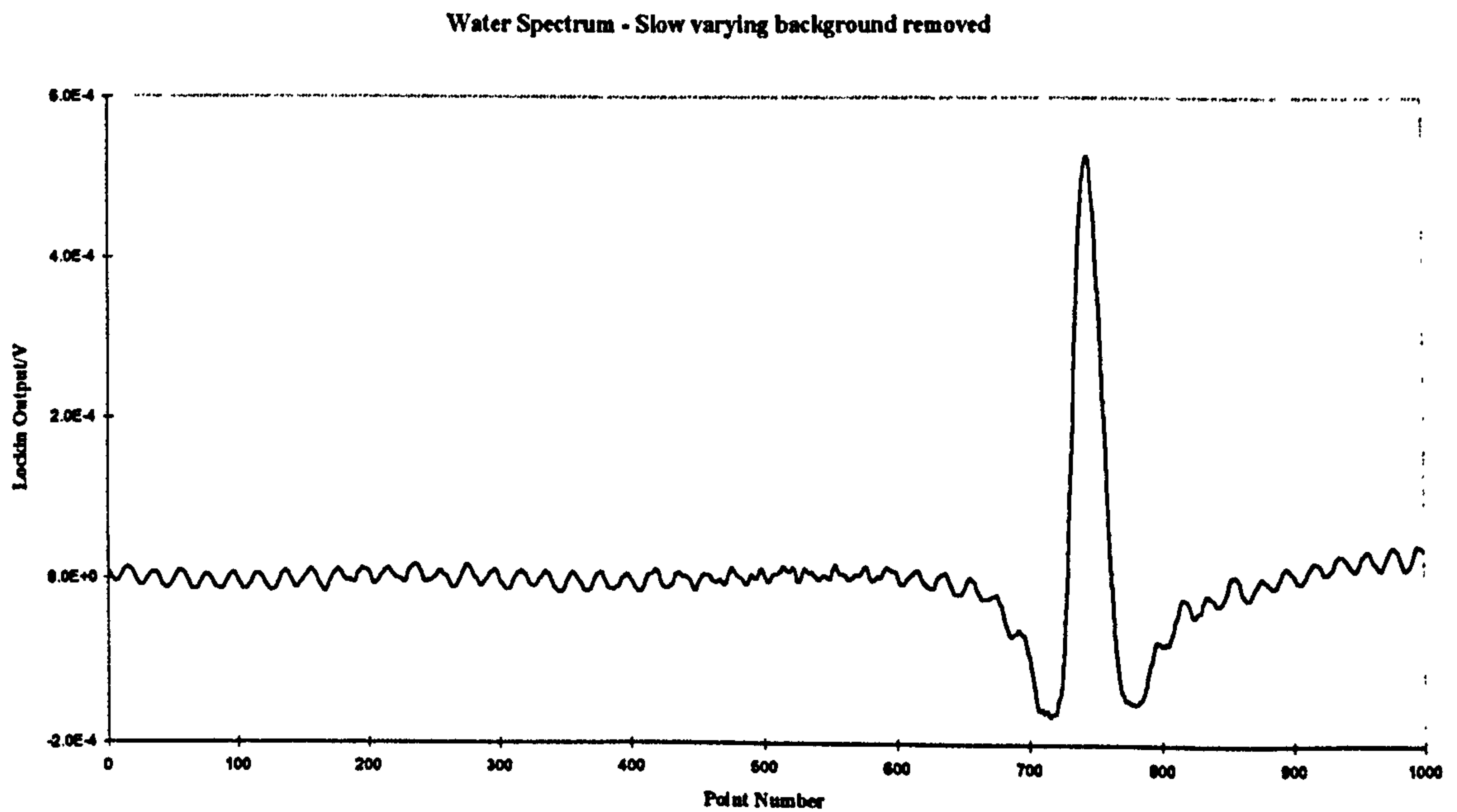


Figure 5.16 - Water Spectrum with low frequency fringing removed.

Figure 5.15 shows the power spectral density of the water spectrum in Figure 5.14. The spectral density gives a measure of the energy at various frequencies. Marked are the clearly distinguishable features corresponding to the high and low frequency fringing and the absorption feature itself.

The feature corresponding to the low frequency fringing was set to zero and the data back-transformed. Figure 5.16 shows the spectrum obtained.

The slowly varying background has all but disappeared. The absorption feature and high frequency fringing were unaffected by this process.

Figure 5.17 shows the spectrum obtained when both the low and frequency components are removed.

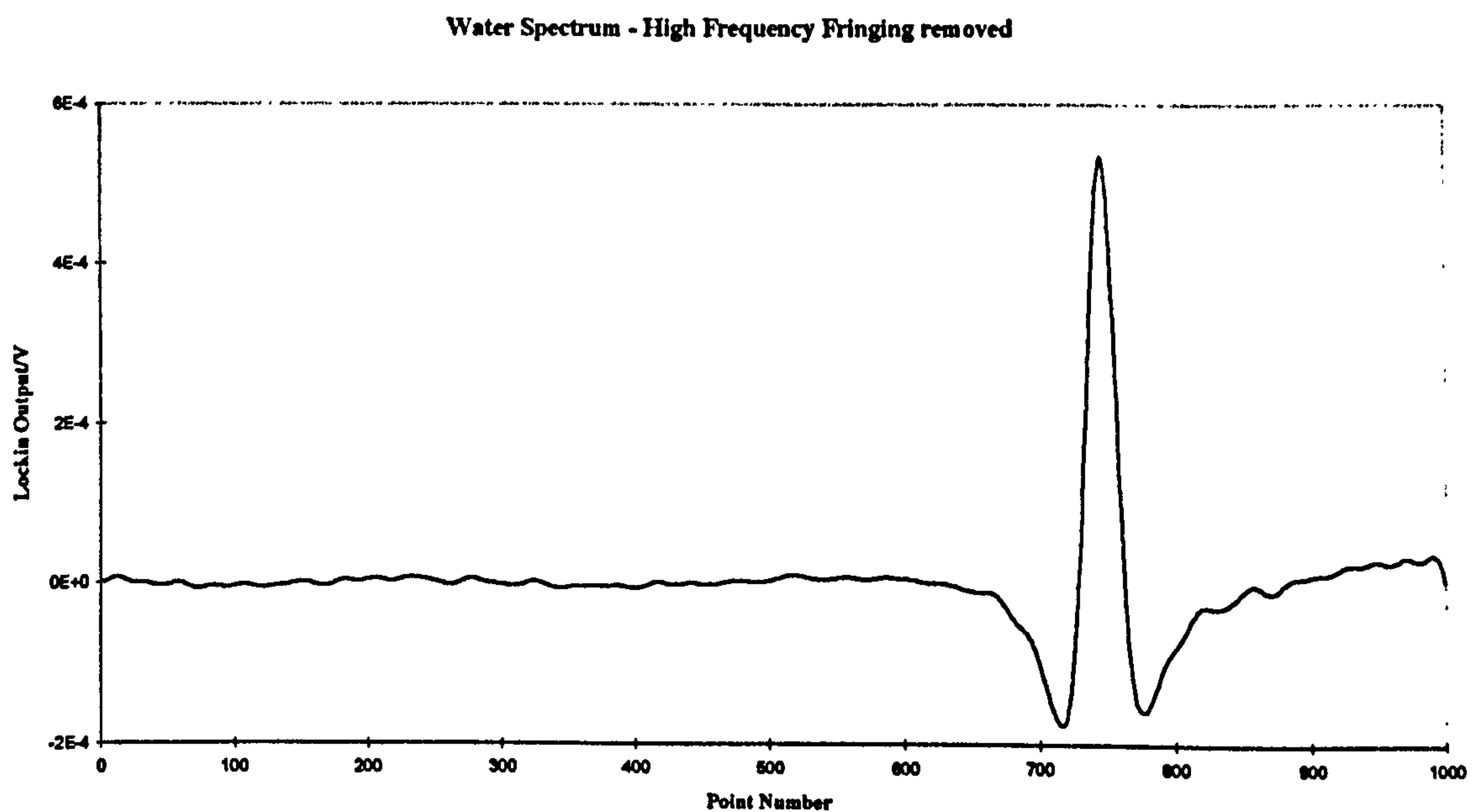


Figure 5.17 - Water Spectrum with low and high frequency fringing removed

The spectrum has been substantially improved by using Fourier transform techniques to remove fringing. The process has been shown to work equally well for fringing with a period that is either substantially smaller or greater than the linewidth of the absorption feature. Importantly, the absorption feature itself is not affected by the Fourier transform removal of the fringing. It should be noted however, that choosing the correct region of the Fourier transformed spectrum to be removed is not always obvious. Usually it is determined through *trial and error*.

5.4.5 Conclusions

Of the three fringe removal techniques used, the Brewster plate spoiler was least suited to TDLAS because it could not remove fringing generated within the multipass cell. Piezo mirror dithering and Fourier transform fringe removal techniques were found to be well suited to TDLAS applications, with both being capable of reducing fringe amplitudes by a factor of twenty or more.

5.5 Instrument Design

5.5.1 Introduction

This section describes a ground based prototype tunable diode laser absorption spectrometer (TDLAS) that has successfully been used to measure methane and nitrous oxide in their natural abundances within the atmosphere (1.78 ppm and 330 ppb respectively).

Where possible commercial electronic and optical components have been used. The decision to use commercial components was taken because in general it proved to be cheaper, but more importantly, drastically reduced the development time. However, some items such as the diode laser mount, evacuable cell and the detector preamplifiers have been designed and built *in house*.

5.5.2 Optical Design

Figure 5.18 shows a schematic of the optical layout. A brief description of the optical train will be given. The main components will then be discussed in more detail.

Optical Train

The optics fit on a 30 x 60 cm² lightweight aluminium honeycomb breadboard (Ealing Cat. no. 54-1953). This gives the optics the required stability while minimising the overall weight of the instrument. The compact design is facilitated by the small optical absorption cell and the thermoelectric temperature controlled laser diode mount. The optical train of the module is as follows.

Infrared light from the laser diode is collimated by an anti reflection coated microscope objective (Newport F-40B, $f = 4.5$ mm, $NA = 0.47$). The 4mm diameter collimated beam passes through a 50/50 pellicle beamsplitter (Ealing Cat. no. 22-3453), allowing a visible trace laser diode (Ealing Cat. no.21-6515) to be brought into the system. This trace beam is an indispensable aid to alignment of the optics. The 50/50 beamsplitter

transmits 50 % in the visible and 85% in the near-infrared (1500-1800 nm). Two irises are set up further along the optical train to ensure both beams are colligned.

The laser then passes through a 90/10 beamsplitter (Ealing Cat no. 22-3420) where 10 % of the laser power is directed through a Brewster reference cell which is filled with a low pressure mixture of methane or nitrous oxide. This cell provides strong absorption features, allowing spectral line positions to be identified. Where laser wavelength stability is a problem, these lines can also be used to line lock the laser. An InGaAs detector (Newport 818-BB-30) is used to detect the reference beam.

The remaining 90 % of the infrared beam is reflected by two aluminium coated beam steering mirrors (Ealing Cat no. 23-6604) before being sent into the Herriott cell. The nearly collimated output beam exits from the cell at an angle of $\sim 6.6^\circ$ relative to the input beam. The beam is collected by an aluminium coated Off Axis Paraboloid (Ealing Cat no. 43-8853) and focused onto a Germanium/InGaAs detector 50mm away.

All components are mounted on Newport VPH series holders and SP series posts. The optical axis height is 7.5cm

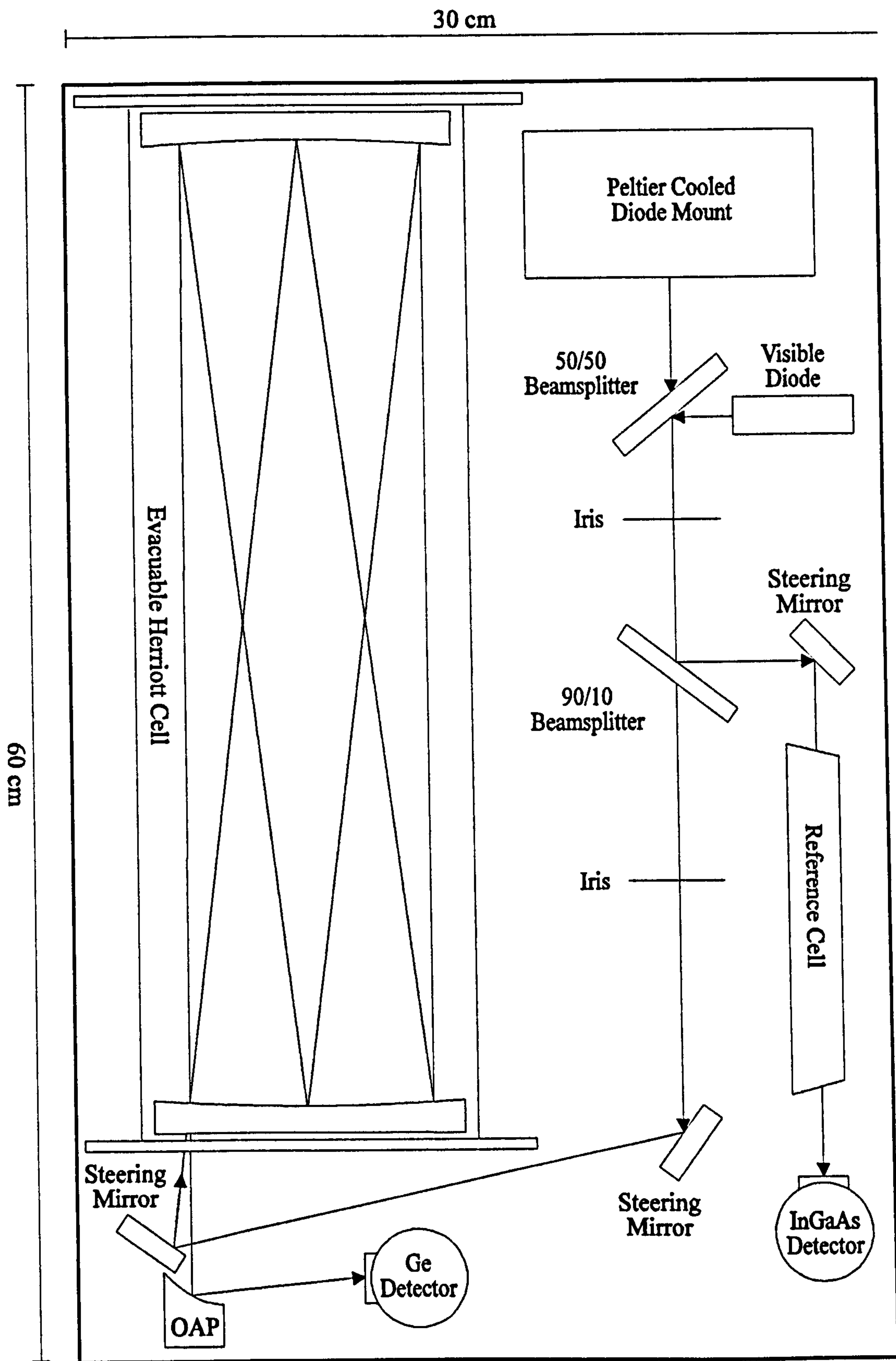


Figure 5.18 - Schematic of the Optical Module

Distributed Feedback (DFB) Near-infrared Lasers

The optical source chosen for the instrument is near-infrared (0.8 - 2.0 μm) DFB laser diodes. The main advantages of DFB devices over their mid-infrared lead salt counterparts have been well documented elsewhere in this thesis (see section 2.2.5)

External Cavity Lasers (ECL) were also considered, however, experience gained at Strathclyde University with an ECL (BTD TSL 1000) has cast doubt on their suitability to TDLAS. It was found that the wavelength stability of these devices was poor because of the highly alignment sensitive external optics. In addition, the inability to wavelength modulate these lasers at high frequencies (>2 kHz) coupled with low output powers resulted in the ECL giving substantially poorer noise performance than DFB lasers.

Two DFB lasers will be used in this instrument set up. A 1.52 μm device from Thomson Semiconductors (MKL 1200 DFB X11/12) for detecting the $3\nu_3$ transitions of nitrous oxide and a 1.65 μm laser from Anritsu (SD3B254F) for detecting the Q branch transitions of the $2\nu_3$ band of methane. The characteristics and specification of both devices are given in Section 3.2.1 and 3.2.2. The Thomson laser comes in a standard can package which is mounted directly in the temperature controlled laser mount. The methane device comes in a butterfly package, has inbuilt thermoelectric coolers and is fibre coupled. It is housed in a die cast box for protection.

Temperature Controlled Laser Mount

The temperature controlled diode mount was designed and built at Strathclyde University. Commercial devices are available (Newport, ILX) but the cost is prohibitive ($> \pounds 2000$). **Figure 5.19** shows a schematic of the mount. The main body is made from Aluminium.

Temperature control is achieved through the use of two 5 W thermoelectric coolers (RS 284-804). These thermoelectric devices use the Peltier effect, which is the change in temperature produced at the junction between two dissimilar metals or semiconductors when a current is passed through that junction. The direction of the

current determines whether the temperature rises or falls. These devices are capable of creating a temperature difference of up to 60°C between the two surfaces. The coolers are attached to the mount with thermally conductive adhesive (RS 850-984). This adhesive also acts as an electrical insulator which ensures the static sensitive diodes are insulated from the outside casing.

The diode itself is mounted on a removable copper plate. This allows diodes for different species to be incorporated quickly and easily into the mount. Copper was chosen because of its superior thermal properties. A curve matched thermister (RS Components) is embedded in this copper plate beside the laser to allow the temperature of the device to be monitored.

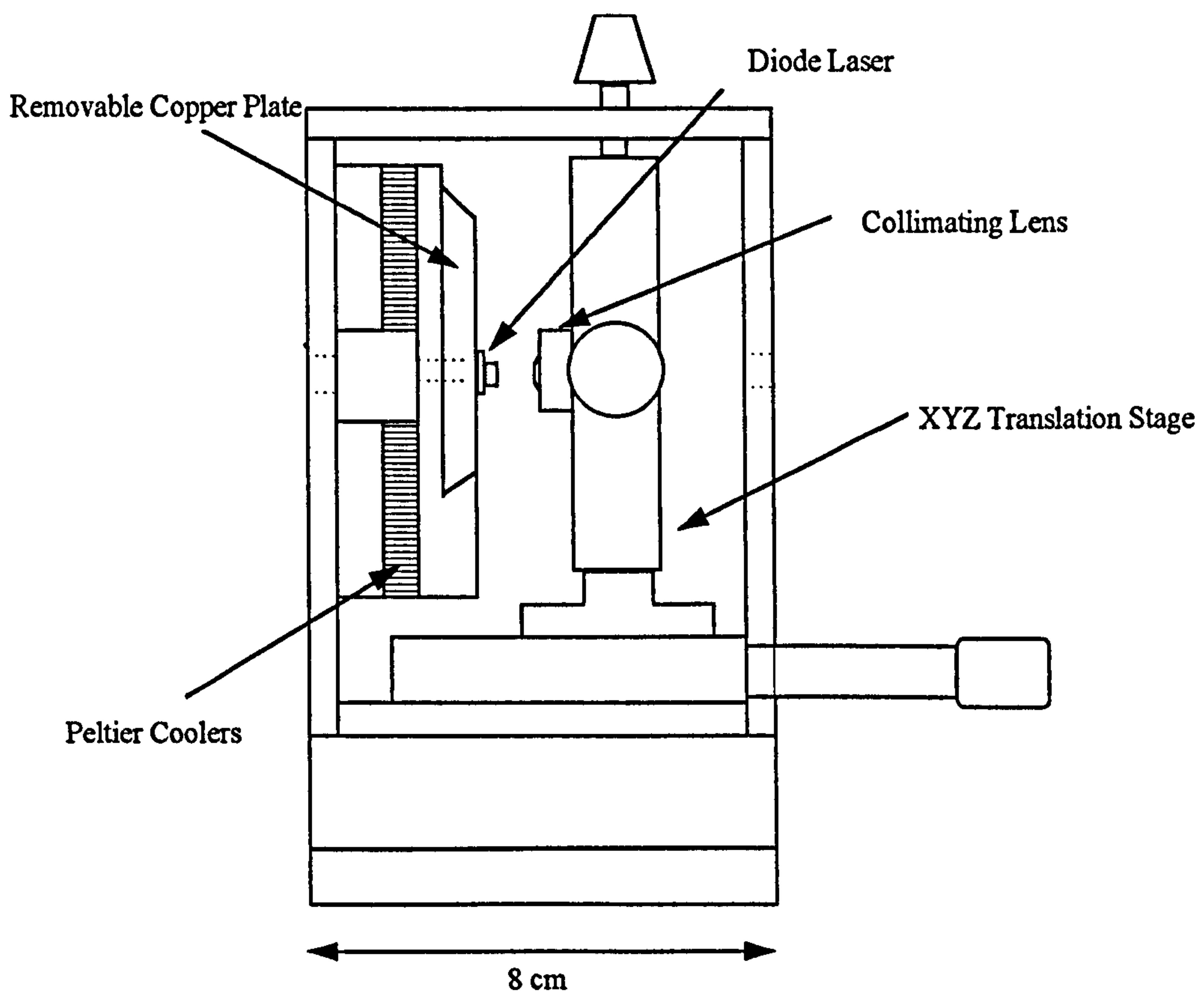


Figure 5.19 - Schematic of Temperature Controlled Diode Mount

The mount also incorporates the collimating optics. An X-Y-Z translation stage (Microcontrol) with 5 µm resolution is used to ensure accurate collimation. Finally, a fibre optic bulkhead connector is situated on the backplate to allow collimation of fibre coupled lasers.

Evacuatable Herriott Cell

Figure 5.20 shows the schematic of the Herriott cell. The main body of the cell is made from four inch inner diameter glass QVF piping. This piping allows a clear unobstructed view of both mirror surfaces facilitating easy optical alignment. The piping is closed at both ends by 20 cm diameter, 1 cm thick aluminium flanges. Viton O-rings provide an evacuable seal and allows for thermal contraction or expansion in the glass. The front of the cell is attached rigidly to the breadboard. The back of the cell has some degree of movement to allow for table contraction or expansion.

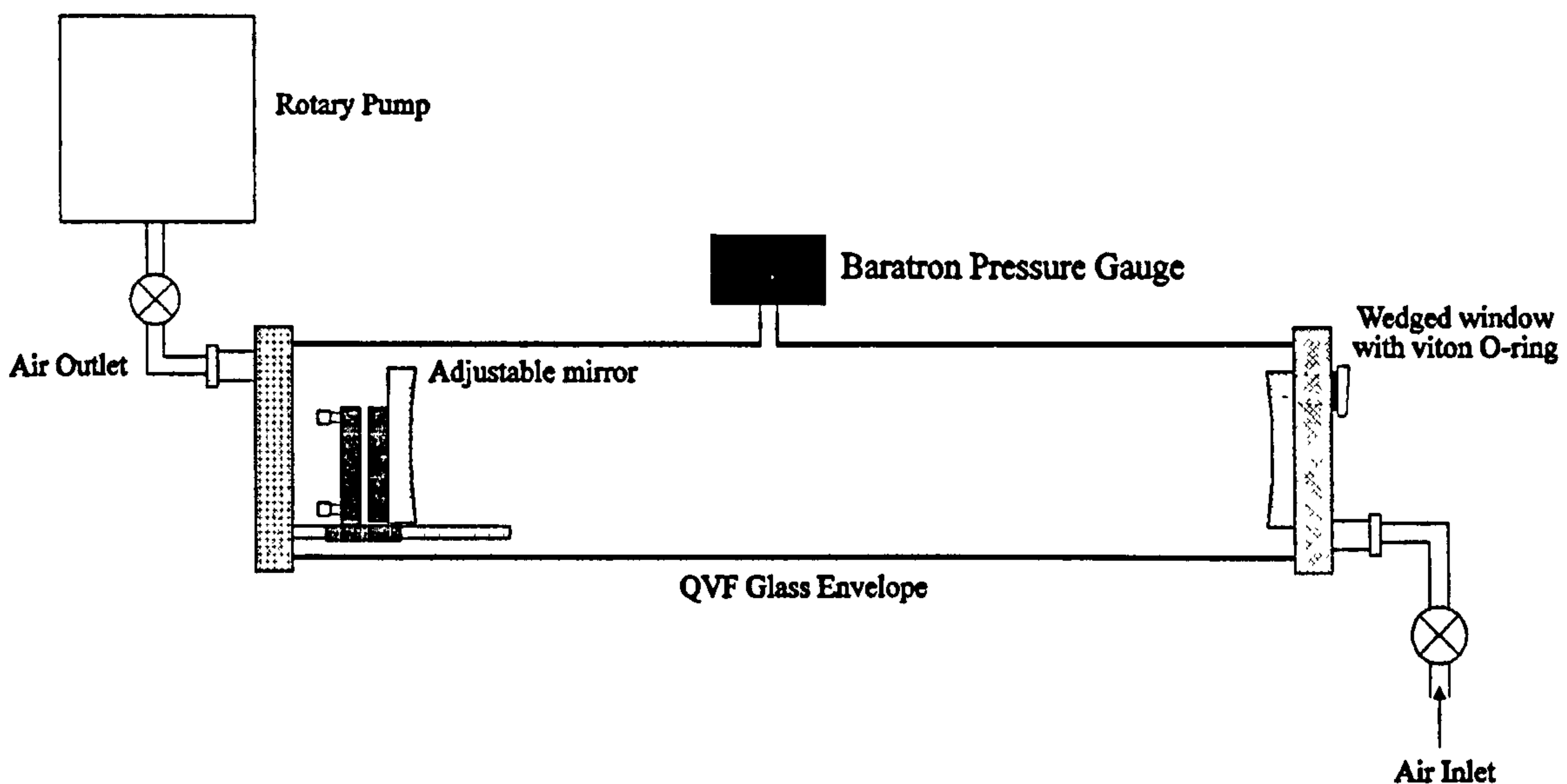


Figure 5.20 - Schematic of the Evacuatable Herriott Cell

The front flange has the coupling hole mirror directly attached to it with epoxy resin. A wedged fused quartz window (unknown origin, 40mm diameter, wedge angle 5°), placed 3.5 cm off axis, allows beam entrance and exit. The wedge spoils any etalon fringing generated between the two window surfaces. A 3cm diameter Viton O-ring provides the seal.

The second flange bears an aluminium slide and rail, to which the back mirror is attached. The rail is 15 cm long. This mirror is mounted on a Kinematic mount

adjustment. A piezoelectric element (unknown origin) is placed on the mirror mount to allow fringe reduction (Section 5.4.3). An electrical feedthrough on the backplate was sealed with epoxy resin. A young tap on the QVF piping allows gas fill for static measurement.

Air is sampled through a 1 metre length of 1/4 inch O.D. PTFE tubing. The flow rate is controlled by a Teflon needle valve attached to the front flange. Pressure is monitored by a 1000 Torr Full scale Capacitance Manometer (MKS Baratron 122AA-01000AB). A rotary pump (Leybold D8B) draws air through the multipass cell with a pumping speed of 161 l/min. The sample line resistance limits the maximum flow rate to 30 standard litres per minute (slm). The flow rate was measured by rapidly injecting small amounts of CH₄ into a stream of carrier gas (dry nitrogen) flowing through the cell and measuring the spectral contrast as a function of time. Figure 5.21 shows the results of the response test on the cell. The time response of the cell (1/e) was approximately 2.5 seconds.

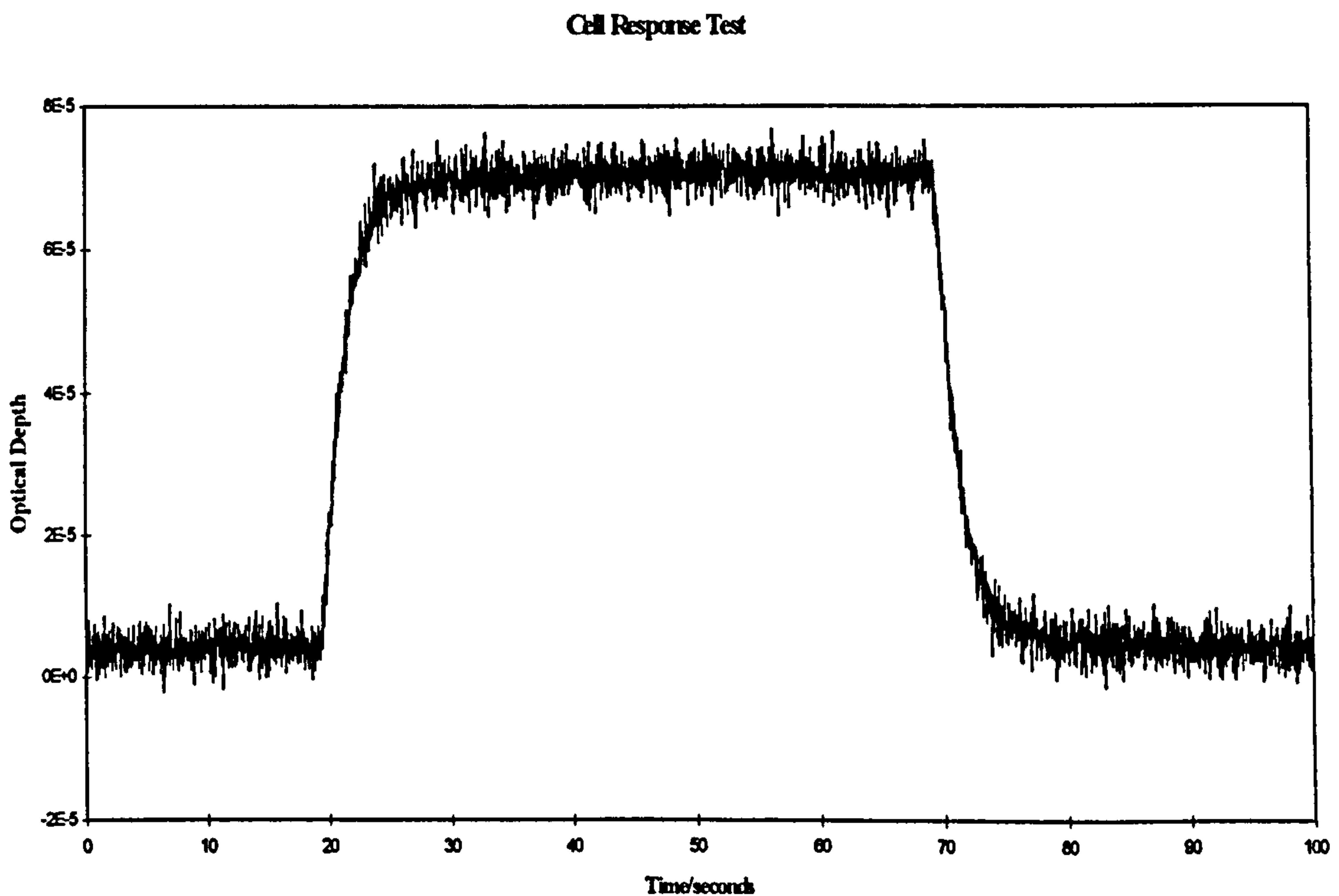


Figure 5.21 - Flow response test of multipass cell

Temperature within the cell is monitored by two curve matched thermistors (RS Components). They have a rated accuracy of ± 0.5 K at 296 K. They are placed at each end of the cell allowing any temperature gradient within the cell to be detected.

The Herriott cell mirrors (Ealing Cat no. 25-9943) have a 60 cm radii of curvature and 7.5 cm diameter. A 48 pass solution provides a 25.1 m pathlength in a 3 litre volume.

A 4mm hole placed 3.5cm off axis on the front mirror allows beam entrance and exit.

Figure 5.22 shows the spot pattern obtained.

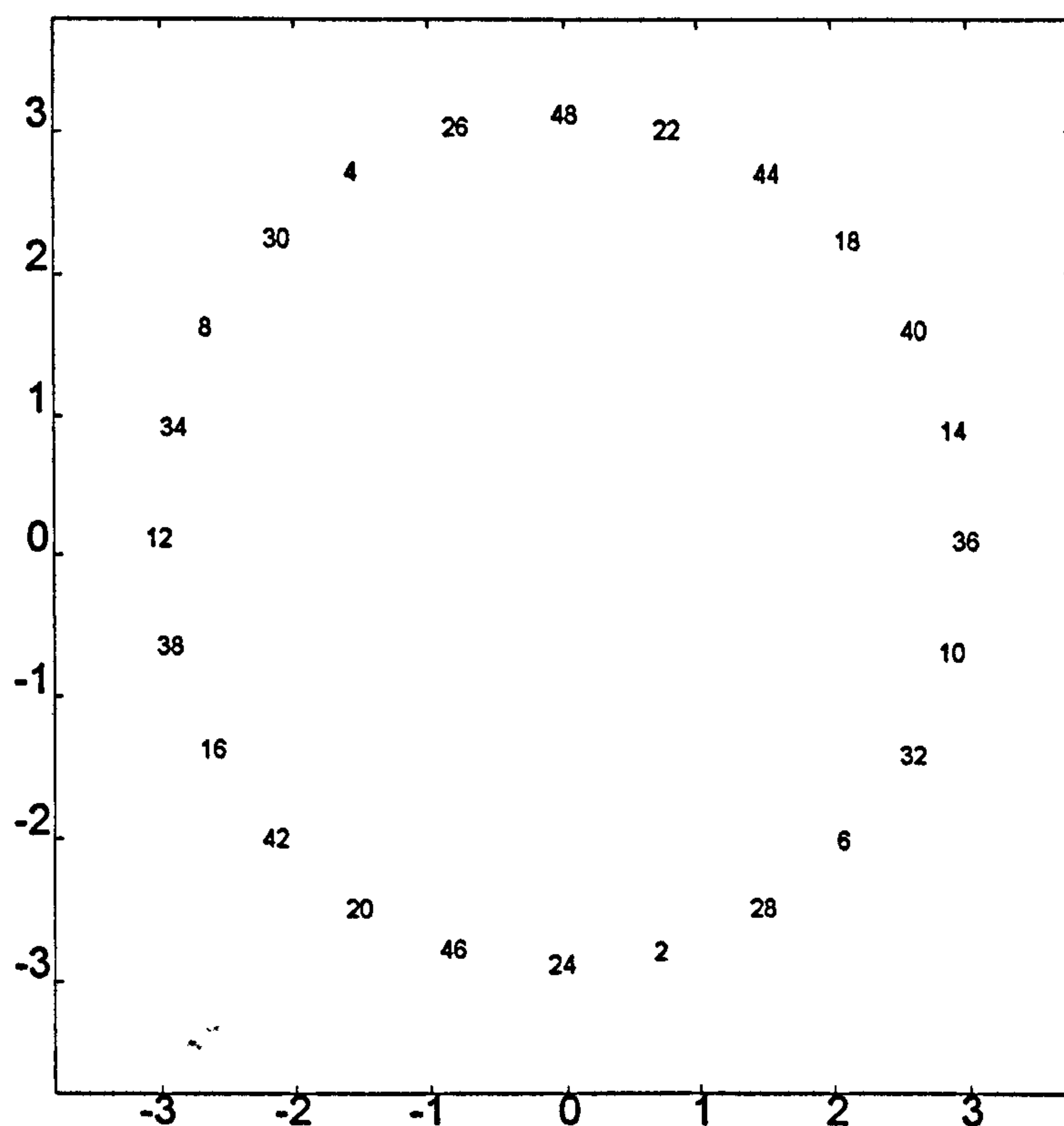


Figure 5.22 - 48 Pass Herriott Cell Solution

This pattern was chosen because it belongs to a subset of solutions that ensures that the FSR of any fringing generated within the cell will be significantly smaller than the width of the feature to be measured. Further details about the choice of spot pattern can be found in Chapter 4.

Detectors

There are two suitable materials available for detection in the region 1 -2 μm . They are Germanium and InGaAs. A Hamamatsu Ge detector (B1720-02) and Newport InGaAs detector (Newport 818-BB-30) were procured to allow detection at 1.65 μm and 1.52 μm respectively. The specification of the two detectors are given below.

Detector	Responsivity	Active Area	Cut Off Frequency	Dark Current
InGaAs	0.6 A/W at 1.5 μm	5 x 10 ⁻³ mm ²	> 1 GHz	< 1 nA
Ge	0.8 A/W at 1.65 μm	1 mm ²	> 10 MHz	< 300 nA

Table 5.4 - Specification of the InGaAs and Ge Detectors

Unlike HgCdTe or InSb mid-infrared detectors both these detectors operate at room temperature.

The main design criteria in choosing a suitable detector include responsivity at the detection wavelength, active area diameter and detection bandwidth. These are now discussed in more detail.

Active area diameter and Detector Bandwidth

The detector area will affect both the bandwidth of the detector and the dark current noise. Theory predicts that the detector area must be minimised to provide low dark current levels and large detection bandwidths. However, it is advantageous to have the detector area sufficiently large to allow it to be underfilled, thus making the system insensitive to misalignments. A compromise between these conflicting design requirements must be found.

Theoretical calculations have shown that for both materials considered here, a good compromise would be to use a 1mm diameter active area. This area dimension can be easily underfilled, will give bandwidths in the tens of MHz region and will exhibit dark current noise equivalent powers (NEP) of 10^{-13} W/Hz^{1/2}.

The Newport InGaAs device does not follow this design compromise. It has an extremely small active area resulting in dark noise levels and detection bandwidths that are superior to the Germanium device. This would seem to be at the expense of alignment sensitivity, however, this detector has a focusing element attached to it which has been designed to ensure that the beam (collimated or diverging up to 20°) will underfill the detector area. This detector would seem to provide the *best of both worlds*.

Other Design considerations

These detectors can be thermoelectrically cooled to reduce dark current (cooling down to 245 K can reduce dark noise by up to three orders of magnitude), however, detailed noise calculations in Section 5.3.5 indicate that the dark noise levels exhibited by these two detectors at room temperature will be an order of magnitude smaller than the calculated thermal/shot noise levels. Cooling is therefore unnecessary.

5.5.3 Electronics

The instrument electronics have all been bought in commercially with the exception of the detector pre amplifiers, which have been designed at Strathclyde University and the National Physical Laboratory. A schematic of the electronics is given in **Figure 5.23**. The majority of the electronics are situated on an equipment rack with dimensions 50 x 50 x 70 cm³. A few components such as the pressure sensor and pre amplifiers are situated on the optical module.

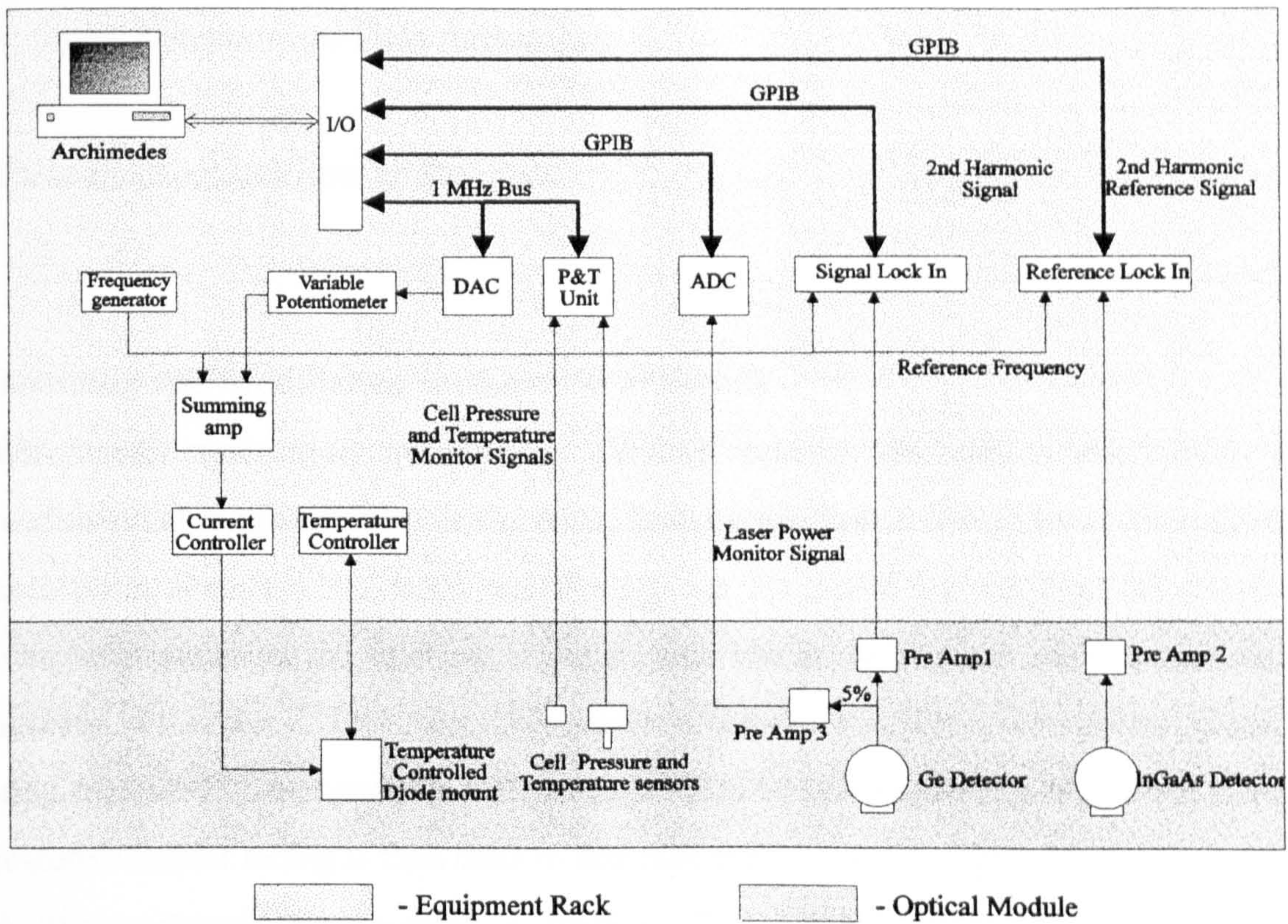


Figure 5.23 - Schematic of the Electronics

Electronic Description

A description of the role these electronics play in the operation of the instrument is given below.

The system is controlled by an Archimedes A310. Control software was developed by Dr Liam Donahoe, a previous research student in the group. A full program listing and detailed description can be found in the relevant part of his thesis, '*Infrared Spectroscopy Related to Atmospheric Chemistry and Physics*' [64].

The control software and electronics perform two primary functions:

- Scanning and modulating the laser wavelength.
- Signal Detection and Data Acquisition

These functions will now be described.

Scanning and Modulating the Laser Wavelength

Wavelength modulation spectroscopy (WMS) requires the laser wavelength to be modulated at high frequency as the mean laser wavelength is slowly tuned through the absorption feature. The laser wavelength can be tuned by varying the junction temperature and/or the injection current. The tuning mechanism of these lasers is detailed in Chapter 2. Changing the junction temperature provides course tuning and is used to shift the laser operating wavelength close to the wavelength of the absorption feature. Current tuning is then used to fine tune the laser wavelength across the feature. A thermoelectric temperature controller (ILX LDT-5412) maintains the laser temperature during the current scan preventing drift in the centre wavelength of the laser. A current controller (ILX-3207B) controls the injection current. The output level of this controller can be adjusted manually or remotely via an analogue voltage control/modulation port.

The computer generates a 0-5 V ramp via the 12 bit DAC (Apple DA101). This ramp scans the current via the analogue voltage control/modulation port (transfer function - 100mA/Volt). The resolution of the ramp is controlled in software (1-4096 steps) and the range of the ramp is determined by the variable potentiometer. This arrangement allows the injection current to be scanned over any region accessible to the diode. Current offset is set manually on the controller.

Modulating the laser wavelength is achieved by modulating the diode current at high frequency. A 200 MHz function generator (RS 610-635) provides the modulating waveform. In order for the laser wavelength to be both modulated and scanned, the voltage ramp and function generator waveform must be superimposed. The summing amplifier (RS Components) is used to combine the ramp and function generator signals.

Signal Detection and Data Acquisition

The detectors detect infrared light and convert it into an electrical signal. The signals from these detectors are first preamplified. The preamplifiers limit the effective bandwidth of the detector, cutting out much of the high frequency noise. They also reduce the effects of unwanted RF interference and cable noise. The amplifiers are ac coupled to ensure full use of the preamplifiers dynamic range. For the Ge detector a 100 k Ω transimpedance pre amp (100 KHz B.W) was used. This was designed at the University of Strathclyde by the Electronic workshop. The preamplifier for the InGaAs detector was a 150 k Ω transimpedance amplifier, again with a 100 kHz bandwidth (B.W). This was designed at the National Physical Laboratory [65].

The lock in amplifiers perform several key roles in the data acquisition process. On arrival at the lockin amplifier the signals are first filtered to remove either the line frequency or the second harmonic of the line frequency, depending on which detection harmonic is being recorded. The signals are further amplified and the Fourier component selected. Phase control of the reference frequency, supplied by the function generator, allows the signal due to the absorption feature to be optimised or the unwanted background signal caused by RAM to be removed (see Section 5.3.5 for more details). After phase sensitive detection the signals pass through variable filters.

Both the filter roll off and time constant can be altered. The time constant selection usually determines the overall detection bandwidth of the instrument. The effective detection bandwidth is given by equation (5.31) [66]:

$$B.W_{eff} = \frac{1}{4T} \quad (5.31)$$

where T is the time constant.

The two lock in amplifiers used here also perform the digitisation of the detected signal. The signal lock in (Stanford Research Systems Model SR510) has 16 bit resolution while the reference lock in (EG&G Princeton Applied Research Model 5209) has 13 bit. GPIB interface is used to transfer the digital Words to the Archimedes. At each ramp step the computer requests the digital Word representing the analogue signal. The computer waits until it has received this word before initiating the next ramp increment. A facility exists enabling the computer to request any number of digital words at each ramp step. This allow the data to be averaged. When the scan is finished the recorded spectrum is displayed on screen. The operator can then choose to save, plot or discard the data before initiating a new scan.

The GPIB interface and Lockin digitisers limit the data transfer rate to seven 16 bit words per second

Additional information recorded during a Scan

Accurate retrieval of concentrations from recorded spectra requires additional information such as the temperature and pressure inside the cell to be recorded. Further details about this can be found in Section 6.2. The outputs from both the temperature and pressure sensors are interfaced to the Archimedes analogue port via the 1 MHz bus. Each is sampled once at the beginning, middle and end of the scan.

It is also necessary to monitor the dc laser power falling on the detector. The detected second harmonic signal is proportional to the gas concentration and the signal intensity arriving at the detector. All spectra must therefore be normalised by the dc laser power. This ensures power fluctuations caused by misalignments or laser drift do not affect the retrieved concentration. A small portion (5%) of the detector signal is sent through

preamp 3 as shown in the schematic in **Figure 5.23**. This preamp is a modified version of the 150 k Ω InGaAs detector. The signal is low pass filtered to remove the high frequency modulation component. It is then digitised by the ADC (Biodata Microlink II Module AN-1) and interfaced to the computer via GPIB connection.

5.6 Conclusions

A theoretical analysis of different detection schemes has been performed. It has been shown that near optimum sensitivities can be achieved using second harmonic wavelength modulation spectroscopy (WMS) in the 50-100 kHz region. Sensitivities equivalent to a minimum detectable absorption of $\sim 1 \times 10^{-7}$ are predicted for both laser systems. Sensitivity can be improved by a factor of two by going to higher modulation frequencies and higher detection harmonics, but the additional cost and increased complication of the detection electronics associated with these detection schemes was not felt to be worthwhile.

A comparison between theoretical harmonic lineshapes and those recorded experimentally was also performed. It was found that the fits for higher harmonic ($n > 2$) were significantly worse than those for 1st or 2nd harmonic detection. This was a consequence of the departure from linear frequency tuning at the increased modulation depths associated with the higher harmonics. This would adversely affect the accuracy of concentration retrievals as described in Section 6.2.

Investigation of different fringe reduction techniques has shown that piezo fringe reduction is particularly suited for Herriott cell applications. Post measurement Fourier Transform techniques have also been shown to be an effective way of improving the sensitivity of the instrument.

The optical and electronic layout of the instrument has also been described. The use of near-infrared technology and a compact multipass cell has allowed the optics to fit on 30x60 cm² breadboard. The complete optical module weighs less than 7 Kg. The use of commercial electronics has prevented them from being miniturised.

The entire instrument (including both lasers) would cost \sim £22,000 to build. This represents a significant saving over the cost of a mid-infrared TDLAS system, where cryogenically cooled detectors and lasers and specialised laser control electronics push up the cost.

Chapter 6 -

Ground Based Tunable Diode Laser

Absorption Spectrometer (TDLAS)

Performance Assessment

6.1 Introduction

This chapter describes the calibration and performance assessment of the ground based TDLAS described in the previous chapter.

The completed instrument was used to successfully measure both methane and nitrous oxide in a laboratory environment. An instrument precision equivalent to 560 pptV of methane and 22 ppbV of nitrous oxide was achieved (Section 6.3). This corresponds to a minimum detectable absorption of between 2×10^{-7} and 1×10^{-7} . The instrument accuracy for these two species has been determined as 5% and 12% of the natural abundance for each species respectively (Section 6.5). The above figures represent the precision and accuracy assuming a 1 Hz bandwidth.

A successful field test of the instrument, measuring methane at a landfill site, was performed, allowing an intercomparison with other methane monitors (Flame Ionisation Detectors) to be carried out (Section 6.4). This intercomparison showed that the methane concentrations measured with the TDLAS were within the measurement uncertainty of the other two instruments, validating both the technique and the calibration procedure used.

This work has allowed:

- The sensitivity achievable with near-infrared technology to be determined
- The performance and suitability of the Herriott multipass cell to be assessed

6.2 Instrument Calibration

6.2.1 Introduction

One of the easiest ways to calibrate a TDLAS, is to determine the system response to a known concentration of gas in the absorption cell. While gas standards available for such calibration techniques provide exceptional accuracy in concentration retrievals at the temperature and pressure they were measured at (typically better than 0.5 %), they do not allow accurate concentration retrieval at different temperatures and pressures. This is a concern in a field instrument where variation in the operating temperature, pressure etc. cannot be controlled as they would be in a laboratory environment. A calibration technique that takes into account the effect that these variable parameters have on the linestrength, lineshape etc. is required.

The ALIAS instrument [11] utilises a calibration procedure that allows accurate concentration retrieval over a wide range of operational conditions. The technique determines the system response as a function of the absorption level over the full range of operational parameters (returned laser power, signal chain gain, gas absorption level, modulation depth, line shape etc.). While this procedure is substantially more complicated than the gas standard method it has several advantages, including being gas unspecific, i.e. any gas having absorption lines in the region accessible by the laser can be used to calibrate the signal channel. A detailed description of this technique is given by May and Webster [67]. A brief overview is given below.

6.2.2 Calibration Procedure

Raw spectra recorded by TDLAS usually have, for abscissa, the point number or some other arbitrary unit not directly related to the actual wavelength. Likewise, the ordinate scale will usually be an arbitrary voltage or a digital number proportional to the lock in amplifier output. In order to extract a gas concentration, this ordinate scale must be converted into spectroscopic units. To achieve this the signal processing chain, which consists of preamplifiers and variable gain stages, must be calibrated.

The calibration procedure will determine the signal chain *response number*, R_i , which will be independent of the spectral region used for calibration or the specific characteristics of the diode. To obtain this response number, both the direct absorption and $2f$ spectra for an isolated absorption line are recorded under identical conditions. The observed direct absorption level for the calibration feature, A (obs), and the peak to peak $2f$ signal size of the feature, H_2 (obs), are measured from the spectra. The response number, R_i , is then evaluated as:

$$R_i = \left(\frac{H_2(\text{obs})A(\text{calc})}{H_2(\text{calc})A(\text{obs})} \right) \left(\frac{\text{d.c.}_g}{\text{a.c.}_g} \right) \left(\frac{1}{P'} \right) \quad (6.1)$$

where A (calc)/ H_2 (calc) is the calculated ratio of the direct absorption level to the peak to peak $2f$ signal size for the modulation index used, P' is the total power level above the detector zero, and a.c._g and d.c._g are the a.c. ($2f$ channel) and d.c. (direct absorption channel) amplifier gain values used for the measurements.

The ratio A (calc)/ H_2 (calc) is determined by solving the integrals describing second harmonic WMS as given in equation (5.8). To calculate this ratio, however, requires an accurate knowledge of the line shape of the species that you are measuring and the modulation index that was being used. Further details of how this is achieved is given later in this Section.

Having determined the response number of the signal chain, conversion into spectroscopic units is achieved by scaling the spectrum on a point by point basis as follows,

$$H_j(\nu) = y_j \left(\frac{1}{R_i P'_j} \right) \left(\frac{\text{d.c.}_g}{\text{a.c.}_g} \right) \quad (6.2)$$

where for a point j in the spectrum, y_j is the observed $2f$ value from the lock in amplifier and R_i is the response number. Normalization to the returned TDL power is accomplished by the factor $1/P'_j$. A gas mixing ratio can then be extracted by comparing the observed peak to peak $2f$ signal to the corresponding peak to peak

amplitude in a synthetic spectrum calculated for the same conditions (pressure, temperature, modulation amplitude) as the atmospheric measurement. The synthetic spectrum can usually be generated in HITRAN using a mixing ratio equal to a value close to that expected for the atmospheric measurement. The MATLAB program discussed in Section 5.3.3 can be used to generate the second harmonic lineshape of this feature.

The volume mixing ratio is given by:

$$\delta(\text{obs}) = \frac{\delta(\text{calc})}{H_2(\text{calc})} H_2(\text{obs}) \quad (6.3)$$

where $H_2(\text{obs})$ is the observed peak to peak $2f$ signal amplitude after conversion to spectroscopic units, and $H_2(\text{calc})$ is the synthetic peak to peak signal amplitude calculated for a mixing ratio of $\delta(\text{calc})$.

The above calibration procedure will only be valid if the following conditions are met:

- The absorption of the calibration feature and the atmospheric feature are in the linear region (optically thin) of the Beer Lambert law.
- The calibration gas is unreactive and is not easily adsorbed onto the cell walls.
- The detectors and gain stages must show no non linearity in their response.
- The modulation amplitude used in measuring $H_2(\text{obs})$ must be accurately known to allow accurate determination of the ratio $A(\text{calc})/H_2(\text{calc})$.
- The synthetic spectrum and recorded spectrum must coincide when least square scaling is employed to retrieve the concentration.

Some of the procedures used to ensure that these conditions are met will now be discussed.

Linearity

Non linearity in the detector response and the adjustable gain stage could cause large discrepancies between the extracted and the actual concentration. It is therefore necessary to ensure that such non linearities do not appear over the range of incident power levels and signal sizes that one would expect to encounter during measurement. To ensure this is the case, the signal chain response must be mapped for a wide variety of incident powers and absorption levels. Figure 6.1 shows the instrument response for methane concentrations between 2 - 20 ppmV. This range more than covers the concentrations that one would expect to encounter at ground level. It is seen that an extremely good linear response was obtained. The deviation from the linear fit is within the uncertainty in the gas standard concentrations used ($\pm 0.5\%$).

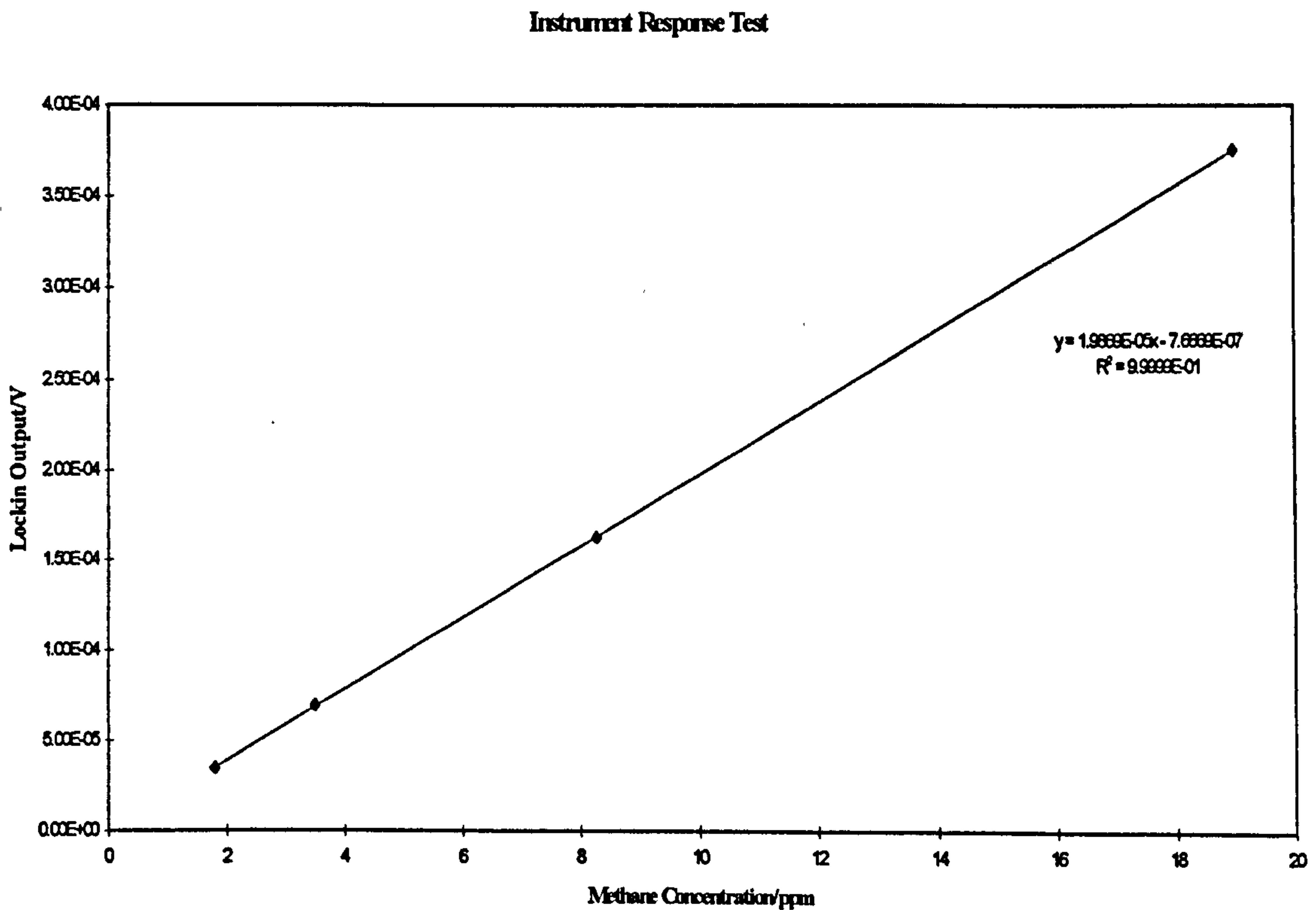


Figure 6.1 - Signal Chain Linear Response Test

Accurate Determination of the Modulation Depth

One of the main factors governing the uncertainty with this calibration technique, is the accuracy to which the modulation depth is known. Figure 6.3 shows how the theoretical peak height of different lineshapes varies with modulation depth. It is seen that small changes in modulation depth can have a significant effect on the peak height, and therefore a significant effect on the extracted concentration. To obtain an accurate value of the modulation depth, a fit between the known theoretical behaviour of the recorded $2f$ lineshape as a function of modulation depth, and the observed behaviour of the calibration feature over the whole range of modulation depths available to the diode, is performed. This fit will produce a scaling constant, C , between the theoretical modulation depth (in cm^{-1}) and the experimental modulation depth, which is usually measured in mA. May and Webster [67] fit the peak height, P (see Figure 6.2), of the second harmonic feature, but it is more convenient to fit the ratio, R (see Figure 6.2), of the top (P) and bottom (N) lobe heights, as it is independent of laser power, signal gain etc. Figure 6.4 shows the theoretical behaviour of the peak ratio, R , for a Doppler and Lorentz line. Voigt lines exhibit behaviour somewhere in between these two cases. It should be noted that the scaling constant obtained from the fitting will only be valid for the modulation frequency used in the fit. The variation of the tuning rate with modulation frequency for both the DFB lasers used in this study (see Section 5.3.5 for a detailed discussion) preclude the constant being used at any other frequency.

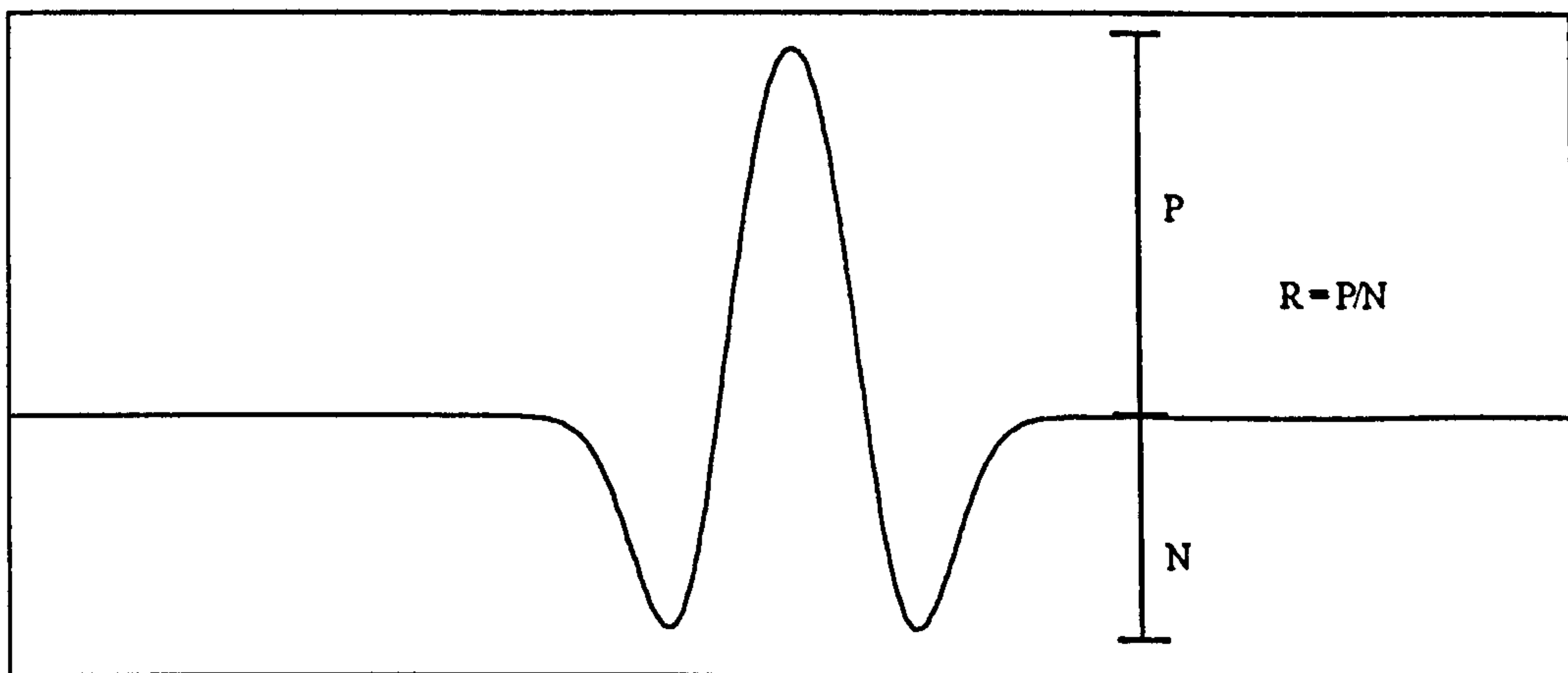


Figure 6.2 - Top and Bottom Lobe Labelling Convention

Figure 6.5 and Figure 6.6 show the fit of both the peak height, P, and the peak ratio, R, for a water line recorded with the 1.52 μm laser. Using a modulation frequency of 35 kHz, the second harmonic spectrum was recorded for a series of different modulation depths ranging between 0.1 and 5. Least squares fitting was then used to determine the scaling constant, C, between the experimental behaviour and that predicted by theory. It is seen that the fit obtained using the peak height is not as good as the fit of the peak ratio, however, the scaling constant predicted by the two methods, 0.00311 cm^{-1}/mA (peak fit) and 0.00313 cm^{-1}/mA (ratio fit), are in good agreement. It is thought that the poorer fit to the peak height, at larger modulation depths, could be caused by a slight Lorentzian component to the recorded lineshape and/or a departure from the linear tuning assumption used in the theoretical model. (see section 5.3.4). Why this should not affect the peak ratio fit, however, is not understood.

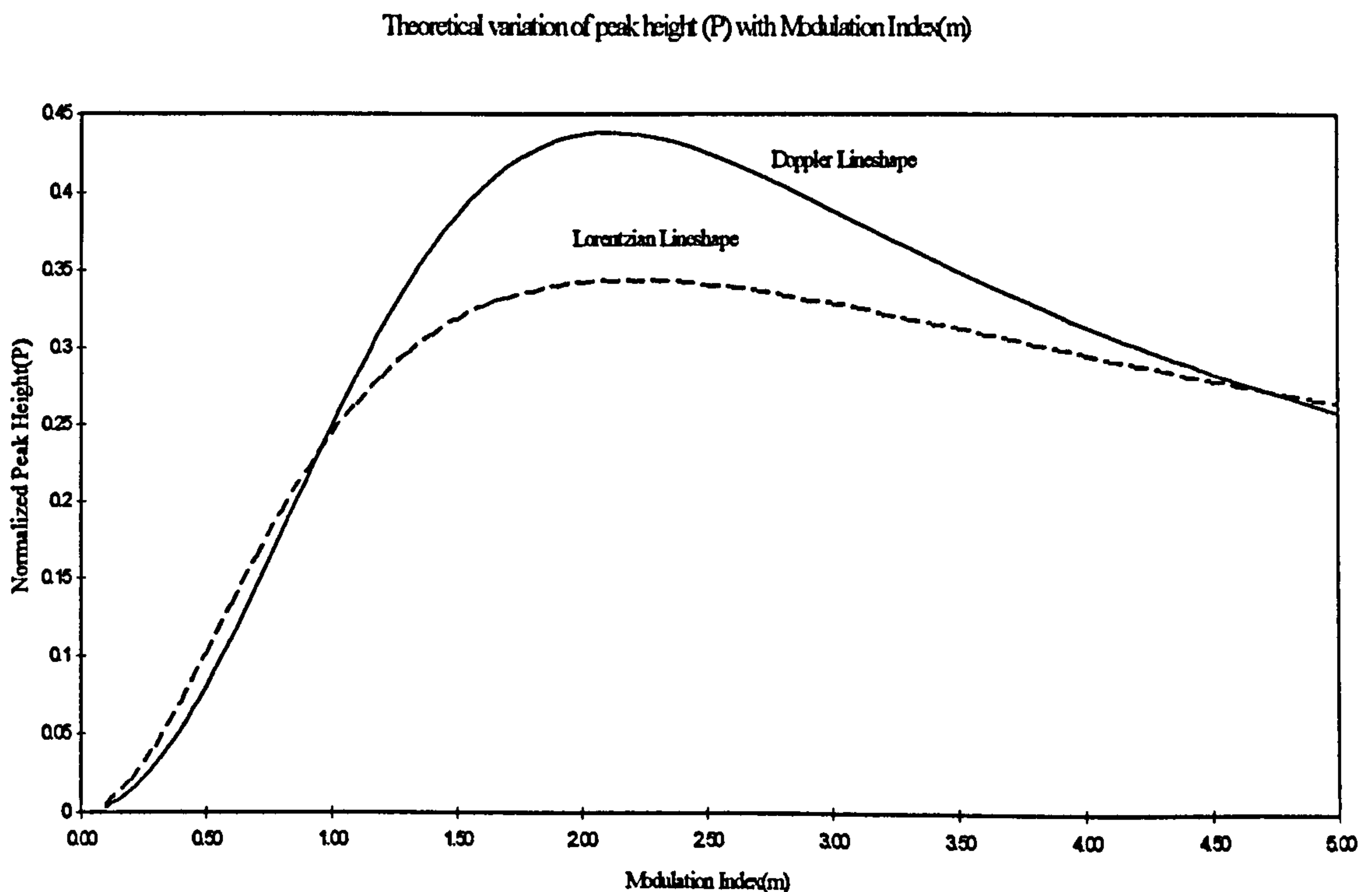


Figure 6.3 - Theoretical variation of P against m

Theoretical Variation of R against Modulation Index(m)

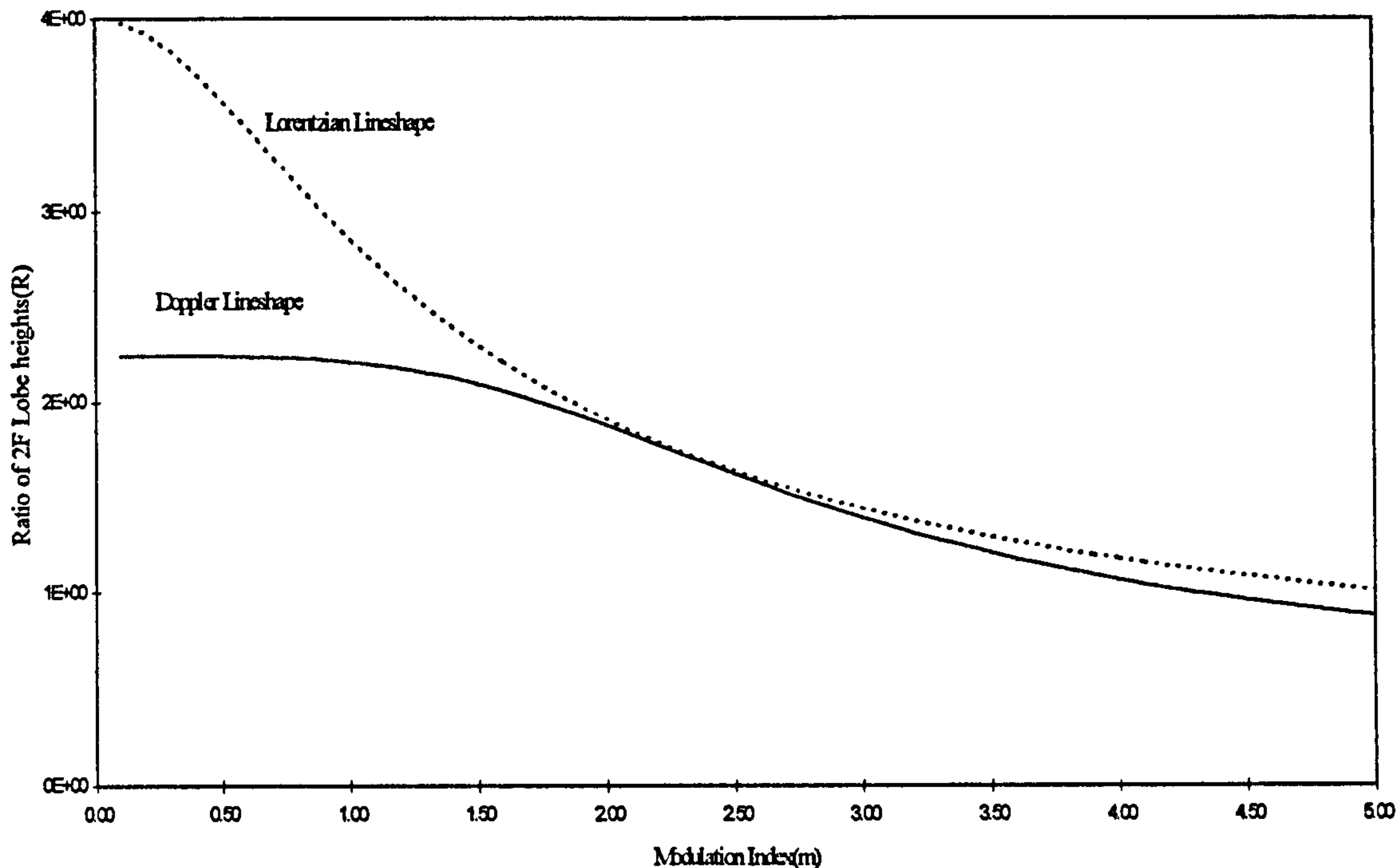


Figure 6.4 - Theoretical variation of R against m

Fit Between Theoretical and Experimental Doppler Peak Heights

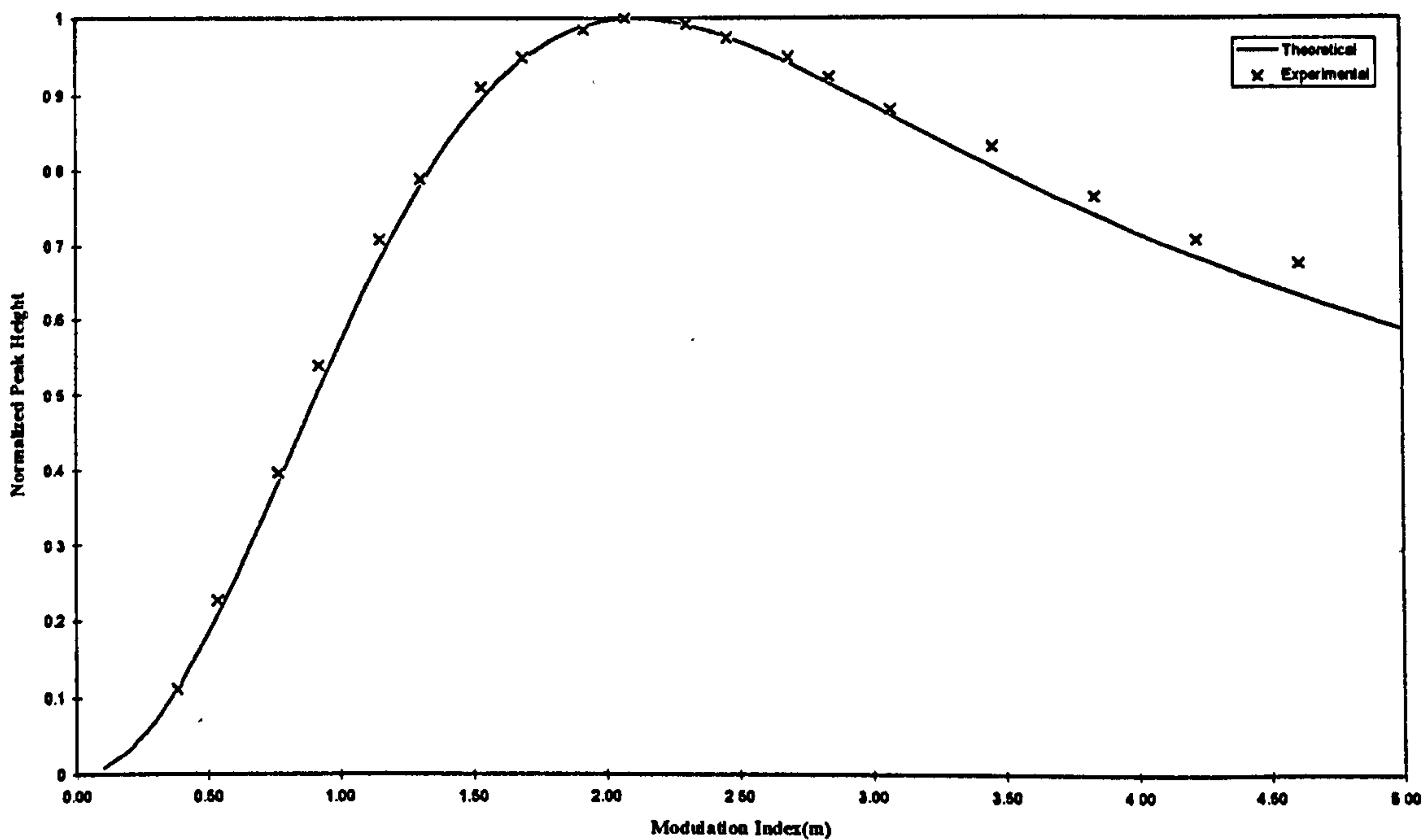


Figure 6.5 - Fit between Theoretical and Experimental Doppler Peak Heights

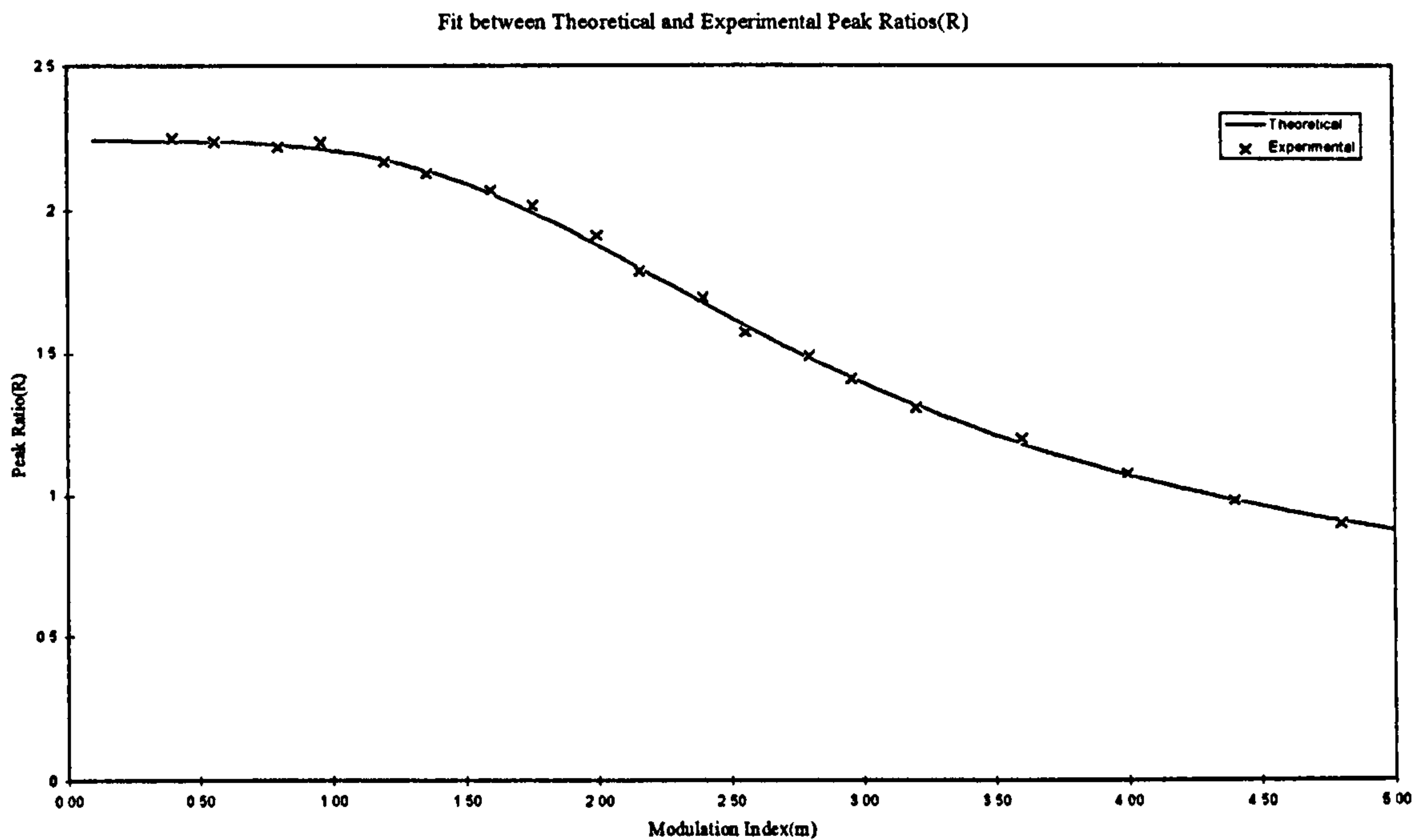


Figure 6.6 - Fit between Theoretical and Experimental Peak Ratios (R)

A more detailed discussion of this fitting procedure is given by Reid and Labrie [59].

Spectrum Registration

When the synthetic and recorded spectra intensities are least squared fitted to determine the gas concentration, non registration will result in errors in the retrieved concentration. Over time the laser will exhibit slight drifts in wavelength resulting in horizontal shifts in the recorded spectra. These spectra must be *lined up* before fitting is done. May and Webster [68] have developed a correlation based procedure to accurately shift any number of spectra in the horizontal axis so that their spectral peaks coincide (to within one data point).

The technique uses the result of autocorrelation to determine the relative direction and magnitude of the horizontal shift between two spectra, which are similar except for signal amplitudes and noise characteristics. The discrete correlation of an N point spectrum is:

$$C(f, f)_j = \sum_{i=1}^N f_j + if_i \quad (6.4)$$

where j represents the lag or horizontal shift between the two spectra. This can be evaluated by using the discrete Fourier transform.

$$C(f, f)_j = F_i F_j^* \quad (6.5)$$

where F_i is the Fourier transform of f_i and F_j^* is the complex conjugate. The autocorrelation is obtained by back transforming this product. This autocorrelation routine has been programmed using MATLAB. The code is listed in Appendix 2.

Figure 6.7 shows two similar spectra of methane (Q(6) transition of $2\nu_3$ band). One is shifted with respect to the other. Figure 6.8 shows the autocorrelation of these two spectra. The peak position gives the magnitude of the shift (in data points) and the sign of its peak value the direction of the shift. Positive is a shift to the right.

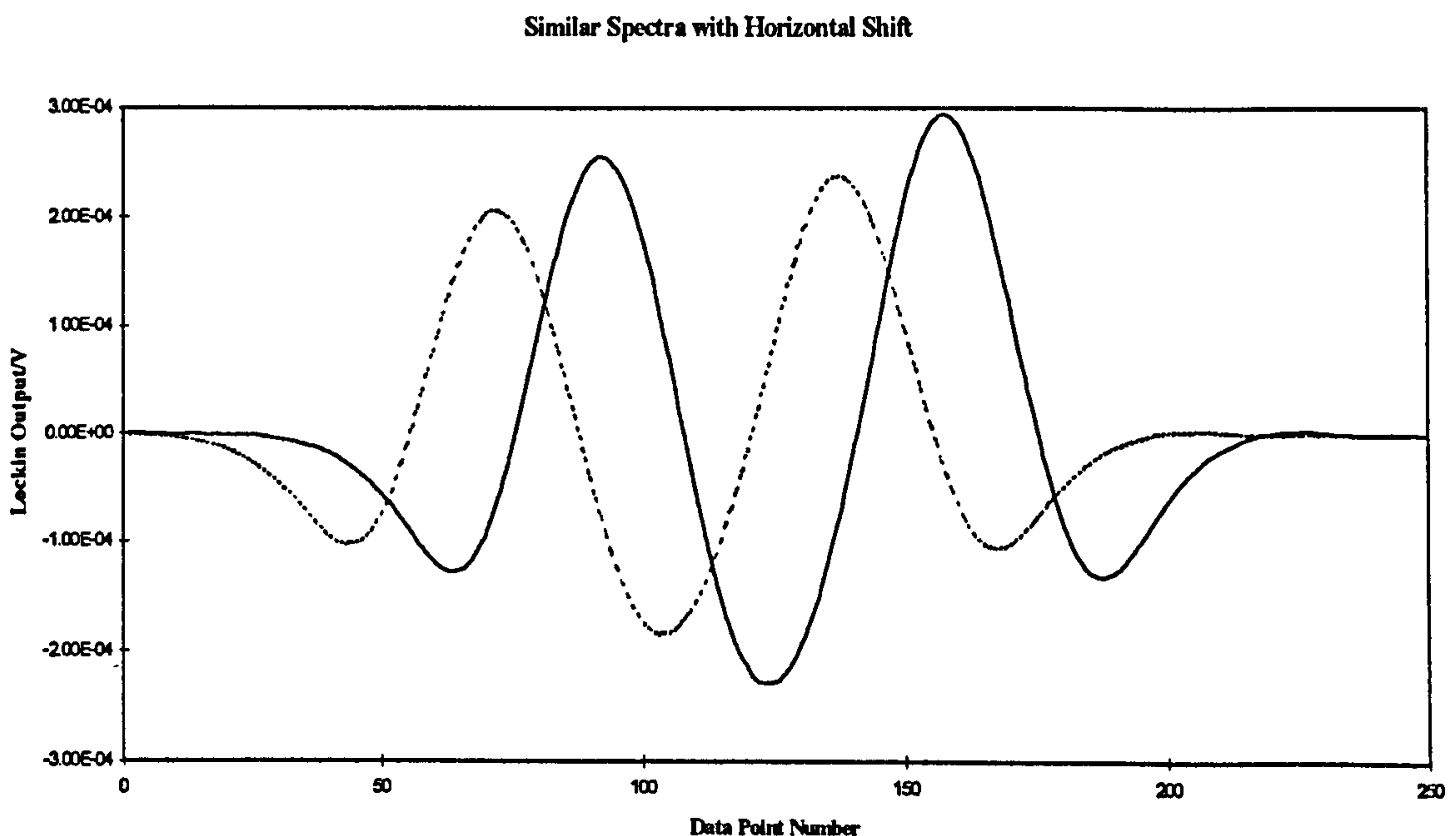


Figure 6.7 - Similar Spectra with Horizontal Shift

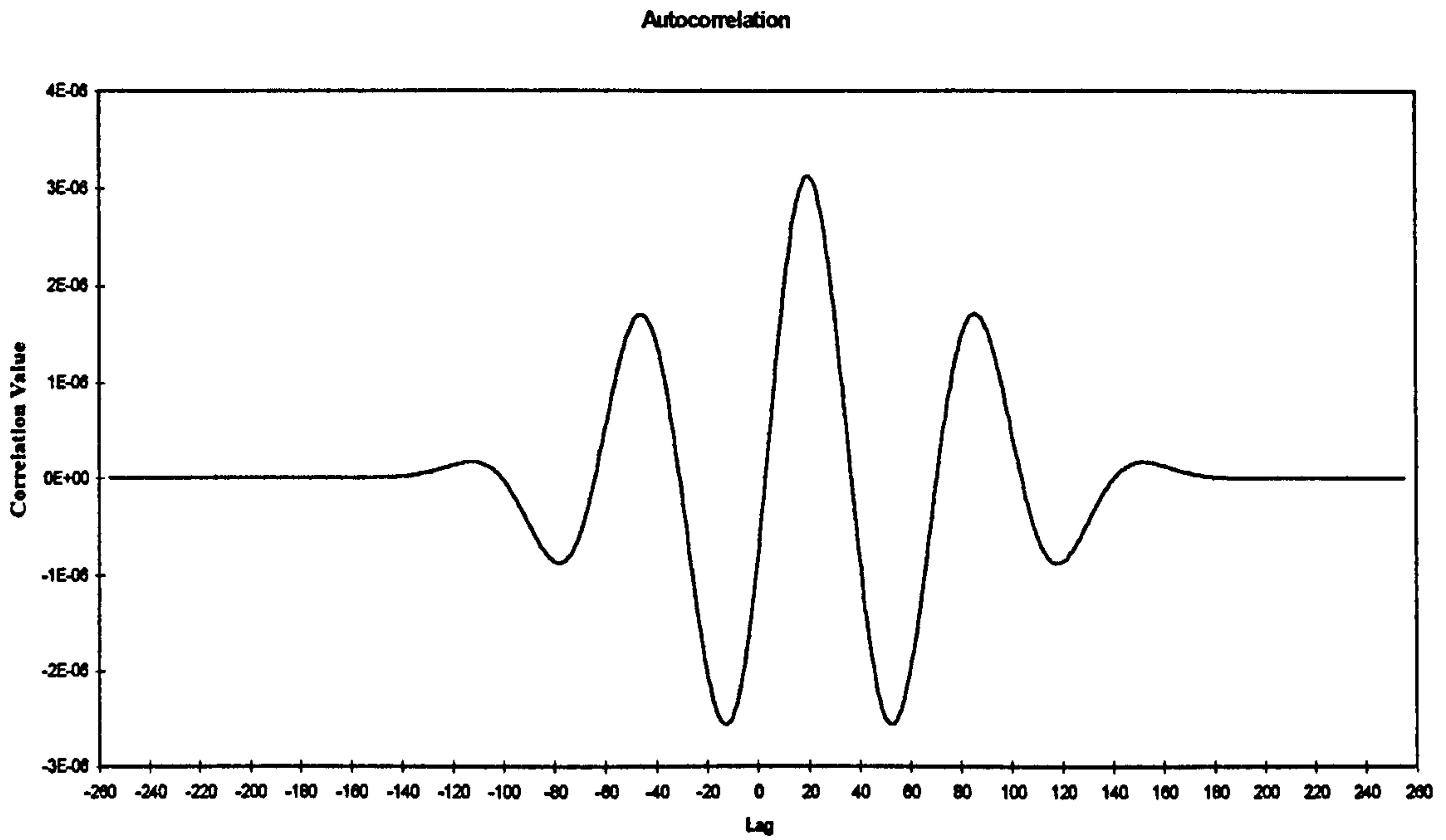


Figure 6.8 - Autocorrelation of Spectra shown in Figure 4.x

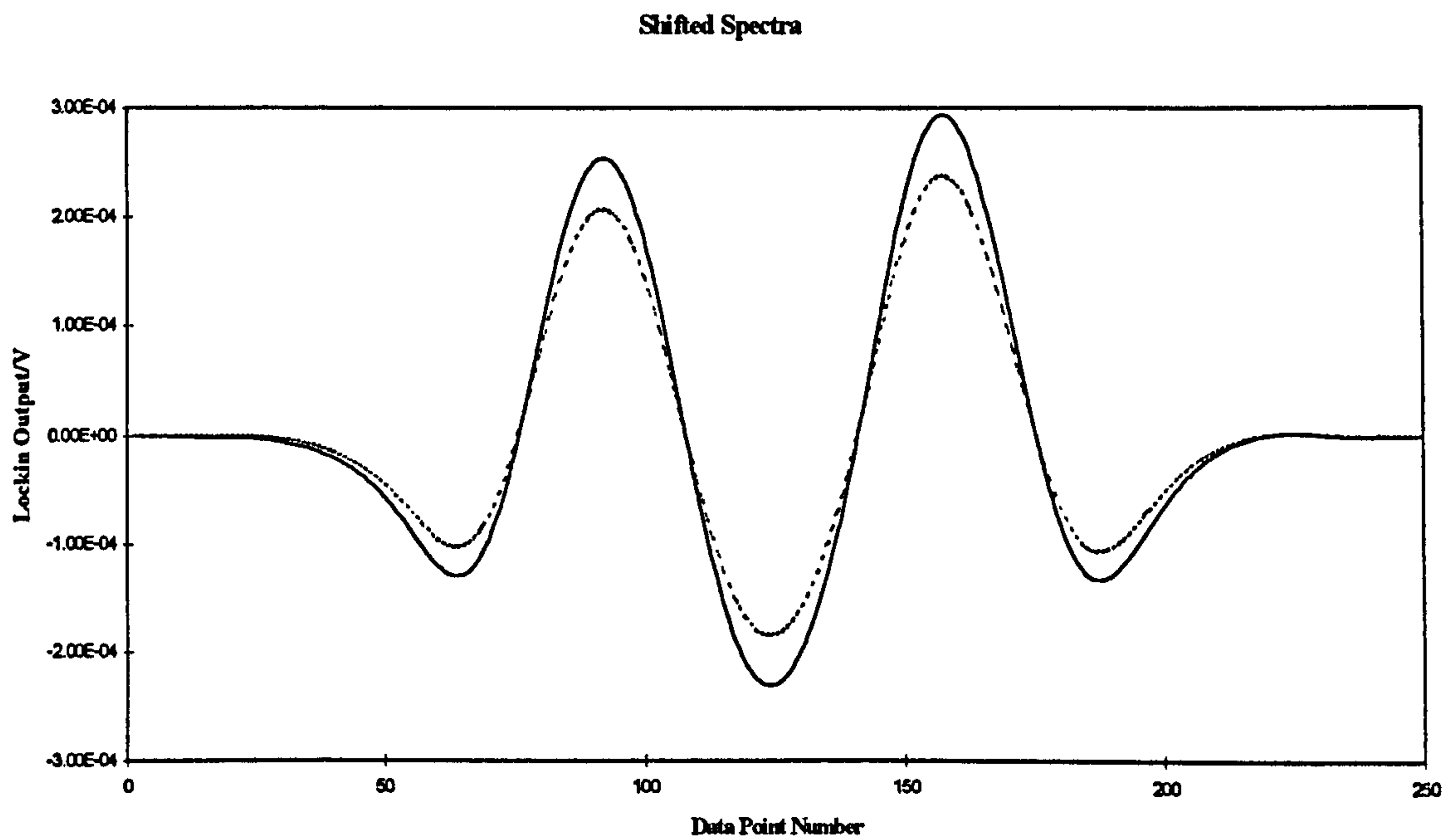


Figure 6.9 - Spectrum Registration

The autocorrelation peak is positive and is positioned at a lag value of 20. This indicates that the spectrum should be shifted 20 points to the right. The shifted spectrum is shown in Figure 6.9.

6.2.3 Pathlength Calibration

The pathlength of the multipass Herriott cell is determined through the use of a primary gas standard from the National Physical Laboratory (100.45 ppm of methane in dry air). Methane was chosen because it is unreactive and is not easily adsorbed onto the walls. The pathlength, l , of the cell is calculated using Beers law:

$$\ln\left(\frac{I}{I_0}\right) = \alpha Pl \quad (6.6)$$

where α is the absorption coefficient of the feature being measured (determined from HITRAN) and P is the sample pressure.

Figure 6.10 shows the pathlength calibration spectrum.

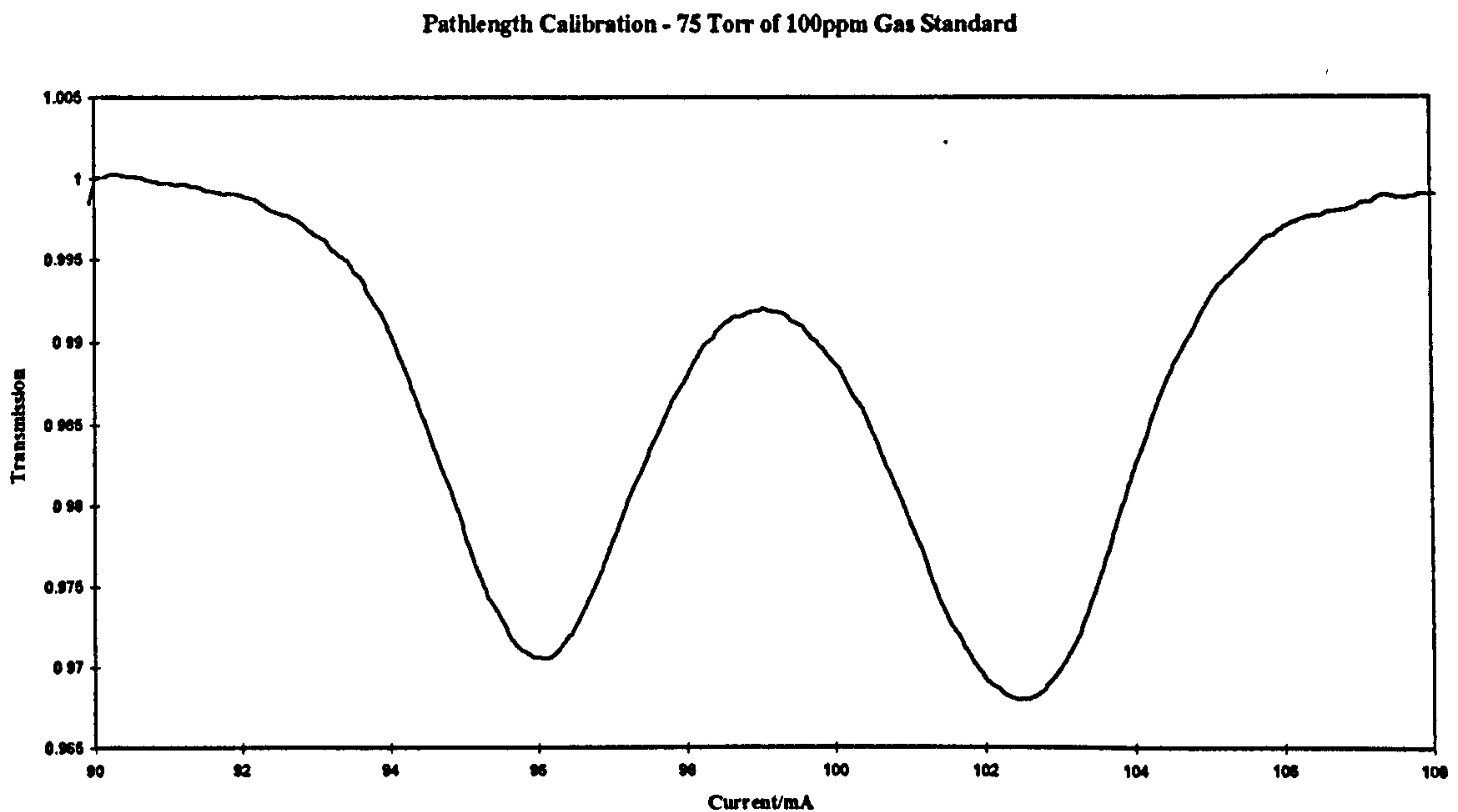


Figure 6.10 - Pathlength Calibration Spectra of Q6 Methane Transition

From this spectra the pathlength has been calculated to be 25.1 (5) m. The figure in brackets represents the standard deviation from a series of twenty repeated measurements. The theoretical pathlength predicted by the computer model for the Herriott Cell was 25.11 m. Thus an excellent agreement was achieved between the measured and theoretical pathlength.

6.2.4 Calibration Results

CH₄

The (6) $2\nu_3$ methane transition was used as the calibration feature. A 100ppm gas standard (NPL Cylinder29) was used at a sample pressure of 100 Torr. This gave a 3.10 % peak absorption with a 25.1 m pathlength. The response number was determined as 6.10.

N₂O

The R(17) transition of the $3\nu_3$ band of N₂O was chosen as the calibration feature. This was a isolated line with no obvious water interference. A 1% mixture of N₂O in Dry Air was used at 60 Torr pressure. This gave a 2.65 % absorption with a pathlength of 25.10 m. The response number was determined to be 0.0106.

6.3 Lab Measurements and Performance Assessment

6.3.1 Introduction

This section outlines work carried out in the laboratory aimed at measuring N₂O and CH₄ in their natural abundance. The spectra recorded have allowed detection limits for each gas to be determined and the instrument performance to be assessed. Noise levels equivalent to a minimum detectable absorption of 2×10^{-7} and 1×10^{-7} in a 1 Hz bandwidth were achieved with the methane and nitrous oxide lasers respectively. Four point averaging and piezo mirror dithering fringe reduction was employed during these measurements. This gives detection limits of 560 pptV for methane and 22 ppbV for nitrous oxide in a 25m pathlength. Additional signal averaging and fringe removal techniques were shown to be able to improve detection limits by a factor of two .

6.3.2 Methane

Before any measurements of methane could be made a survey spectrum of the wavelength region accessible to the Anritsu diode (section 3.2) was carried out to determine suitable lines for measurement.

Spectrum Survey

Figure 6.11 shows the HITRAN simulation of air at a sample pressure of 100 Torr in the region accessible to the diode. The diode covers the entire Q branch of the $2\nu_3$ band. It is seen that there are a few strong water lines in this region. Interference from these water lines rule out using the Q(1), Q(2), Q(4), Q(5), Q(7) and Q(10) transitions. Of the remaining features the best is the Q (6) multiplet. It is the strongest feature in this region and is free from interference. The multiplet consists of six individual transitions with a combined strength of 3.4×10^{-21} cm molecule⁻¹ at 296 K [23]. Figure 6.12 shows each of these individual transitions. The combined lineshape of the transitions is also shown. Calculations have assumed a 25.1 m pathlength and a 100 Torr sample of air with methane present at atmospheric abundance (~ 1.76 ppmV). Under these conditions the peak absorption of the Q(6) multiplet is 6×10^{-4} .

Hitran Simulation Of 100 Torr Air

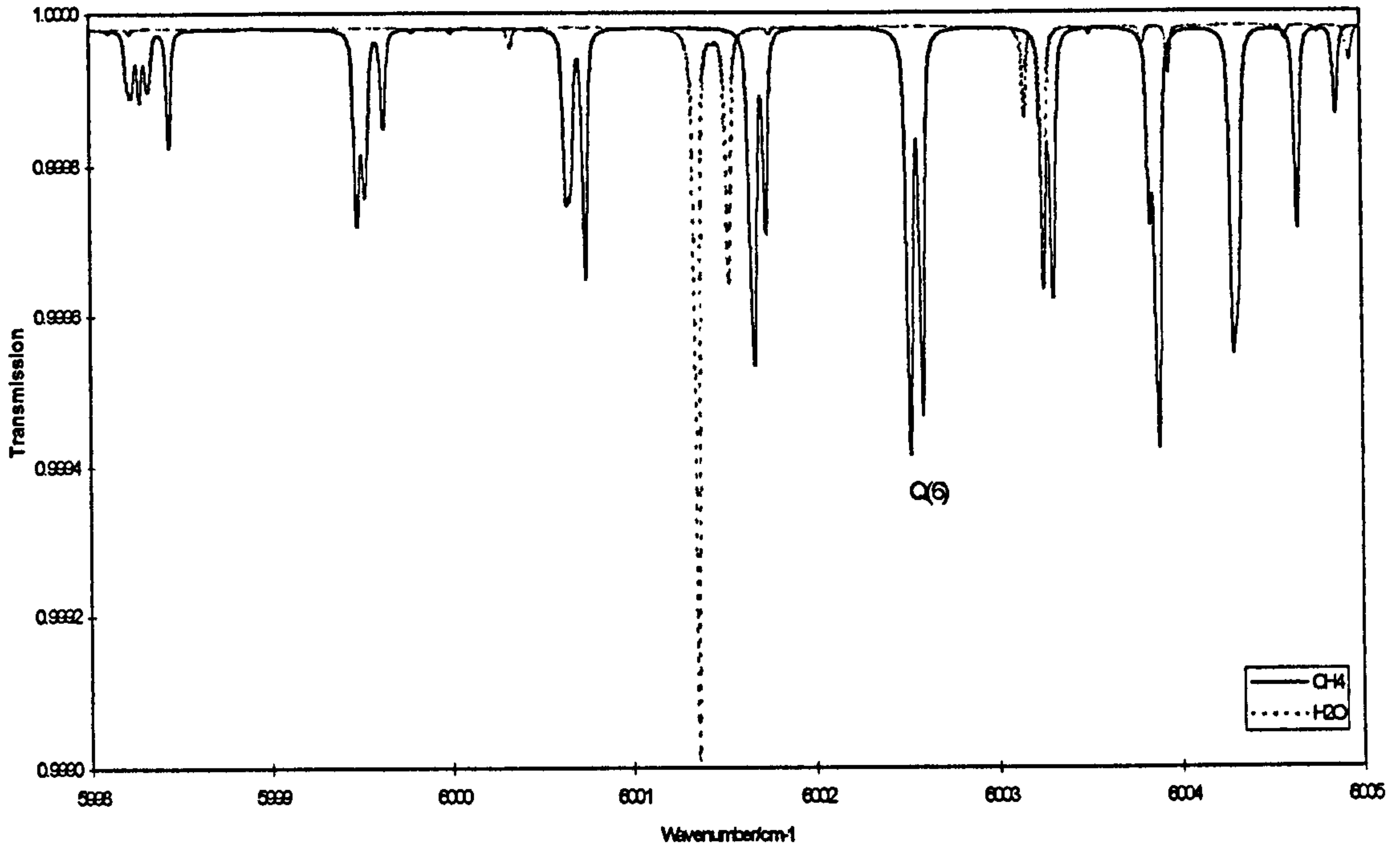


Figure 6.11 - Hitran Simulation of Air (P = 100 Torr, l = 25.1 m)

Q6 Transitions

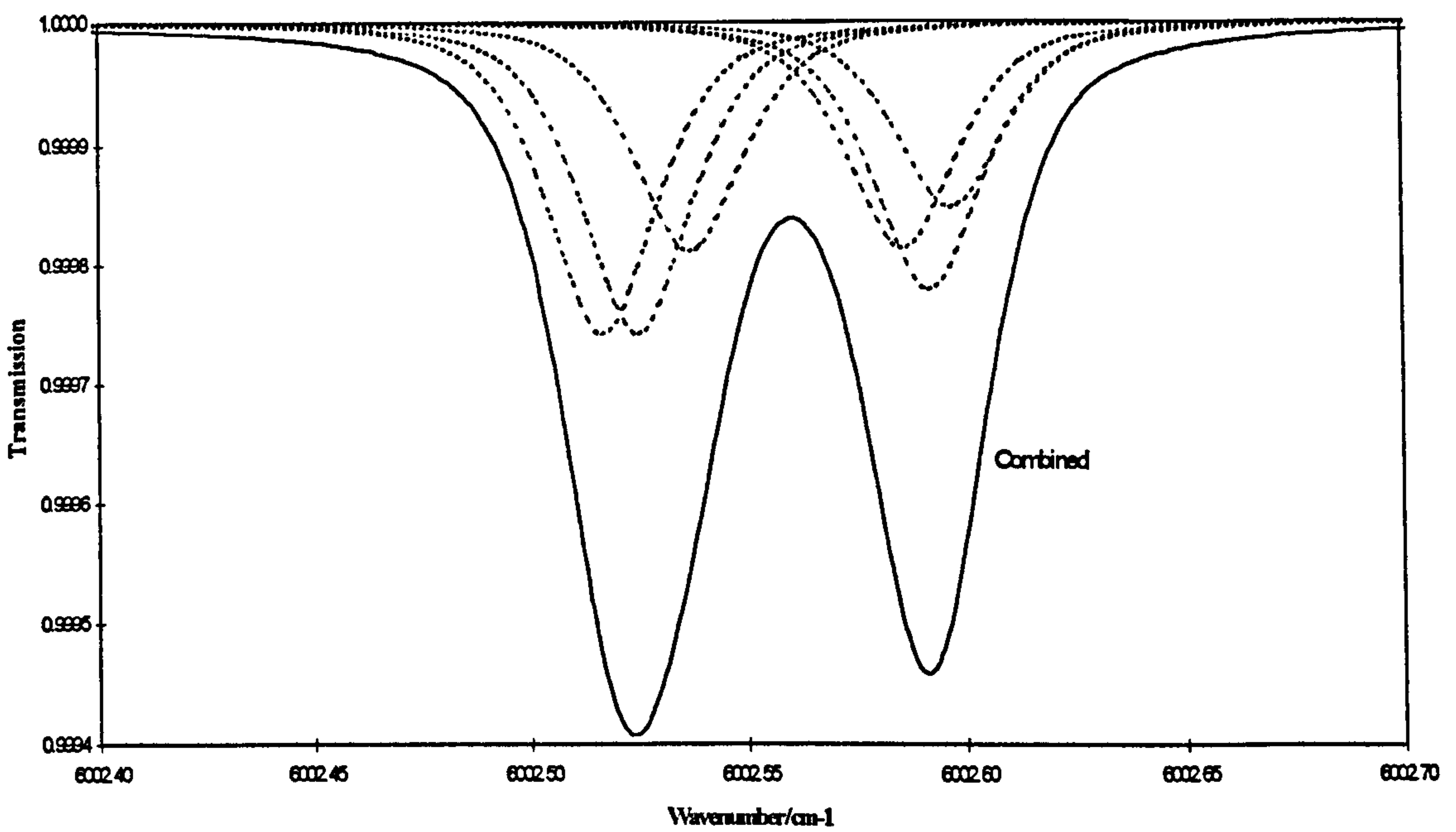


Figure 6.12 - Transmission Spectra of Q(6) Multiplet (100 Torr, 25.1m pathlength)

Methane Spectra Recorded in Lab

Figure 6.13 shows an example of a typical spectrum of methane recorded in the laboratory. Air from outside was sampled via the inlet tube. A sample pressure of 100 Torr was chosen for measurement. At this pressure the Lorentz and Doppler components of the line are approximately equal ($\gamma_{D_{\text{HWHM}}} = 0.00924\text{cm}^{-1}$, $\gamma_{L_{\text{HWHM}}} = 0.00845\text{cm}^{-1}$). This provides a good compromise between higher pressure measurements where the absorptions are greater because there is more gas in the cell and low pressure measurements where interference from nearby lines is minimised.

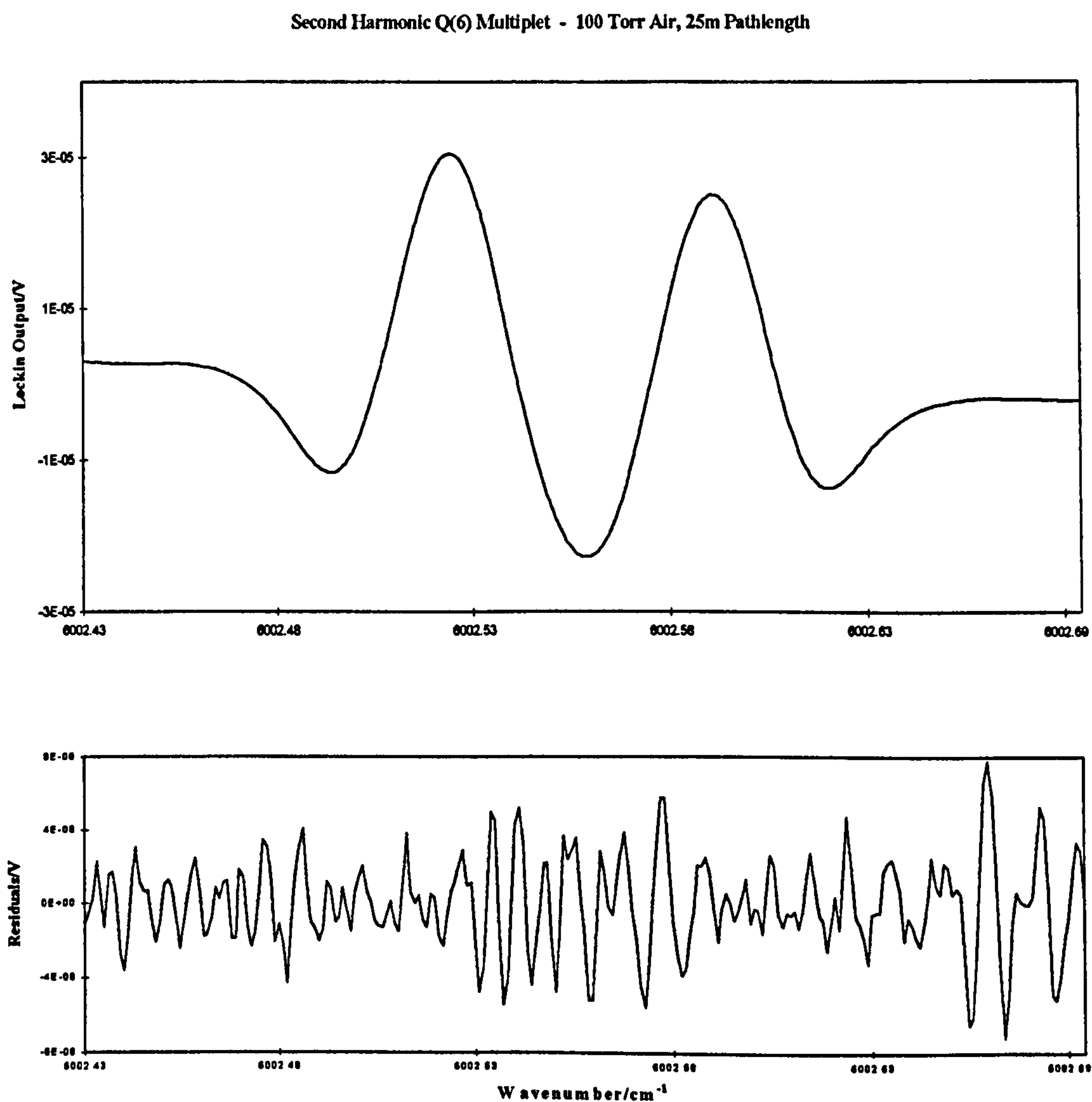


Figure 6.13 - Lab Spectra of Q (6) Multiplet and Residuals

The laser current was tuned between 85 and 110 mA, corresponding to laser wavelengths of 1665.7 cm^{-1} and 1665.43 cm^{-1} respectively. The temperature controller was set to $7.88 \text{ k}\Omega$ ($31.2 \text{ }^\circ\text{C}$). 250 data points were recorded across this region with four points being averaged at each ramp step. The lockin time constant was set to 300 ms. This gave an effective detection bandwidth of $\sim 0.8 \text{ Hz}$ (see Section 5.5 for more details).

Figure 6.13 also shows the noise residuals. The residuals were obtained by Fourier transforming the data, removing the noise components and back transforming (see section 5.4.4). The original data set was then subtracted from the Fourier transformed data. The rms residual amplitude is $\sim 2 \times 10^{-8} \text{ V}$. This noise level corresponds to a minimum detectable absorption of 2×10^{-7} in a 1Hz bandwidth. The residuals appear to have a repeatable pattern, indicating that they were caused by optical fringing. The FSR of this fringing corresponds to a pathlength difference of $\sim 1 \text{ m}$. The source of this fringing was not identified, however, the fringe period is sufficiently different to the period of the absorption feature to allow it to be removed using fringe reduction techniques. This technique of determining noise levels will not show up fringing with a FSR close to the width of the absorption feature as such fringing can not be removed by Fourier Transform techniques. To determine if such fringing did exist, the spectrum was recorded again but the Herriott cell was evacuated. This would remove the large absorption feature due to methane and allow closer inspection of the noise on the baseline. Figure 6.14 shows the spectrum obtained with the cell evacuated.

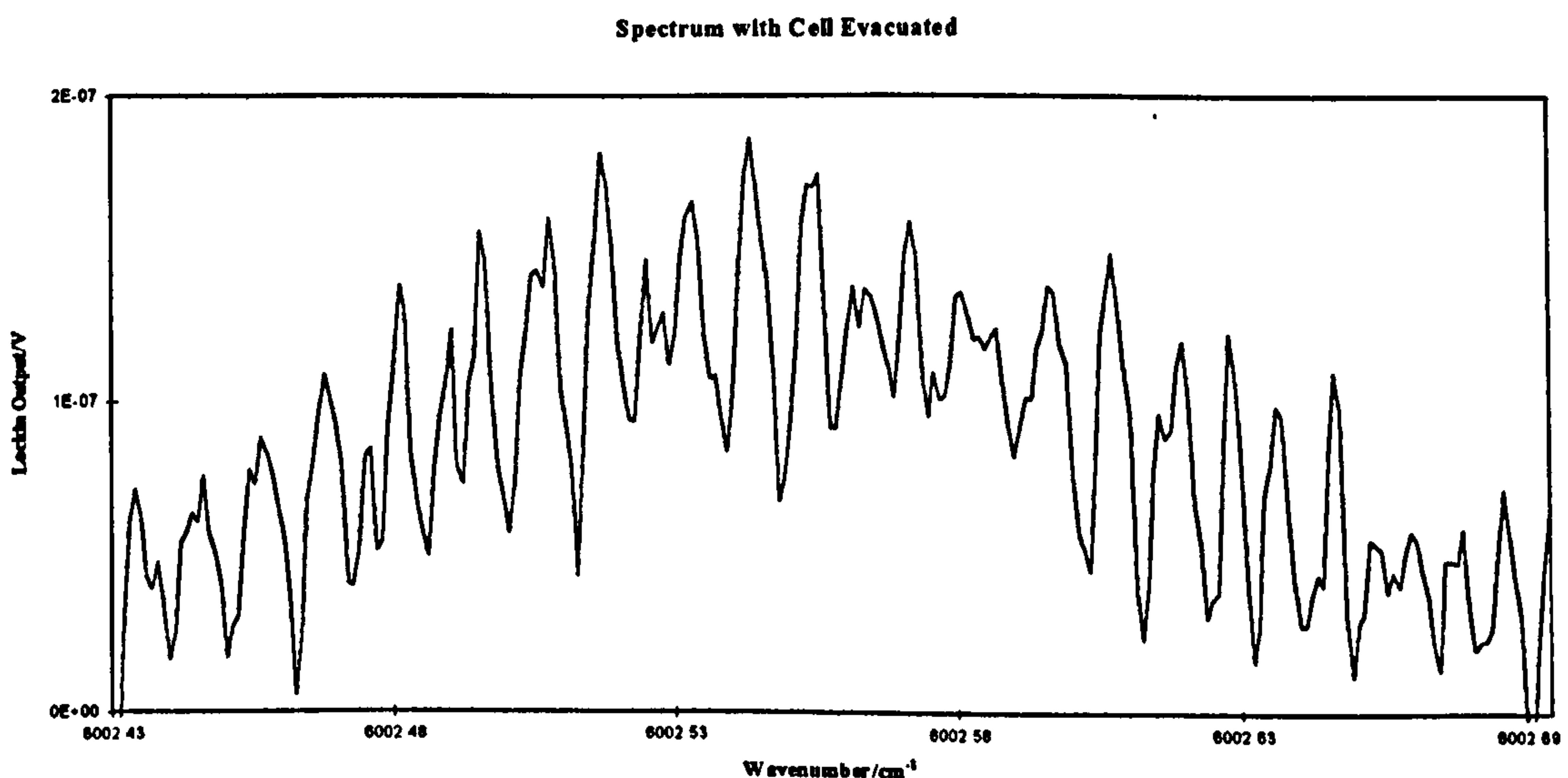


Figure 6.14 - Spectrum with the Herriott cell Evacuated

The rms noise on this spectrum is very similar to that determined using Fourier transform techniques, however, it is seen that the base line is not flat. The broad feature on the baseline is in fact caused by methane in the optical path outside the cell (0.7m). HITRAN was used to model the absorption of methane at one atmosphere and 0.7 m pathlength. The feature is equivalent to an absorption of 1.5×10^{-6} , however, it is seen that it is a factor of 400 smaller than the spectrum of methane shown in **Figure 6.14**. This is an order of magnitude greater than what would be predicted by the size of the absorptions for the two features. The difference can be accounted for if the change in the effective modulation depth is considered. The atmospheric feature is considerably under modulated.

Concentration Retrieval

The calibration technique for this instrumentation is described extensively in Section 6.2. This technique has been used to determine the concentration of methane in the atmosphere. The technique determines the transfer function between the intensity of the second harmonic feature and the size of the direct absorption associated with that feature. A gas mixing ratio can then be extracted by least square fitting the $2f$ signal amplitude to a synthetic second harmonic spectrum calculated for the same conditions (pressure, temperature, modulation amplitude) as the atmospheric measurement. **Figure 6.15** shows a recorded and synthetically generated spectrum that have been least square fitted. The synthetic second harmonic spectrum was generated assuming a concentration of 1.7 ppmV. The least square fitting determined the scaling constant between these two spectra as being 1.060. This indicates that the measured methane concentration was 1.80 ppm. This is a good agreement with the accepted ambient concentration of methane which is 1.76ppm [3].

Measurements were then taken at 3 minute intervals for approximately an hour, to see how the methane concentration varied. **Figure 6.16** shows the variation of the retrieved concentration with time. The concentration was found to fluctuate by a surprisingly large amount (between 1.8 ppm to 2 ppm). To ensure that these changes were real, a repeatability test was carried out. Over a similar time period, several spectra of a 8.1 ppmV methane gas standard (National Physical Laboratory) were recorded. **Figure 6.17** shows the variation in the retrieved concentration of the gas

standard. It is seen that very little change in concentration was observed. The mean concentration was determined to be 8.09 ppm with a standard deviation (1σ) less than 0.15 % of this value. It was concluded that the variation in ambient methane concentrations shown in Figure 6.16 were real.

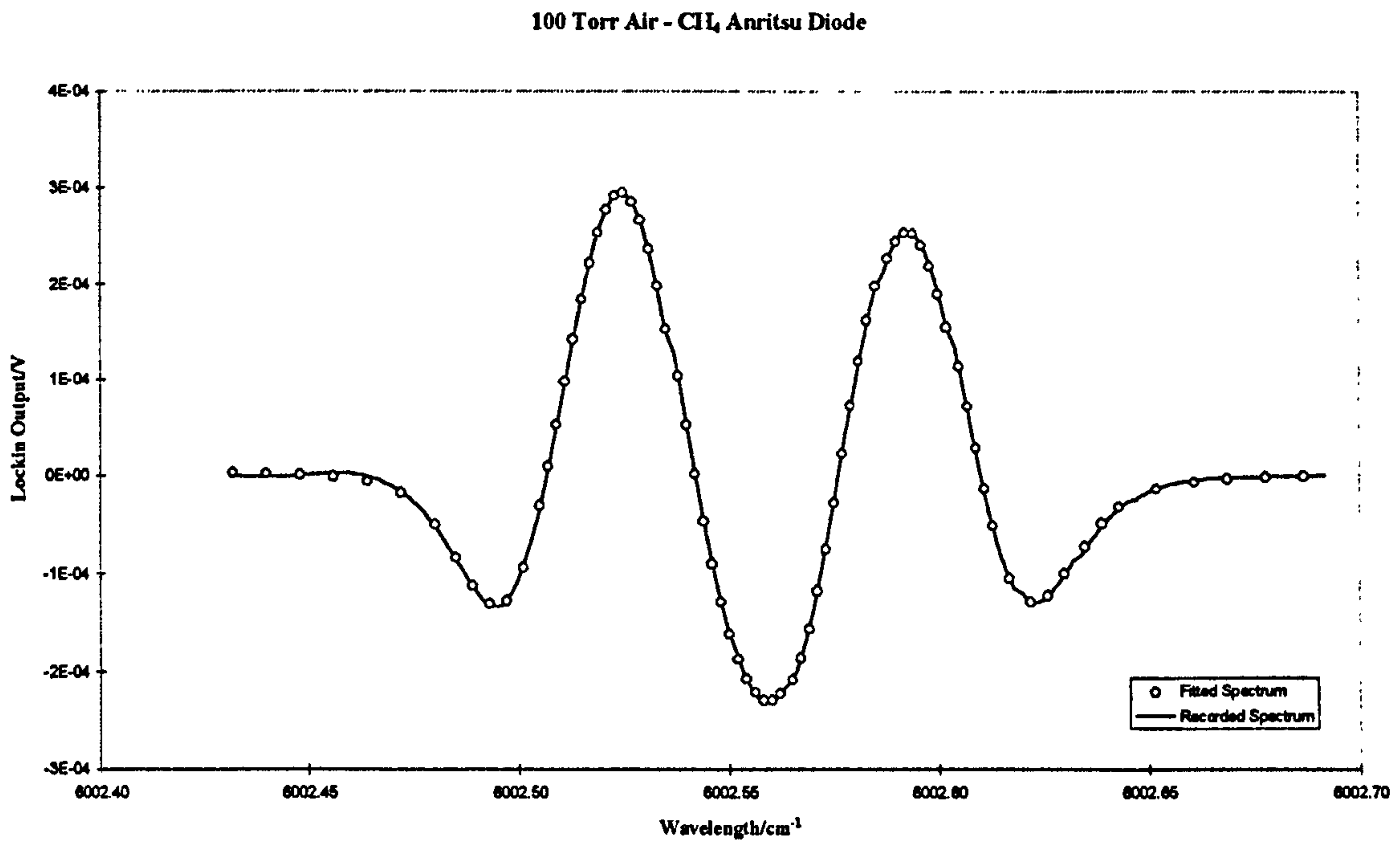


Figure 6.15 - Least Square Fitted Recorded and Synthetic Spectrum

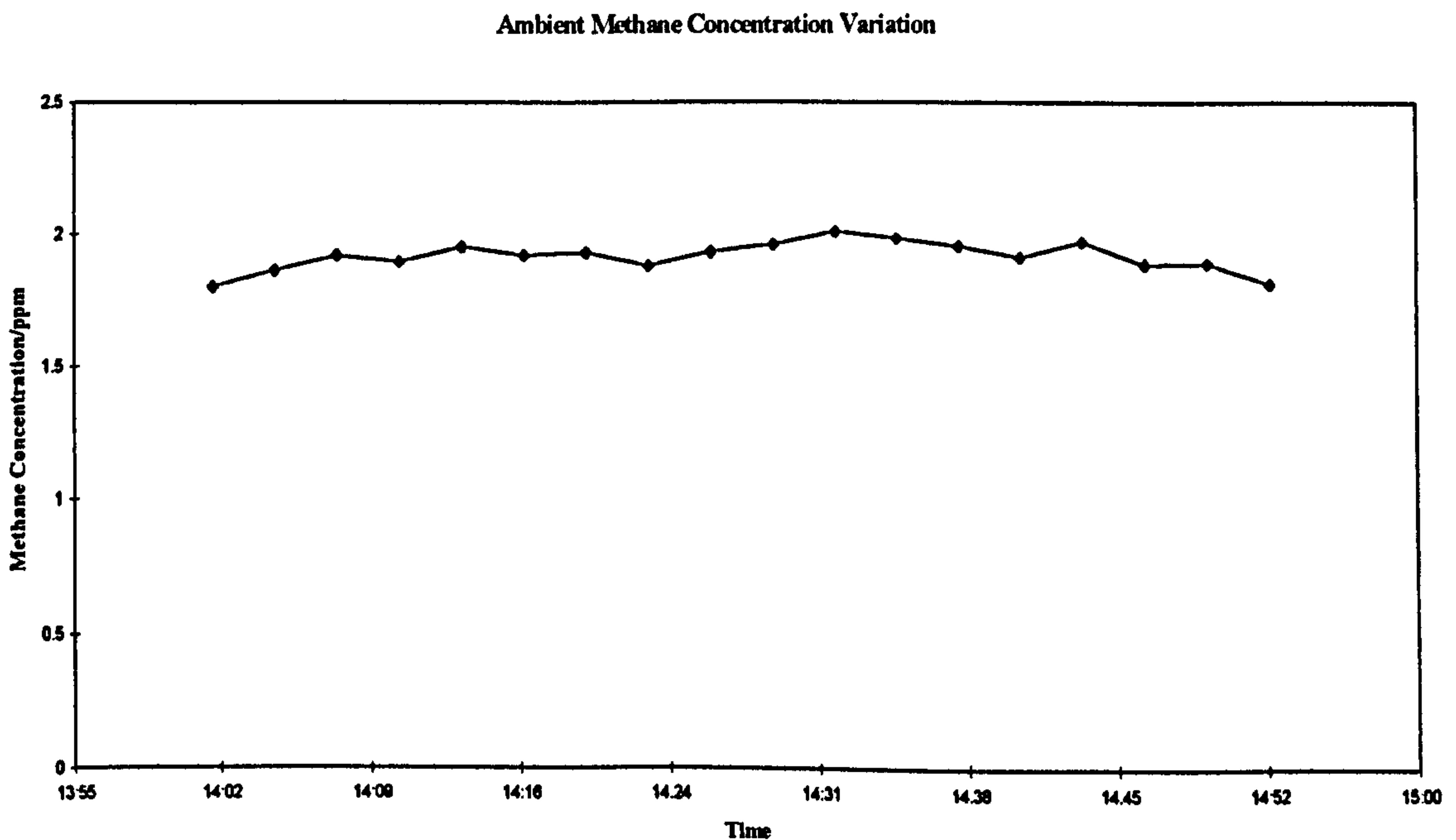


Figure 6.16 - Variation in Ambient Methane Concentration

Repeatability Measurement

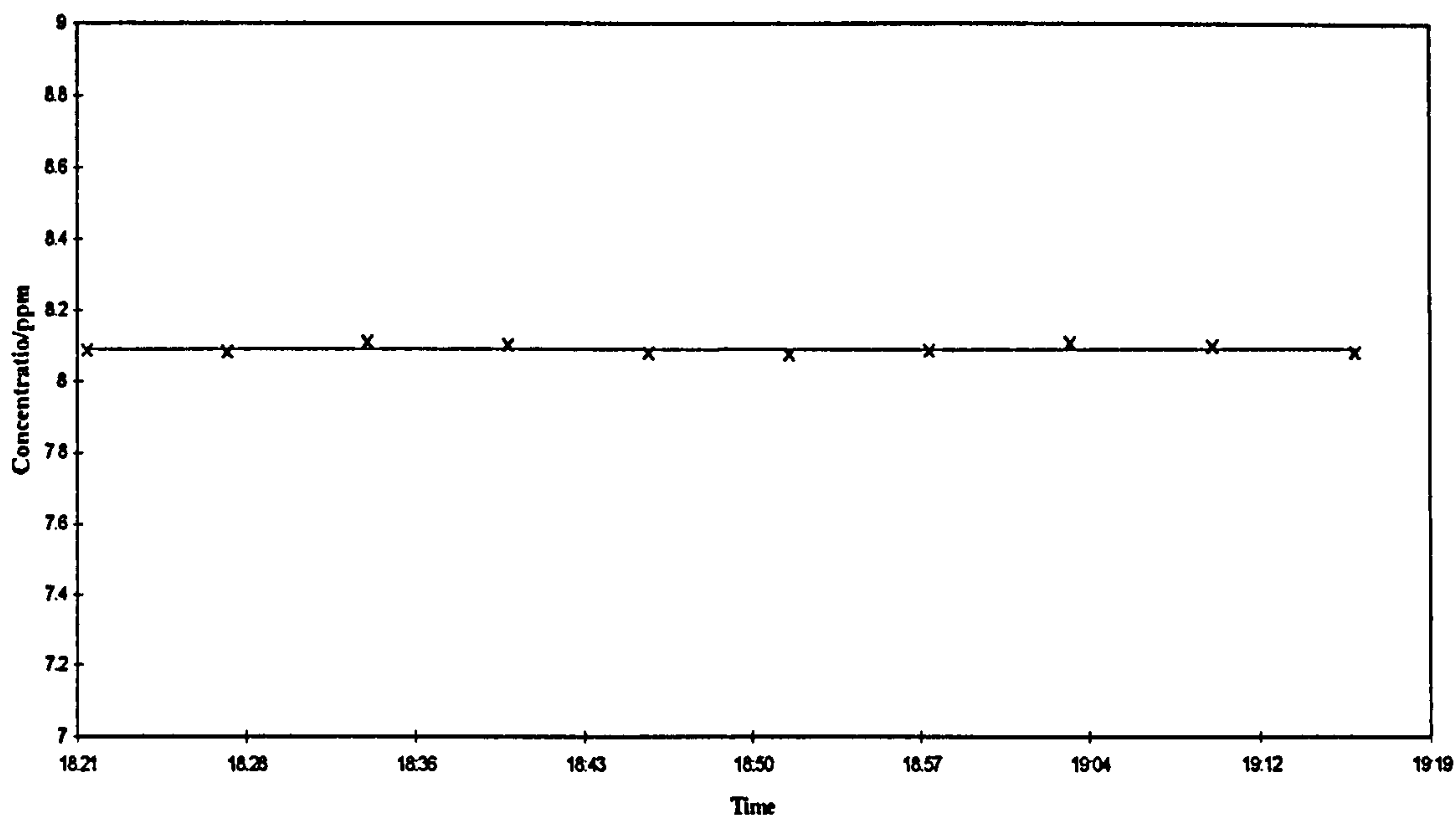


Figure 6.17 - Repeatability Test for the retrieved concentration of a 8.1 ppmV gas standard

The elevated levels of ambient methane recorded and the variability in the retrieved concentration indicated that there was a source of methane near to where the measurements were taken. It is thought that the local park (Bushy Park, Teddington), which has a large population of grazing deer, was the most likely candidate.

Species Interference

Figure 6.18 shows the residuals between the experimentally recorded spectrum and synthetic spectrum used to determine the ambient methane concentration (see Figure 6.15). It is clear that there is an absorption feature at $\sim 6002.45 \text{ cm}^{-1}$. A closer inspection of HITRAN has allowed this feature to be identified. It is the R (4) transition of the 10002 - 40015 band of CO_2 . It has a linestrength of 7.5×10^{-27} and is found at 6002.447 cm^{-1} . For a 100 Torr air sample and 25 m pathlength it has a peak absorption of 5×10^{-7} [23].

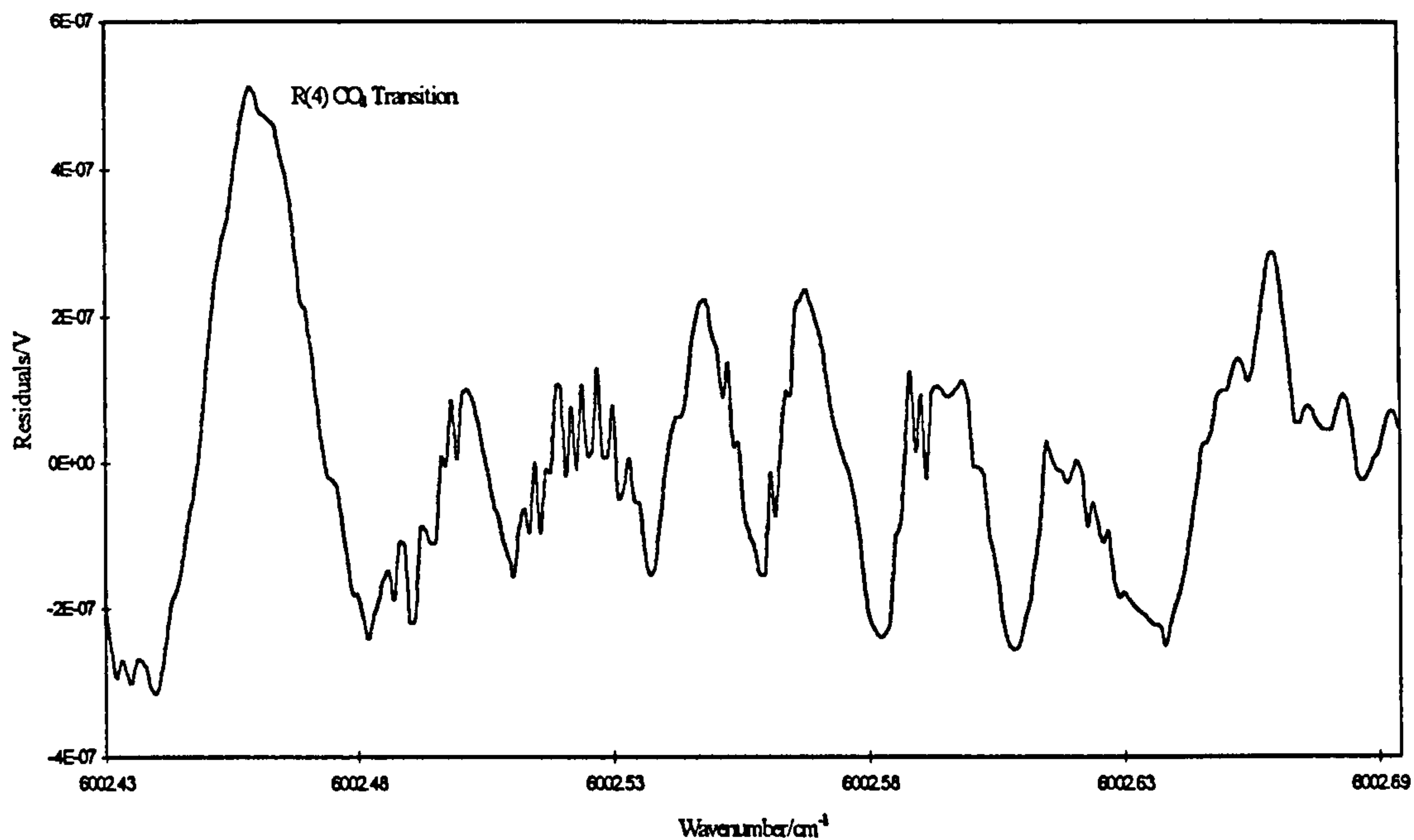


Figure 6.18 - Residuals between Experimental Spectrum and Synthetic Spectrum

General Comments and Conclusions

The day to day sensitivity that could be achieved typically varied between 2×10^{-7} and 1×10^{-6} . This sensitivity was determined by looking at the residuals between the original data and the data with the noise removed using Fourier transform techniques. The sensitivity was confirmed by the identification of the R(4) CO₂ transition which has a 5×10^{-7} absorption [23].

The variation in the day to day sensitivity is thought to be due to variations in the effectiveness of the piezo mirror dithering fringe reduction technique. This is thought to be caused by shifts in the alignment of the optics. Once sensitivity started to deteriorate, it continued to do so. The only way to re-establish the original sensitivity levels was to realign the entire optical system. Improved mounts for the optical components would reduce this problem.

The sensitivities achieved here (without averaging) are within a factor of three to those predicted by theory (see Section 5.3.5). Signal averaging and Fourier transform fringe removal techniques could improve sensitivity further, allowing an absorption of 1×10^{-7}

to be detected. Low frequency optical fringing prevented any further improvement in sensitivity.

The instrument sensitivity allows detection of methane at 560 ppt mixing ratios. This assumes a 25 m pathlength and a 1Hz bandwidth. This is comparable with the best sensitivities achieved using other techniques. The ALIAS instrument [11] measured CH₄ concentrations at high altitudes on the ER-2 aircraft using mid-infrared lasers. Detection sensitivities equivalent to 50 ppt were reported. These figures, however, represented the sensitivity achieved with a 3 second integration time and a 100m pathlength. Aerodyne Research Inc. have developed a mid-infrared TDLAS for ground based measurements of methane [48] . Detection sensitivities approaching 2ppb were achieved using a 32 meter pathlength. A near-infrared open path TDLAS developed by Uehara [69] achieved detection limits of ~ 2ppb for a 25 metre pathlength.

It has been shown that near-infrared technology can provide comparable sensitivities to mid-infrared TDLAS. This is despite linstrengths that are two orders of magnitude weaker.

6.3.3 Nitrous Oxide

Spectrum Survey

Nitrous Oxide is present in the atmosphere at concentrations of approximately 330 ppbV [3]. The transitions of the $3\nu_3$ band accessible to the Thomson laser diode are not listed in the HITRAN database. Line positions, linestrengths and pressure broadening coefficients measured at Strathclyde University have therefore been used to generate a synthetic spectrum of nitrous oxide. A HITRAN simulation of air in the same region has been superimposed to allow suitable lines for detection to be chosen. **Figure 6.19** shows the simulated spectra, for a 60 Torr air sample and 25.1 m pathlength.

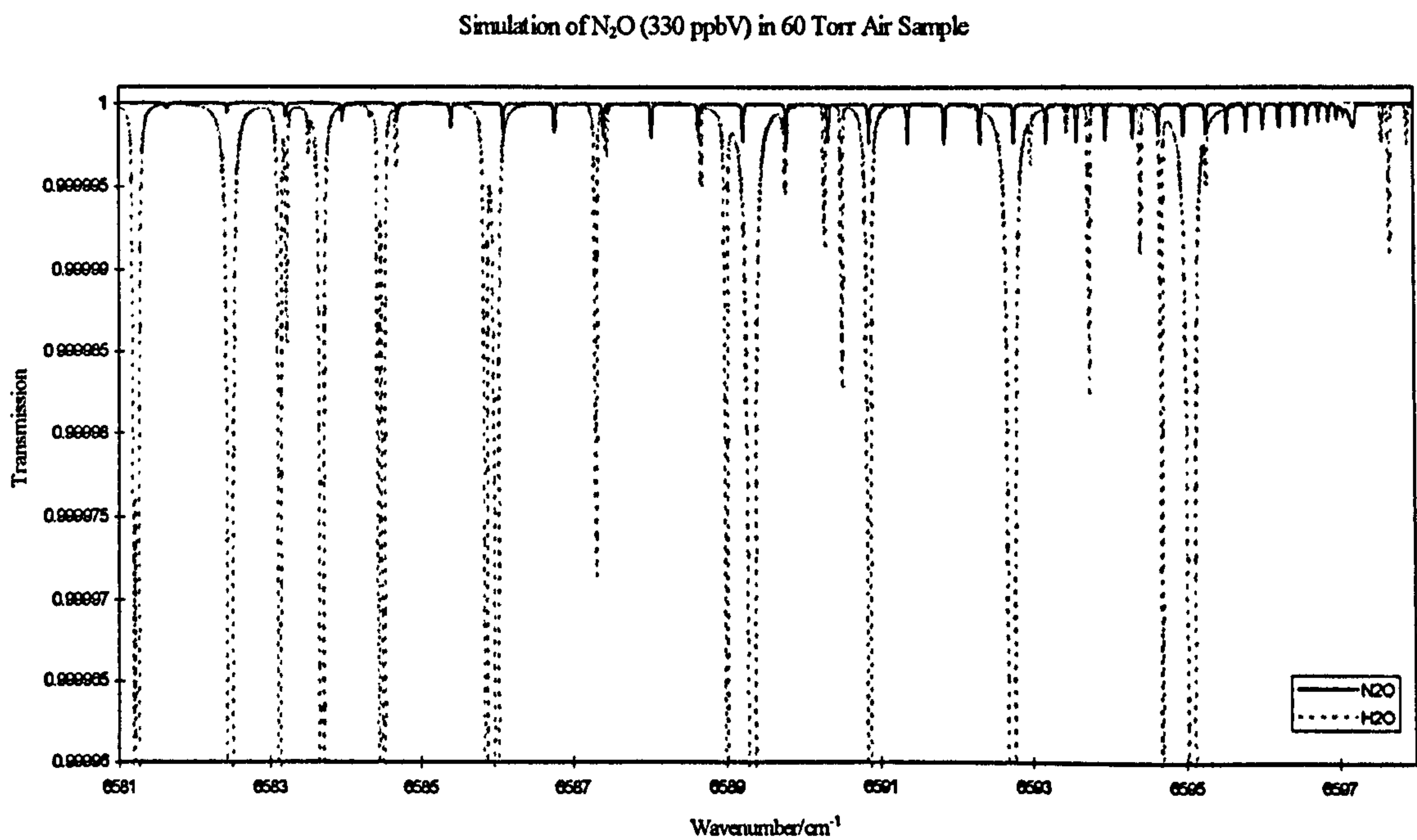


Figure 6.19 - Simulated Spectrum of N₂O at 330 ppbV in at 60 Torr Air Sample

It is clear from the simulated spectrum that there are many strong water lines in this region. The strongest of these lines has a peak absorption of $\sim 2\%$. The water lines are found to interfere with many of the N₂O transitions preventing them from being used. The strongest nitrous oxide lines are found at $\sim 6592\text{ cm}^{-1}$. Fortunately a few N₂O transitions in this region do not appear to have any significant interference. **Figure 6.20** shows this region in more detail.

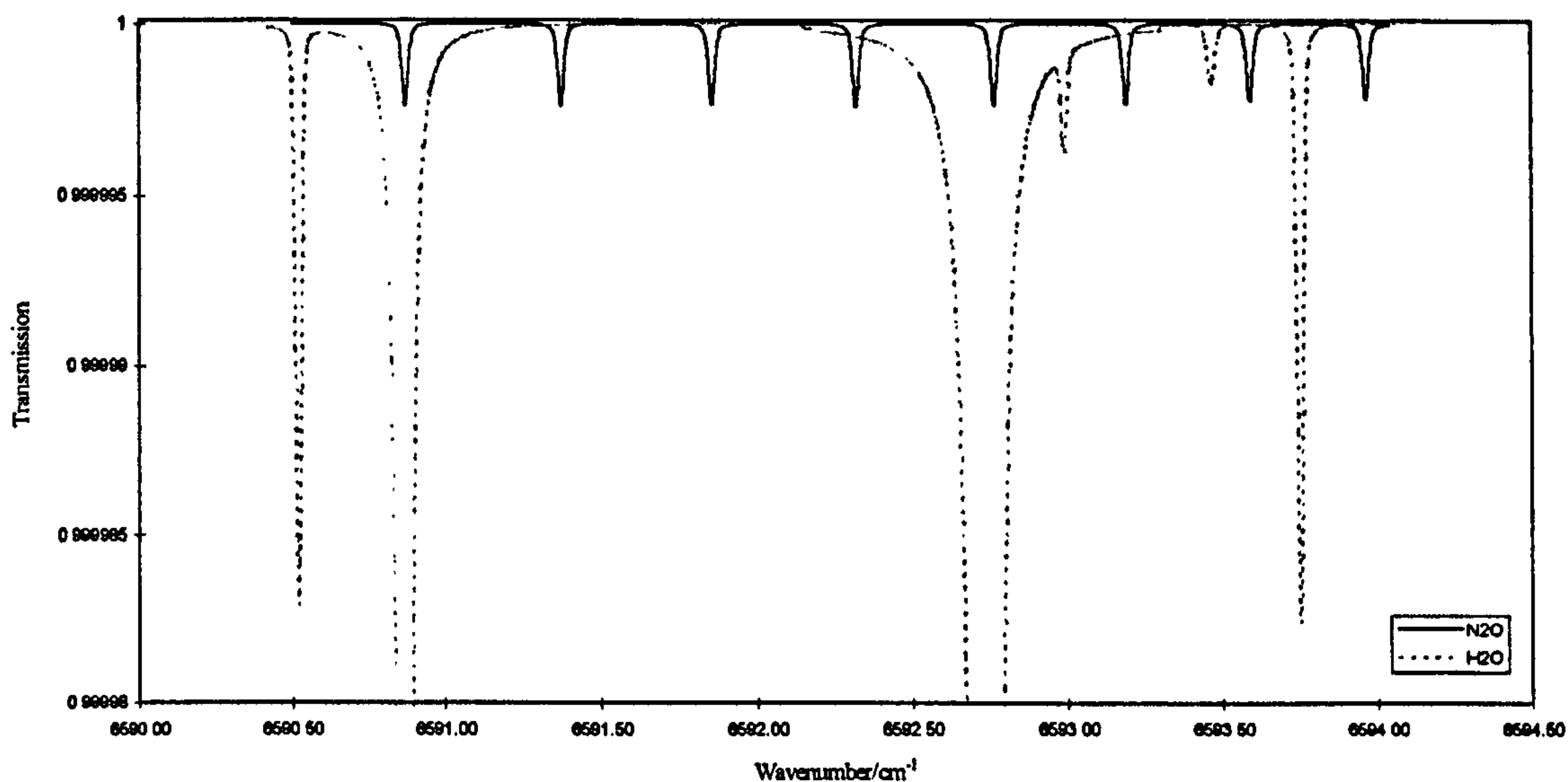


Figure 6.20 - Strongest lines in the $3 \nu_3$ R branch

The strongest line in this region has a line strength of 1.81×10^{-23} . Assuming an air sample pressure of 60 Torr and an absorption pathlength of 25.1 m, the strongest N₂O transition (R(17)) has a peak absorption of 1.5×10^{-6} . The R(17) transition also appears to be free from interference. This transition was therefore chosen as the best candidate for trace gas measurement.

Nitrous Oxide Spectra Recorded in the Lab

The peak absorptions of the nitrous oxide transitions are more than two orders of magnitude weaker than those seen with methane. As such detection of N₂O would be significantly more difficult than CH₄. Initial attempts at recording the N₂O lines in this region failed. The sensitivity of the instrument with the Thomson diode was found to be nearly two orders of magnitude worse than the Anritsu laser. The noise seemed to have a definite fringing pattern on it, however, no matter how often the optics were realigned the magnitude of these features and their FSR did not change. Only after several weeks of investigation was the source of this *fringing* found. The noise was in fact electronic rather than optical. A fault had developed in the signal channel lock in amplifier. A spectral analysis of the lockin output showed that it had a large high frequency component at about 70 MHz. The lockin digitiser was sampling this high frequency component giving an aliasing effect similar to that seen on Fourier

Transform Spectrometers (FTS). The frequency of the *fringing* was found to vary as the sampling rate of the instrument was changed. The reference lock in amplifier and signal amplifier were swapped over to eliminate this problem. The magnitude of this noise source was estimated to be equivalent to an absorption of 5×10^{-5} .

Another source of noise was also identified. The InGaAs detector used with the 1.52 μm laser was generating optical noise. It is not sure whether this noise was caused by fringing between the lens attached to this detector and another optical component or whether the lens was in some way causing optical feedback into the laser. The latter of these two explanations is thought to be the more likely because the noise had no apparent fringe FSR. The laser did not have an optical isolator and would thus be susceptible to feedback. The problem, however, was eliminated by replacing the InGaAs device with the Ge detector. The magnitude of the noise associated with the detector was estimated to be equivalent to an absorption of 1×10^{-6} .

Figure 6.21 shows the spectrum of 60 Torr of Air between 6001 cm^{-1} and 6004 cm^{-1} after these modifications had been made. The diode temperature was set to $19.87 \text{ k}\Omega$ ($10.5 \text{ }^\circ\text{C}$) and the laser current was tuned between 80 and 85 mA. 300 points were recorded with four points being averaged at each ramp step. The reference cell channel was also logged, enabling easier identification of the nitrous oxide transitions. The signal from the reference channel is shown below the main spectrum. The R(17) transition is clearly visible on both channels, however, it is seen that the reference cell feature is significantly broader. This is because the cell was filled with a 150 Torr $\text{N}_2\text{O}/\text{Air}$ mixture, whereas the Herriott cell was filled with 60 Torr of air. The Herriott cell was operated at a reduced sample pressure so as to reduce interference from other lines (water). At this pressure the lineshape is Voigt with similar Doppler and Lorentz components ($\gamma_{\text{D}_{\text{HWHM}}} = 0.00600 \text{ cm}^{-1}$, $\gamma_{\text{L}_{\text{HWHM}}} = 0.00528 \text{ cm}^{-1}$). In addition, the spectra were slightly under modulated, again so as to minimise line interference. An absorption feature at the edge of the spectrum is also visible. This line could not be identified but the day to day variability in the relative strength of the feature compared to the R (17) transition meant that it was most likely to be water. The noise residuals are shown in Figure 6.22. The rms noise is $4 \times 10^{-7} \text{ V}$. This corresponds to a minimum detectable absorption of 1×10^{-7} . There was no apparent fringe pattern on the noise.

R(17) N₂O Transition (60 Torr Air Sample)

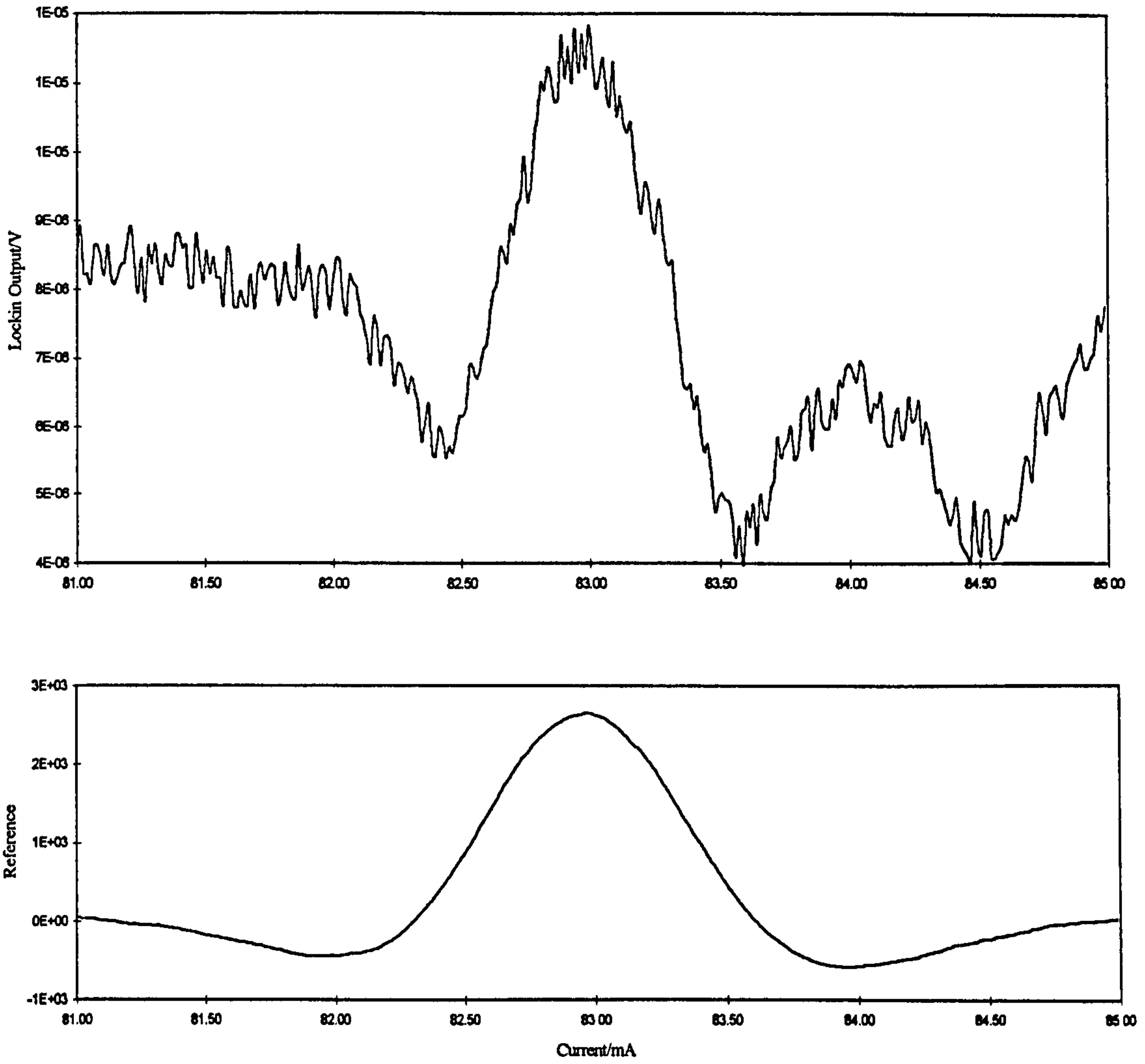


Figure 6.21 - Spectrum of Ambient Nitrous Oxide (R(17) transition)

Noise Residuals

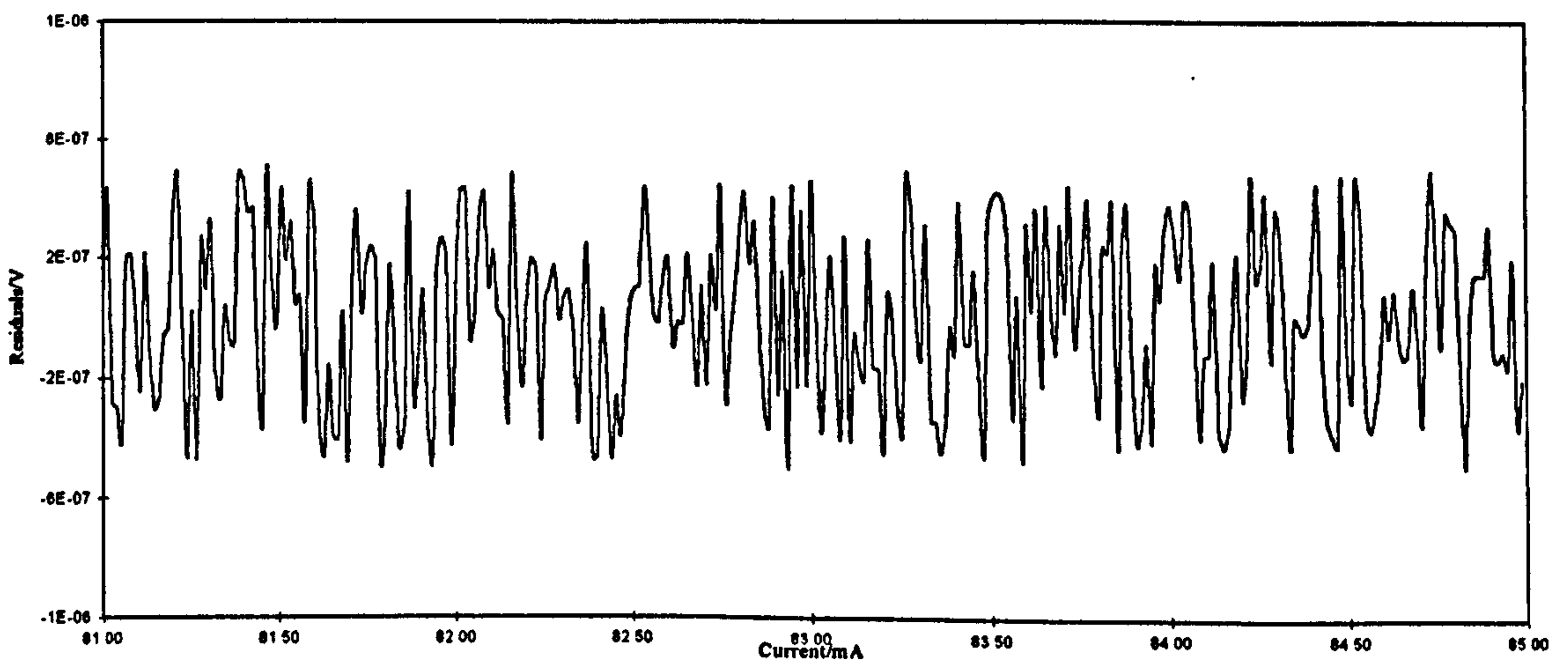


Figure 6.22 - Noise Residuals

The other lines in this region, that did not appear to suffer interference from water, namely the R(21) and R(16) transitions, were also recorded. They are shown in **Figure 6.23** and **Figure 6.24** respectively.

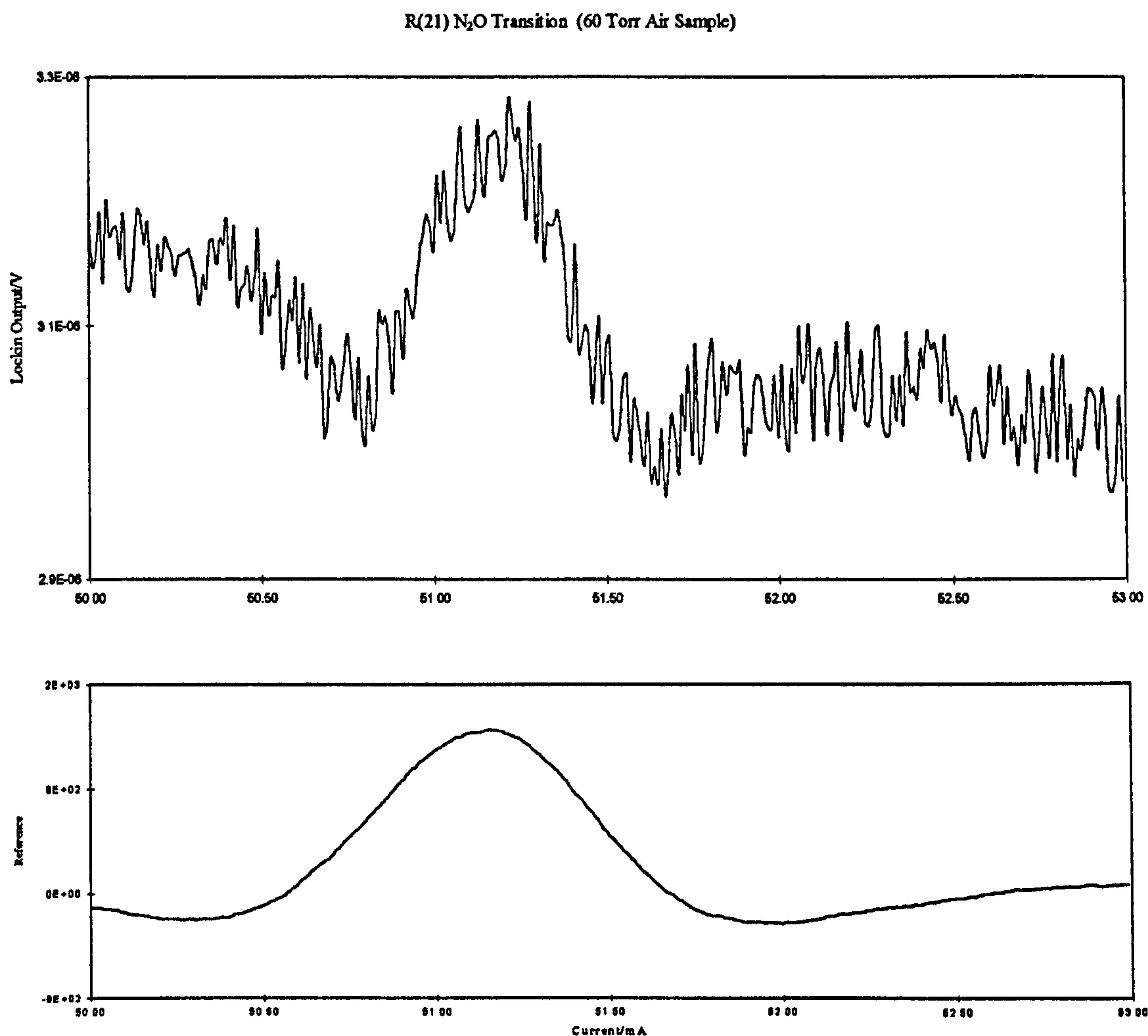


Figure 6.23 - R(21) N₂O Transition (P= 60 Torr, l = 25.1 m)

Again 300 points were taken in each spectrum with four points being averaged at each ramp step. The diode temperature was 10.5°C. Sensitivity limits for both the R(16) and R(17) spectra were similar, however the spectrum of the R(21) transition exhibited poorer sensitivity. This was partly due to a slightly weaker linestrength (1.63 cm molecule⁻¹ against 1.81 cm molecule⁻¹), but in the main was a result of reduced power

falling on the detector at the lower laser currents (~50 mA) the feature was recorded at.

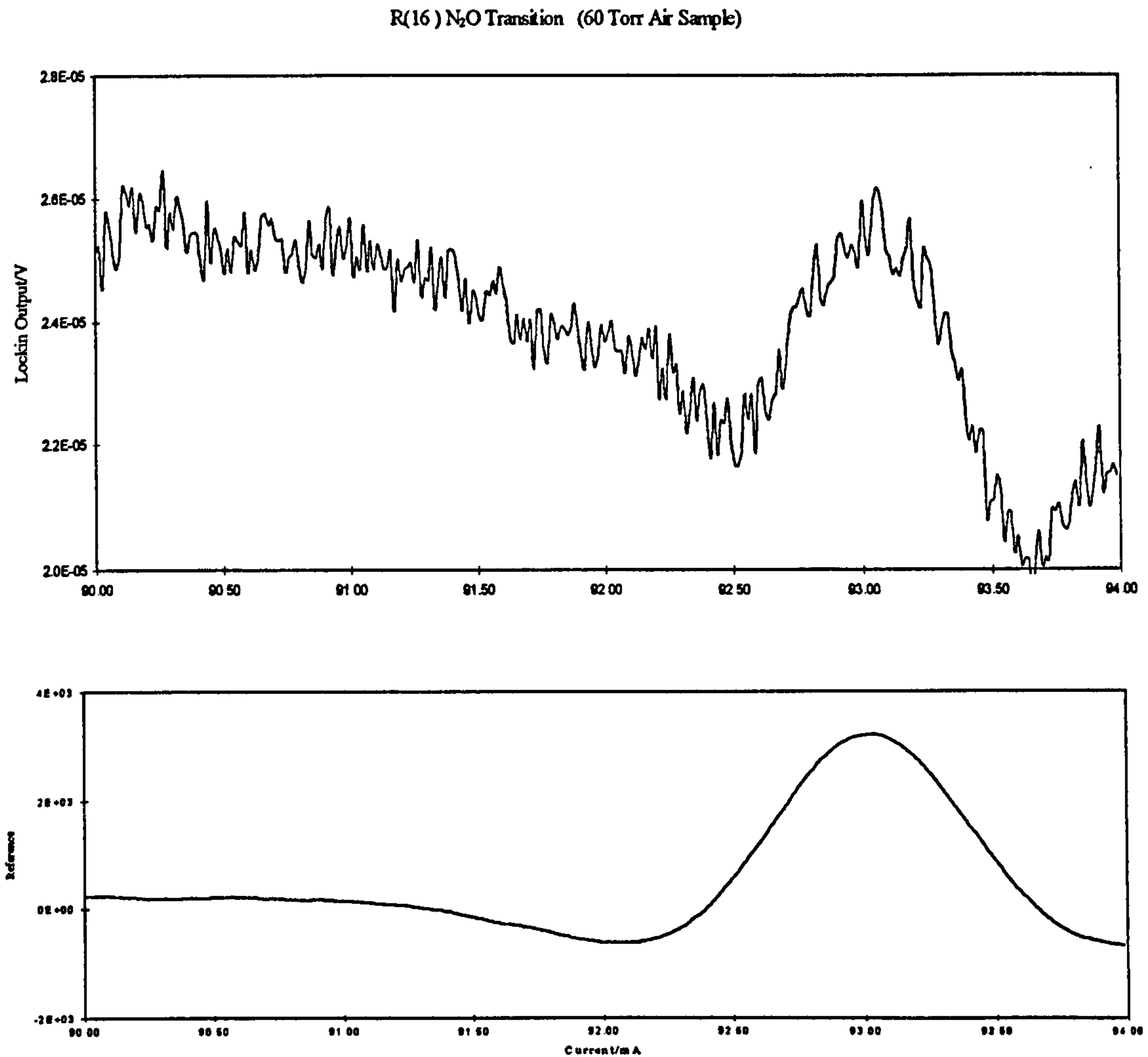


Figure 6.24 - R (16) N₂O Transition (P= 60 Torr, l = 25.1 m)

The noise levels exhibited here would limit the instrument precision to approximately 22 ppbV. This would be precise enough for detecting nitrous oxide as a pollutant from industry but would not provide the required sensitivity for flux measurements (section 5.2). Fourier transform noise removal techniques (section 5.4) were used to try and remove the high frequency noise component from the R(17) spectrum in Figure 6.21. The resulting spectrum is shown in Figure 6.25. The sloping baseline has been subtracted by polynomial fitting. The signal to noise has been substantially improved, however, close inspection of the baseline shows that a low frequency component is

present. A synthetic spectrum of the R(17) transition was subtracted from the recorded spectrum to allow a closer inspection of the baseline. The recorded and synthetic spectrum and the residuals are shown in **Figure 6.26**.

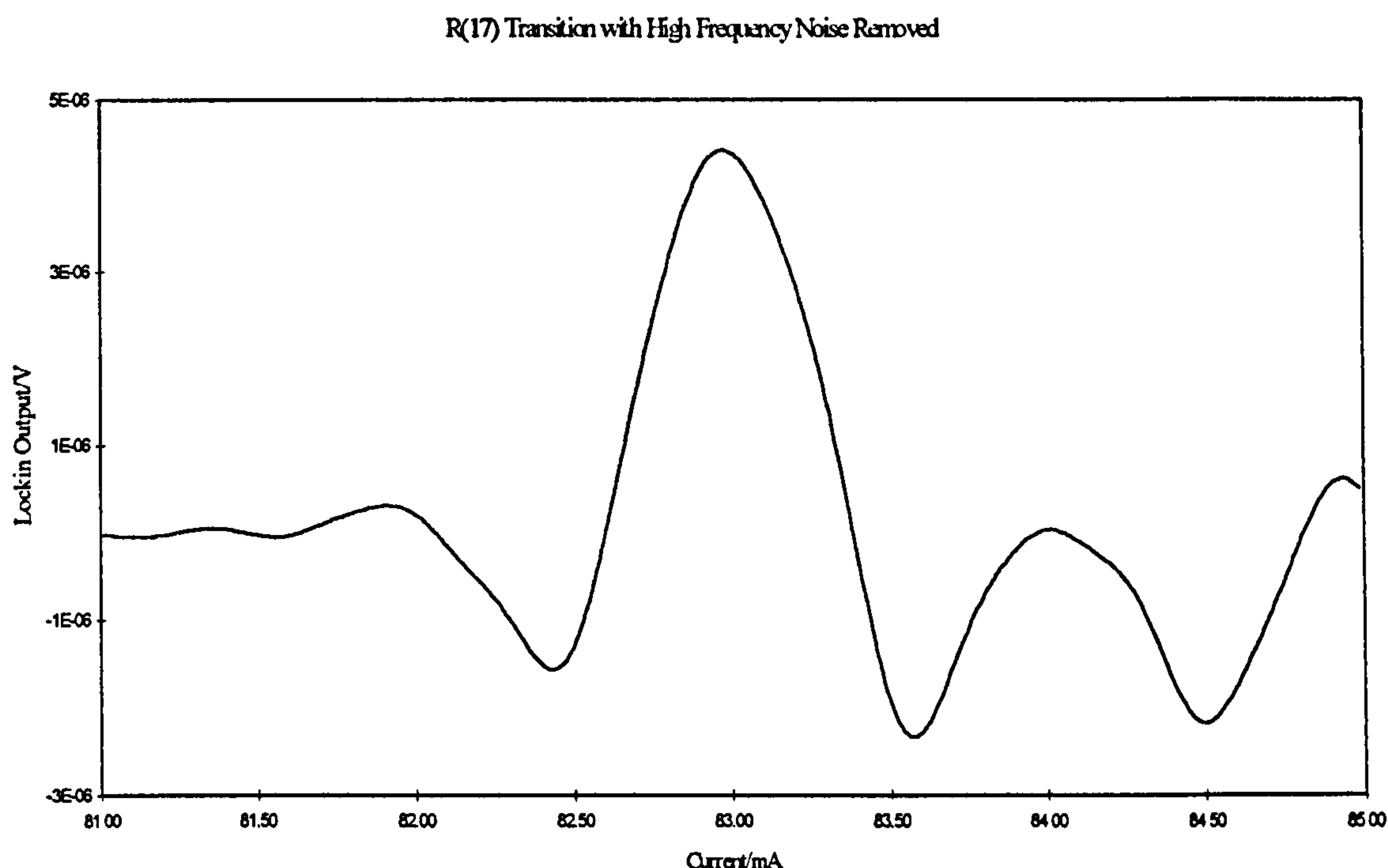


Figure 6.25 - R(17) N₂O Transition with High Frequency Noise Removed

The residuals show up the slowly varying background. It is thought that some of this variation in the background is a result of absorption features. The most obvious being the broad peak at $\sim 6591.86 \text{ cm}^{-1}$. This could not be identified but again is likely to be water or carbon dioxide. The remaining background variation is thought to be fringing. The FSR corresponds to a path difference of 0.5 m. As with the methane laser the source of this fringing was not identified. The magnitude of this slow fringing is equivalent to a minimum detectable absorption of $\sim 6 \times 10^{-8}$. This will give a N₂O detection limit of $\sim 15 \text{ ppbV}$.

Recorded and Synthetic Spectrum of the R(17) Transition

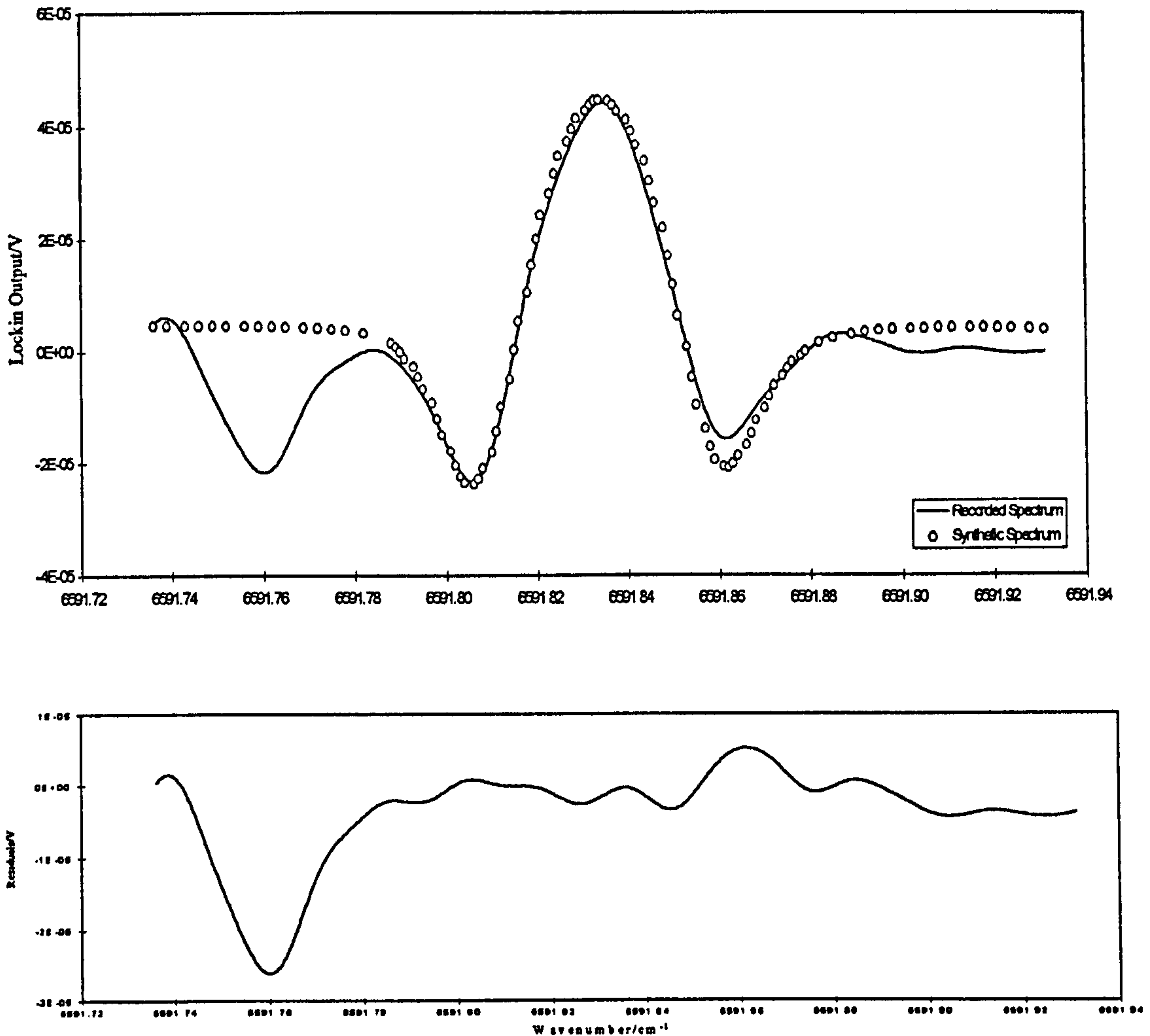


Figure 6.26 - Recorded and Synthetic Spectrum of the R (17) Transition

Concentration Retrieval

The procedure for concentration retrieval for nitrous oxide is identical to that used for methane. A synthetic spectrum of the R(17) transition, calculated assuming a sample pressure of 60 Torr, a pathlength of 25.1 m and concentration of 330 ppb/V, was least square fitted to the recorded spectrum as is shown in Figure 6.26. The scaling constant between the two spectra was 1.09. This gives an ambient nitrous oxide concentration of 360 ppb. Repeated measurements were taken over a period of an hour to check the variability in the retrieved concentration. This is shown in Figure 6.27.

Variability in Nitrous Oxide Concentration

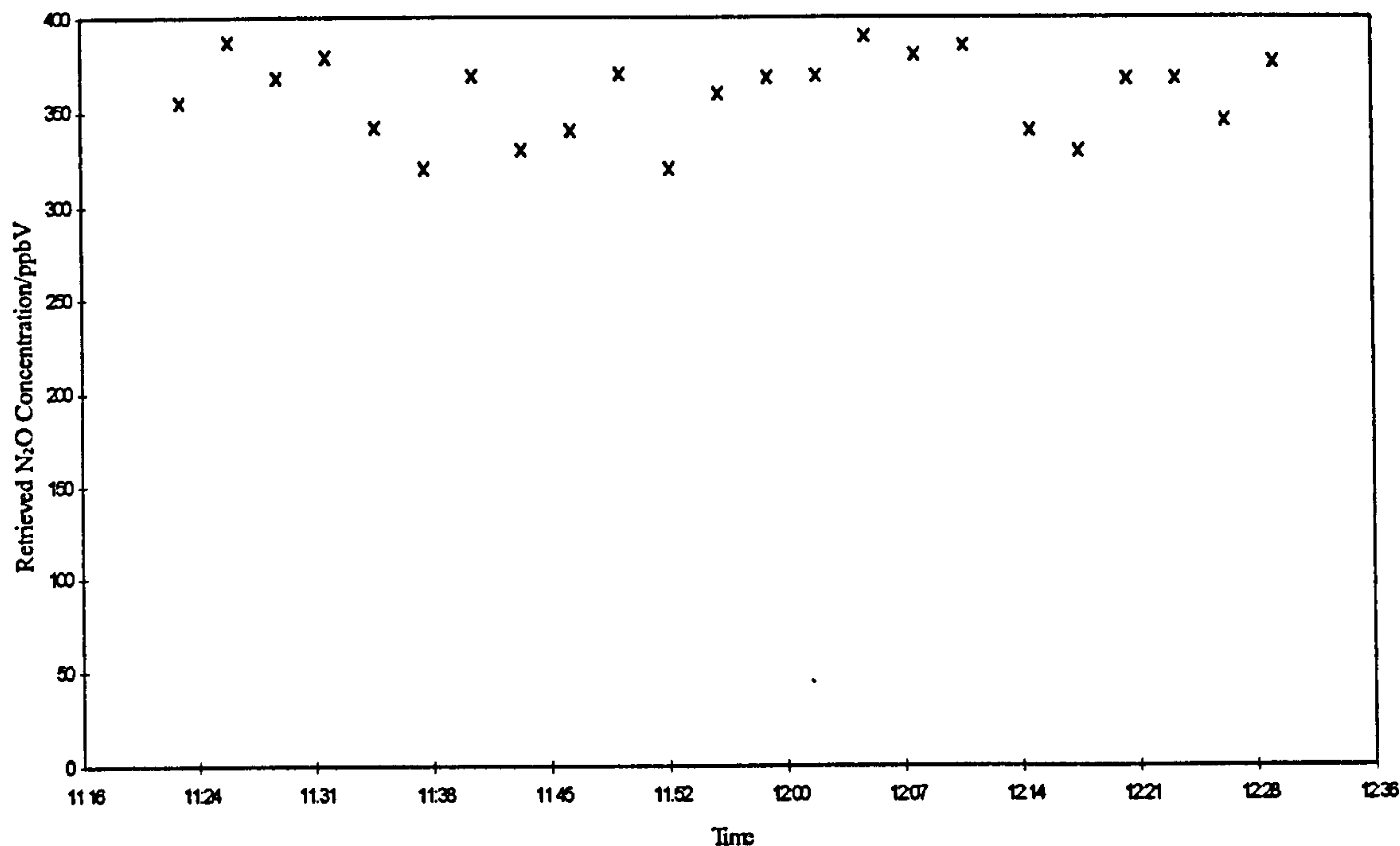


Figure 6.27 - Variation in the Retrieved N₂O Concentration with Time

The mean concentration during this period was 358 ppb. This is about 8% above the accepted ambient concentration (333 ppbV) [3]. The standard deviation of the retrieved concentration was equivalent to ~ 22ppbV of N₂O. The concentration of N₂O at ground level is known to be fairly stable [3], it was therefore concluded that the observed changes in the retrieved concentration were not real. The variability is also greater than what one would expect if the uncertainty due to instrumental noise (fringing) is taken into account. It was concluded that the additional uncertainty arose from the large sloping background present in all the recorded spectra (see **Figure 6.21**). This background was removed by a polynomial fit procedure which required the user to identify points on the spectrum that he/she thought represented the background signal. Repeatability tests showed that variations of up to 3 % were observed in the retrieved concentration depending on what points on the spectrum were nominated as the background. It is thought that problems in defining the background also resulted in the 8% discrepancy between the mean concentration and the accepted level.

General Comments and Conclusions

As experienced with the methane laser, the day to day sensitivity was found to vary between 1×10^{-7} and 1×10^{-6} . Again the variation is thought to be due to variations in the level of optical fringing. The Thomson laser exhibited slightly better sensitivities than the Anritsu laser. Two factors are thought to be responsible: the Thomson diode had a higher power output than the Anritsu laser (5mW against 1mW). The Anritsu laser was more prone to intensity fluctuations as a consequence of being fibre coupled (variations in intensity of about 0.1 % were observed when the fibre was moved). Sensitivities were again extremely close to those predicted by theory.

Fourier transform noise removal techniques were shown to improve detection sensitivities to an equivalent absorption of 7×10^{-8} . Sensitivity could not be improved further because of the presence of low frequency fringing. In addition to noise on the spectra, the large background signal created an additional uncertainty of about 3 % in the retrieved concentration.

The sensitivities shown here will allow nitrous oxide mixing ratios of the order of 22 ppb to be detected. This is more than an order of magnitude worse than the best sensitivities achieved by mid-infrared TDLAS. ALIAS reported sensitivities of 50 ppt [11]. ATLAS, a mid-infrared instrument also flown on the ER-2 aircraft achieved an instrument precision equivalent to ~ 5 ppb [33]. The inability of the instrument to match the sensitivity of the mid-infrared systems is due to the weak linestrengths of the N_2O transitions recorded. The $3 \nu_3$ band is the second overtone and as such has linestrengths more than three orders of magnitude weaker than those used in the mid-infrared.

6.3.4 Wavelength Stability

A test was performed to assess the wavelength stability of the instrument. In the mid-infrared poor wavelength stability results in the narrow absorption feature from the reference cell being used to line lock the laser wavelength. This increases the cost and complexity of the instrument. It was hoped that in the near-infrared this would not be necessary.

The wavelength of the methane laser was monitored with a Burleigh wavemeter (WA 1000, 0.001 cm^{-1} resolution) over a period of ~ 15 hours. The temperature of the diode and the surrounding air were also logged to ascertain if there was any correlation. Figure 6.28 shows how the wavelength and the diode temperature varied over this period.

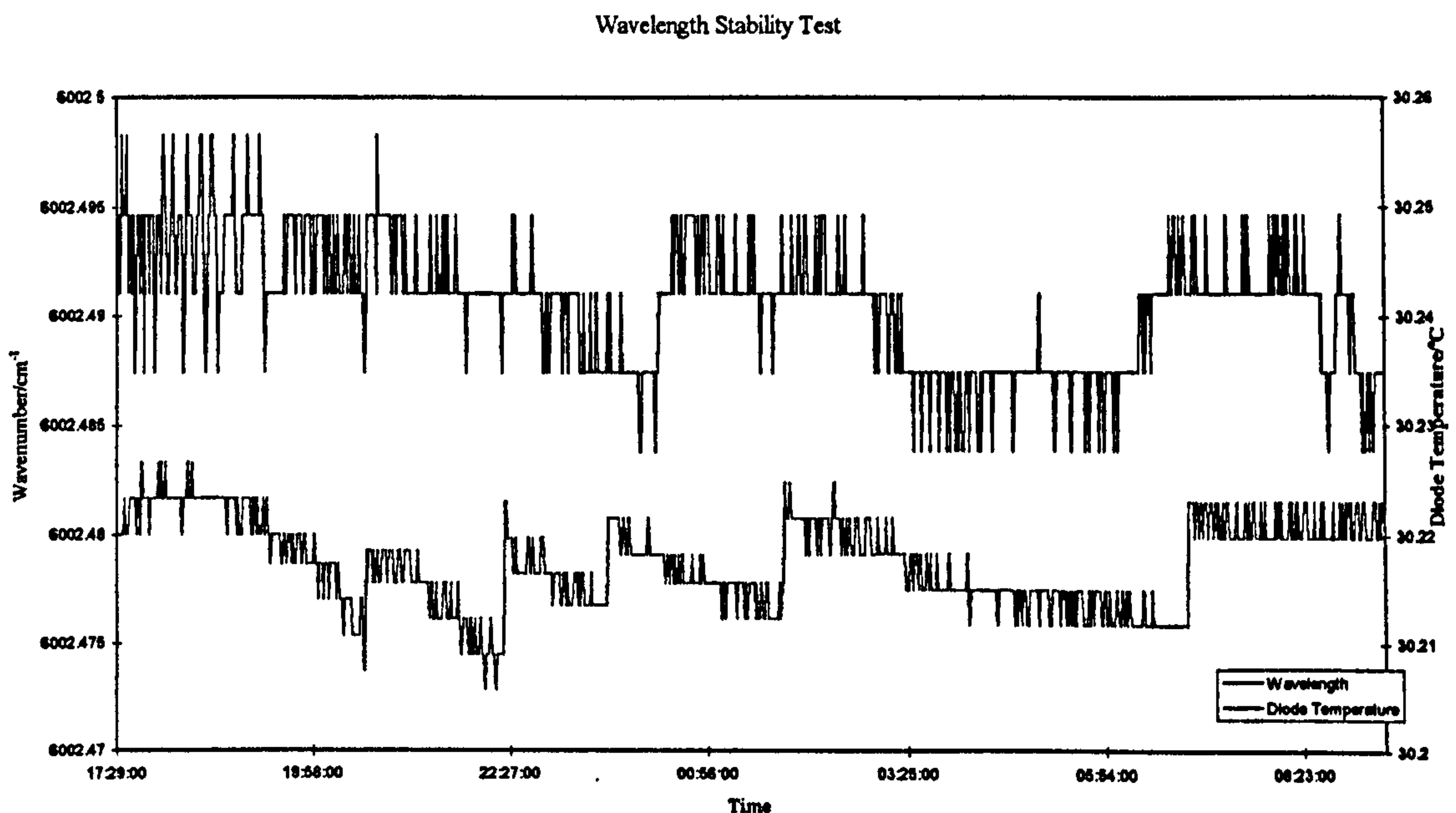


Figure 6.28 - Wavelength Stability Test

It is seen that the diode temperature and the operating frequency of the diode are directly correlated. The surrounding air temperature during this period did not show any obvious correlation with either the wavelength or the diode temperature. This tells us that variations in the surrounding air temperature were not responsible for the wavelength drift. The correlation between the diode temperature and wavelength

shows that the drift was caused by the temperature controller and not by drifts in laser operating current.

The maximum drift over the 15 hour period was 0.007 cm^{-1} . This is about 2-3 % of a typical wavelength scan when recording a spectrum. Importantly, it is seen that the wavelength of the laser remained fairly constant for periods of hours at a time. The temperature of the diode during this period changed by less than $0.01 \text{ }^\circ\text{C}$. It is concluded that the instrument wavelength stability is sufficiently good not to require the use of line locking techniques.

6.3.5 Projected Performance for other Gas Species

Unlike in the mid-infrared where laser quality can be *patchy* and achievable sensitivities can vary significantly from diode to diode, it has been shown that two lasers from different manufacturers and operating at different wavelengths can provide similar performance. This reproducibility is a consequence of the mass production techniques used to manufacture these devices for optical communication purposes. The similarity in the performance allows the projected detection sensitivities for other gases to be made with confidence.

Table 6.1 details the projected minimum detectable mixing ratios for different gases which are of interest either as industrial pollutants or in atmospheric chemistry. The calculations have assumed a sample pressure of 100 Torr, a twenty five metre pathlength and a minimum detectable absorption of 1×10^{-7} in a 1 Hz bandwidth. Lines not referenced have been measured in this study (Section 3.3).

Molecule	Line Position	Line Strength	Doppler Width HWHM	Lorentzian Width HWHM	Minimum Detectable Mixing Ratio
	(nm)	(cm molecule ⁻¹)	(cm ⁻¹)	(cm ⁻¹)	(ppbV)
CH ₄ [23]	1651	1×10^{-21}	0.0093	0.0083	0.56
CH ₄ [69]	1330	5×10^{-23}	0.0116	0.0083	5
N ₂ O	1517	1.81×10^{-23}	0.0061	0.0084	22
N ₂ O [23]	1984	6×10^{-23}	0.0047	0.0084	6
CO ₂ [7]	1577	1.6×10^{-23}	0.0058	0.0079	24
CO [7]	1567	2.3×10^{-23}	0.0077	0.0087	19
NH ₃	1512	1×10^{-21}	0.0099	0.0079	0.47
HCl [7]	1747	1.2×10^{-20}	0.0059	0.0121	0.04
HBr [7]	1341	2.1×10^{-23}	0.0051	0.0106	21
HF [7]	1330	1.3×10^{-20}	0.0104	0.0044	0.02
HI [7]	1541	3.1×10^{-22}	0.0035	0.0079	1
H ₂ S [7]	1578	1.3×10^{-22}	0.0067	0.0112	4
H ₂ O [7]	1365	2.1×10^{-20}	0.0106	0.0125	0.03

Table 6.1 - Projected Sensitivity assuming a minimum detectable absorption of 1×10^{-7}

6.3.6 Conclusions

A spectral survey for both methane and nitrous oxide has been carried out allowing suitable transitions free from interference to be identified. A performance assessment of the instrument in both methane and nitrous oxide detection configurations has shown that:

- Near-infrared technology can allow absorptions as small as 1×10^{-7} to be detected.
- Sensitivities were within a factor of three to those predicted by theory.
- Detection sensitivities equivalent to 560 ppt and 22 ppb mixing ratios for methane and nitrous oxide respectively could be detected.
- Sensitivities could be improved further through signal averaging and Fourier transform noise removal techniques (typically by a factor of two).
- The ultimate limit to the attainable sensitivity was found to be optical fringing resulting from path differences of the order of a metre.
- The performance of the instrument is comparable to that obtained with the best of the mid-infrared TDLAS.
- The low level of fringing encountered, confirms the suitability of the Herriott Cell for pathlength enhancement.
- Excellent wavelength stability removed the need for laser line locking techniques.

So far only the instrument precision has been considered. There are many more measurement uncertainties that will affect the accuracy of the retrieved concentration. The error budget for the instrument is detailed in Section 6.5.

6.4 Field Test

6.4.1 Introduction

While the performance of the instrument had been fully assessed in the laboratory (Section 6.3), it was felt that it would be worthwhile assessing the performance in an environment the instrument would typically be used in. To this end, a field test of the instrument at a landfill site was arranged. It was hoped that the large variability in the methane concentration typical encountered at such a site would allow both the instrument sensitivity and measurement bandwidth to be determined. In addition, an intercomparison with two other methane monitors (Flame Ionisation Detectors) was arranged. This would hopefully give some idea of how accurate the concentration retrieval process was and highlight any instrumental drifts.

6.4.2 Field Site and Experimental Details

The landfill site, situated in the south west of England, covered an area of approximately six hectares. The site had been used from the 1970's up until 1990. The bulk of the waste was of the household type. After tipping had stopped a 30 cm layer of soil was used to cap the site. The soil, being permeable, was expected to emit methane uniformly across the entire site area. This site was chosen because it did not contain any hazardous waste, was easily accessible and had already been used by the National Physical Laboratory for measurement of methane fluxes.

For intercomparison purposes, two flame ionisation detectors (FID) were used. The operation of these instruments is based on the principle of 'flame ionisation' whereby the presence of hydrocarbons in a hydrogen flame produces an electrical (ionisation) current effect which can be detected and amplified by suitable electronic circuits. The R.E. GAS_TEC was a portable FID which had a detection limit equivalent to 0.1 ppm of methane and had a two to three second response time. The Horiba APHA-350E had a significantly slower response time (60 seconds), but exhibited superior detection limits (0.01 ppm) and excellent long term stability (~ 1% over the period of a day).

For the purpose of the field test, the TDLAS was incorporated into a mobile gas detection unit from the National Physical Laboratory. Power was supplied by a 1 kilowatt petrol generator. A UPS was used, enabling controlled shut down in the event of a generator failure.

Measurements were taken downwind from the site at intervals of 30 seconds. Each spectrum consisted of 250 points with four points being averaged at each ramp step. Each scan took approximately 15 seconds to record. This scan time represented a compromise between obtaining reasonable signal to noise and a fast scan rate. The lock in time constant was reduced to 100ms for these measurements, ensuring the bandwidth was wide enough so as not to affect the recorded lineshape.

For the field test it was decided to monitor pressure, temperature and laser power continuously (every 0.1 seconds) rather than at the beginning, middle and end of the scan (see section 5.5). A DT600 data logger was used to capture this data. At the end of each scan when the data was being saved to hard disk the laser beam was blocked allowing the detector zero level to be determined. As with the laboratory measurements, the sample pressure inside the cell was maintained at 100 Torr. At this pressure, the Lorentzian and Doppler components are approximately equal. A 100ppmV methane gas standard (National Physical Laboratory - Cylinder 29) was used for instrument calibration. A calibration was performed at the beginning and end of the field test allowing instrument drift to be assessed.

6.4.3 Results

Sensitivity

A typical spectrum obtained during the field test is shown in **Figure 6.29**. The laser was scanned between 85 and 110 mA and the diode temperature was set at 7.88 k Ω (32.5 °C). The rms residual value between this spectrum and a synthetic spectrum was equivalent to an absorption of 6×10^{-7} assuming a 1Hz bandwidth. This is equivalent to a minimum detectable gas mixing ratio of 1.7ppbV. This is only slightly worse than the best sensitivity that was achieved in a laboratory environment (equivalent to a minimum detectable absorption of 2×10^{-7}). The reduction in sensitivity is attributed to an increase in noise in the power supply from the electrical generator.

Example of a Typical Spectrum Recorded at Land Fill Site

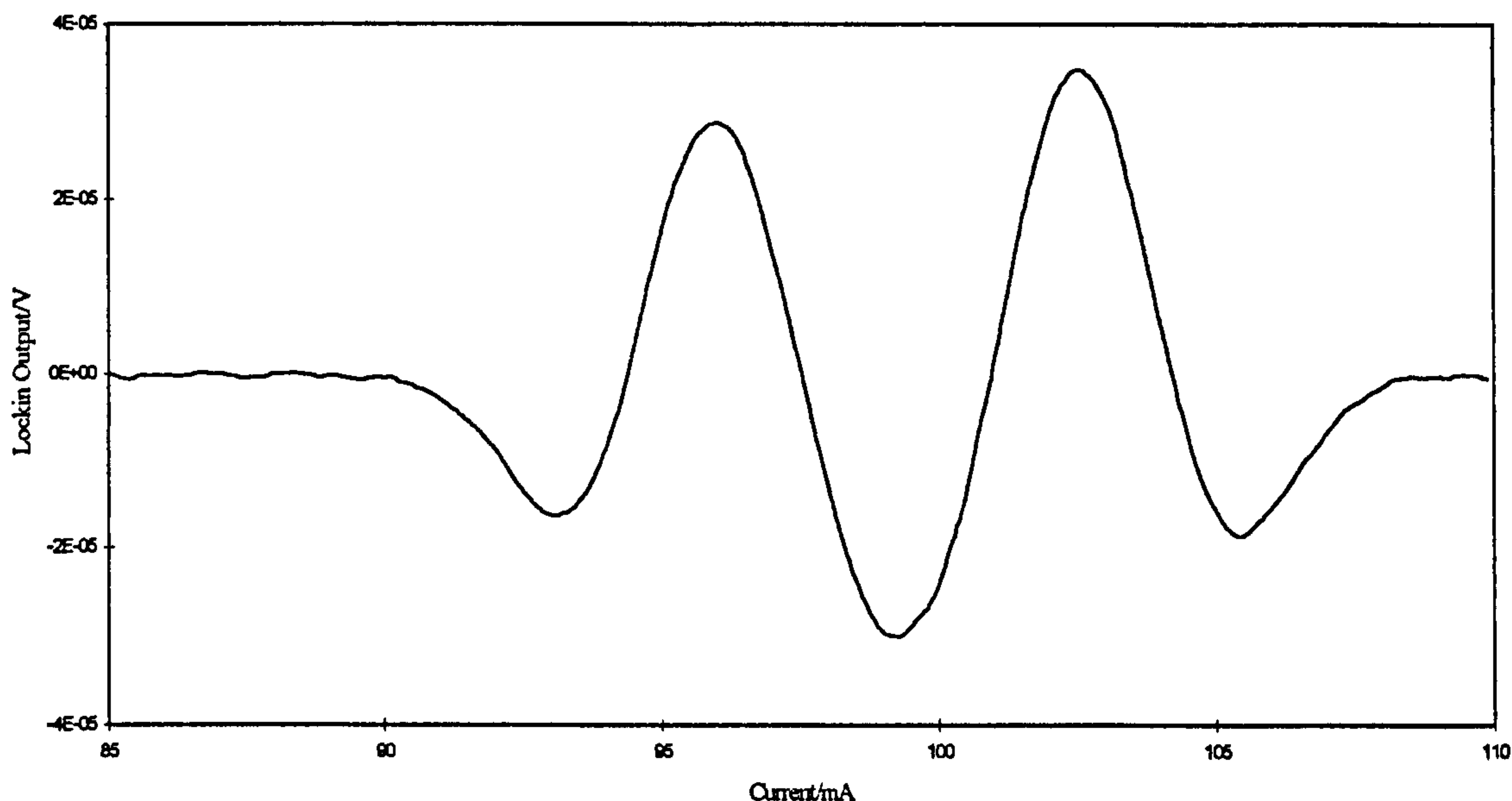


Figure 6.29 - Example of a Typical Spectrum Recorded at Landfill Site

Pressure, Temperature and Laser Power Measurement

As stated earlier, the cell pressure, air temperature and laser power were monitored every 0.1 seconds. The data was monitored continuously, during the 15 second period of the scan and also during the 15 seconds dead time between the end of the scan and the beginning of the next scan. During this dead time the laser beam was blocked allowing the detector zero level to be determined. This allowed the absolute power level of the laser to be retrieved for normalisation of the recorded spectra (Section 6.2). Over the period of the day, the absolute power changed by approximately 1%. This was probably caused by a drift in the alignment of the optics. Temperature varied between 22 °C and 26.5 °C, while the sample pressure stayed within 2% of its set level (100 Torr).

Intercomparison

Intercomparison measurements were taken in four blocks over a period of ~ 4 hours. Each block representing approximately 30 minutes of data.. About half way through the first measurement block the power generator failed. It could not be restarted and a controlled shut down was initiated. On start up, the instrument was recalibrated. Consequently, the first data block was discarded. The remaining three sets of data were taken without mishap.

The Horiba FID was used to take simultaneous methane concentration measurements in all three of the remaining sets of data. The GAS_TEC was being used for measurement elsewhere and an intercomparison was performed in the last data set only. **Figure 6.30** and **Figure 6.31** show the intercomparison for the first two data sets (Block 2 and Block 3).

It is seen that the retrieved methane concentrations are similar for both instruments, with an average methane concentration of approximately 3ppmV being recorded. The general trends are also well reproduced, with the large peaks and troughs occurring in the same regions. However, it is seen that some of the finer features in the TDLAS measurements do not show up in the Horiba data. This is a consequence of the FID's slower time response. Close inspection of the data shows that the features recorded with the Horiba FID are smeared out and appear to lag slightly behind the TDLAS measurements. A good example of this is seen in the feature recorded at ~ 14:10 PM in the Block 2 data. The TDLAS starts to respond to a sudden increase in methane concentration some 3-4 seconds before the FID. In the TDLAS the methane concentration is shown to be declining back to original levels about 60 seconds after the initial increase, however, in the FID data the decline is not apparent for approximately 90 seconds. This smearing out of the data is also thought to be responsible for the slightly higher methane concentration recorded by the FID. It is apparent, that where there are features that exhibit a large increase in the methane concentration, there is a ghost contribution from that feature for some time afterwards. Inspection of a *quiet* region in the data confirms this explanation. At approximately 14:50 PM in the Block 3 data, very little change in the methane concentration is observed for about three minutes. During this period it is seen that both instruments are in excellent agreement.

Field Test Intercomparison Data - Block 2

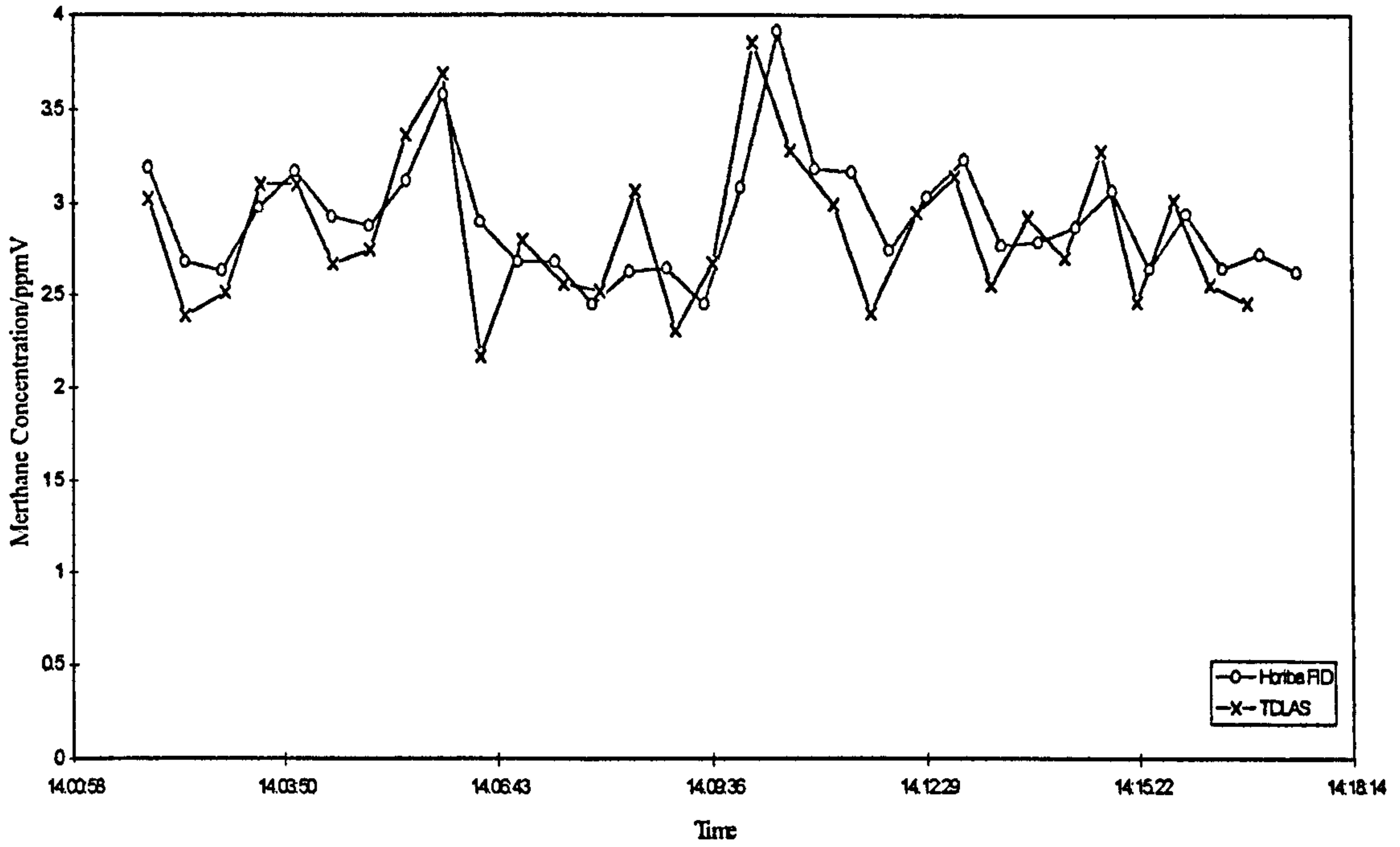


Figure 6.30 - TDLAS and Horiba FID Intercomparison (Block 2)

Field Test Intercomparison Data - Block 3

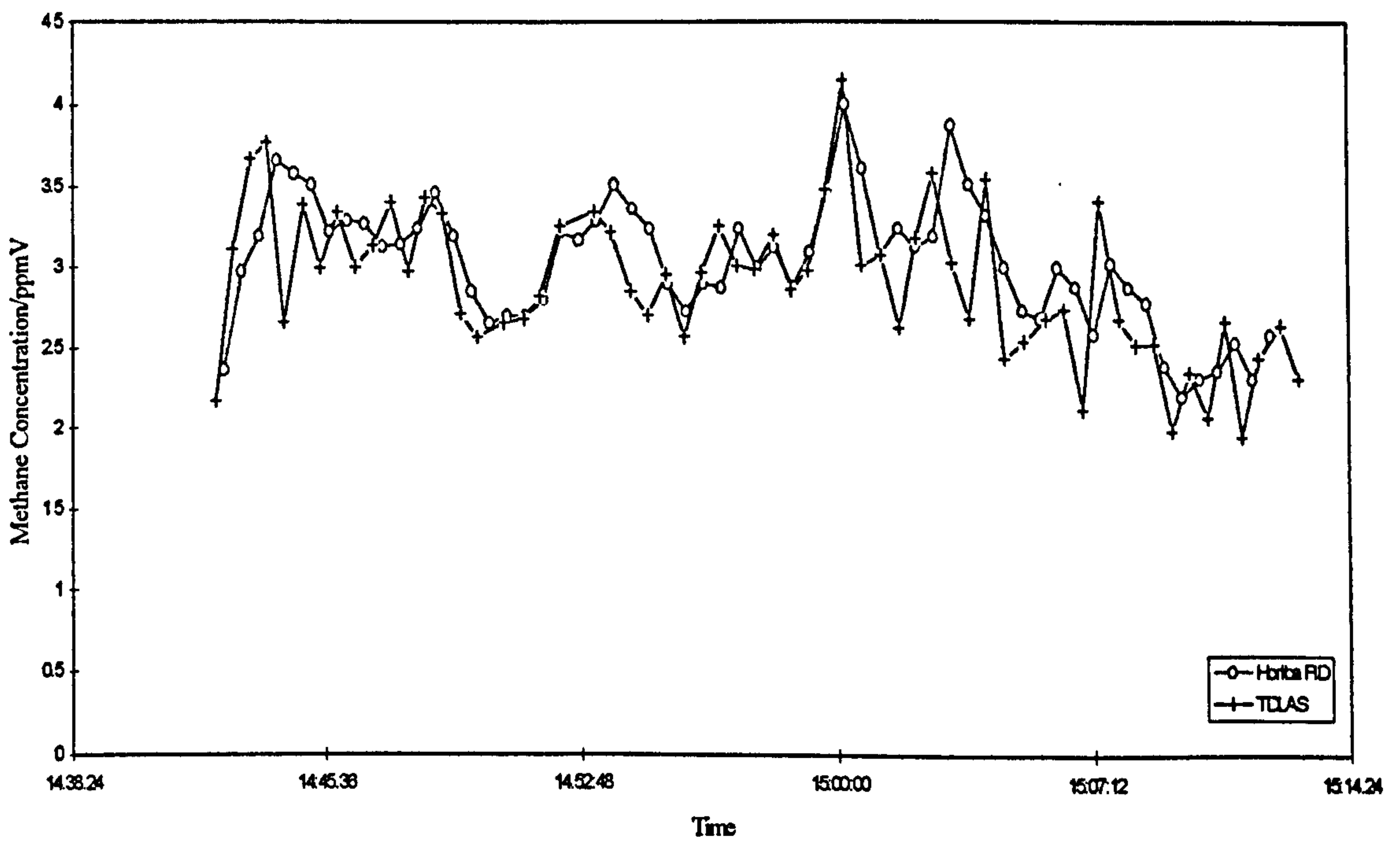


Figure 6.31 - TDLAS and Horiba FID Intercomparison (Block 3)

For the final block of data the TDLAS, Horiba FID and the GAS_TEC were used to record the methane concentration simultaneously. The data is shown in **Figure 6.32**. It is clear that the TDLAS and GAS_TEC provide excellent agreement. This is a consequence of the similar response time for both these instruments. (between 2-3 seconds). Residuals between the retrieved concentrations have a rms value of less than 0.1 ppmV. This is well within the measurement uncertainty of the GAS_TEC (0.2 ppm). Again, the Horiba concentrations were slightly elevated and the features smeared out.

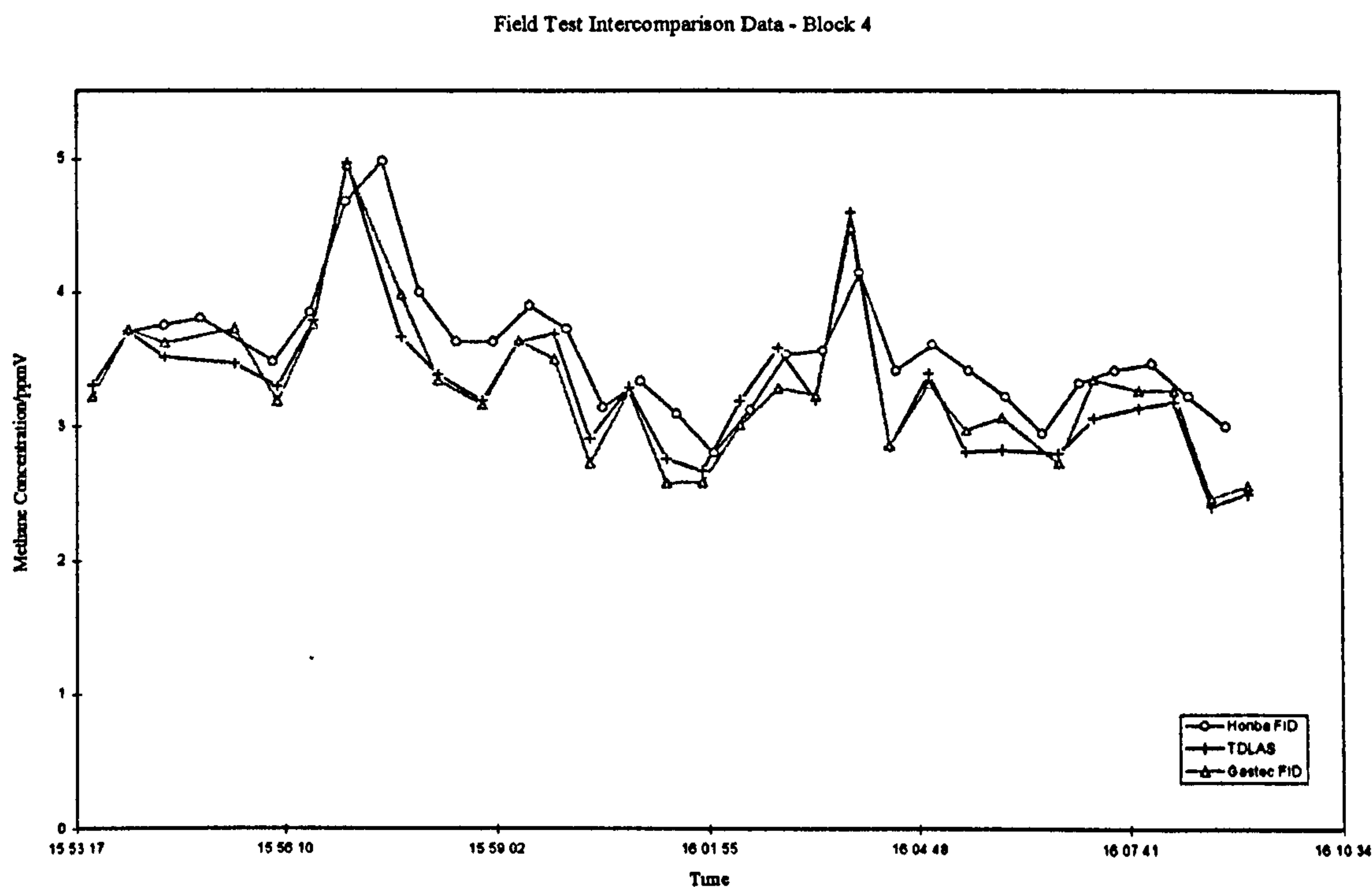


Figure 6.32 - TDLAS, Horiba FID and GAS_TEC Intercomparison (Block 4)

In all three data sets the TDLAS results were in excellent agreement to those obtained with the FID's. Over time no obvious systematic offset in retrieved concentration was observed, indicating that there was little drift in the instrument.

Effect of Varying Methane Concentration during a Scan

For many of the spectra recorded it was clear that the methane concentration was changing as the scan was taken. This changing concentration was most obviously

manifested in the spectra by the variability of the ratio of the two peaks associated with the absorption feature. **Figure 6.33** shows an example of a spectrum taken where this change is particularly obvious. A spectrum where the concentration is not changing is also shown to highlight the difference.

Affect of Varying Concentration on a Recorded Spectrum

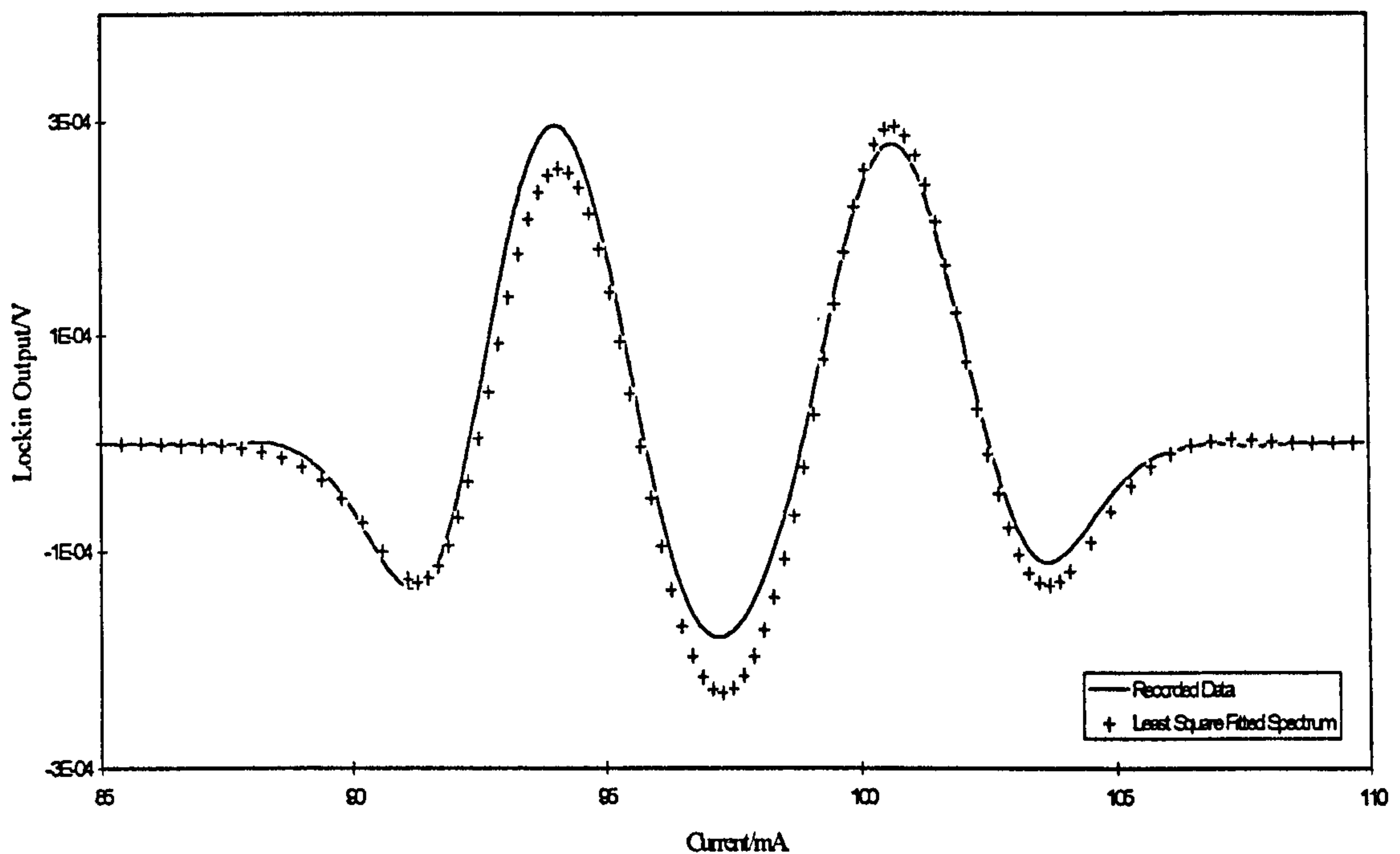


Figure 6.33 - Affect of varying concentration on a recorded scan

It is seen that the peak intensities have been *flipped over*, with the first peak larger than the second. The ability to detect these changes in concentration has been used to determine the concentration at different time intervals during the scan. The scan was split up into five sections, each one corresponding to a peak or trough on the absorption feature. These sections of the spectrum were each separately least square fitted to the corresponding sections on a synthetic spectrum. In this way concentrations at ~ 2 second intervals could be determined. **Figure 6.34** shows how the concentration varied during the scan. Also shown is the retrieved concentration from the GAS_TEC FID over the same period. It is seen that a good agreement between the two was obtained.

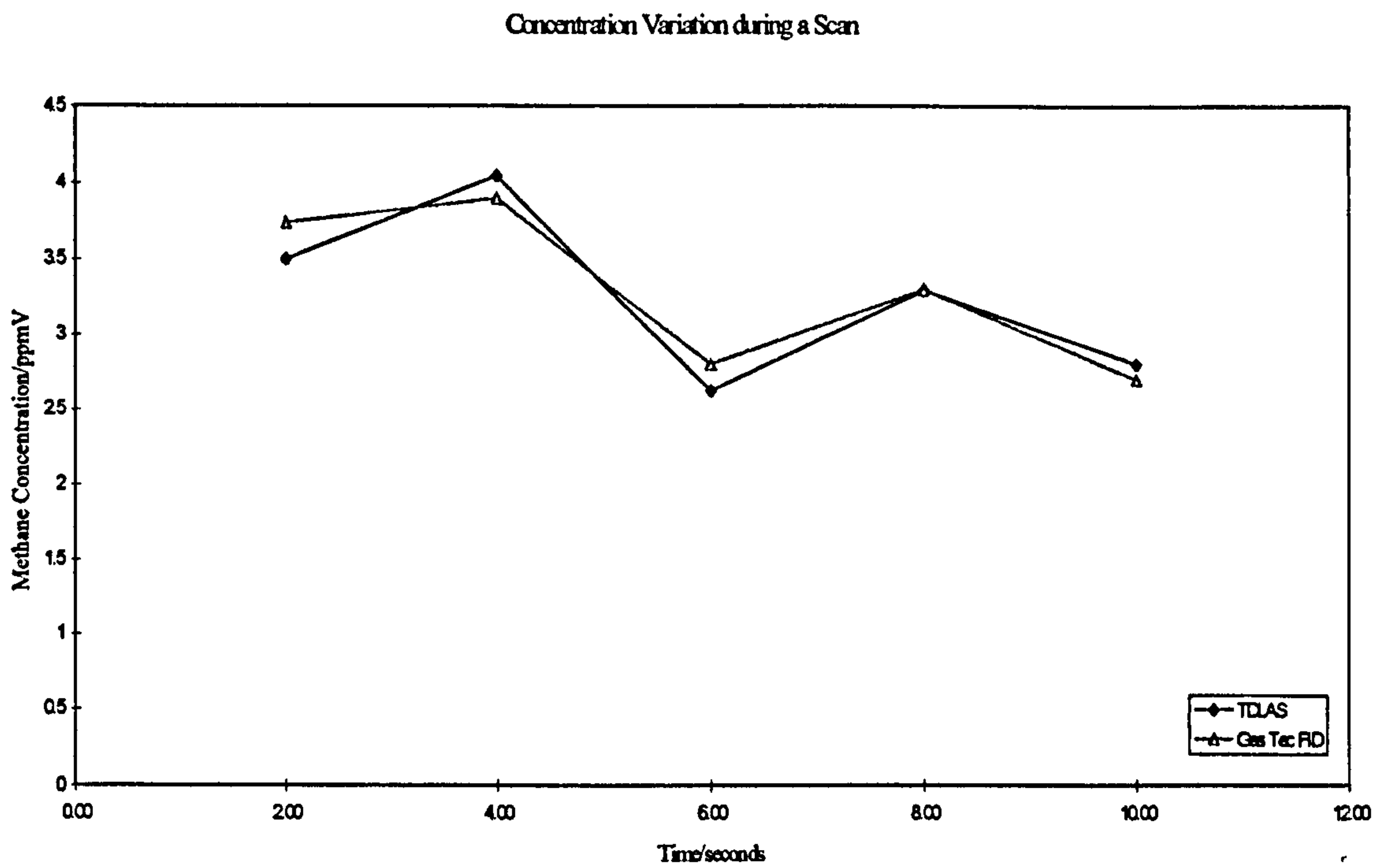


Figure 6.34 - Concentration Variation during a Scan

Calibration

As stated previously (Section 6.4.2), the instrument was calibrated before and after the set of measurements were taken. The response numbers obtained (Section 6.2), were found to agree to within 0.2 % of each other. This confirms the conclusion made earlier (Section 6.4.3) that little to no drift was observed in the measurements.

6.4.4 Conclusions

The field test and intercomparison has shown that:

- TDLAS sensitivities equivalent to a minimum detectable absorption of 6×10^{-7} could be achieved in the field.
- Excellent agreement (within measurement uncertainties) between the TDLAS and Flame Ionisation Detectors was achieved.
- Little to no drift in the instrument was observed over a period of 4 hours
- Variations in the methane concentration during the period of the scan, are shown up clearly in the recorded spectrum. This has allowed concentrations at 2 second intervals to be determined from a scan of 15 seconds duration.
- The good agreement between the TDLAS and GAS_TEC indicate similar response times (2-3 seconds). This agrees well with the measured cell response time of ~ 2.5 seconds (Section 5.5).
- The small variation in the absolute power falling on the detector, during the field test, confirmed the excellent stability of the Herriott cell.

6.5 Error Budget

6.5.1 Introduction

In this section, the combined error budget of the instrument for both the N₂O and CH₄ diodes has been determined. The main sources of error affecting the instrument sensitivity and the accuracy of the concentration retrievals, have been considered. They are discussed in two sections: instrumental noise sources, leading to noise on the recorded 2f spectra (instrument precision) and sources of analytical errors affecting the retrieval of gas concentrations (instrument accuracy). The combined uncertainty from both these sources is evaluated in Section 6.5.4.

6.5.2 Instrumental Noise Sources

This section describes the main sources of instrumental noise. The contribution from each source has been assessed experimentally using the ground based TDLAS described previously (Chapter 5). All noise sources are given in terms of an equivalent minimum detectable absorption (MDA) assuming a 1Hz Bandwidth. A summary of the findings are given in Table 6.3.

Optical Fringing

It was shown in Section 5.4 that without any active fringe reduction technique employed, fringing was found to limit sensitivity to a noise level of 1×10^{-6} . This was achieved by careful design of the optical train (i.e. using reflective optics where possible, using a wedged window and a suitable choice of multipass cell and spot pattern etc.) Fringe reduction techniques (Section 5.4), in particular piezo mirror dithering and Fourier Transform removal, reduced this fringe level by more than a factor of ten ($< 1 \times 10^{-7}$). The exact contribution of optical fringing was determined by

subtracting a synthetic spectrum from the recorded data. In most cases a low frequency fringing effect was observed with a magnitude equivalent to an absorption of $\sim 7 \times 10^{-8}$.

TDL (1/f), Detector - Amplifier and Residual Amplitude Modulation Noise

A detailed study of 1/f noise from the laser diode, noise from the detector-amplifier combination and noise caused by residual amplitude modulation was carried out for a variety of different detection schemes in Section 5.3. The results of this study, for the detection scheme chosen (2nd harmonic WMS nominally at 70 kHz), are given below

1/f noise

The 1/f noise spectrum of both lasers was measured using the MI TF2371 Spectrum Analyser. An example of the noise spectrum obtained for the methane diode is shown in Figure 5.1. The nitrous oxide diode had a 1/f noise contribution of $1.5 \times 10^{-7} \text{ V} / \sqrt{\text{Hz}}$ at a modulation frequency of 70 kHz. This is equivalent to a MDA of 1×10^{-7} . The methane laser had excess noise equivalent to a MDA of 9×10^{-8} ($3 \times 10^{-8} \text{ V} / \sqrt{\text{Hz}}$).

Detector - Amplifier Noise

The detector - amplifier noise was determined experimentally and theoretically. Shot noise and detector thermal noise are the two main sources of noise in detector-amplifier combinations. Theoretical calculations for both detector-amplifier combinations are given below.

Noise Type	Ge Detector - Preamplifier	InGaAs Detector - Preamplifier
Thermal	1.3×10^{-10}	7×10^{-10}
Shot	1.4×10^{-8}	3.4×10^{-8}
Combined	1.4×10^{-8}	3.4×10^{-8}

Table 6.2 - Thermal and Shot Noise Levels

The combined noise from both detector/amplifier combinations was measured on the spectrum analyser (MI TF2371). Noise levels equivalent to a minimum detectable absorption of 4×10^{-8} and 6×10^{-8} for the Ge and InGaAs detectors were measured respectively. In each case the measured noise is approximately twice as big as predicted by theory. The discrepancy is felt to be caused by noise generated in the spectrum analyser itself (estimated to be equivalent to a minimum detectable absorption of 3×10^{-8}).

RAM

Residual Amplitude Noise (RAM), resulting from simultaneous amplitude modulation as the laser frequency is modulated was estimated theoretically to be equivalent to a MDA of 8×10^{-8} for the methane laser and 6×10^{-8} for the nitrous oxide diode. No direct measurement of the RAM contribution was made.

TDL Current/Temperature Controller

The current controller (ILX LDX-3207) has a rated noise performance of 0.01 mA rms at an operating current of 100 mA. To verify this, the noise was measured across a 10Ω resistor (typical resistance of a laser diode) with the spectrum analyser (MI TF2371). The noise was measured as $7 \times 10^{-7} \text{ V} / \sqrt{\text{Hz}}$ which corresponds to 0.007 mA at 100 mA. This is equivalent to a minimum detectable absorption of 6×10^{-9} in the case of the CH_4 laser and 8×10^{-9} for the N_2O diode.

Noise from the temperature controller will be negligible because the time constant associated with thermal changes in the diode mount is large compared with the time required to scan the laser through the feature of interest.

Lock In Amplifier

The lock in amplifier (SR5209) has a quoted input noise of $7 \text{ nV} / \sqrt{\text{Hz}}$. The signal source impedance (50Ω) will contribute an additional $1 \text{ nV} / \sqrt{\text{Hz}}$. The lock in amplifier noise is equivalent to a MDA of 7×10^{-9} .

Noise Type	Noise Contribution/MDA (CH ₄ Laser)	Noise Contribution/MDA (N ₂ O Laser)
Fringing	7×10^{-8}	7×10^{-8}
1/f	1.5×10^{-7}	1×10^{-7}
Detector/Amplifier	1.4×10^{-8}	3.4×10^{-8}
RAM	8×10^{-8}	6×10^{-8}
Current Controller	6×10^{-9}	8×10^{-9}
Temperature Controller	Negligible	Negligible
Lock In Amplifier	3×10^{-9}	7×10^{-9}

Table 6.3 - Summary of Noise Sources

Combined Instrument Noise

The overall instrument noise can be determined by summing the contribution from the different sources discussed above. The combined noise can also be measured directly by measuring the noise on the signal from the instrument output. Noise on the instrument output was measured using two different techniques. The first uses a facility on the lock in amplifier which will measure the noise (RMS deviation from the mean) on the instrument signal at the chosen reference frequency. The second measures the noise on a recorded spectrum. Table 6.4 summarises the findings for all three techniques.

Measurement Method	Combined Noise/MDA (CH ₄ Laser)	Combined Noise/MDA (N ₂ O Laser)
Individual Summation	1.7×10^{-7}	1.5×10^{-7}
Lock In	1×10^{-7}	9×10^{-8}
Recorded Spectra	4×10^{-7} (no averaging)	3×10^{-7} (no averaging)

Table 6.4 - Combined Noise Measurements

It is seen that all methods are in reasonable agreement. Additional noise on the recorded spectrum could in part be due to digitisation noise and RF noise picked up in cables which neither of the other two methods take into account. The smaller noise levels given by the lock in measurement can be explained by the lack of any optical fringing contribution with this technique. For this measurement the noise is determined at a fixed wavelength and therefore a constant current and temperature setting. (100 mA and 31.5 °C for the methane diode and 100 mA and 10.5 °C for the nitrous oxide diode). Fringing will only be generated if the wavelength is scanned.

6.5.3 Analytical Errors

There are many possible sources of analytical errors, including the accuracy of the line parameters and absorption pathlength used in retrieving the concentrations, the measurement of pressure and temperature and interference from nearby lines. These sources will usually be the dominant source of error on the absolute accuracy of the instrument.

Line Parameters

Methane

The line parameters used to generate the synthetic spectrum are taken from 1992 HITRAN Molecular Database [23]. The linestrengths listed for the $2\nu_3$ methane band were taken from work carried out by Margolis [24]. The estimated uncertainty in the linestrengths is 3.5 %.

Pressure broadening coefficients for the ν_3 methane band were measured by Pine [71]. For the Q branch a typical air broadening coefficient is $0.6 \text{ cm}^{-1}/\text{MPa}$. The uncertainty is estimated to be 0.8%.

Nitrous Oxide

The $3\nu_3$ nitrous oxide line parameters are not listed in HITRAN. The linestrengths and pressure broadening coefficients measured at Strathclyde University have been used to generate synthetic spectra. More details about these line parameter measurements can be found in Section 3.3 The uncertainty in the linestrength is estimated to be 5%. The pressure broadening coefficient uncertainty is estimated to be 3%.

Pressure and Temperature

Pressure is recorded using a MKS Baratron (122AA-01000AB). The uncertainty in pressure measurements is specified as 1% at a pressure of 100 Torr.

The temperature of the gas is measured inside the cell with curve matched thermistors (RS Components). The specified uncertainty is specified as 1K at 296K.

The effect of uncertainties in both pressure and temperature measurements on concentration retrievals are quantified in Section 6.5.4.

Absorption Pathlength

The absorption pathlength is determined using the Beer Lambert law (see Section 6.2.3). As such the uncertainty in the pathlength will arise from uncertainties in the linestrength, pressure and the measurement of the absorbance. Details of how the overall uncertainty is determined are given in Section 6.5.4. An additional uncertainty in the retrieved concentration will be caused by the pathlength associated with the optical train outside the multipass cell. For this instrument, the distance covered by the beam outside the cell is 0.7 m. The absorption feature associated with the additional pathlength will be significantly pressure broadened and will increase the observed absorbance by less than 0.1 % (see section 6.3.2).

6.5.4 Combined Uncertainty in Concentration Retrieval

The combined uncertainty in concentration retrievals, resulting from both instrumental noise sources and analytical errors is calculated below. The combined uncertainty in the retrieved concentration, $\delta(\text{obs})$, can be derived from equation (6.3). It is shown to be:

$$\frac{\sigma\delta(\text{obs})}{\delta(\text{obs})} = \left[\left(\frac{\sigma H_2(\text{obs})}{H_2(\text{obs})} \right)^2 + \left(\frac{\sigma H_2(\text{calc})}{H_2(\text{calc})} \right)^2 + \left(\frac{\sigma\delta(\text{calc})}{\delta(\text{calc})} \right)^2 \right]^{1/2} \quad (6.7)$$

$\left(\frac{\sigma\delta(\text{calc})}{\delta(\text{calc})} \right)$ will be zero as this is a user chosen parameter. If we examine the remaining terms on the right hand side:

$\left(\frac{\sigma H_2(\text{obs})}{H_2(\text{obs})} \right)$ is the fractional uncertainty in the peak height of the measured second harmonic spectrum when converted into spectroscopic units. From equation (6.2) it can be shown that:

$$\left(\frac{\sigma H_2(\text{obs})}{H_2(\text{obs})} \right) = \left[\left(\frac{\sigma y}{y} \right)^2 + \left(\frac{\sigma R_i}{R_i} \right)^2 + \left(\frac{\sigma P}{P} \right)^2 \right]^{1/2} \quad (6.8)$$

$\left(\frac{\sigma y}{y} \right)$ is the fractional uncertainty in the peak height of the raw spectrum. This is evaluated by determining the noise levels on the 2f signal. The fractional uncertainty for both methane and nitrous oxide in their natural abundance is listed below.

$\left(\frac{\sigma_{R_i}}{R_i}\right)$ is the uncertainty in the response number used to convert the raw data into spectroscopic units. The main uncertainty in determining R_i , assuming the modulation depth is known, will be the uncertainty associated in measuring the absorbance of the calibration feature. A typical calibration feature will have a 1-2 % absorption. An uncertainty of 0.5% would be associated with this measurement assuming a typical minimum detectable absorption of 1×10^{-4} .

$\left(\frac{\sigma_P}{P}\right)$ is the fractional uncertainty in the measured power at the detector. The uncertainty in the power measurement will be negligible ($< 0.1\%$)

	N₂O	CH₄
$\left(\frac{\sigma_y}{y}\right)$	10%	0.1%
$\left(\frac{\sigma_{R_i}}{R_i}\right)$	0.5%	0.5%
$\left(\frac{\sigma_P}{P}\right)$	<0.1%	<0.1%
$\left(\frac{\sigma_{H_2(\text{obs})}}{H_2(\text{obs})}\right)$	10%	0.5%

Table 6.5 Uncertainties associated with the peak height of a spectrum converted into spectroscopic units.

The combined uncertainty in the peak height of the measured spectrum when converted into spectroscopic units is 10% and 0.5% for nitrous oxide and methane respectively. The large uncertainty in the nitrous oxide measurement is mainly due to the poor signal to noise ratio of the recorded spectrum.

$\left(\frac{\sigma_{H_2(\text{calc})}}{H_2(\text{calc})}\right)$ is the fractional uncertainty in the calculated second harmonic synthetic spectrum. The uncertainty arises from two sources:

$$\left(\frac{\sigma_{H_2(\text{calc})}}{H_2(\text{calc})}\right) = \left[\left(\frac{\sigma_B}{B}\right)^2 + \left(\frac{\sigma_{2f}}{2f}\right)^2 \right]^{1/2} \quad (6.9)$$

The first term on the right hand side is the uncertainty associated with generating the synthetic direct absorption spectrum. This results from uncertainties in the linestrengths and pressure broadening coefficient used and measurements of pressure and temperature. The second term is the additional uncertainty created by generating the second harmonic component of the synthetic direct absorption feature. The main uncertainty in this conversion will be due to errors in the modelled modulation depth. Both of these terms will now be evaluated.

a) $\left(\frac{\sigma_B}{B}\right)$ has been defined as the fractional uncertainty in the calculated simulated spectrum, which can be defined as follows:

$$B(\nu) = \alpha(\nu) \cdot \delta(\text{calc}) \cdot l \quad (6.10)$$

The uncertainty is therefore:

$$\left(\frac{\sigma_B}{B}\right) = \left[\left(\frac{\sigma_\alpha}{\alpha}\right)^2 + \left(\frac{\sigma_l}{l}\right)^2 \right]^{1/2} \quad (6.11)$$

It should be noted that there is no uncertainty in the concentration, $\delta(\text{calc})$, used in calculating the synthetic spectrum as this is a user chosen parameter.

Considering the first term on the right hand side, $\left(\frac{\sigma_\alpha}{\alpha}\right)$, it can be shown that [72]:

$$\alpha(\nu, T, p) = S(T) \cdot g(\nu - \nu_0, T, p) \cdot N(T) \quad (6.12)$$

where $S(T)$ is the absorption linestrength, $g(\nu - \nu_0, T, p)$ is the lineshape function and $N(T)$ is the number density of the gas. Each of these three factors will now be considered.

$S(T)$, the absorption linestrength, can be defined as [72]:

$$\begin{aligned} S(T) &= S(T_{\text{REF}}) \left[\frac{Q(T_{\text{REF}})}{Q(T)} \right] \exp \left[1.439 \nu \frac{(T - T_{\text{REF}})}{TT_{\text{REF}}} \right] \\ &= S(T_{\text{REF}}) \Delta T \end{aligned} \quad (6.13)$$

where $S(T_{\text{REF}})$ is the linestrength at a reference temperature of 296K and $Q(T)$ is the total internal partition sum, which is approximated by the following polynomial expansion in T [72].

$$Q(T) = a_0 + a_1 T + a_2 T^2 + a_3 T^3 \quad (6.14)$$

The coefficients of this polynomial expansion for the molecule of interest can be found in the HITRAN database [23]. They are listed below.

From equation (6.13) it can be seen that the uncertainty in the linestrength at a given temperature, T , is given by:

$$\left(\frac{\sigma_{S(T)}}{S(T)} \right) = \left[\left(\frac{\sigma_{S(T_{\text{REF}})}}{S(T_{\text{REF}})} \right)^2 + \left(\frac{\sigma_{\Delta(T)}}{\Delta(T)} \right)^2 \right]^{1/2} \quad (6.15)$$

where

$\left(\frac{\sigma_{S(T_{\text{REF}})}}{S(T_{\text{REF}})} \right)$ is the uncertainty in the linestrength at 296 K.

This is taken from the HITRAN database [23] in the case of methane and has been estimated in Section 3.3 for nitrous oxide.

$\left(\frac{\sigma_{\Delta(T)}}{\Delta(T)}\right)$ can be found by differentiating $\Delta(T)$ with respect to the temperature, T , and multiplying it by the uncertainty in the measured temperature, σ_T , i.e.

$$\left(\frac{\sigma_{\Delta(T)}}{\Delta(T)}\right) = \frac{1}{\Delta(T)} \cdot \frac{d\Delta}{dT} \cdot \sigma_T \quad (6.16)$$

The uncertainty in the measured temperature using curve matched thermistors (RS 76549) is specified as 1K at 296K. $\frac{d\Delta}{dT}$ has been evaluated as $0.8\%K^{-1}$ for N_2O and $0.1\%K^{-1}$ for CH_4 .

	N_2O	CH_4
$\left(\frac{\sigma_{S(T_{REF})}}{S(T_{REF})}\right)$	5%	3.5%
$\left(\frac{\sigma_{\Delta(T)}}{\Delta(T)}\right)$	0.27%	0.03%
$\left(\frac{\sigma_{S(T_{REF})}}{S(T_{REF})}\right)$	5%	3.5%

Table 6.6 - Uncertainties associated with the Linestrength

	N_2O	CH_4
a_0	-9.5291	-17.475
a_1	15.719	0.95375
a_2	-1.2063×10^{-2}	3.9758×10^{-3}
a_3	5.3781×10^{-5}	-8.1837×10^{-7}

Table 6.7 - Polynomial Expansion Coefficients for Temperature Dependence of Partition sum

$g(\nu - \nu_0, T, p)$ is the lineshape factor. At the sample pressures used for measurement the lineshapes are Voigt. An uncertainty in the lineshape will be caused by the uncertainty in the Lorentzian component of the Voigt line. The Lorentzian component is determined by the product of the pressure broadening coefficient and the sample pressure. As such, the uncertainty in the Lorentzian component will be:

$$\left(\frac{\sigma_{\gamma_L}}{\gamma_L}\right) = \left[\left(\frac{\sigma_P}{P}\right)^2 + \left(\frac{\sigma_{\gamma_{\text{broad}}}}{\gamma_{\text{broad}}}\right)^2 \right]^{1/2} \quad (6.17)$$

where $\left(\frac{\sigma_P}{P}\right)$ is the uncertainty in the pressure measurement and $\left(\frac{\sigma_{\gamma_{\text{broad}}}}{\gamma_{\text{broad}}}\right)$ is the uncertainty in the pressure broadening coefficient. Additional uncertainties in the lineshape can be introduced by collisional narrowing and line mixing effects. However, they have been shown to be negligible at the pressures used (50-100 Torr) [71].

	N₂O	CH₄
$\left(\frac{\sigma_P}{P}\right)$	1%	1%
$\left(\frac{\sigma_{\gamma_{\text{broad}}}}{\gamma_{\text{broad}}}\right)$	3%	0.8%
$\left(\frac{\sigma_{\gamma_L}}{\gamma_L}\right)$	3.1%	1.3%
$\left(\frac{\sigma_g}{g}\right)$	2.5%	1%

Table 6.8 - Uncertainties associated with the Lineshape

$N(T)$, the number density, is defined as follows:

$$N(T) = N_0 \left(\frac{296}{T} \right) \quad (6.18)$$

where N_0 is Loschmidts' number. It has been shown by Martin [73] that the uncertainty in the number density is:

$$\left(\frac{\sigma_N}{N} \right) = \left[\left(\frac{\sigma_P}{P} \right)^2 + \left(\frac{\sigma_T}{T} \right)^2 \right]^{1/2} \quad (6.19)$$

Table 6.9 list the uncertainties associated with the number density.

	N₂O	CH₄
$\left(\frac{\sigma_P}{P} \right)$	1%	1%
$\left(\frac{\sigma_T}{T} \right)$	0.3%	0.3%
$\left(\frac{\sigma_N}{N} \right)$	1%	1%

Table 6.9 - Uncertainties associated with the Number Density

The second term on the right hand side of equation (6.11), $\left(\frac{\sigma_l}{l}\right)$, is the fractional uncertainty in the absorption pathlength. The optical pathlength is determined by measuring the absorption of a gas standard. The pathlength is then calculated by using the Beer Lambert Law. The uncertainty will be given by:

$$\left(\frac{\sigma_l}{l}\right) = \left[\left(\frac{\sigma_A}{A}\right)^2 + \left(\frac{\sigma_\alpha}{\alpha}\right)^2 + \left(\frac{\sigma_{CH_4}}{C}\right)^2 \right]^{1/2} \quad (6.20)$$

where $\left(\frac{\sigma_A}{A}\right)$ is the uncertainty in the measured absorbance, $\left(\frac{\sigma_\alpha}{\alpha}\right)$ is the uncertainty in the absorption coefficient and $\left(\frac{\sigma_{CH_4}}{C}\right)$ is the uncertainty in the partial pressure of the gas standard (100 ppm methane/air mixture NPL Cylinder 29 - 100.1 ppm +/- 0.5%).

$\left(\frac{\sigma_A}{A}\right)$	0.5%
$\left(\frac{\sigma_\alpha}{\alpha}\right)$	3.5%
$\left(\frac{\sigma_{CH_4}}{C}\right)$	0.5%
$\left(\frac{\sigma_l}{l}\right)$	3.6%

Table 6.10 - Uncertainties associated with the optical pathlength

It is seen that the largest uncertainty in the optical path arises from the uncertainty in the absorption coefficient. The uncertainties on the absorption coefficient were assessed previously and result mainly from uncertainties in the linestrengths.

b) $\left(\frac{\sigma_{2f}}{2f}\right)$, is defined as the uncertainty in generating the second harmonic component of the synthetic spectrum created in HITRAN. In Section 5.3 it was shown that residuals between the calculated second harmonic component and the component predicted by theory could be reduced to less than 0.2%.

Table 6.11 lists the combined uncertainty in generating the second harmonic synthetic spectrum. The main uncertainty arose from the linestrengths used.

	N ₂ O	CH ₄
$\left(\frac{\sigma_{2f}}{2f}\right)$	0.2%	0.2%
$\left(\frac{\sigma_B}{B}\right)$	5.2%	6.7%
$\left(\frac{\sigma_{H_2(\text{calc})}}{H_2(\text{calc})}\right)$	5.2%	6.7%

Table 6.11 - Uncertainties associated with generating a synthetic second harmonic spectrum

6.5.5 Overall Uncertainty and Conclusions

Table 6.12 lists the overall uncertainties (1σ) in concentration retrievals of methane and nitrous oxide. In nitrous oxide the main uncertainty arises from the poor signal to noise ratio of the recorded spectra. In methane the uncertainty is dominated by the uncertainty in the linestrengths used in generating the synthetic spectrum. These uncertainties should be multiplied by a coverage factor of two to give a confidence level of 95%.

	N₂O	CH₄
$\left(\frac{\sigma_{H_2}(\text{obs})}{H_2(\text{obs})}\right)$	10%	0.5%
$\left(\frac{\sigma_{H_2}(\text{calc})}{H_2(\text{calc})}\right)$	6.7%	5.2%
$\frac{\sigma\delta(\text{obs})}{\delta(\text{obs})}$	12%	5%

Table 6.12 - Uncertainties associated with Concentration Retrievals for N₂O and CH₄

A major source of uncertainty for both N₂O and CH₄ measurements was the uncertainty in the linestrength. This highlights the need for an improvement of the spectral parameters in the near-infrared, which has largely been ignored by spectroscopists in the past.

6.6 Future Developments and Conclusions

It has been shown that near-infrared technology and second harmonic WMS in the 50 - 100 kHz region will allow absorptions as small as 1×10^{-7} to be detected. This performance was achieved with two different laser systems, one for methane detection and the other for nitrous oxide. Instrument precision equivalent to 560 ppt of methane and 22 ppb of nitrous oxide was achieved, with measurement uncertainties of 5% and 12% respectively. In the case of methane, this is comparable to the best sensitivities achieved in the mid-infrared. Sensitivity for nitrous oxide was limited by the weak line strengths associated with second overtone transitions.

A field test and intercomparison with two Flame Ionisation detectors showed that the retrieved concentrations from the TDLAS were within the measurement uncertainties of the other two instruments. It also showed that instrumental drift was negligible and highlighted the inherent stability of the Herriott cell.

This work has successfully been used in a three way proposal between the University of Strathclyde, Cambridge University and the National Physical Laboratory, to develop and build the next generation near-infrared TDLAS for measurements on a balloon platform [52]. The instrument will measure H_2O , CO_2 and CH_4 and will provide important information about troposphere/stratosphere transport mechanisms. The next generation multipass arrangement, the astigmatic Herriott cell, has already been designed and tested for this instrument (Section 4.3). This cell has all the inherent stability properties of the conventional Herriott cell but allows an increased pathlength to be supported in a smaller cell mode volume. The next generation electronic system has been designed and built at the National Physical Laboratory. The electronics have been substantially miniaturised (they fit in a $30 \times 30 \times 30 \text{ cm}^3$ rack), have low power consumption (0.25 Amps from a 28 V d.c supply) and allow spectra to be recorded once every second. Initial testing has shown that the electronics provide similar noise performance to the commercial set up described here.

Future developments in TDLAS instrumentation include the extension of DFB laser technology out to wavelengths between 2 - 4 μm . Laboratory results indicate that III-V antimonide lasers could provide 1-2 mW single mode operation at temperatures accessible to Peltier coolers ($-60 \text{ }^\circ\text{C}$). [12] This would allow the fundamental

transitions of many gas species to be detected. This could improve detection levels by two or more orders of magnitude.

Chapter 7-

Coolable Herriott Cell

7.1 Introduction

This chapter describes the design, construction and operation of a coolable Herriott Cell. The cell can be operated in two cooling modes. The first involves the circulation of pure ethanol between a liquid nitrogen heat exchanger and the cell jacket. Using this technique the cell can be cooled down to a temperature of $\sim 200\text{K}$. This is similar to the temperatures one would encounter in the upper troposphere/lower stratosphere. As such, the cell would have a useful application in assessing the temperature dependence of the molecular line parameters (line widths, line strengths and absorbance cross sections) for gas species that play an important role in atmospheric chemistry.

The second cooling technique operates in a collisional cooled (or diffusive trapping) mode. The cell temperature is cooled down to $\sim 77\text{K}$ by circulating liquid nitrogen around the jacket. A small quantity of condensable spectroscopic gas is introduced into a higher pressure of non condensable gas (usually helium) contained in the cooled cell. The helium is cooled to the cell wall temperatures through collisions. The spectroscopic gas molecules are rapidly cooled to this temperature via collisions with the helium, and remain trapped in a non equilibrium super cooled state during slow diffusion through the helium. The main application of this technique is to simplify congested spectra by changing the Boltzmann populations of the vibrational and rotational states.

A Herriott multipass optical arrangement is used to enhance the absorption pathlength within the cooled cell. The enhanced pathlength, combined with near-infrared TDLAS technology allow absorptions as small as 1×10^{-5} to be detected.

This work was carried out jointly with Kevin Smith, a member of the group at Strathclyde University.

7.2 Coolable Herriott Cell Spectrometer

7.2.1 Coolable Cell

Figure 7.1 shows a schematic of the coolable cell.

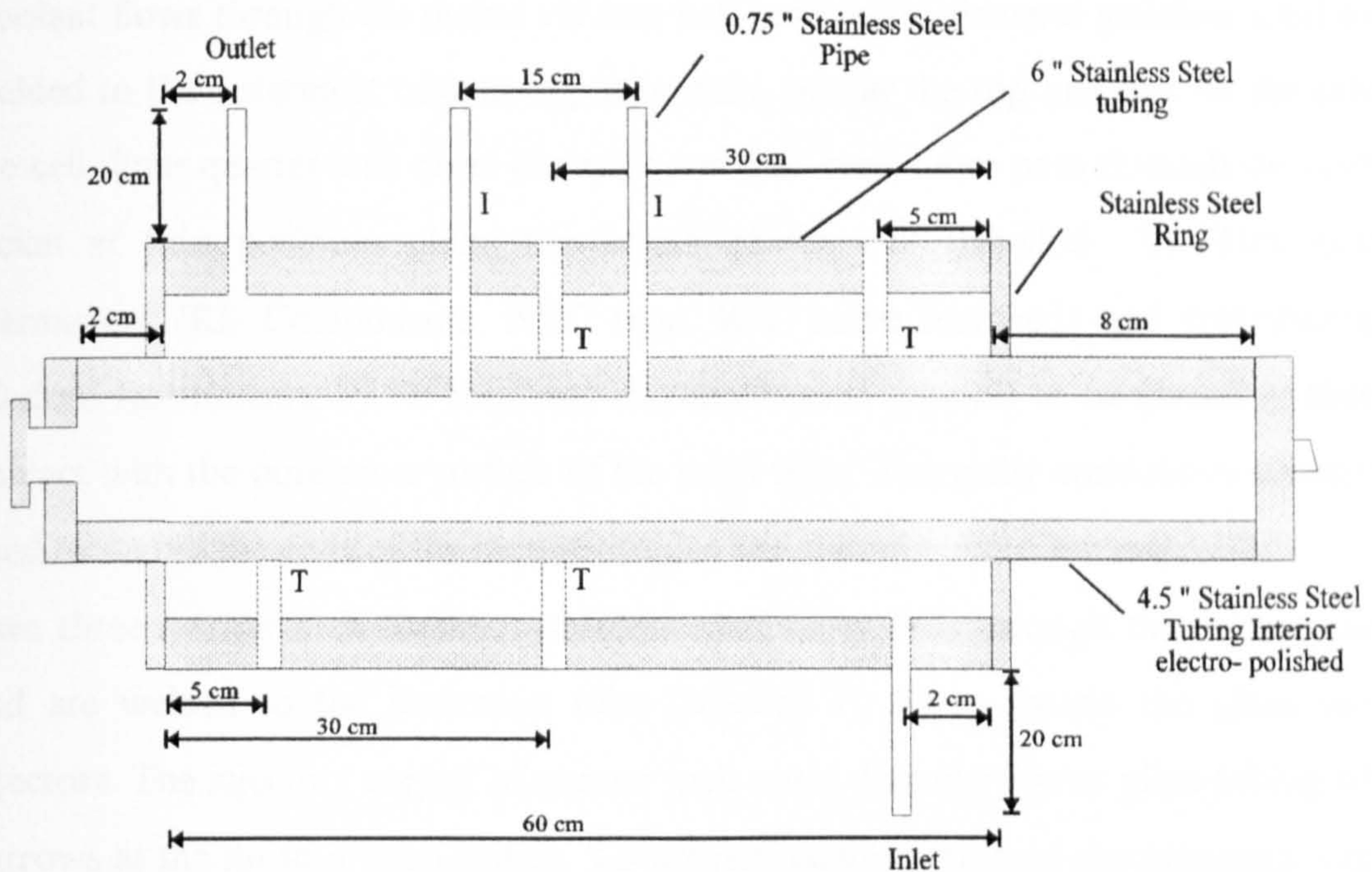


Figure 7.1 - Schematic of the Coolable Cell

The main body of the cell is constructed from concentrically welded stainless steel tubing. An annulus at each end of the tubes forms a jacket of approximately five litres volume. The ends of the inner tube protrude from the outer tube by 2 cm and 8 cm. The 8cm end is closed with a welded stainless steel disc. The disc has a 1.9 cm diameter aperture set 2.3 cm off centre to allow the entrance and exit of the infra-red beam. The aperture is countersunk at a diameter of 2.9 cm and 0.4 cm to house a one inch diameter 5° wedged calcium fluoride window. A viton O-ring provides an evacuable seal. A nichrome wire heater maintains the temperature of the window and O-ring above 276K. This ensures that atmospheric water vapour from outside the cell

does not condense onto the window and maintains the O-ring at its specified operating temperature.

The 2cm end has a 10cm internal diameter welding flange attached. A removable end plate is secured to this flange by 18 bolts. A vacuum seal is provided by a copper gasket, which is specified to operate within the temperature range 3 to 723 K. At the centre of the end plate is a 5 cm diameter pumping aperture, which has an Edwards NW-50 type stainless steel flange. A nichrome wire heater is used to ensure a suitable operating temperature (263 - 473 K) for the fluoroelastomer O-ring vacuum seal.

Coolant flows through the jacket via two half inch outer diameter stainless steel tubes welded to the outermost tube at opposite ends, one at the top and one on the side of the cell. Four quarter inch outer diameter stainless steel tubes pass through the cooling jacket at four positions along the length of the cell (labelled T). This enables thermistors (RS Components, NTC type, R-T curve matched) and thermocouples (Oxford Instruments, PTFE insulated chromel/alumel type-K) to be placed in thermal contact with the outermost surface of the inner tube. Thermally conductive adhesive is used to secure the ends of the thermocouples and thermistors to the cell.

Two three quarter inch diameter stainless steel tubes pass through the cooling jacket and are welded to the innermost tube (labelled I). These house the glass vapour injectors. The injectors consist of quarter inch outer diameter Pyrex glass tubing which narrows at the tip to a 1mm orifice. To prevent condensation of spectroscopic vapour in the injectors an electrical heater surrounds each injector tip. Each heater consists of ~ 5cm of nichrome resistance wire in thermal contact with an aluminium foil cryotube. PTFE tape secures the heaters in place and provide electrical insulation from the cryotubes. Thermistors are attached to the top and bottom of the heater assemblies to allow the injector temperatures to be monitored during operation. Figure 7.2 shows a schematic of the vapour injectors. The injectors are housed in half inch outer diameter Pyrex glass tubing. Vacuum seals between the quarter and half inch Pyrex tubes and the half inch Pyrex tubes and the welded stainless steel feed throughs are located at the end furthest from the body and are formed from epoxy resin. A PTFE foam ring is placed between the two Pyrex tubes preventing convection between the cell and the inside of the half inch glass tube when the heaters are operational. The injector tips protrude past the interior surface of the cell by a few millimetres.

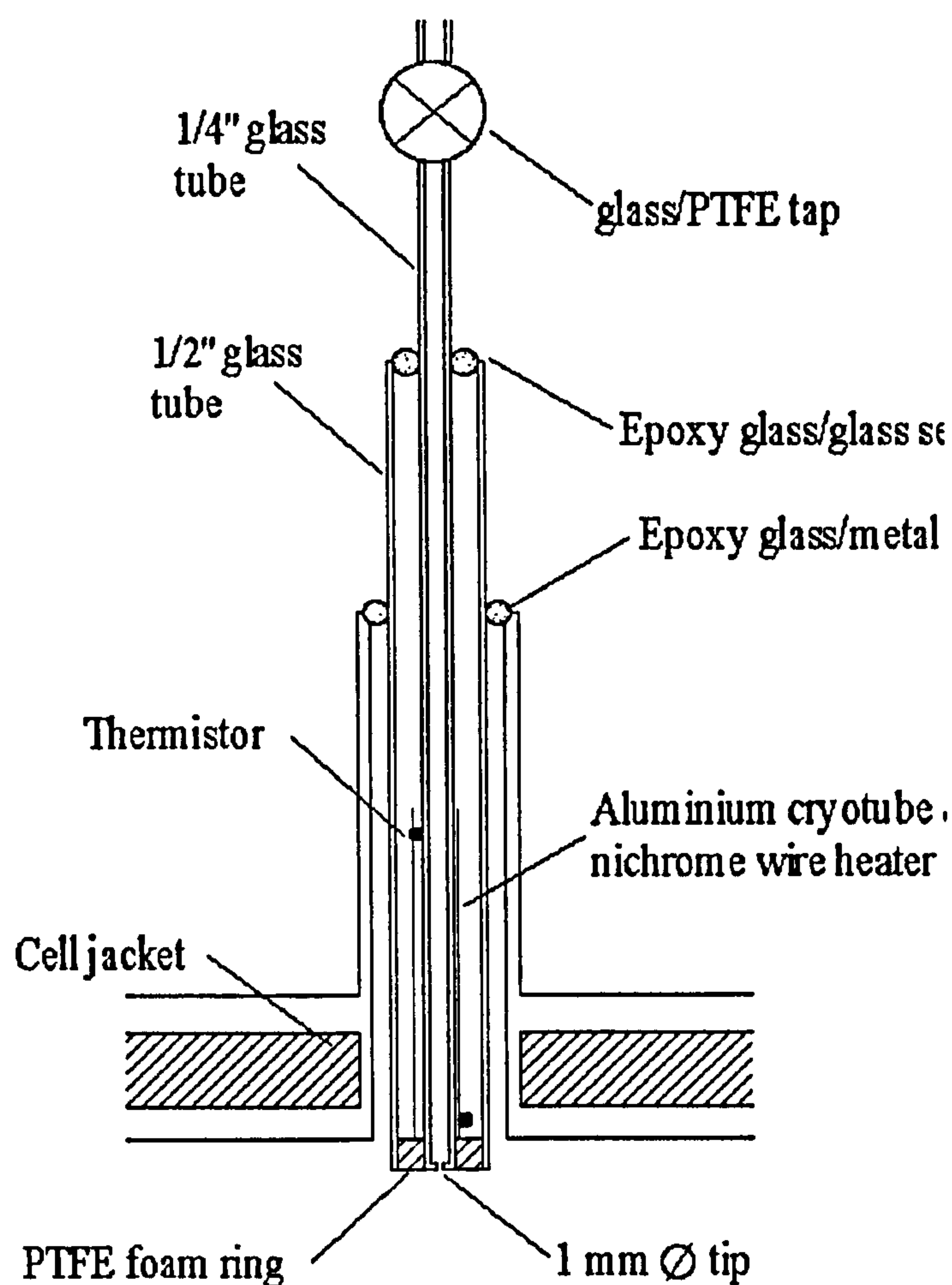


Figure 7.2 - Schematic of the Vapour Injectors

7.2.2 Herriott Cell

Pathlength enhancement is achieved with a Herriott multipass cell. The advantages of the Herriott cell over other multipass cell designs has been well documented elsewhere in the thesis (Chapter 4). Two inch diameter spherical concave mirrors with 30 cm radii of curvature (Ealing Cat. No. 34-9753) provide a 22 pass solution at 34.27 cm separation. A 4mm diameter coupling hole, placed 2 cm off axis, allows beam entrance and exit. The mirrors had a protected aluminium coating giving a quoted reflectivity of $\sim 95\%$ at $1.5 \mu\text{m}$.

Figure 7.3 shows the modelled spot pattern of this solution.

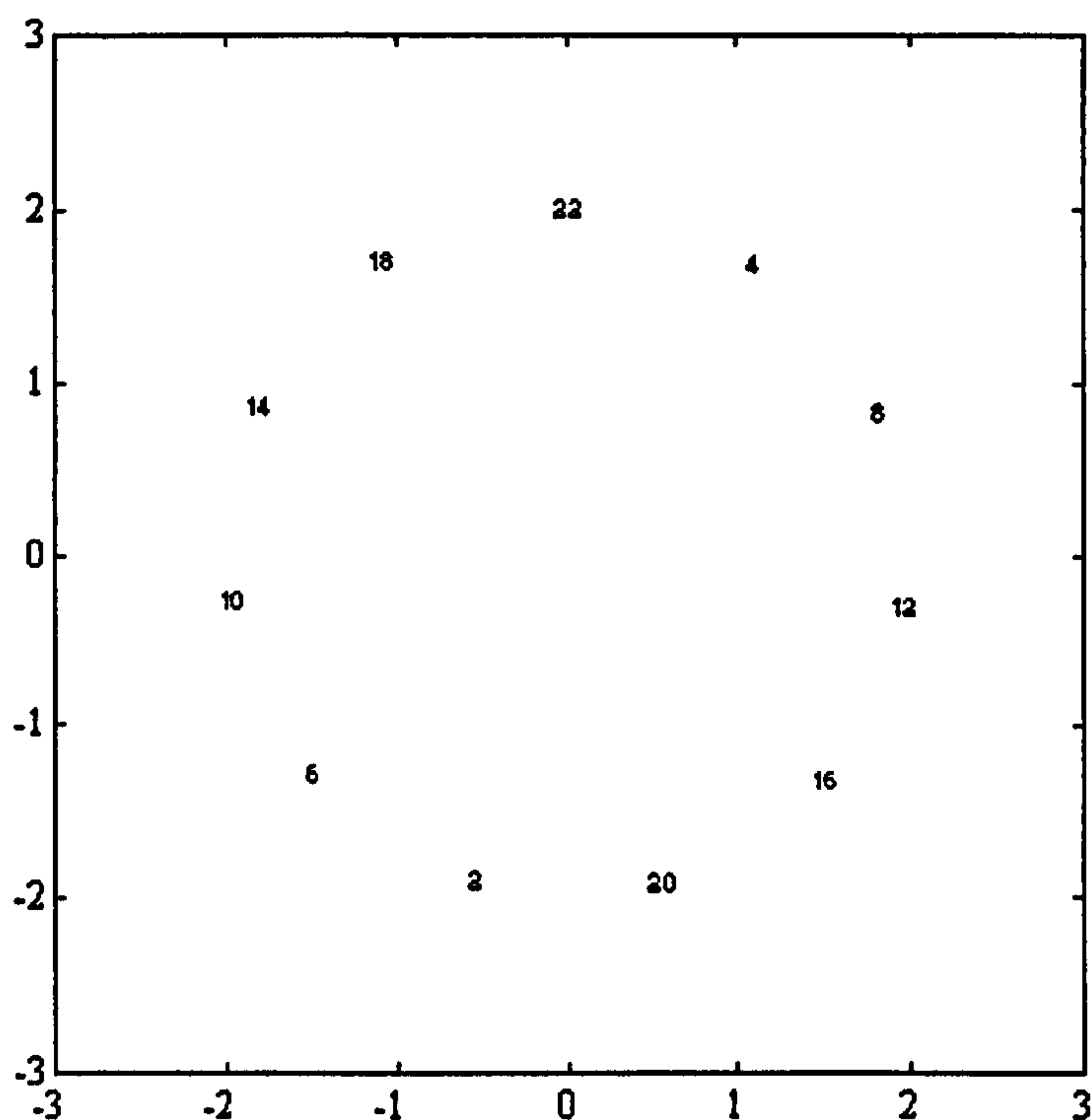


Figure 7.3 - Modelled Spot Pattern ($N = 22$, $R = 30\text{cm}$, $d = 34.27\text{ cm}$)

It was shown in Section 4.2 that the position of the exit spot depends only on the separation between the two mirrors (assuming the radii of curvature does not change). It is therefore important that the mirror separation is preserved over the range of operating temperatures encountered (77K to 288 K). To this end, the mirrors were mounted in a balanced frame made from INVAR 36 and Stainless Steel. **Figure 7.4** shows a schematic of the Herriott cell frame. The frame consists of three INVAR bars, 50 cm long, secured at each end by 9.6cm diameter 2 cm thick circular stainless steel plates. The frame sits on four PTFE feet allowing easy transfer of the frame in and out of the coolable cell. A third stainless steel plate is mounted on the INVAR bars and can be moved to and locked at any position along the frame. A clearance slot (not shown), 2.5 cm wide and 1 cm deep, has been machined into one of the end plates to allow it past the injector tips. The Herriott cell mirrors are mounted, one on the movable slide and one on the end plate, with three locating bolts. PTFE foam pads cushion the mirror and allow for thermal expansion or contraction.

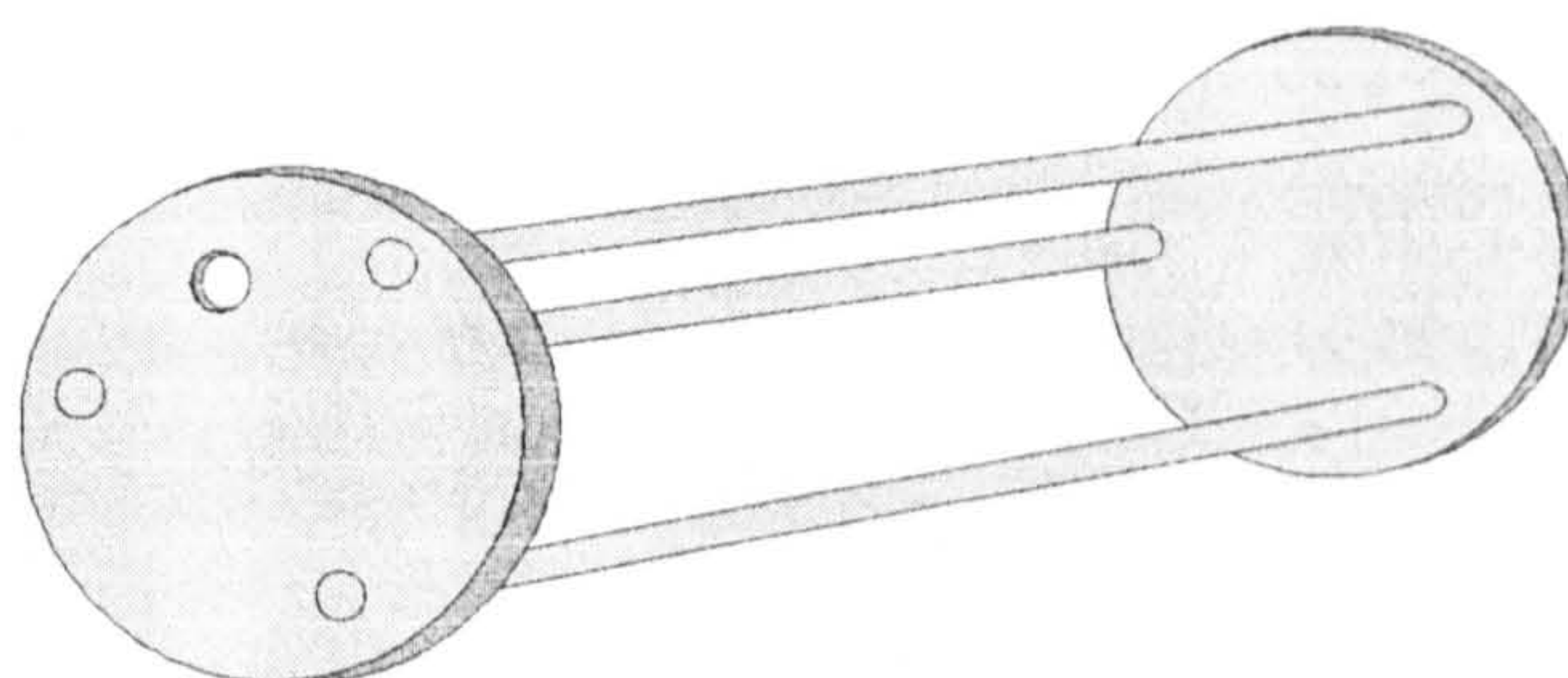
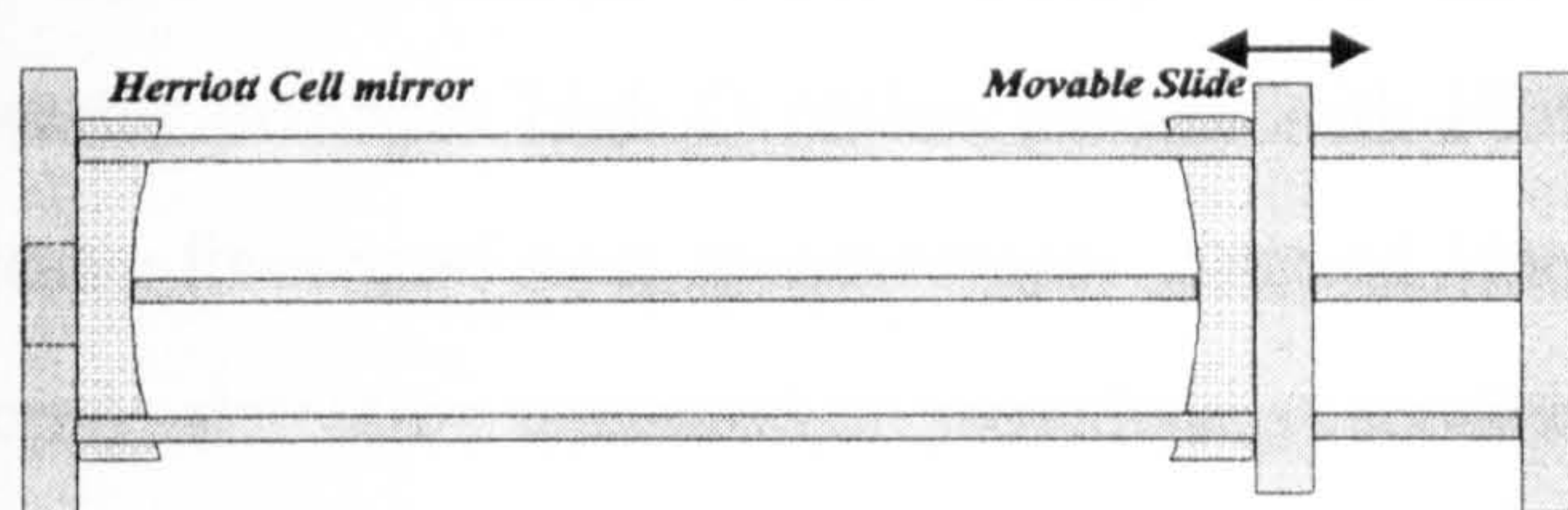


Figure 7.4 - Schematic of the INVAR Herriott Cell frame

In a balanced frame of this design the contraction in the length of the INVAR bars is in theory cancelled out by the contraction in the stainless steel end plates thus keeping the mirrors separation constant over a wide range of operating temperatures. The effect of temperature variations on the frame dimensions and mirror radii of curvature has been modelled, allowing any movement in the exit spot to be quantified. For a temperature variation of 200 K the exit spot is shown to move by 238 μm . The dimension of the coupling hole has been made large enough so that such movements can easily be tolerated. These calculations have assumed a homogeneous temperature distribution within the cell.

7.2.3 Spectrometer

The spectrometer operates in the near-infrared, recording absorptions resulting from rotational-vibrational transitions. A near-infrared DFB tunable diode laser operating between 6580 cm^{-1} and 6610 cm^{-1} , was used as a source. The optical train and electronic layout of the spectrometer are identical to the spectrometer described in Section 5.5.2 and Section 5.5.3 in all respects except that the reference cell has been

replaced with a spherical mirror Fabry-Perot Interferometer (TecOptics). The interferometer provides high Q etalon fringes with FSR 7.5 GHz, allowing recorded spectra to be linearized post measurement. Second Harmonic wavelength modulation spectroscopy detection was used to provide high sensitivity.

7.3 Cell Operation and Results

As was stated previously, the cell can be operated either in collisionally cooled or ethanol cooled mode. Each will now be described.

7.3.1 Ethanol Cooling

In ethanol cooled mode, pure ethanol is circulated between a liquid nitrogen heat exchanger and the cell jacket. Ethanol is used because of its extremely low freezing point (157 K). Figure 7.5 shows a schematic of the cooling circuit.

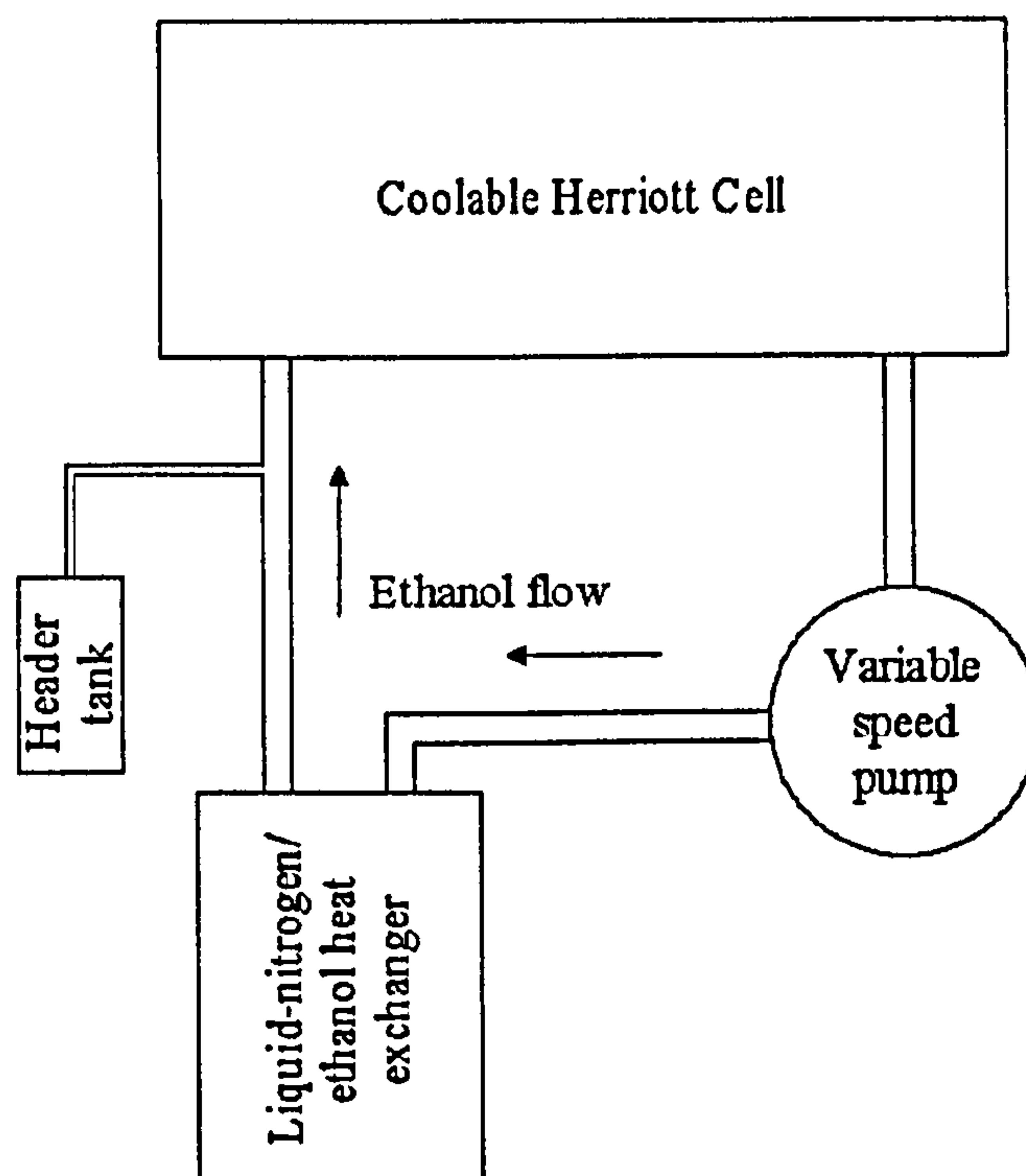


Figure 7.5 - Schematic of the Ethanol Cooling Circuit

The ethanol is circulated between the heat exchanger and cell jacket by a variable speed encapsulated magnetic pump (max. flow rate ~ 50l/min). The pump is placed on the warm side of the heat exchanger. The transfer of the ethanol to and from the cell is via quarter inch outer diameter PTFE tubing. The tubing was connected to the half inch stainless steel inlet/outlet pipes with Swagelock parallel threaded connectors and

adapters. The PTFE pipes have been kept as short as possible and insulated with high density foam so as to reduce heat loss to a minimum.

Figure 7.6 shows a schematic of the heat exchanger.

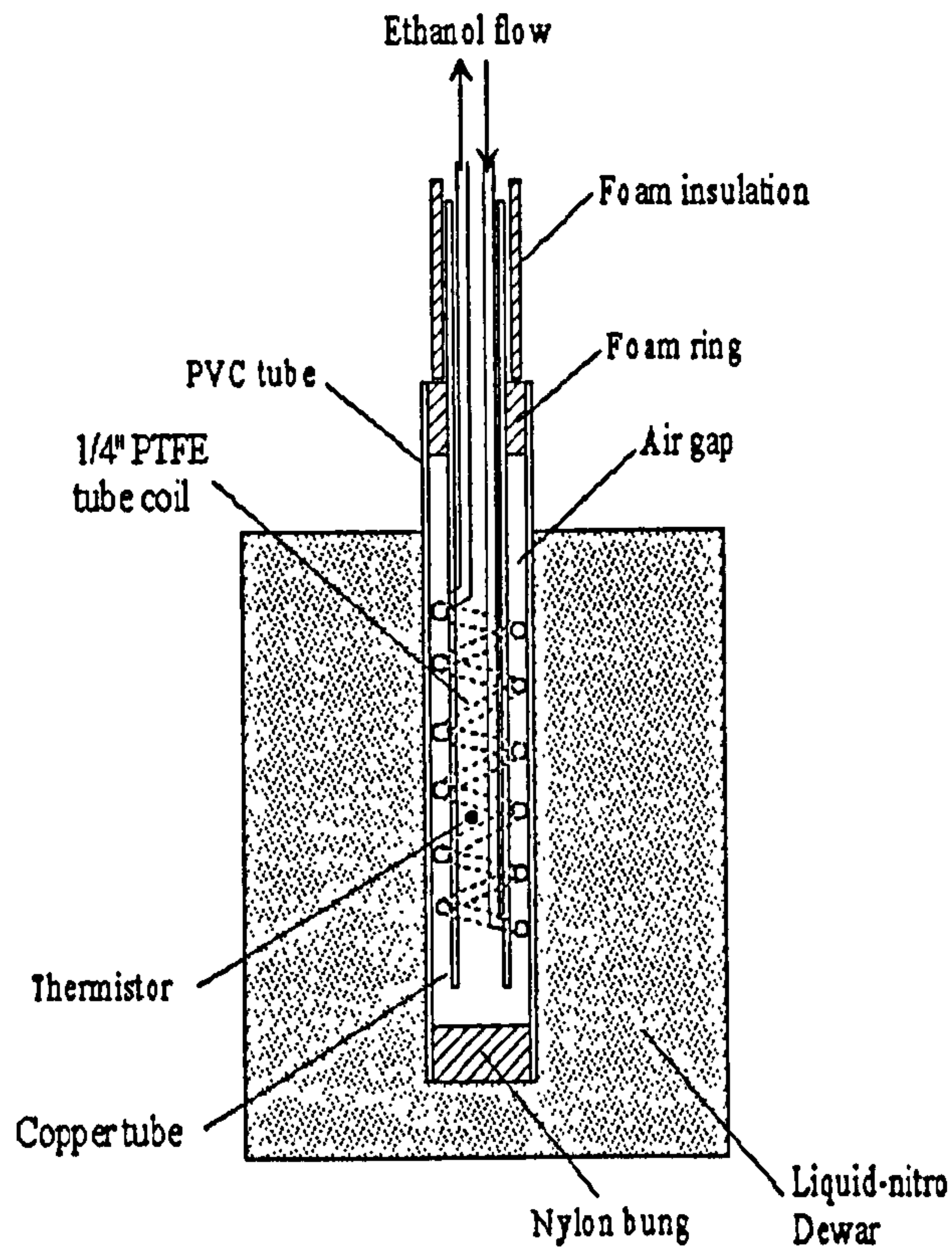


Figure 7.6 - Schematic of the Heat Exchanger

The heat exchanger consists of a 4 m length of quarter inch outer diameter PTFE tubing coiled round a 2.6 cm diameter copper pipe. The pipe and tubing are contained in a 75 cm long, 4.2 cm diameter PVC tube. The tube is plugged at the bottom with a 25 mm thick conical nylon disc. The disc ensures that no liquid nitrogen can seep inside the tube where direct contact with the quarter inch PTFE tubing could potentially freeze the ethanol contained inside. A thermistor was placed in the centre of the copper tube to monitor the temperature of the heat exchanger. The gap between the copper pipe and PVC tube is air filled. The top of the gap is filled with high density insulation foam to prevent convection losses. Swagelock connectors are provided to attach the heat exchanger to the cooling circuit.

The ethanol header tank is maintained at room temperature and allows for thermal expansion/contraction of the ethanol in the circuit. The tank was connected to the cooling circuit via narrow diameter nylon tubing.

During measurement the entire coolable cell was placed inside a wooden box which was packed with polystyrene insulation placed inside plastic bags for easy removal.

7.3.2 Ethanol Cooling Results

Carbon 12 Acetylene ($^{12}\text{C}_2\text{H}_2$) gas was used to assess the performance of the coolable cell. The measurements reported here are strictly qualitative and primarily intended to demonstrate the success of the technique.

Acetylene spectra were recorded at approximately 10 degree intervals between 297 and 200 K. The cell was cooled to approximately 5K below the required temperature, the circulation of the coolant was stopped, and the cell was allowed to slowly warm up to the measurement temperature. This technique minimised any temperature gradient across the length of the cell. The cell was filled from a 1 litre glass bulb of acetylene via a clean glass line attached to one of the gas injectors. A sample pressure of 1 Torr was used. After each measurement the cell was evacuated and refilled.

The laser was scanned between 43 and 100 mA at a temperature of 288 K. Under these operating conditions the laser frequency was tuned between 6591 and 6588 cm^{-1} . Second harmonic detection at 10 kHz was used. The R(14) transition (see Figure 7.7) was used to optimise the modulation depth ($m = 2.2$). Optimisation involves increasing the modulation amplitude until the peak of the $2f$ feature is at a maximum (more details of this technique can be found in section 5.3). During cooling the modulation amplitude had to continually be reduced to maintain the same effective modulation index. This is a consequence of the temperature dependence of the Doppler width. (section 2.1.8)

Figure 7.7 shows a survey spectrum of this region taken at room temperature. The two strong lines at 63 mA and 101 mA have been identified as the R (15) and (R14) $\nu_1 + \nu_3$ transitions. Two weaker line, at 60 mA and 107 mA, have also been identified. They are the P(12) and P(11) $\nu_1 + \nu_2 + \nu_4 + \nu_5$ transitions. The strongest features shown correspond to an absorption of $\sim 6\%$.

Figure 7.8 shows the same spectrum, linearized and wavelength calibrated (see Section 3.3.3 for further details). The spectrum has been expanded allowing the weaker transitions in the spectrum to be seen. These weaker lines are believed to hot band transitions and fundamental transitions of the singly substituted ^{13}C isotope.

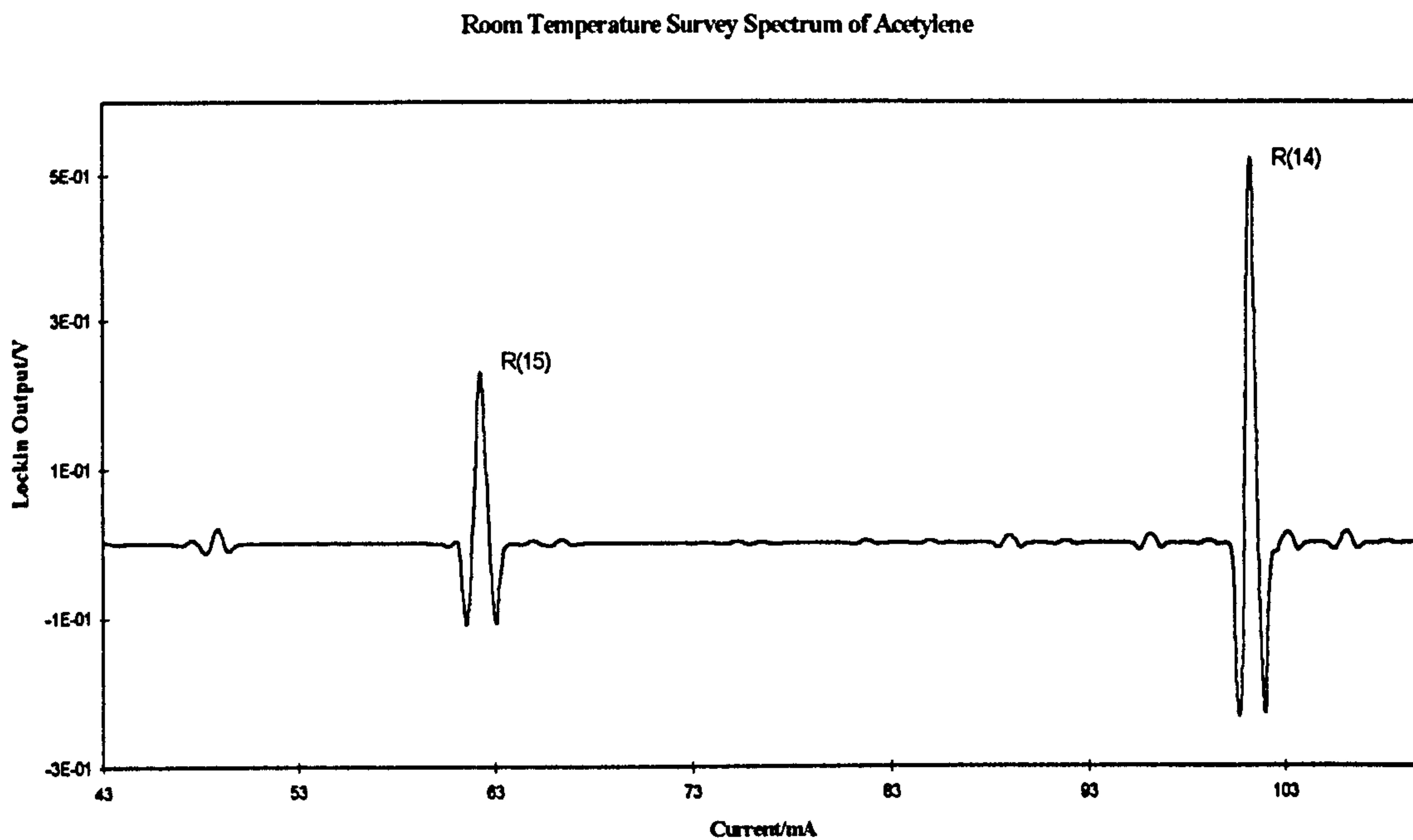


Figure 7.7 - Room Temperature Survey Spectrum of Acetylene

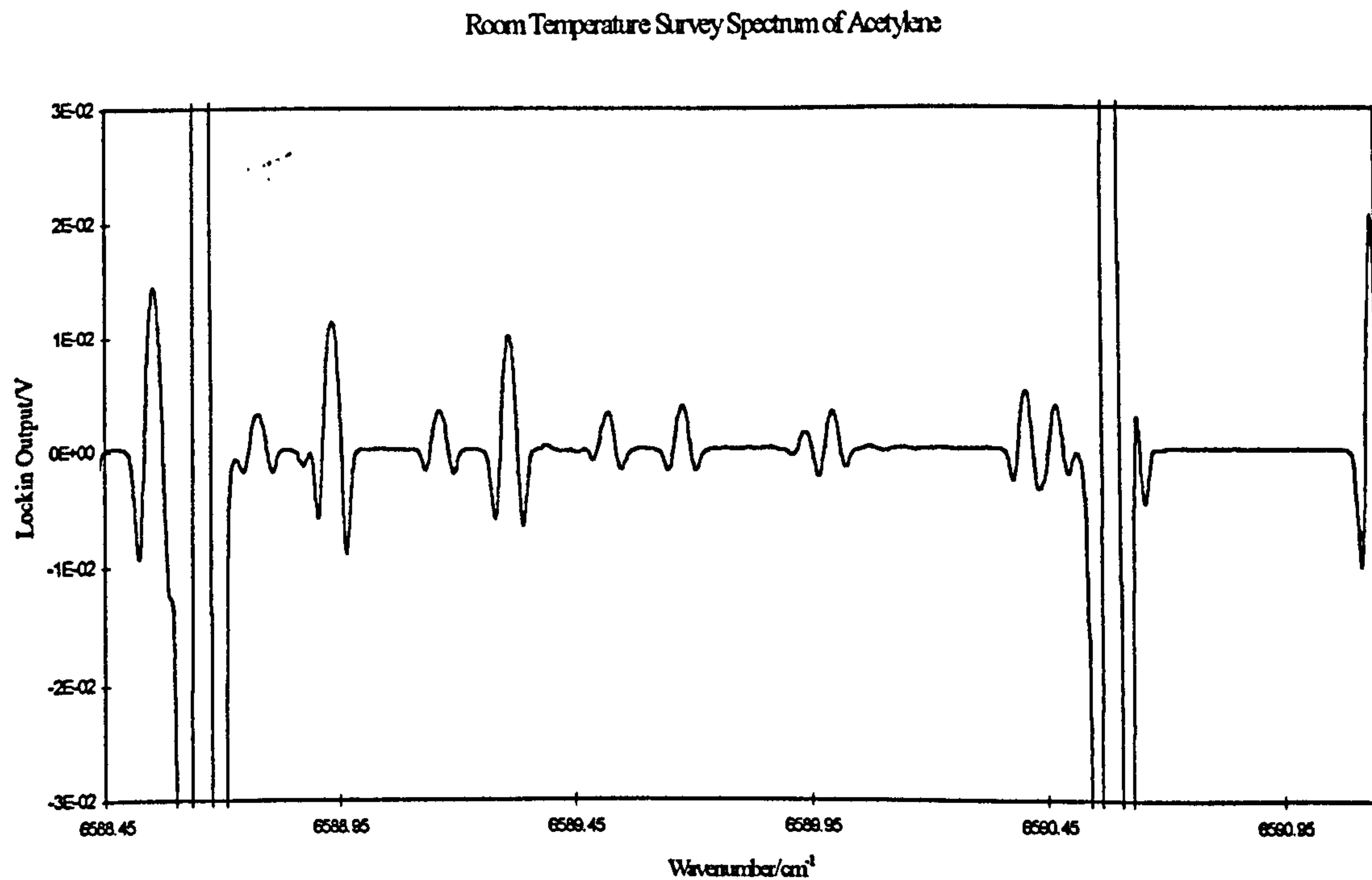


Figure 7.8 - Room Temperature Survey Spectrum of Acetylene (linearized, wavelength calibrated)

Figure 7.9 shows acetylene spectra recorded at temperatures of 216, 256 and 296 K. The strongest feature shown here corresponds to an absorption of 0.1 %. Each spectra took approximately one minute to record. During this time the cell temperature was found to change by less than 0.1 K indicating very good thermal stability. The spectra have been normalised by the absolute laser power falling on the detector so as to preserve the intensity information. It is apparent that the intensity of the feature near 6589.5 cm^{-1} increases at lower temperature indicating that it is an overtone transition with low J number. It is thought that it is most likely to belong to singly substituted C_{13} acetylene. The absorptions near 6589.68 and 6589.95 cm^{-1} decrease at lower temperature. It is concluded that these transitions are likely to be hot bands or high J overtone transitions. None of the transitions have been identified.

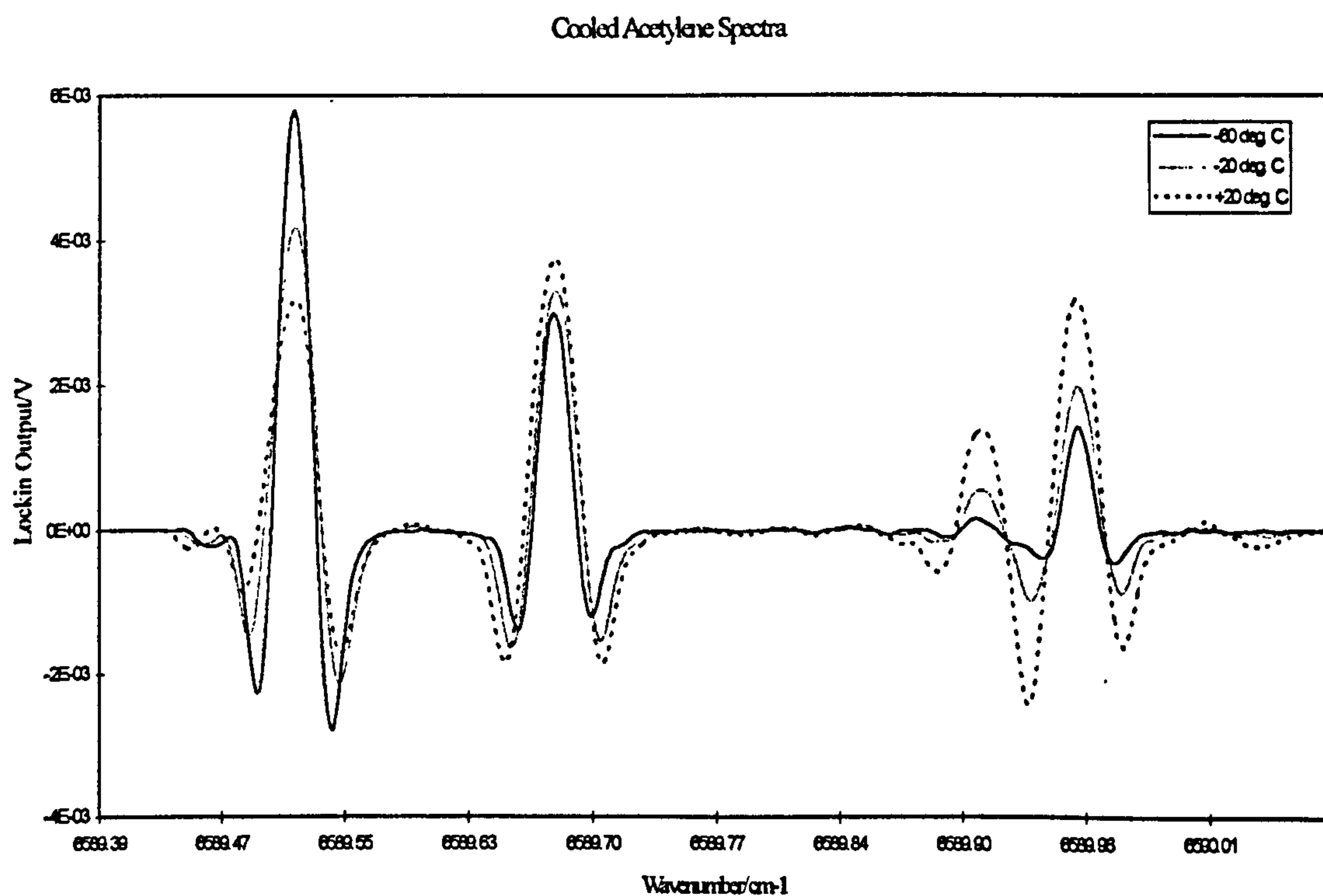


Figure 7.9 - Cooled Acetylene Spectra

The natural log of the line intensity of an isolated unblended hot band transition at 6588.1 cm^{-1} was plotted against the reciprocal of the cell temperature. This is shown in Figure 7.10. As predicted by theory, a linear relationship between these two parameters was found. This indicates that the cell temperature measured at the

outermost surface of the cooling jacket is in good agreement with the temperature of the gas inside.

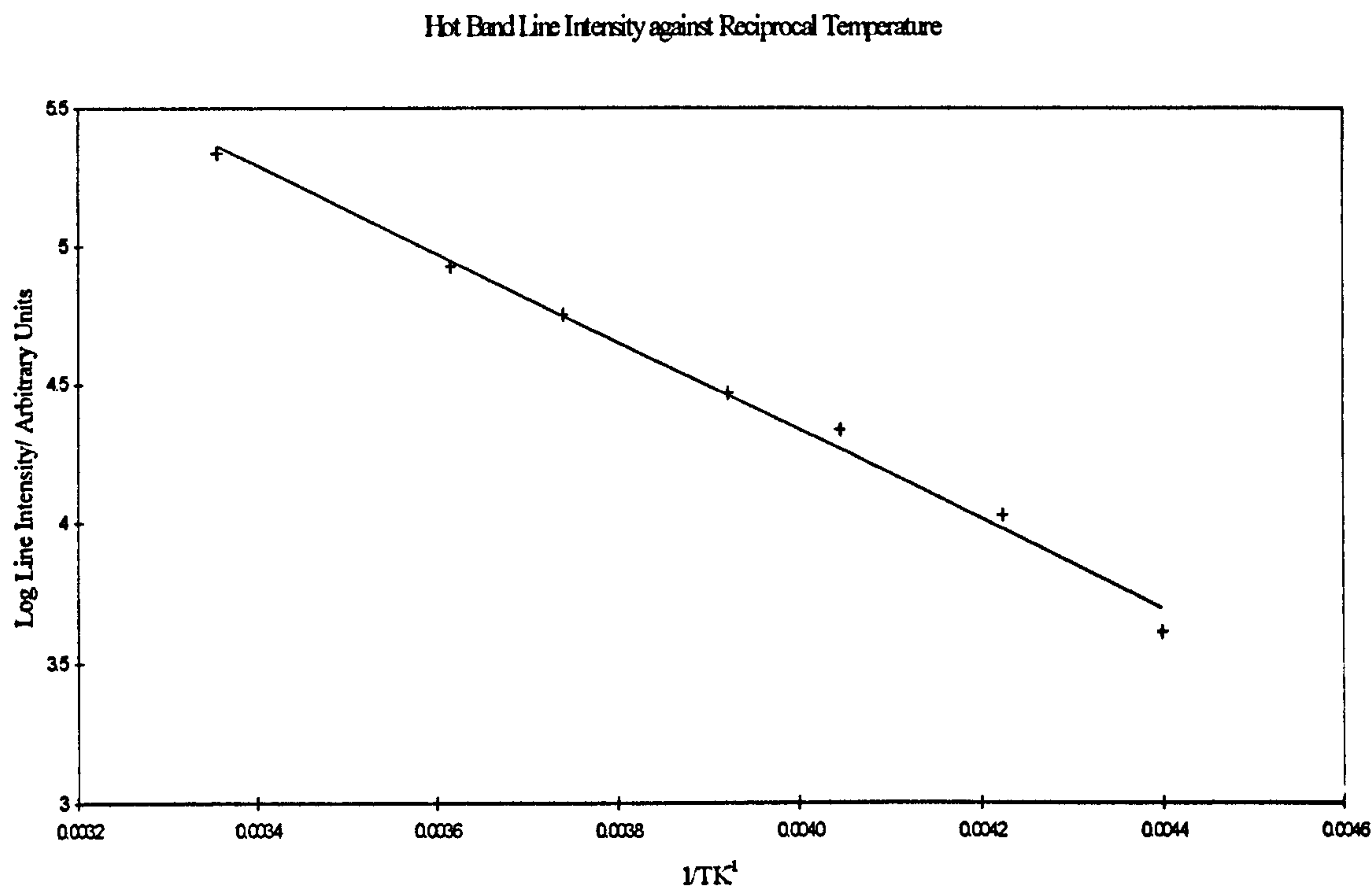


Figure 7.10 - Natural Log of Line Intensity against Reciprocal Temperature

7.3.3 Collisional Cooling

Figure 7.11 shows the schematic of the collisional cooling circuit.

A 120 l pressurised Dewar (Oxford Instruments) was used to flow liquid nitrogen through the cell jacket, cooling the cell to $\sim 77\text{K}$. Transfer to the cell is via quarter inch outer diameter PTFE tubing. Excess nitrogen vapour and liquid escaped from the cell through the top exit pipe which was open to air. A control valve was placed between the Dewar and the cell to enable fine control of the coolant flow rate. A nitrogen cylinder is used for additional pressurising of the Dewar as it empties.

Before cooling, the cell is filled with ~ 20 Torr of helium ensuring that even cooling throughout the cell. takes place. During the cooling process the helium pressure in the cell decreases to ~ 9 Torr.

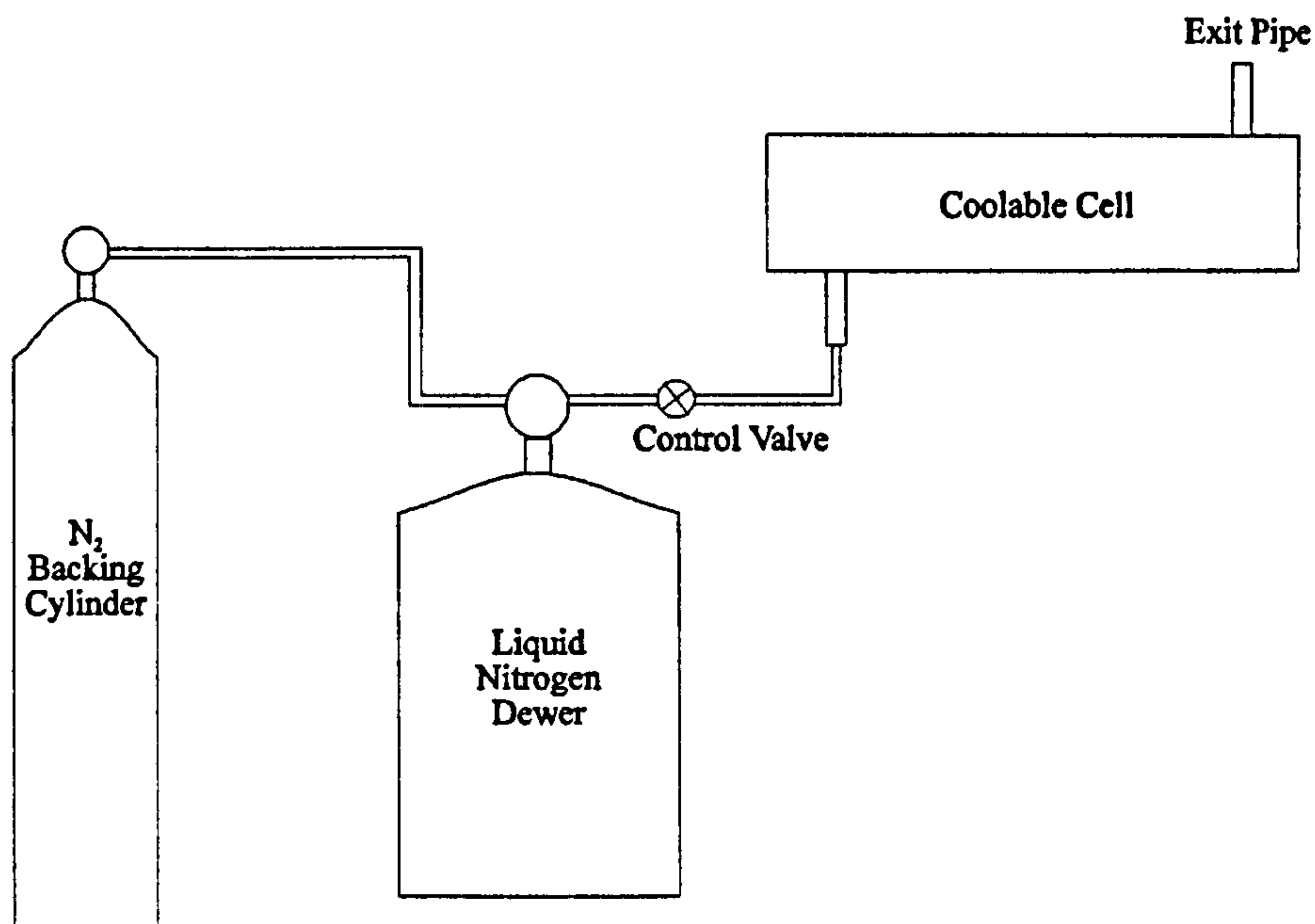


Figure 7.11 - Schematic of the Collisionally Cooling Circuit

7.3.4 Collisional Cooling Results

Carbon 12 Acetylene ($^{12}\text{C}_2\text{H}_2$) gas was used to assess the performance of the coolable cell in collisionally cooled operating mode. Again the measurements reported here are strictly qualitative and primarily intended to demonstrate the success of the technique.

Figure 7.12 shows a schematic of the collisionally cooled gas handling system. A 10% mixture of acetylene in helium was made in a 8 litre reservoir at a pressure of 700 Torr. The helium buffer gas is used to reduce the possibility of molecular clusters of the spectroscopic gas from forming (further details are given later). The pressure in the reservoir is monitored with a 1000 Torr full scale baratron gauge (MKS). The connection between the cell injector and the reservoir was made via a clean glass line. The mixture was injected into the cell such that the absorption of a *strong* feature was maximised. This was achieved by monitoring the infrared absorption signal on an oscilloscope. The other injector was attached to a rotary pump to establish an equilibrium vapour pressure within the cell. The optimum flow rate was determined to be $\sim 0.5 \mu\text{mol s}^{-1}$.

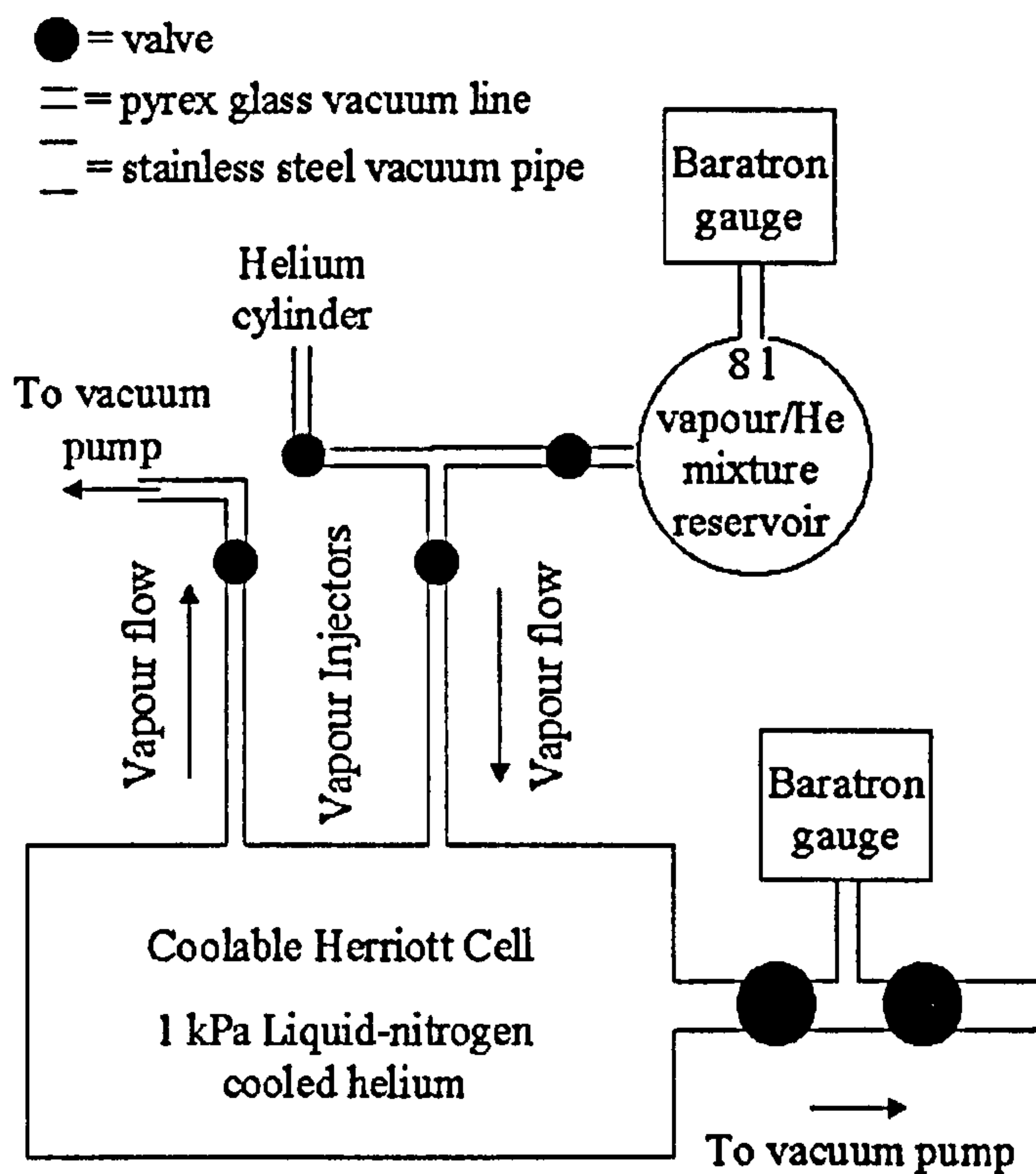


Figure 7.12 - Schematic of the collisionally cooled gas handling system

During cooling the electrical heater currents in the vapour injectors, at the cell window and vacuum aperture were adjusted to maintain room temperature. A typical current load of 1 Amp at 15 Volts was required.

Figure 7.13 shows a collisionally cooled spectra of acetylene taken in the same region as the ethanol cooled spectra. Also shown on this figure is the ethanol cooled spectrum of acetylene taken at 216K. The intensity of the 216 K spectrum has been scaled so that the intensities of the lines at 6589.5 cm^{-1} are comparable. The collisionally cooled feature has an absorption equivalent to $\sim 6 \times 10^{-5}$. The broad feature at 6589.3 cm^{-1} is absorption due to water vapour in the optical path outside the cell. The water line is not evident in the 216 K spectrum because of the intensity scaling factor and the coincidence with an acetylene line.

It is apparent that the transitions at 6589.3 , 6589.7 and 6589.95 cm^{-1} have all but disappeared, while the low J overtone at 6589.5 cm^{-1} is still clearly visible.

Collisionally Cooled Acetylene Spectra

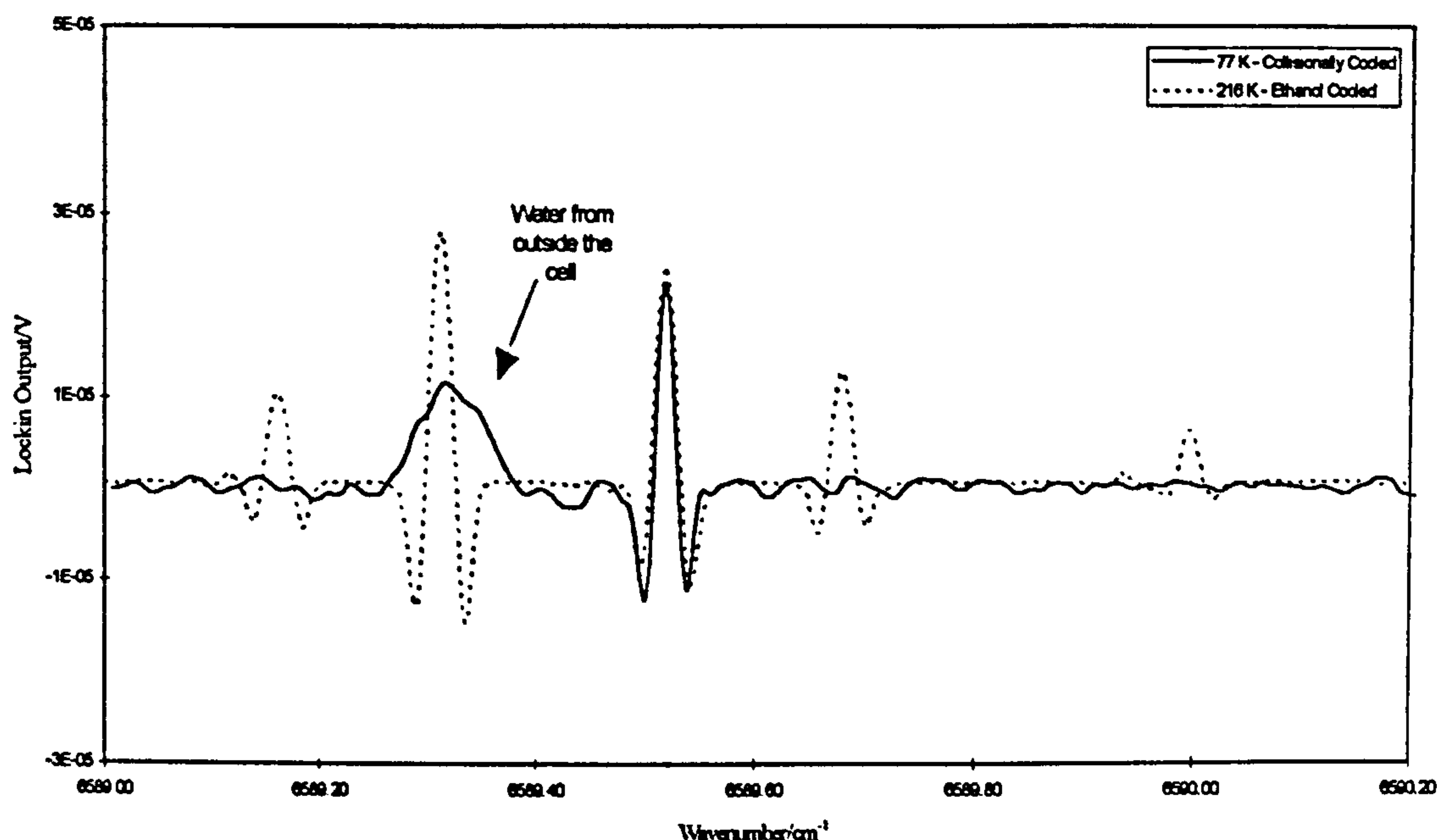


Figure 7.13 - Collisionally Cooled Spectrum of Acetylene

It is concluded from these spectra that the collisional cooling has been successful with significant changes being observed in the Boltzmann populations of the vibrational and rotational states of the spectroscopic gas.

Molecular Clusters

When a 15 % acetylene/helium mixture was flowed into the cell, strong absorptions from cooled vapour were observed for a few seconds. However, after this time the signal at the detector completely disappeared. After a few minutes the signal would return to original levels. The loss of power was attributed to high concentrations of molecular clusters being formed in the optical path. The absorption features of these clusters are expected to be sufficiently broad (several wavenumbers) to *black out* the laser across the entire measurement frequency range.

7.4 Conclusions

A coolable Herriott cell capable of operating in ethanol cooled and collisionally cooled modes has been constructed. Successful testing of this instrument, recording the spectra of acetylene at 6590 cm^{-1} in both cooling modes, has shown:

- Operating temperatures between 77 and 297 K can be achieved.
- The temperature of the cell remained stable to within 0.1 K during the period of the measurement (~ 1 minute).
- The alignment of the Herriott cell was preserved over the entire range of operating temperatures (77-298K).
- Near-infrared diodes and enhanced pathlengths provide sensitivities equivalent to a minimum detectable absorption of $\sim 1 \times 10^{-5}$. This is a factor of 100 better than what has been achieved with mid-infrared FTS measurements [74].
- Most of the weaker transitions in the acetylene spectrum in this region belong to hot bands or high J overtone transitions.
- C_2H_2 molecular clusters are formed when acetylene/helium mixtures above $\sim 10\%$ are used

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Appendices

Appendix 1 - Laser Specification

1.65 μm DFB LD MODULE

SD3B254F

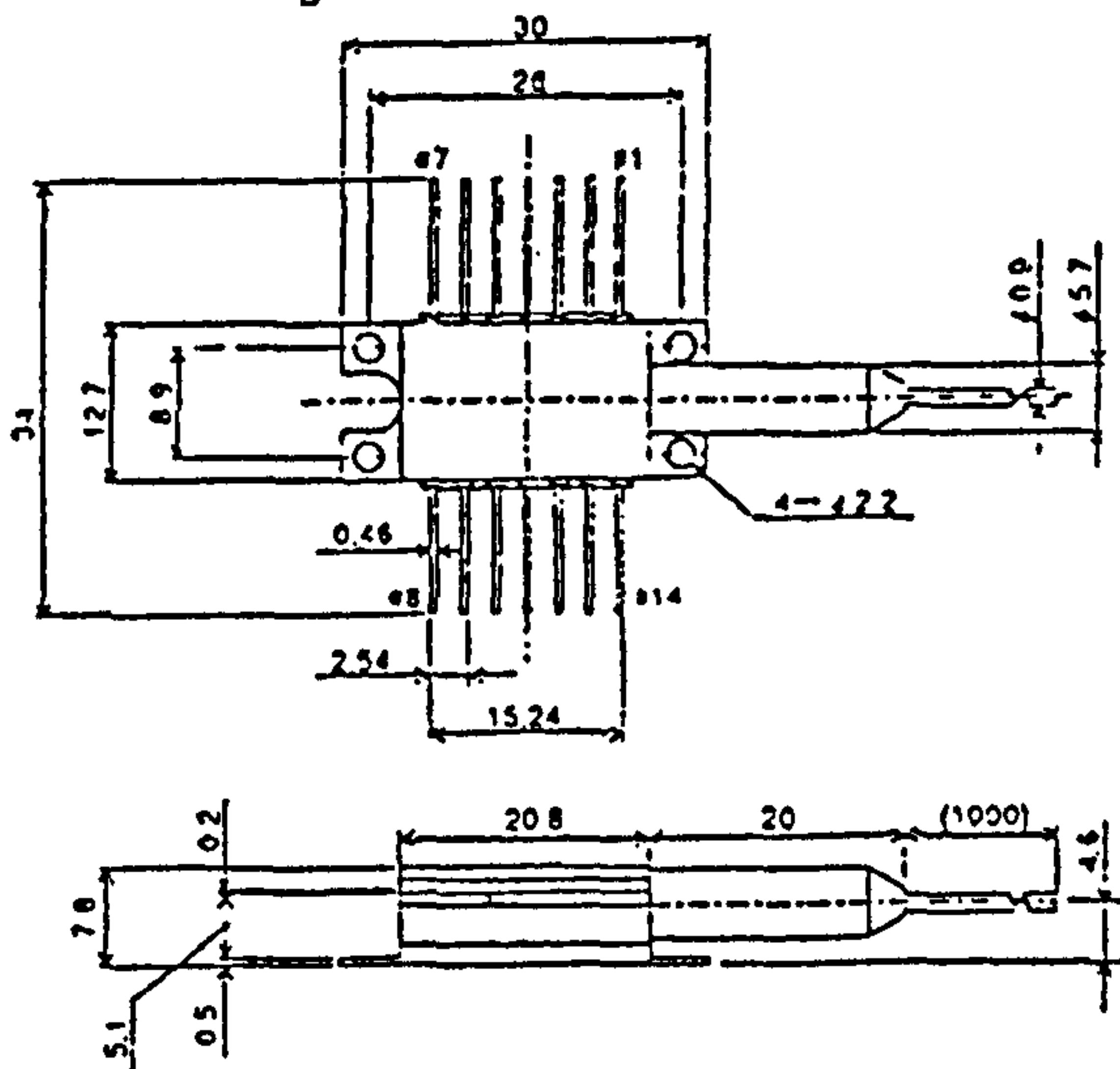
The SD3B254F is a InGaAsP/InP distributed feedback (DFB) laser diode developed as a light source for methane gas detection.

FEATURES

- Single longitudinal mode
- Internal optical Isolator
- Internal monitor PD and thermo-electric cooler
- Single mode fiber pigtail type (SMF 10/125 μm)

APPLICATION

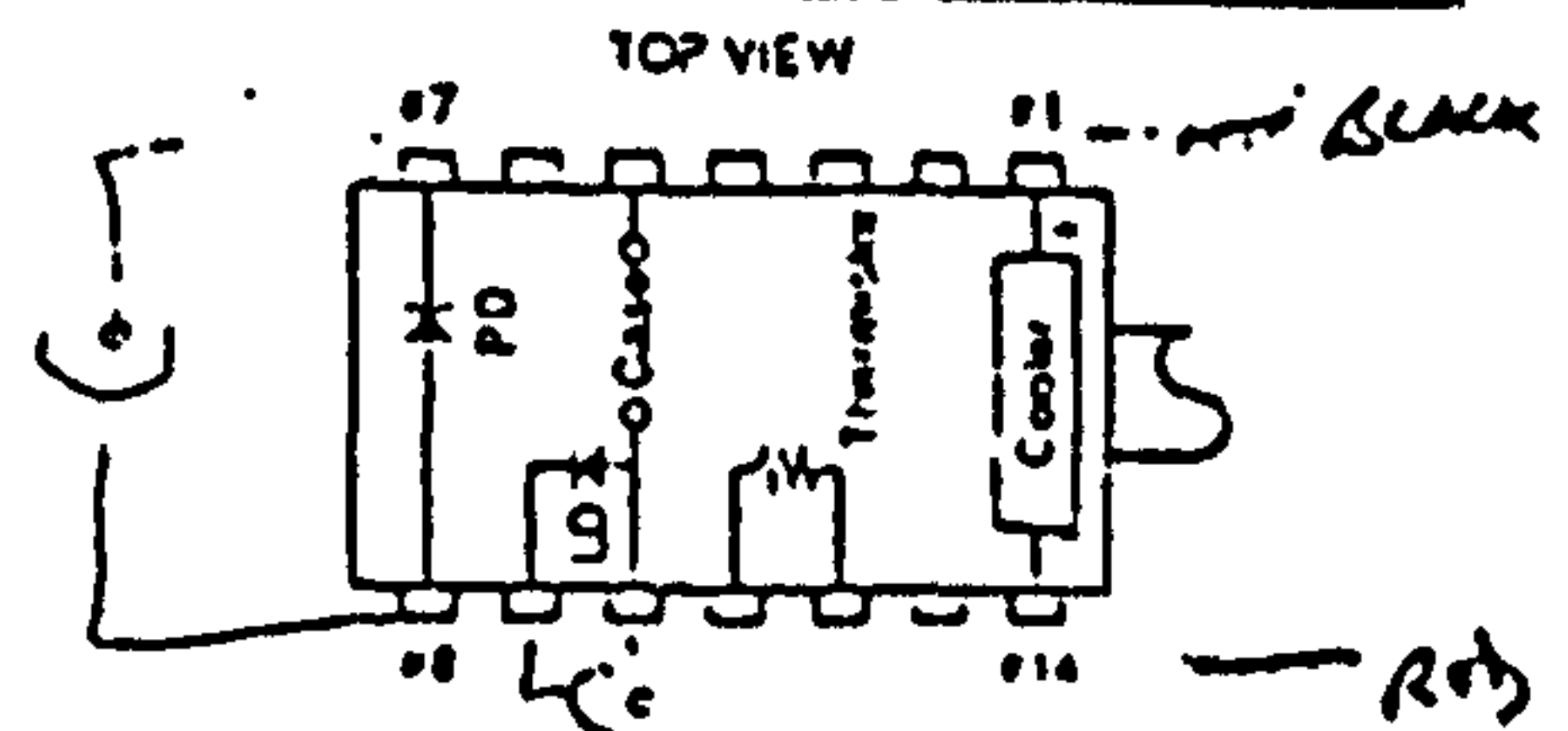
- Methane gas detection



< PACKAGE OUTLINE (Unit:mm) >

ABSOLUTE MAXIMUM RATINGS (T_{LD}=25°C)

Item	Symbol	Rating	Unit
LD Reverse Voltage	V _r	2	V
LD Forward Current	I _r	150	mA
PD Reverse Voltage	V _{rs}	20	V
PD Forward Current	I _{rs}	10	mA
Operating Case Temperature	T _c	-20 to 70	°C
Storage Temperature	T _{stg}	-40 to 85	°C
Cooler Current	I _c	2.3	A



NO	FUNCTION	NO.	FUNCTION
1	Cooler anode	8	PD anode
2	NC	9	LD cathode
3	NC	10	LD anode, case
4	NC	11	Thermistor
5	Case	12	Thermistor
6	NC	13	NC
7	PD cathode	14	Cooler cathode

< PIN CONFIGURATION >

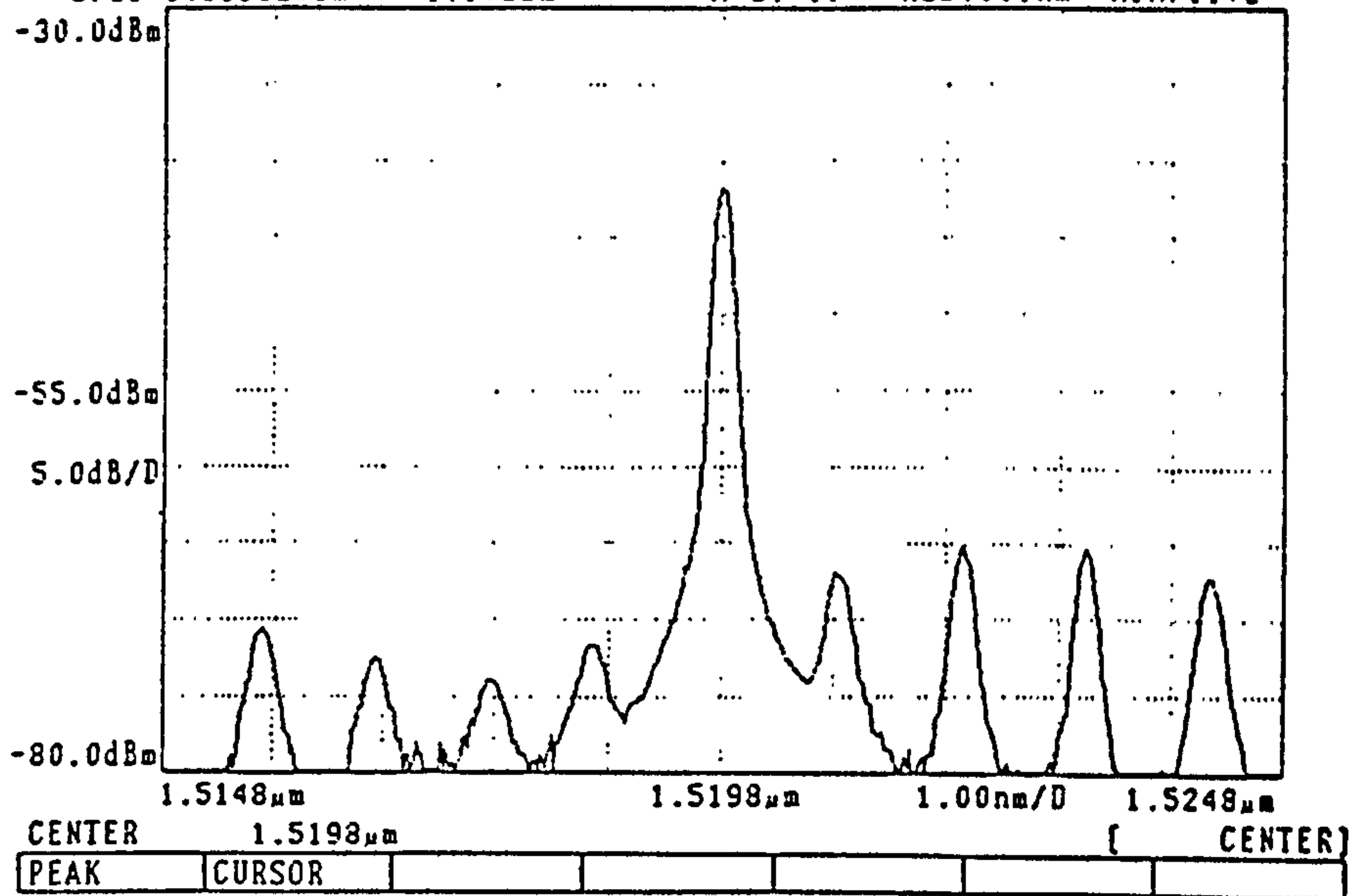
OPTICAL AND ELECTRICAL CHARACTERISTICS (T_{LD}=25°C, T_c=-20 to 70°C)

Item	Symbol	Test Condition	Min.	Typ.	Max.	Unit
Forward Voltage	V _f	I _r = 100 mA		1.5		V
Threshold Current	I _{th}			20	50	mA
Optical Output Power	P _i	I _r = 100 mA	1			mW
Peak Wavelength *1	λ_p	P _i = 1 mW	1663		1668	nm
Side Mode Suppression Ratio	SMSR	P _i = 1 mW	30	35		dB
Spectral Half Width	Δf	P _i = 1 mW		10		MHz
Rise Time	t _r				1.0	ns
Fall Time	t _f				1.0	ns
Monitor Current	I _m	P _i = 1 mW, V _{rs} = 5 V	100	400		μA
PD Dark Current	I _d	V _{rs} = 5 V			10	μA
Cooler Voltage	V _c	I _r = 100 mA, T _c = 70°C		1.6		V
Cooler Current	I _c	I _r = 100 mA, T _c = 70°C		1.1		A
Thermistor Resistance	R _{th}	T _{LD} = 25°C	9.5	10	10.5	K Ω
Optical Isolation	R _o	T _c = 25°C		50		dB

*1 The peak wavelength can be tuned to one of absorption lines shown below by controlling the temperature of lasers within 25 \pm 15°C.

Absorption line	R(9)	R(8)	R(7)	R(6)	R(5)	R(4)	R(3)	O branch
Wavelength (nm)	1637.7	1640.4	1642.9	1645.6	1648.3	1651.0	1653.8	1655.5-1665.8

MKL 1200DFB XII/12 50 mA 1995- 4-29 11:11:06
SPEC 1.519920 μ m -41.67dBm AVG: 10 RES:0.1nm ADAPTIVE



Date : 28/04/95

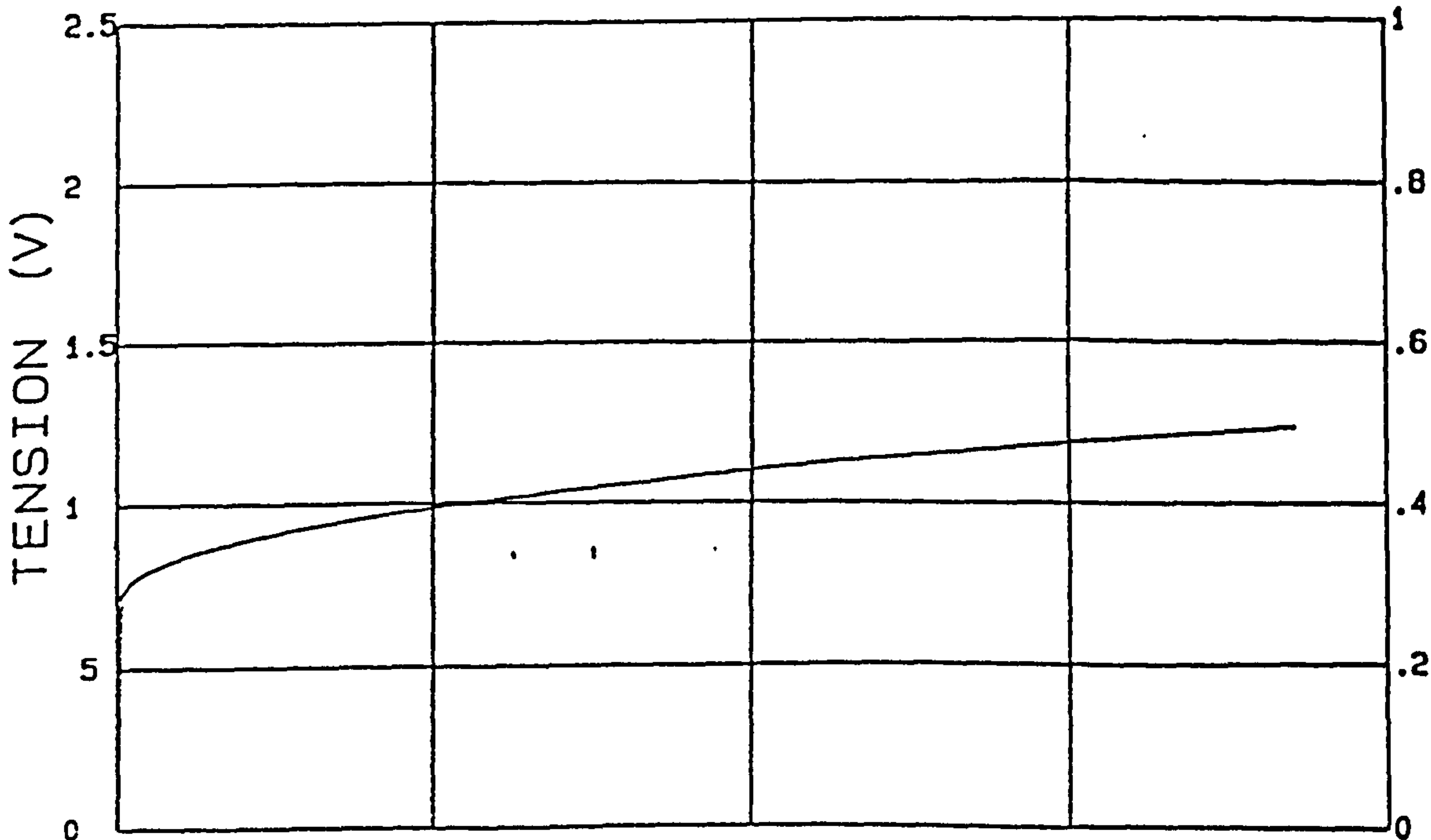
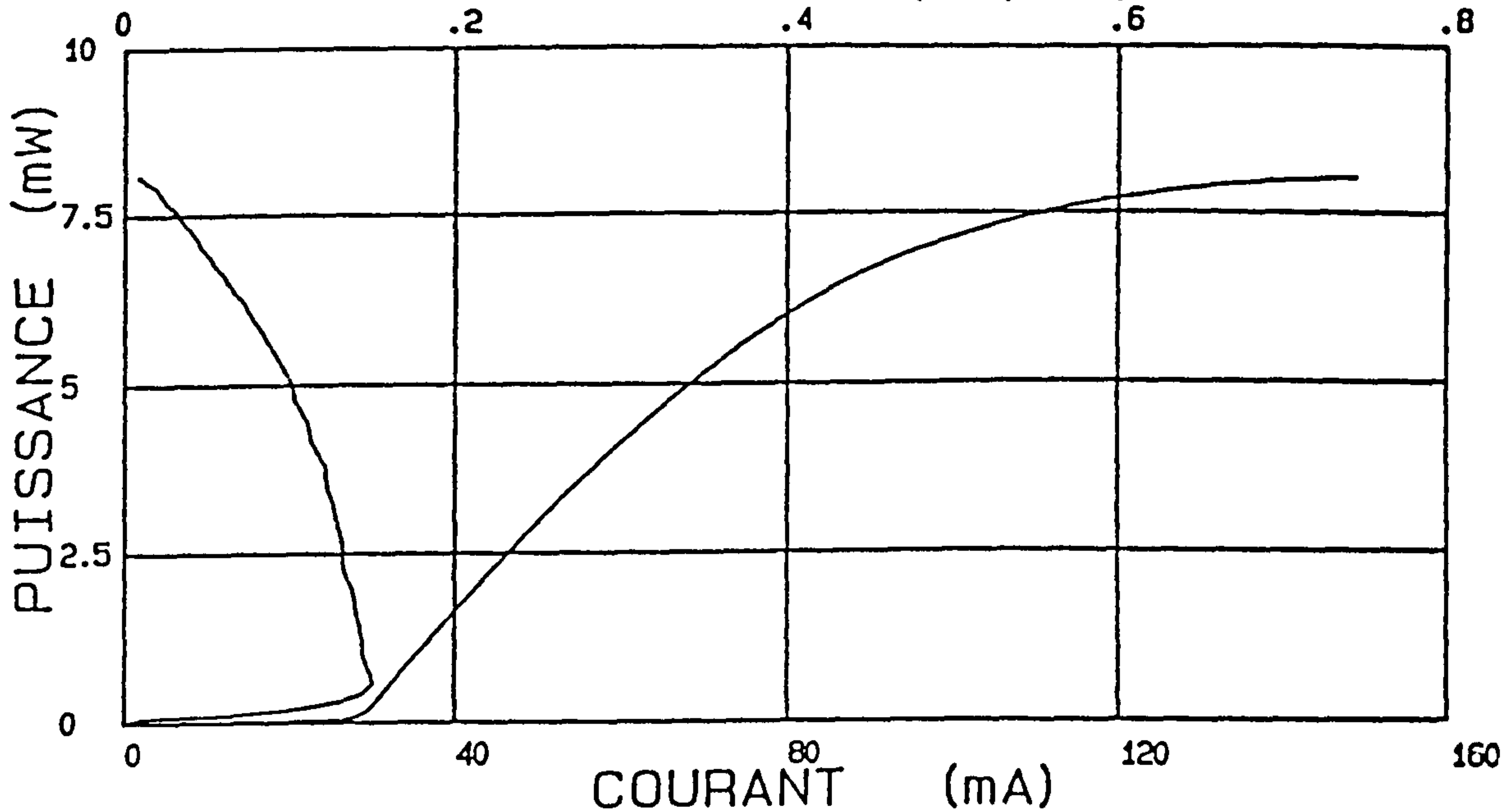
$\lambda = 1550 \text{ nm}$

25 °C

K1158A 00/00C

			APRES DEVERMINAGE					
	Continu	Impulsion	Continu		Impulsion	Rs Reg (Ω)	Continu	Impu
Is (mA)	28.9					2.52		NO K
R2m (mW/mA)	.135		0	$\frac{\Delta I_s}{I_{s0}}$	4mW	I (XmW) (mA)	58.09	
P2s (mW)	.161				3mW	I (KmW) (mA)	49.7	
R1m (W/A)	.007		0	$\frac{\Delta R_{1m}}{R_{1m0}}$	2mW	I (XmW) (mA)	42.25	
P1s (mW)	.011				1mW	I (XOmW) (mA)	34.85	
R1m/R2m	.05		0	$\frac{\Delta R_{2m}}{R_{2m0}}$		Vmax (V)	1.25	
Rth x T/T0						Δ		

RENDEMENT (mW/mA)



THOMSON-CSF SEMICONDUCTEURS SPECIFIC

D. 1. 5. 2

Appendix 2 - Software

VTDLDAQ.M

MATLAB routine to:

Load spectrum from disk
Load background from disk
Polynomial fit the background
Fourier Transform Spectra to remove high frequency noise components
Wavelength linearize spectrum
Combine overlapping spectra

HERRIOTT.M

MATLAB routine to:

Model paraxial ray passing through Herriott Cell
Model paraxial ray passing through Astigmatic Herriott Cell

SECHARM.M

MATLAB routine to:

Load line parameters from file and generate direct spectrum with either Doppler, Lorentz or Voigt lineshape.
Load real direct spectrum from disk
Combine real and simulated spectrum
Generate the nth harmonic component of simulated or real direct spectrum

DATALOAD.M

MATLAB routine to:

Load real and simulated spectrum
Autocorrelate spectra to remove any shift
Least square fit, simulated and real spectrum

```

function vtdldaq(option)
%*****
%* Filename: VTDLDAQ.M
%* Version: 1.07
%* Update: 2nd February 1996
%* Usage: vtdldaq(<option>)
%*
%* (C) Liam Donohoe
%* Dept. Physics & Applied Physics
%* University of Strathclyde
%* Glasgow
%*
%* Updated 14/3/96 by I F Howieson
%* Now includes facility to allow: background fit
%* : data filtering
%* : frequency linearization
%* : combine spectra
%* File which contains routines for displaying and manipulating
%* TDLDAQ spectra. ....
%* ..... The various routines are represented by 'options'
%* which allow a matlab-style self-contained recursive function.
%*
%* Possible <option>'s are:
%*
%* 'start' - initialises environment
%*
%* 'loadSpec' - loads TDLDAQ spectrum from disc
%* 'loadBgnd' - loads spectrum into background
%*
%* 'quit' - cleans up workspace and terminates
%*
%* Requires the following user files (and user toolboxes):
%*
%*
%*****
%
% ! define paths for default location of TDLDAQ files !
%*****
DefaultPath = 'c:\apps\matlab\work\tdldaq\*. *';
%*****
%
% check input argument and declare globals
%*****
if ( nargin < 1 )
option = 'start';
end;

global FigH AcqStatH bkgnd_X bkgnd_Y offset dataOffset OFFSET_H EDIT_H
global versionID version DOS_frame filename spectrumSource startTime
global diodeTempK diodeData Vdata Tdata MCdata channels samples
global cellTempK STDcellTK dataPoints transM etalon scanVariant scanMin

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global scanMax xUnits yUnits absorption AcqStatH plotAxes axesH
global cellPressT STDcellPT ab freq1 tran1 tran2 etalonH transmH
global scanM transma xpt1 xpt2 data ref datab auto

% initialise cell statistics...
cellTempK=0.0;
STDcellTK=0.0;
cellPressT=0.0;
STDcellPT=0.0;

% define colours
WHITE = [ 1 1 1];
LIGHTGREY = [0.7529 0.7529 0.7529];
%*****
% 'start' - initialise environment and generate figure with menus *
%*****
if(strcmp(option,'start'))

FigH = figure('Visible','Off'); % instantiate a hidden figure...

% label the figure
set(FigH,'Name','TDLDAQ Spectral Display Utilities - Version 1.07');
set(FigH,'NumberTitle','off');

gtools; % add a graph tools menu to it

% generate a menu for selecting filing options
FilingM = uimenu('Label','Filing');

% generate analysis menu options
mh(1) = uimenu('FilingM','Label','Load Spectrum');
set(mh(1),'Callback','vtdldaq(''loadSpec'')');
mh(2) = uimenu('FilingM','Label','Load Background');
set(mh(2),'Callback','vtdldaq(''loadBgnd'')');
mh(3) = uimenu('FilingM','Label','Acquisition Statistics','Separator','On');
set(mh(3),'Callback','vtdldaq(''acqStat'')');
mh(4) = uimenu('FilingM','Label','X-Y Data -> File','Separator','On');
set(mh(4),'Callback','vtdldaq(''xyData'')');
mh(8) = uimenu('FilingM','Label','Quit','Separator','On');
set(mh(8),'Callback','vtdldaq(''quit'')');
mh(9) = uimenu('FilingM','Label','Combine','Separator','On');
mh(10) = uimenu(mh(9),'Label','Spectrum 1');
set(mh(10),'Callback','vtdldaq(''spectrum1'')');
mh(11) = uimenu(mh(9),'Label','Spectrum 2');
set(mh(11),'Callback','vtdldaq(''spectrum2'')');
mh(12) = uimenu(mh(9),'Label','Combine');
set(mh(12),'Callback','vtdldaq(''combine'')');
mh(13) = uimenu('FilingM','Label','loadxy','Separator','On');
set(mh(13),'Callback','vtdldaq(''loadxy'')');

% generate a menu for selecting analysis options
AnalysisM = uimenu('Label','Analysis');

% generate analysis menu options
mh(10) = uimenu('AnalysisM','Label','Offset');

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mh(11) = uimenu(mh(10),Label,'X Data');
set(mh(11),Callback,'vtdldaq("xOffset");');
mh(12) = uimenu(mh(10),Label,'Y Data');
set(mh(12),Callback,'vtdldaq("yOffset");');
mh(13) = uimenu(AnalysisM,Label,'Ratio Background','Separator','On');
set(mh(13),Callback,'vtdldaq("ratioBkgnd");');
mh(14) = uimenu(AnalysisM,Label,'Fit Background');
set(mh(14),Callback,'vtdldaq("fitdata");');
mh(15) = uimenu(AnalysisM,Label,'Absorption Coefficients');
set(mh(15),Callback,'vtdldaq("absCoeff");');
mh(16) = uimenu(AnalysisM,Label,'Filter Data','Separator','On');
set(mh(16),Callback,'vtdldaq("filtdata");');
mh(17) = uimenu(AnalysisM,Label,'Linearize','Separator','On');
set(mh(17),Callback,'vtdldaq("linearize");');
mh(18) = uimenu(AnalysisM,Label,'Spectrum Range','Separator','On');
set(mh(18),Callback,'vtdldaq("specrange");');
mh(19) = uimenu(AnalysisM,Label,'Data -> Workspace','Separator','On');
set(mh(19),Callback,'vtdlgtbl');
mh(20) = uimenu(AnalysisM,Label,'Update Display','Separator','On');
set(mh(20),Callback,'vtdldaq("dispSpec");');
mh(21) = uimenu(AnalysisM,Label,'Close');
set(mh(21),Callback,'vtdldaq("close");');

set(FigH,'visible','on'); % ...show the figure

FIT_NUMBER = 1;

%*****
% generate display parameters
%*****
elseif(strcmp(option,'spectrum1'))

[DOS_frame, path] = uigetfile(DefaultPath,'Enter input filename');

% concatenate path and filename strings
FilePath = [path, DOS_frame];

% open data file for reading
[sfid, ErrorMessage] = fopen(FilePath, 'r');

% check that no file errors occurred
if (sfid==-1)
    disp(ErrorMessage)
    return
end
data=[];
data = fscanf(sfid,'%f');
fclose(sfid);
b = size(data);
datapoints = b(1);
freq1 = data(1:datapoints./2);
tran1 = data(2:datapoints);

%freq2 = data(1:datapoints./2);
%tran2 = data((datapoints./2)+1:datapoints);

vtdldaq('dispSpec2');

%*****
% 'dispSpec' - displays the spectrum
%*****
elseif(strcmp(option,'dispSpec2'))

axesH = getobjh(FigH,'axes');
if (length(axesH) > 0)
    delete(axesH);
end

% instantiate lower residual axes
left = 0.23;
bottom = 0.11;
width = 0.5475;
height = 0.35;
etalonPosition = [left bottom width height];
etalonH = axes('Position', etalonPosition);

% plot etalon channel
plot(freq2, tran2)

% automate the residual axes aspect-ratio
set(etalonH, 'AspectRatib', [-nan -nan])

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```

versionID = rdstr(sfid);
version = str2num( versionID(9:12) );
if (version > 1.03)
    headerStrings = 42;
end
if (version == 1.05)
    headerStrings = 44;
end
if (version > 1.06)
    headerStrings = 47;
end

i=1;
while (i<length(headerStrings))
    rdstr(sfid);
    i=i+1;
end

%
fileName = rdstr(sfid);
if (version > 1.02)
    spectrumSource = rdstr(sfid);
end
if (version < 1.06)
    startTime = [rdstr(sfid), rdstr(sfid), rdstr(sfid)].
else
    startTime = [rdstr(sfid), 'rdstr(sfid)', ''];
end
rdstr(sfid);
day = rdstr(sfid);
day = day(1:length(day)-2);
month = rdstr(sfid);
month = month(1:3);
if(strcmp(month,'Jan'))
    month = '1';
elseif(strcmp(month,'Feb'))
    month = '2';
elseif(strcmp(month,'Mar'))
    month = '3';
elseif(strcmp(month,'Apr'))
    month = '4';
elseif(strcmp(month,'May'))
    month = '5';
elseif(strcmp(month,'Jun'))
    month = '6';
elseif(strcmp(month,'Jul'))
    month = '7';
elseif(strcmp(month,'Aug'))
    month = '8';
elseif(strcmp(month,'Sep'))
    month = '9';
elseif(strcmp(month,'Oct'))
    month = '10';
elseif(strcmp(month,'Nov'))
    month = '11';
elseif(strcmp(month,'Dec'))
    month = '12';
end

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year = rdstr(sfid);
year = year(3:4);
startTime = [startime, day, '/', month, '/', year];
end
if (version <= 1.06)
    day = str2num( startime(5:6) );
    day = num2str(day);
    month = startime(7:9);
    if(strcmp(month,'Jan'))
        month = '1';
    elseif(strcmp(month,'Feb'))
        month = '2';
    elseif(strcmp(month,'Mar'))
        month = '3';
    elseif(strcmp(month,'Apr'))
        month = '4';
    elseif(strcmp(month,'May'))
        month = '5';
    elseif(strcmp(month,'Jun'))
        month = '6';
    elseif(strcmp(month,'Jul'))
        month = '7';
    elseif(strcmp(month,'Aug'))
        month = '8';
    elseif(strcmp(month,'Sep'))
        month = '9';
    elseif(strcmp(month,'Oct'))
        month = '10';
    elseif(strcmp(month,'Nov'))
        month = '11';
    elseif(strcmp(month,'Dec'))
        month = '12';
    end
    year = startime(12:13);
    hr = str2num( startime(15:16) );
    if (hr >= 12)
        if (hr > 12)
            hr = hr - 12;
        end
        ampm = 'pm';
    else
        ampm = 'am';
    end
    hr = num2str(hr);
    min = str2num( startime(18:19) );
    sec = str2num( startime(21:22) );
    if (sec >= 30)
        min = min + 1;
    end;
    if (min > 9)
        min = num2str(min);
    else
        min = ['0',num2str(min)];
    end
    startTime = [hr, ':', min, ampm, ' - ', day, '/', month, '/', year];
end

```



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%
diodeTempK = str2num( rdstr(sfid) );
if (version > 1.03)
    diodel = str2num( rdstr(sfid) );
end
ldata = fscanf(sfid, '%d', [1 3]);
if (version <= 1.03)
    diodel = str2num( rdstr(sfid) );
end
vdata = fscanf(sfid, '%f', [1 3]);
if (version > 1.03)
    Tdata = fscanf(sfid, '%f', [1 3]);
    MCdata = fscanf(sfid, '%f', [1 4]);
end
channels = fscanf(sfid, '%d', [1 5]);
samples = fscanf(sfid, '%d', [1 2]);
cellTempK = fscanf(sfid, '%f', [1 1]);
if (version > 1.04)
    rdstr(sfid);
    STDcellTK = fscanf(sfid, '%f', [1 1]);
else
    STDcellTK = 0.0;
end
if (version > 1.06)
    cellPressT = fscanf(sfid, '%f', [1 1]);
    rdstr(sfid);
    STDcellPT = fscanf(sfid, '%f', [1 1]);
end
if (version == 1.02)
    MCdata = fscanf(sfid, '%f', [1 4]);
elseif (version < 1.02)
    MCdata = fscanf(sfid, '%f', [1 3]);
end
dataPoints = fscanf(sfid, '%d', [1 1]);
dataLeft = dataPoints;
data=[];
while (dataLeft >= 100)
    data = [data; fscanf(sfid, '%f', [2 100])];
    dataLeft = dataLeft - 100;
end
if (dataLeft > 0)
    data = [data; fscanf(sfid, '%f', [2 dataLeft])];
end
transm = data(:,1);
etalon = data(:,2);
fclose(sfid);

%
% Generate spectral information and display parameters
%
if (version == 1.02)
    if (ldata(2) - ldata(1))
        spectrumSource = 'LMS';
    end
    if (vdata(2) - vdata(1))
        spectrumSource = 'BT&D';
    end
    if ((channels(1) == -1) & (channels(2) == -1))
        plotAxes = 2;
    else
        plotAxes = 1;
    end
    if ( strcmp(spectrumSource, 'LMS') | strcmp(spectrumSource, 'LS-3') )
        scanVariant = ldata(1):ldata(3) ldata(2);
        scanMin = ldata(1);
        scanMax = ldata(2);
        if ((plotAxes==2) & (channels(1) == -1))
            yUnits = 'Transmission';
        else
            yUnits = 'Etalon';
        end
        xUnits = 'Current / mA';
    end
    if ( strcmp(spectrumSource, 'LX') | strcmp(spectrumSource, 'LX_RAMP') )
        scanVariant = ldata(1):ldata(3) ldata(2);
        scanMin = ldata(1);
        scanMax = ldata(2);
        if ((plotAxes==1) & (channels(1) == -1))
            yUnits = 'Transmission';
        else
            yUnits = 'Etalon';
        end
        xUnits = 'Current / mA';
    end
    if ( strcmp(spectrumSource, 'LDC') )
        scanVariant = ldata(1):ldata(3) ldata(2);
        scanMin = ldata(1);
        scanMax = ldata(2);
        if ((plotAxes==1) & (channels(1) == -1))
            yUnits = 'Transmission';
        else
            yUnits = 'Etalon';
        end
        xUnits = 'Current / mA';
    end
    if ( strcmp(spectrumSource, 'LDC_T') )
        scanVariant = Tdata(1):Tdata(3):Tdata(2);
        scanMin = Tdata(1);
        scanMax = Tdata(2);
        if ((plotAxes==1) & (channels(1) == -1))
            yUnits = 'Transmission';
        else
            yUnits = 'Etalon';
        end
        xUnits = 'Temperature / Centigrade';
    end
end
end

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if ( strcmp(spectrumSource,'BT&D') | strcmp(spectrumSource,'ETALON') )
    scanVariant = Vdata(1); Vdata(3); Vdata(2);
    scanMin = Vdata(1);
    scanMax = Vdata(2);
    if ( (plotAxes==1) & (channels(1)~=1) )
        yUnits = 'Transmission';
    else
        yUnits = 'Etalon';
    end
    xUnits = 'Voltage / volts';
end
if ( strcmp(spectrumSource,'RALMC') )
    scanVariant = MCdata(1) MCdata(3) MCdata(2);
    scanMin = MCdata(1);
    scanMax = MCdata(2);
    yUnits = 'Etendue';
    xUnits = 'Wavelength / microns';
end
if ( strcmp(spectrumSource,'JY125') | strcmp(spectrumSource,'LS3MC') )
    scanVariant = MCdata(1) MCdata(3) MCdata(2);
    scanMin = MCdata(1);
    scanMax = MCdata(2);
    yUnits = 'Etendue';
    xUnits = 'Wavenumber / cm-1';
end
if ( strcmp(spectrumSource,'TRANSM') )
    scanVariant = Idata(1) Idata(3) Idata(2);
    scanMin = Idata(1);
    scanMax = Idata(2);
    xUnits = 'Time / secs';
    yUnits = 'Absolute Intensity';
end
if ( strcmp(spectrumSource,'THERMOS') )
    scanVariant = Idata(1) Idata(3) Idata(2);
    scanMin = Idata(1);
    scanMax = Idata(2);
    xUnits = 'Time / secs';
    yUnits = 'Temperature / Kelvin';
end
if ( strcmp(spectrumSource,'BARATRON') )
    scanVariant = Idata(1) Idata(3) Idata(2);
    scanMin = Idata(1);
    scanMax = Idata(2);
    xUnits = 'Time / secs';
    yUnits = 'Pressure / Torr';
end
yLength = length(transm);
xLength = length(scanVariant);
if (yLength < xLength)
    scanVariant = (scanVariant(1:yLength)/x
end
if (yLength > xLength)
    transm = transm(1:xLength);
    etalon = etalon(1:xLength);
end
vtdldaq('dispSpec');
%*****
% 'xOffset' - offsets X-data by specified amount
%*****
elseif(strcmp(option,'xOffset'))
    offset = 'x-data';
    vtdldaq('initOffset');
%*****
% 'yOffset' - offsets Y-data by specified amount
%*****
elseif(strcmp(option,'yOffset'))
    offset = 'y-data';
    vtdldaq('initOffset');
%*****
% 'initOffset' - generates dialog box for offsets
%*****
elseif(strcmp(option,'initOffset'))
    if (strcmp(offset,'x-data'))
        OName = 'Offset X-data by ...';
    elseif (strcmp(offset,'y-data'))
        OName = 'Offset Y-data by ...';
    end
    % instantiate a new figure without a menu bar
    left = 307;
    bottom = 400;
    width = 180;
    height = 95;
    OPosn = [left bottom width height];
    MBSStatus = 'none';
    OFFSET_H = figure('Position',OPosn,'MenuBar','MBSStatus');
    % prevent it from being resized
    RStatus = 'off';
    set(OFFSET_H,'Resized',RStatus);
    % name it
    NTSStatus = 'off';
    set(OFFSET_H,'NumberTitle',NTSStatus);
    set(OFFSET_H,'Name',OName);
    % give it a grey background
    OColour = LIGHTGREY;
    set(OFFSET_H,'Color',OColour);
    % define dialogbox and label for axes
    EAxisPosn = [40 60 100 20];
    EDIT_H = uicontrol(OFFSET_H,'Style','Edit','Position',EAxisPosn);

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set(EDIT_H, 'BackgroundColor', WHITE, 'String', '0.0');

% define OK push-button
OKPosn = [40 15 100 30];
OKHString = 'OK';
OKH = uicontrol(OFFSET_H, 'Style', 'PushButton', 'Position', OKPosn);
set(OKH, 'String', OKHString, 'Callback', 'vidldaq(''killOffset'');');

%*****
% 'killOffset' - deletes offset dialog box
%*****
elseif(strcmp(option, 'killOffset'))

% get the axis limits...
dataOffset = str2num(get(EDIT_H, 'String'));

% remove the dialog box...
delete(OFFSET_H);

if (strcmp(offset, 'x-data'))
    scanVariant = scanVariant + dataOffset;
elseif (strcmp(offset, 'y-data'))
    transm = transm + dataOffset;
end

vidldaq('dispSpec');

%*****
% 'loadBkgnd' - loads spectrum into background
%*****
elseif(strcmp(option, 'loadBkgnd'))

vidldaq('loadSpec');
bkgnd_X = scanVariant;
bkgnd_Y = transm;

%*****
% 'absCoeff' - calculates and plots absorption coefficients
%*****
elseif(strcmp(option, 'absCoeff'))

ab = -log(transm);
fig;
plot(scanVariant, ab)

%*****
% 'fidata' - fits background and ratios it with spectrum
%*****
elseif(strcmp(option, 'fidata'))

if plotAxes == 2
    [x,y] = ginput(15);
    x = round((1+(x-ldata(1))/ldata(3)));

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c = polyfit(x,transm(x),3);
xdata = l.dataPoints;
y = (c(1).*(xdata.^3))+c(2).*(xdata.^2)+c(3).*(xdata.^1)+c(4).*(xdata.^0)+c(5);
y = flipud(rot90(y));

transm = transm./y;
%transma(xpt1:xpt2) = transm;
%transm = transma;
%scanVariant = scanm;
vidldaq('dispSpec');
else
    [x,y] = ginput(4);

x = round((1+(x-ldata(1))/ldata(3)));
%x = round(x)
c = polyfit(x,transm(x),2)
xdata = l.dataPoints
y = (c(1).*(xdata.^2))+c(2).*(xdata.^1)+c(3).*(xdata.^0)+c(4).*(xdata.^0)+c(5);
y = flipud(rot90(y))
size(y)
save tets y -ascii
transm = transm./y;
vidldaq('dispSpec');

end
%*****
% 'spectrange' - defines spectral range for filtering etc.
%*****
elseif(strcmp(option, 'spectrange'))
    if plotAxes == 1
        [x,y] = ginput(2);
        ldata(1) = x(1);
        ldata(2) = x(2);
        ldata(3) = (scanVariant(2)-scanVariant(1));
        xpt1 = 1+((x(1)-0)/(ldata(3)));
        xpt2 = 1+((x(2)-0)/(ldata(3)));
        scanm = scanVariant;
        scanVariant = [ldata(1).ldata(3).ldata(2)];
        transma = transm;
        transm = transm(xpt1:xpt2);
        dataPoints = 1+(xpt2-xpt1);
        vidldaq('dispSpec');
    else
        [x,y] = ginput(2);
        xpt1 = 1+((x(1)-ldata(1))/ldata(3));
        xpt2 = 1+((x(2)-ldata(1))/ldata(3));
        scanMin = x(1);
        scanMax = x(2);
        ldata(1) = x(1);
        ldata(2) = x(2);
        scanVariant = ldata(1).ldata(3).ldata(2);
        transm = transm(xpt1:xpt2);
        etalon = etalon(xpt1:xpt2);
    end
end

```



```

dataPoints = 1+(xpt2-xpt1);
vtdldaq('dspSpec');

end
%*****
% 'linearize' - linearizes data using etalon fringes
%*****
elseif(strcmp(option,'linearize?'))
    deriv = diff(etalon);
    z = fft(deriv,20000);
    b = zeros(10000,1);
    z(5001:15000)=b;
    d = ifft(z);
    deriv = real(d(1:dataPoints));

    s = 1;
    for i = 1:dataPoints-1;
        if deriv(i+1) * deriv(i) <= 0
            if etalon(i) > max(etalon)/14
                zer(s) = i;
                s=s+1;
            end
        end
    end
    zer;
    s=s-2;
    wavnum = [0.75:(s.*7.5)];
    c = polyfit(zer,wavnum,3);
    x=1:dataPoints;
    X = (c(1).*(x.^3)) + (c(2).*(x.^2))+(c(3).*(x))+c(4);
    x2 = 0:((s.*7.5)/(dataPoints-1)):(s.*7.5);
    transm = interp1(X,transm,x2);
    transm = rot90(transm);
    scanVariant = x2;
    plotAxes=1;
    channels(1)=1;
    channels(2)=2;
    xUnits=Frequency/GHz;
    yUnits=Transmission;
    vtdldaq('dspSpec');

%*****
% 'filterdata' - uses fft to filter out high frequency noise
%*****
elseif(strcmp(option,'filterdata?'))
    transm = transm-1;
    y = fft(transm,20000);
    %z = fft(etalon,20000);
    a = zeros(18000,1);
    %b = zeros(10000,1);
    Y(1001:19000)=a;
    %z(5001:15000)=b;

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c = ifft(y);
%d = ifft(z);
transm = 1+real(c(1:dataPoints));
%etalon = real(d(1:dataPoints));

vtdldaq('dspSpec');

%*****
% 'conce' - uses fft to filter out high frequency noise
%*****
elseif(strcmp(option,'conce?'))
    data = transm;
    size(data)
    load call181 -ascii
    ref = call181;
    size(ref);

    start2 = ref(1);
    end2 = ref(250);
    int2 = (end2-start2)/249;
    back2 = start2-int2;
    back2 = flipud(rot90(back2));
    ref = ref-back2;

    y = fft(data,20000);
    z = fft(ref,20000);
    a = zeros(17000,1);
    Y(1501:18500)=a;
    Z(1501:18500)=a;
    b = ifft(y);
    c = ifft(z);
    data1 = real(b(1:dataPoints));
    re = (data-data1)/2;
    save resi re -ascii
    rms = sqrt((sum((re.^2)))/dataPoints)
    %percent = rms/(max(data1)-min(data1))
    %plot(data-data1);
    data = data1;
    ref = real(c(1:250));

    refpad = zeros(512,1);
    refpad(1:250)=ref;
    dalapad = zeros(512,1);
    dalapad(1:250) = data;
    fref = fft(refpad);
    conji = conj(fft(dalapad));
    auto = fftshift(real(fft(conji.*fref)));

    k=1;
    for l = 2:512
        if abs(auto(l)) > abs(auto(k))

```

```

k=i;
else
q=i;
end
end
f=-256:255;
n=f(k);

if n < 0
datapad = zeros(250,1);
datapad(1+25:250+n)=data((-n)+1+25:250);
data = datapad;
else
datapad = zeros(250,1);
datapad(n+1:250)=data(1:250-n);
data = datapad;
end

%Peak Finder and Wavelength Callibration
%l = 1;
%for n = 90:5:200
%dataq = data(n-25:n);
%refq = ref(n-25:n);
%r = nls(dataq,refq);
%concoq(l) = (((18.975/r)+(19.991/r))/2)/1.3772;
%l=l+1;
%end
%plot(concoq);

s=1;
x = nls(data,ref)
data=x.*data;
l = (x/2.122);
m = (x/12.1066);
conco=18.875/x
conc = (((18.975/m)+(3.498/m))/2);
conco = (((((18.975/x)+(19.991/x))/2)/1.3772)/3);
f = ref - data;
save ff -ascii;
di = diff(ref);
for r = 40:210
if di(r+1) .* di(r) < 0
zer(s) = r
s = s+1;
end
end
wavnum = [6002.625953,6002.5929619,6002.55938416,6002.5249267,6002.488563];
c = polyfit(zer,wavnum,1);
x=1:250;
X = (c(1) .* x) + c(2);
plot(X,data);
hold on;

plot(X,ref,'r');
%save X X -ascii
save scanvar scanVariant -ascii
save dataf data -ascii;
save reff ref -ascii;
%save conc concoq -ascii;

%*****
% 'ratioBkgnd' - ratios spectrum with background
%*****
elseif(strcmp(option,'ratioBkgnd'))
if (length(bkgnd_X)~=length(scanVariant))
bkgnd_Y = spline(bkgnd_X,bkgnd_Y,scanVariant);
bkgnd_X = scanVariant;
end
transm = transm / bkgnd_Y;
vidldaq('dispSpec');

%*****
%'dispSpec' - displays the spectrum
%*****
elseif(strcmp(option,'dispSpec'))
axesH = getobjh(FigH,'axes');
if (length(axesH) > 0)
delete(axesH);
end
if (plotAxes==2)
% instantiate lower residual axes
left = 0.23;
bottom = 0.11;
width = 0.5475;
height = 0.1825;
etalonPosition = [left bottom width height];
etalonH = axes('Position',etalonPosition);
% plot etalon channel
plot(scanVariant,etalon)
% automate the residual axes aspect-ratio
set(etalonH,'AspectRatio',[-nan -nan])
% label the residual axes
xlabel(xUnits);
ylabel(Etalon)
% instantiate upper fit axes
left = 0.23;
bottom = 0.38;
width = 0.5475;

```

```

height = 0.5475;
transmPosition = [left bottom width height];
transmH = axes('Position', transmPosition);

% plot spectral fit
plot(scanVariant, transm)
ylab('Transmission')

% automate the fit axes aspect-ratio
set(transmH, 'AspectRatio', [nan nan])

else
if (channels(1) == -1)
plot(scanVariant, transm)
elseif (channels(2) == -1)
plot(scanVariant, etalon)
end

xlabel(xUnits);
ylabel(yUnits);
end

% label the fit axes
tlab('FileName', ' ', startTime)

%*****
% 'AcqStat' - display the acquisition statistics
%*****
elseif(strcmp(option,'AcqStat'))

% instantiate a new figure without a menu bar
left = 342;
bottom = 250;
width = 340;
height = 400;
ASPosn = [left bottom width height];
MBStatus = 'none';
AcqStatH = figure('Position', ASPosn, 'MenuBar', 'MBStatus');

% prevent it from being resized
RStatus = 'off';
set(AcqStatH, 'Resize', RStatus);

% name it
NTStatus = 'off';
ASName = 'Spectrum Acquisition Statistics';
set(AcqStatH, 'NumberTitle', NTStatus);
set(AcqStatH, 'Name', ASName);

% give it a grey background
ASColour = LIGHTGREY;
set(AcqStatH, 'Color', ASColour);

set(0, 'DefaultAxesFontName', 'Courier');
step = 16;

width = 260;
Label = ['MS-DOS File: ', 'DOS_filename'];
LPosn = [40 (height-(3*step)) width 20];
LH(1) = uicontrol('AcqStatH','Style','Text','Position',LPosn);
set(LH(1), 'BackgroundColor', LIGHTGREY, 'HorizontalAlignment', 'Left', 'String', Label);

Label = ['RISC-OS File: ', 'fileName'];
LPosn = [40 (height-(4*step)) width 20];
LH(2) = uicontrol('AcqStatH','Style','Text','Position',LPosn);
set(LH(2), 'BackgroundColor', LIGHTGREY, 'HorizontalAlignment', 'Left', 'String', Label);

Label = ['Version: ', num2str(version)];
LPosn = [40 (height-(6*step)) width 20];
LH(3) = uicontrol('AcqStatH','Style','Text','Position',LPosn);
set(LH(3), 'BackgroundColor', LIGHTGREY, 'HorizontalAlignment', 'Left', 'String', Label);

Label = ['Source: ', 'spectrumSource'];
LPosn = [40 (height-(7*step)) width 20];
LH(4) = uicontrol('AcqStatH','Style','Text','Position',LPosn);
set(LH(4), 'BackgroundColor', LIGHTGREY, 'HorizontalAlignment', 'Left', 'String', Label);

Label = ['Start Time: ', 'startTime'];
LPosn = [40 (height-(8*step)) width 20];
LH(5) = uicontrol('AcqStatH','Style','Text','Position',LPosn);
set(LH(5), 'BackgroundColor', LIGHTGREY, 'HorizontalAlignment', 'Left', 'String', Label);

Label = ['Diode Temp: ', 'num2str(diodeTempK), ' K'];
LPosn = [40 (height-(10*step)) width 20];
LH(6) = uicontrol('AcqStatH','Style','Text','Position',LPosn);
set(LH(6), 'BackgroundColor', LIGHTGREY, 'HorizontalAlignment', 'Left', 'String', Label);

Label = ['Scan Variable: ', 'xUnits'];
LPosn = [40 (height-(12*step)) width 20];
LH(7) = uicontrol('AcqStatH','Style','Text','Position',LPosn);
set(LH(7), 'BackgroundColor', LIGHTGREY, 'HorizontalAlignment', 'Left', 'String', Label);

Label = ['Scan Minimum: ', 'num2str(scanMin)'];
LPosn = [40 (height-(13*step)) width 20];
LH(8) = uicontrol('AcqStatH','Style','Text','Position',LPosn);
set(LH(8), 'BackgroundColor', LIGHTGREY, 'HorizontalAlignment', 'Left', 'String', Label);

Label = ['Scan Maximum: ', 'num2str(scanMax)'];
LPosn = [40 (height-(14*step)) width 20];
LH(9) = uicontrol('AcqStatH','Style','Text','Position',LPosn);
set(LH(9), 'BackgroundColor', LIGHTGREY, 'HorizontalAlignment', 'Left', 'String', Label);

Label = ['Data Points: ', 'int2str(dataPoints)'];
LPosn = [40 (height-(15*step)) width 20];
LH(10) = uicontrol('AcqStatH','Style','Text','Position',LPosn);
set(LH(10), 'BackgroundColor', LIGHTGREY, 'HorizontalAlignment', 'Left', 'String', Label);

Label = ['Temperature: ', ''];
if (cdfTempK==0 & STDcdfTK==0)
Label = [Label, 'N / A'];
else
Label = [Label, Y, num2str(cdfTempK), '+/- ', num2str(STDcdfTK), ' K'];
end

```



```

%.....
% 'quit' - cleans up workspace and terminates
%.....
elseif(strcmp(option,'quit'))
    delete(FigH);
end

Label = ['Pressure:'];
if (ceil(PressT/10) & STDceilPT==0)
    Label = [Label,'N/A'];
else
    Label = [Label, num2str(ceil(PressT), '+/- ', num2str(STDceilPT), ' Torr)];
end
LPosn = [40 (height-(18*step)) lwidth 20];
LH(12) = uicontrol(AcqStatH,'Style','Text','Position',LPosn);
set(LH(12),'BackgroundColor','LIGHTGREY','HorizontalAlignment','Left','String',Label);

set(0,'DefaultAxesFontName','TimesNewRoman');

% define OK push-button
OKPosn = [120 15 100 30];
OKHString = 'OK';
OKH = uicontrol(AcqStatH,'Style','PushButton','Position',OKPosn);
set(OKH,'String','OKHString','Callback','vliddaq('KillStats'),?);

%.....
% 'KillStats' - terminates acquisition statistics display
%.....
elseif(strcmp(option,'KillStats'))
    delete(AcqStatH);
end

%.....
% 'xyData' - saves data to file in flat ascii 'x y1 y2' format
%.....
elseif(strcmp(option,'xyData'))
    [filePath] = uiputfile('','Save X-Y Data As?');
    % concatenate path and filename strings
    filePath = [path, file];
    if (channels(1)~=1 & channels(2)~=1)
        %scanVariant= flipud(rot90(scanVariant))
        xyData = [scanVariant transm etalon];
    elseif (channels(1)~=1)
        %scanVariant = freq2;
        scanVariant= flipud(rot90(scanVariant));
        transm = ((transm));
    end
    xyData = {transm}
    elseif (channels(2)~=1)
        xyData = {scanVariant etalon};
    end
end

eval([save 'filePath','xyData -ascii']);

```

```

%%
%% Ray Tracing Program for Herriott Cell
%%
%% Iain F Howieson 1995
%%
%%
%% Filename: HERRJOTT.M
%%
%% Input parameters are:
%%
%% 'd' - mirror separation
%% 'a' - mirror rotation angle
%% 'e' - error in mirror radius
%% 'nmx' - integer that defines the pattern
%%          of transits through the cell
%% 'nmy' - integer that defines the pattern
%%          of transits through the cell
%% 'n' - number of passes
%% 'x' - x coordinate of the injection
%%          beam on the back mirror
%% 'y' - y coordinate of the injection
%%          beam on the back mirror
%% 'x0' - initial x injection beam coordinate
%% 'y0' - initial y injection beam coordinate
%%
%%
function Zn = Herriott(d,a,e,nmx,nmy,n,x,y,x0,y0)

% Work out Radius of curvature for X and Y axis
Rx = 30%*67.425d/(1-cos(pi*nmx/n))
Ry = 30%*77.274d/(1-cos(pi*nmy/n))

%Rx = 50/(1-cos(pi*84/202))
%Ry = 50/(1-cos(pi*78/202))

% Calculate Injection Slopes for Circular Spot Pattern
%a = (x0.^2)/d.^2)/30;
%b = ((y0.^2)-(a.*d).^2)/.5)/y0)/d;
%ax = x0-(a.*d)
%ay = y0-(b.*d)
% Work out radii assuming error e

Rxe = Rx/(1+e)
Rye = Ry/(1-e)

%%
%%
%% Work out initial X and Y slopes
dx = (x-x0)/d;
dy = (y-y0)/d;
%dx = (y0/Rx)^(sqrt(((2.*Rx)-d)/d))
%dy = (sqrt(y0.^2 - ((dx*d).^2))-y0)/d

% Define initial ray vector(Zo)
Zo = [x0,dx;y0,dy];

% Define free space propagation vector(D)
D = [1 d 0 0;0 1 0 0;0 0 1 d;0 0 0 1];

% Define mirror reflection matrix(R)
R = [1 0 0 0;-2/Rxe 1 0 0;0 0 1 0;0 0 -2/Rye 1];

% Calculate half rotation angle(a) in radians(o)
o = (pi/360)*a;

% Define mirror rotation matrices (Tp),(Tm),(T2m)
Tp = [cos(o) 0 sin(o) 0 0 cos(o) 0 sin(o);-sin(o) 0 cos(o) 0 0 -sin(o) 0 cos(o)];
Tm = [cos(-o) 0 sin(-o) 0 0 cos(-o) 0 sin(-o);-sin(-o) 0 cos(-o) 0 0 -sin(-o) 0 cos(-o)];
T2m = [cos(-2*o) 0 sin(-2*o) 0 0 cos(-2*o) 0 sin(-2*o);-sin(-2*o) 0 cos(-2*o) 0 0 -sin(-2*o) 0 cos(-2*o)];

% Define ray matrix for one complete cycle(C)
C = Tp*R*D*T2m*R*D*Tp;

```

```
%C = R*D
```

```
% Generate Point matrix(Zn)
```

```
for i = 1:n/2
```

```
    Zn(i) = C*i*Zo;
```

```
end
```

```
%Z = R*D*Zn(,(n/2)-1))
```

```
% Plot X and Y coordinates
```

```
plot(Zn(1,:),Zn(3,:),'ro')
```

```
xl = 3 + 0.8;
```

```
axis([-xl xl -xl xl])
```

```
axis('equal')
```



```

global Scanend n versionID version DOS_frame fileName XZ h V G data
global plotAxes axesH ModInd ModH pts SpecH StartH EndH no

```

```

% define colours
WHITE = [1 1 1];
LIGHTGREY = [0.7529 0.7529 0.7529];

```

```

% .....
% 'start' - initialise environment and generate figure with menus
% .....

```

```

if(strcmp(option,'start'))

```

```

    FigH = figure('Visible','Off'); % instantiate a hidden figure...

```

```

    % label the figure
    set(FigH,'Name','2nd Harmonic Display Utilities');
    set(FigH,'NumberTitle','off');

```

```

    gtools; % add a graph tools menu to it

```

```

    % generate a menu for selecting filing options
    SecondM = uimenu('Label','2F');

```

```

    % generate analysis menu options
    mh(1) = uimenu(SecondM,'Label','Simulated');
    mh(2) = uimenu(mh(1),'Label','Load Data');
    set(mh(2),'Callback','secharm('loaddata')');
    mh(3) = uimenu(SecondM,'Label','Real');
    mh(4) = uimenu(mh(3),'Label','Load Spec');
    set(mh(4),'Callback','secharm('loadspec')');
    mh(5) = uimenu(SecondM,'Label','Combined');
    mh(6) = uimenu(mh(5),'Label','Load Spec');
    set(mh(6),'Callback','secharm('loadspec')');
    mh(7) = uimenu(SecondM,'Label','Quit','Separator','On');
    set(mh(7),'Callback','secharm('quit')');

```

```

    set(FigH,'visible','on'); % ...show the figure

```

```

    FIT_NUMBER = 1;

```

```

    % .....
    % Load File
    % .....

```

```

    elseif(strcmp(option,'loaddata'))

```

```

        [DOS_frame, path] = uigetfile(DefaultPath,'Enter input filename');

```

```

        % concatenate path and filename strings
        FilePath = [path, DOS_frame];

```

```

        % open data file for reading
        [sfd, ErrorMessage] = fopen(FilePath, 'r');

```

```

function secharm(option)
% .....

```

```

%* Filename: SecondHam.M
%* Version : 1.01
%* Update : 14th March 1996
%* Usage : secharm(<option>)
%*

```

```

%* (C) Iain Howison
%* Dept. Physics & Applied Physics
%* University of Strathclyde
%* Glasgow
%*

```

```

%* File which contains routines for generating and displaying
%* Second Harmonic Spectra
%* . . . . .
%* The various routines are represented by 'options'
%* which allow a matlab-style self-contained recursive function.
%*

```

```

%* Possible <option>'s are:
%*

```

```

%* 'start' - initialise environment
%*

```

```

%* 'simulated' - load line parameters from file and generate
%* direct spectrum with either Doppler-Lorentz
%* or Voigt lineshape. Then generate
%* corresponding 2F spectrum.
%*

```

```

%* 'real' - loads real spectrum and generates 2F
%*

```

```

%* 'combined' - combines real and simulated spectra and
%* generates 2F
%*

```

```

%* 'quit' - cleans up workspace and terminates
%*

```

```

%* .....
%* ! define paths for default location of Secharm files !
%* .....
DefaultPath = 'c:\apps\matlab\work\secharmo\*.*';
%* .....
%* check input argument and declare globals
%* .....

```

```

if (nargin < 1)
    option = 'start';
end;

```

```

global FigH ParamH ChoiceH TempH MassH MinH MaxH NoH Dop Lor Scanstart

```

```

% check that no file errors occurred
if (sfid==1)
    disp(ErrorMessage)
    return
end
data=[];
data = fscanf(sfid,'%f');
fclose(sfid);
setcharm('param');

%.....
% Load File
%.....
checkbox(stcamp(option,'loadspec'))

[DOS_frame, path] = uigetfile(DefaultPath,'Enter input filename');

% calculate path and filename strings
FilePath = [path, DOS_frame];

% open data file for reading
[sfid, ErrorMessage] = fopen(FilePath, 'r');

% check that no file errors occurred
if (sfid==1)
    disp(ErrorMessage)
    return
end
data=[];
data = fscanf(sfid,'%f');
fclose(sfid);
setcharm('specinfo');

%.....
% create the spec info window
%.....
else if (stcamp(option,'specinfo'))

Size = size(data);
S = Size(1);
I = S/2;
data = reshape(data,2,I);
data = rot90(data);
data = flipud(data);
Size = size(data);
pts = Size(1);
start = data(1);
End = data(pts);

ScaleFig = gca;

% CurrentScale= axis;

% instantiate a new figure without a menu bar
left = 307;
bottom = 250;
width = 220;
height = 210;
ASPosn = [left bottom width height];
MBSStatus = 'none';
SpecH = figure('Position',ASPosn, 'MenuBar', MBSStatus);

% prevent it from being resized
RStatus = 'off';
set(SpecH, 'Resize', RStatus);

% name it
NTSStatus = 'off';
ASName = 'Spectral Information';
set(SpecH, 'NumberTitle', NTSStatus);
set(SpecH, 'Name', ASName);

% give it a grey background
ASColour = LIGHTGREY;
set(SpecH, 'Color', ASColour);

% define editbox and label for Dopp HW
TempLabel = LineWidth/cm-1;
TempDefault = num2str(200);
TempLPosn = [20 170 120 20];
TempLH = uicontrol('SpecH', 'Style', 'Text', 'Position', TempLPosn);
set(TempLH, 'BackgroundColor', WHITE, 'String', TempLabel);
TempH = uicontrol('SpecH', 'Style', 'Edit', 'Position', TempPosn);
set(TempH, 'BackgroundColor', WHITE, 'String', TempDefault);

% define label for Scan Start
StartLabel = 'Scan Start/cm-1';
StartDefault = num2str(start);
StartLPosn = [20 130 120 20];
StartPosn = [140 130 60 20];
StartLH = uicontrol('SpecH', 'Style', 'Text', 'Position', StartLPosn);
set(StartLH, 'BackgroundColor', WHITE, 'String', StartLabel);
StartH = uicontrol('SpecH', 'Style', 'Edit', 'Position', StartPosn);
set(StartH, 'BackgroundColor', WHITE, 'String', StartDefault);

% define editbox and label for Scan end
EndLabel = 'Scan End/cm-1';
EndDefault = num2str(End);
EndLPosn = [20 90 120 20];
EndPosn = [140 90 60 20];
EndLH = uicontrol('SpecH', 'Style', 'Text', 'Position', EndLPosn);
set(EndLH, 'BackgroundColor', WHITE, 'String', EndLabel);
EndH = uicontrol('SpecH', 'Style', 'Edit', 'Position', EndPosn);
set(EndH, 'BackgroundColor', WHITE, 'String', EndDefault);

% define editbox and label for Modulation Index
ModLabel = 'Mod. Index';
ModDefault = num2str(2.2);

```



```

ModLPosn = [20 50 120 20];
ModPosn = [140 50 60 20];
ModLH = uicontrol(SpecH, 'Style', 'Text', 'Position', ModLPosn);
set(ModLH, 'BackgroundColor', 'WHITE', 'String', ModLabel);
ModH = uicontrol(SpecH, 'Style', 'Edit', 'Position', ModPosn);
set(ModH, 'BackgroundColor', 'WHITE', 'String', ModDefault);

% define OK push-button
OKPosn = [60 10 100 30];
OKHString = 'OK';
OKH = uicontrol(SpecH, 'Style', 'PushButton', 'Position', OKPosn);
set(OKH, 'String', OKHString, 'Callback', 'secharm("killspec");');

%*****
% 'killspec' - deletes spec dialog box
%*****
checkbox1=strncmp(option, 'killspec')

% get the parameters...
Dop = str2num(get(TempH, 'String'));
Scanstart = str2num(get(StartH, 'String'));
Scanend = str2num(get(EndH, 'String'));
ModInd = str2num(get(ModH, 'String'));
Scanstart = data(1);
Scanend = data(2);
n = pts;
Lor = 0;
V = data(:,1);
G = data(:,2);
% remove the dialog box...
delete(SpecH);
secharm('deniv');

%*****
% create the axis fparam window
%*****
else if (strcmp(option, 'param'))

ScaleFig = gca;

%
CurrentScale=axis;

% return to rescale dialog box
% figure(AxiScaleH)

% instantiate a new figure without a menu bar
left = 307;
bottom = 250;
width = 220;
height = 240;
ASPosn = [left bottom width height];
MBStatus = 'none';

ParamH = figure('Position', ASPosn, 'MenuBar', 'MBStatus');

% prevent it from being resized
RStatus = 'off';
set(ParamH, 'Resize', RStatus);

% name it
NTStatus = 'off';
ASName = 'Spectral Parameters';
set(ParamH, 'NumberTitle', NTStatus);
set(ParamH, 'Name', ASName);

% give it a grey background
ASColour = LIGHTGREY;
set(ParamH, 'Color', ASColour);

%*****
% create the edit box and push-button that initiates controls
%*****

% define editbox and label for Dopp HW
TempLabel = 'Doppler H W :';
TempDefault = num2str(0.06);
TempLPosn = [20 210 120 20];
TempPosn = [140 210 60 20];
TempLH = uicontrol(ParamH, 'Style', 'Text', 'Position', TempLPosn);
set(TempLH, 'BackgroundColor', 'WHITE', 'String', TempLabel);
TempH = uicontrol(ParamH, 'Style', 'Edit', 'Position', TempPosn);
set(TempH, 'BackgroundColor', 'WHITE', 'String', TempDefault);

% define editbox and label for Lorentz HW
MassLabel = 'Lorentz H W :';
MassDefault = num2str(0.1);
MassLPosn = [20 180 120 20];
MassPosn = [140 180 60 20];
MassLH = uicontrol(ParamH, 'Style', 'Text', 'Position', MassLPosn);
set(MassLH, 'BackgroundColor', 'WHITE', 'String', MassLabel);
MassH = uicontrol(ParamH, 'Style', 'Edit', 'Position', MassPosn);
set(MassH, 'BackgroundColor', 'WHITE', 'String', MassDefault);

% define editbox and label for Scan Start
MinLabel = 'Scan Start/cm-1 :';
MinDefault = num2str(5999);
MinLPosn = [20 150 120 20];
MinPosn = [140 150 60 20];
MinLH = uicontrol(ParamH, 'Style', 'Text', 'Position', MinLPosn);
set(MinLH, 'BackgroundColor', 'WHITE', 'String', MinLabel);
MinH = uicontrol(ParamH, 'Style', 'Edit', 'Position', MinPosn);
set(MinH, 'BackgroundColor', 'WHITE', 'String', MinDefault);

% define editbox and label for Scan End
MaxLabel = 'Scan End/cm-1 :';
MaxDefault = num2str(6001);
MaxLPosn = [20 120 120 20];
MaxPosn = [140 120 60 20];
MaxLH = uicontrol(ParamH, 'Style', 'Text', 'Position', MaxLPosn);

```



```

set(MaxLH, 'BackgroundColor', WHITE, 'String', MaxLabel);
MaxH = uicontrol('ParentH', 'Style', 'Edit', 'Position', MaxPosn);
set(MaxH, 'BackgroundColor', WHITE, 'String', MaxDefault);

% define editbox and label for No. of Pts
NoLabel = No. of Pts ;
NoDefault = num2str(600);
NoLPosn = [20 90 120 20];
NoPosn = [140 90 60 20];
NoLH = uicontrol('ParentH', 'Style', 'Text', 'Position', NoLPosn);
set(NoLH, 'BackgroundColor', WHITE, 'String', NoLabel);
NoH = uicontrol('ParentH', 'Style', 'Edit', 'Position', NoPosn);
set(NoH, 'BackgroundColor', WHITE, 'String', NoDefault);

% define editbox and label for Modulation Index
ModLabel = Mod. Index ;
ModDefault = num2str(2.2);
ModLPosn = [20 60 120 20];
ModPosn = [140 60 60 20];
ModLH = uicontrol('ParentH', 'Style', 'Text', 'Position', ModLPosn);
set(ModLH, 'BackgroundColor', WHITE, 'String', ModLabel);
ModH = uicontrol('ParentH', 'Style', 'Edit', 'Position', ModPosn);
set(ModH, 'BackgroundColor', WHITE, 'String', ModDefault);

% define OK push-button
OKPosn = [60 10 100 30];
OKHString = 'OK';
OKH = uicontrol('ParentH', 'Style', 'PushButton', 'Position', OKPosn);
set(OKH, 'String', OKHString, 'Callback', 'setharm("killparam");');

% killparam - deletes offset dialog box
% killparam' - deletes offset dialog box
% killparam' - deletes offset dialog box
elseif(strcmp(option, 'killparam'))

% get the parameters...
Dop = str2num(get(TempH, 'String'));
Lor = str2num(get(MassH, 'String'));
Scanstart = str2num(get(MinH, 'String'));
Scanend = str2num(get(MaxH, 'String'));
n = str2num(get(NoH, 'String'));
Modlnd = str2num(get(ModH, 'String'));

% remove the dialog box...
delete(ParamH);
setharm('choice');

% Doppler, Lorentz, Voigt Choice
% Doppler, Lorentz, Voigt Choice
elseif(strcmp(option, 'choice'))

% instantiate a new figure without a menu bar
left = 307;
bottom = 250;
width = 120;
height = 140;
ASPosn = [left bottom width height];
MBStatus = 'none';
ChoiceH = figure('Position', ASPosn, 'MenuBar', MBStatus);

% prevent it from being resized
RStatus = 'off';
set(ChoiceH, 'Resize', RStatus);

% name it
NTSStatus = 'off';
ASName = 'Line Type';
set(ChoiceH, 'NumberTitle', NTSStatus);
set(ChoiceH, 'Name', ASName);

% give it a grey background
ASColour = LIGHTGREY;
set(ChoiceH, 'Color', ASColour);

% define Doppler push-button
DoppPosn = [10 95 100 30];
DoppHString = 'Doppler';
DoppH = uicontrol(ChoiceH, 'Style', 'PushButton', 'Position', DoppPosn);
set(DoppH, 'String', DoppHString, 'Callback', 'setharm("Doppler");');

% define Lorentz push-button
LorPosn = [10 55 100 30];
LorHString = 'Lorentz';
LorH = uicontrol(ChoiceH, 'Style', 'PushButton', 'Position', LorPosn);
set(LorH, 'String', LorHString, 'Callback', 'setharm("Lorentz");');

% define Voigt push-button
VoiPosn = [10 15 100 30];
VoiHString = 'Voigt';
VoiH = uicontrol(ChoiceH, 'Style', 'PushButton', 'Position', VoiPosn);
set(VoiH, 'String', VoiHString, 'Callback', 'setharm("Voigt");');

% Calculate Doppler Spectrum
% Calculate Doppler Spectrum
% Calculate Doppler Spectrum
elseif(strcmp(option, 'Doppler'))

delete(ChoiceH);
Size = size(data);
S = Size(1);
r = S/2;
data = reshape(data, 2, r);
data = rot90(data);
data = flipud(data);
Size = size(data);
nlines = Size(1);

% DOPPLER WIDTH

```

```

gammad = Dop;
Lor = 0;
sechamf('deriv');

%*****
% Calculate Voigt Spectrum
%*****
elseif(strcmp(option,'Voigt'))

delete(ChoiceF);
Size = size(data);
S = Size(1);
r = S/2;
data = reshape(data,2,r)
data = rot90(data);
data = flipud(data);
Size = size(data);
nlines = Size(1);

gammad = Dop;
gammal = Lor;

% VOIGT PROFILE
Interval = (Scanend - Scanstart)/n;
for i=1:n,
V(i)=Scanstart + (i * Interval);
end

for j=1:nlines
for i=1:n
wd = 2 * gammad;
wl = 2 * gammal;
wv = (0.5346 * wf) + (((0.2166 * (wl.^2)) + (wd.^2)).^0.5);
Ivo = 1./wv.* (1.065 + (0.447 * (wl/wv)) + (0.058 * ((wl/wv).^2)));
dN = V(i) - data(j,1);
voigtfunct = real(((1 - (wl/wv)) * (exp(-2.2772 * ((dN/wv).^2))) + ((wl/wv)/(1 + (4 * ((dN/wv).^2)))) + ((0.016 * (1 - (wl/wv)) * (wl/wv)) * ((exp(-0.4 * ((dN/wv).^2.25)) - (10/(10 + ((dN/wv).^2.25))))));
Voigt(i) = data(j,2) * voigtfunct;
G(j,i)=[Voigt(i)];
end
end

%ADDS ALL THE VOIGT PROFILES TOGETHER AND NORMALIZES THEM
%G = sum(G);
%a = max(G);
%a = G/a;
%Lor = sqrt((Dop.^2)+(Lor.^2))
%Dop = 0;
V = flipud(rot90(V));
G = flipud(rot90(G));
G = G;

save ish4 V G -ascii

gammad = Dop;
Lor = 0;

%DOPPLER PROFILE
Interval = (Scanend - Scanstart)/n;
for i=1:n
V(i)=Scanstart + (i * Interval);
end

for j=1:nlines
for i=1:n
Dopp(i) = -data(j,2) * (sqrt(log(2)/pi)) * (exp(-log(2) * (((V(i)-data(j,1))/gammad).^2)))/(gammad);
G(j,i)=[Dopp(i)];
end
end

%ADDS ALL THE DOPPLER PROFILES TOGETHER AND NORMALIZES THEM
G = sum(G);
a = max(G);
G = (G/a);
sechamf('deriv');

%*****
% Calculate Lorentz Spectrum
%*****
elseif(strcmp(option,'Lorentz'))

delete(ChoiceF);
Size = size(data);
S = Size(1);
r = S/2;
data = reshape(data,2,r)
data = rot90(data);
data = flipud(data);
Size = size(data);
nlines = Size(1);

%DOPPLER WIDTH
Dop = 0;
gammal = Lor;

%LORNTZ PROFILE
Interval = (Scanend - Scanstart)/n;
for i=1:n;
V(i)=Scanstart + (i * Interval);
end
for j=1:nlines
for i=1:n
Lorentz(i) = data(j,2) * gammal / (pi * (((V(i)-data(i)).^2) + (gammal).^2));
G(j,i)=[Lorentz(i)];
end
end

%ADDS ALL THE LORENTZ PROFILES TOGETHER AND NORMALIZES THEM
G = sum(G);

```

```

plot(V,G)
%*****
% Calculate 2F Spectrum
%*****
elseif(strcmp(option,'deriv'))
    gamma = Dop+Lor
    moddepth = (ModInd*gamma)
    modint = (moddepth/(Scanend - Scanstart))^n;
    modint = floor(modint);
    x1 = V;
    y1 = G;
    start = modint;
    end1 = n - modint;
    for j = 1 end1 - modint - 1
        theta = 0.01*pi;
        x = (moddepth * (-cos(theta))) + x1(modint + 1 + j);
        y = interp1(x1,y1,x,'cubic');
        for i = 1:315
            P(i) = (y(i)^2*cos(2*theta(i)));
            end
            h(i) = trapz(theta,P);
            XZ(i) = x1(modint + 1 + j);
            end
            h = 2*real(h)/pi
        secham('dispSpec');
        %*****
        % 'dispSpec' - displays the spectrum
        %*****
        elseif(strcmp(option,'dispSpec'))
            axesH = getobj(FigH,'axes');
            if (length(axesH) > 0)
                delete(axesH);
            end
            % instantiate lower residual axes
            left = 0.23;
            bottom = 0.11;
            width = 0.5475;
            height = 0.365;
            etalonPosition = [left bottom width height];
            etalonH = axes('Position', etalonPosition);
            % plot direct channel
            plot(V,G)

```

```

% automate the residual axes aspect-ratio
set(etalonH, 'AspectRatio', [-nan -nan])

% label the residual axes
xlabel('Wavelength/cm-1');
ylabel('Direct')

% instantiate upper fit axes
left = 0.23;
bottom = 0.57;
width = 0.5475;
height = 0.365;
transmPosition = [left bottom width height];
transmH = axes('Position', transmPosition);

% plot spectral fit
plot(XZ,h)
ylabel('Second Harmonic')
save sav sav -ascii;
P = max(h)
N = min(h)
R = P/N
% automate the fit axes aspect-ratio
set(transmH, 'AspectRatio', [-nan -nan])
end
end
end

```



```

function dataload(option)
%*****
% ! define paths for default location of Secham files 1 *
%*****
DefaultPath = 'c:\apps\matlab\work\secharmo\*. *';
%*****
% check input argument and declare globals *
%*****
if (nargin < 1)
    option = 'start';
end,
% check input arguments...
global data ref datab
% define colours
WHITE = [1 1 1];
LIGHTGREY = [0.7529 0.7529 0.7529];
%*****
% 'start' - initialise environment and generate figure with menus *
%*****
if(strcmp(option,'start'))
    [DOS_fname, path] = uigetfile(DefaultPath,'Enter input filename for data');
% concatenate path and filename strings
FilePath = [path, DOS_fname];
% open data file for reading
[sfid, ErrorMessage] = fopen(FilePath, 'r');
% check that no file errors occurred
if (sfid==~-1)
    disp(ErrorMessage)
    return
end
data=[];
data = fscanf(sfid, '%f');
fclose(sfid);
databload('getref');
size(data)
elseif(strcmp(option,'getref'))
    [DOS_fname, path] = uigetfile(DefaultPath,'Enter input filename for ref data');
% concatenate path and filename strings
%FilePath = [path, DOS_fname];
% open data file for reading
%[sfid, ErrorMessage] = fopen(FilePath, 'r');

```

```

% check that no file errors occurred
%if (sfid==~-1)
% disp(ErrorMessage)
% return
% end
%ref=[];
%ref = fscanf(sfid, '%f');
%fclose(sfid);
load call181 -ascii
ref = call181;
% Removes Background from Reference and Measured Background
databload('background');
elseif(strcmp(option,'background'))
    start1 = data(1);
    end1 = data(250);
    int = (end1-start1)/249;
    back = start1:int:end1;
    back2 = flipud(rot90(back));
    data = data-back;
    size(data)
    start2 = ref(1);
    end2 = ref(250);
    int2 = (end2-start2)/249;
    back2 = start2:int2:end2;
    back2 = flipud(rot90(back2));
    ref = ref-back2;
    y = fft(data,20000);
    z = fft(ref,20000);
    a = zeros(17000,1);
    y(1501:18500)=a;
    z(1501:18500)=a;
    b = ifft(y);
    c = ifft(z);
    data = real(b(1:250));
    ref = real(c(1:250));
    databload('auto')
elseif(strcmp(option,'auto'))
    refpad = zeros(512,1);
    refpad(1:250)=ref;
    databpad = zeros(512,1);
    databpad(1:250) = data;
    fbref = fft(refpad);
    conji = conj(fft(databpad));
    auto = fftshift(real(fft(conji.*fbref)));
    sized(auto)
    save auto auto -ascii
j=1;
for i = 2:512

```

```

plot(X,ref,r);
%save X X -ascii
%save dataf data -ascii;
%save reff ref -ascii;
%save conc conoq -ascii;
save data data -ascii
save ref ref -ascii
end
end

```

```

if abs(auto(i)) > abs(auto(i))
j=i;
else
q=i;
end
end
f = 2.56.255;
n = f(i);

```

```

if n < 0
datapad = zeros(250,1);
datapad(1+25.250+n) = data((-n)+1+25.250);
data = datapad;
else

```

```

datapad = zeros(250,1);
datapad(n+1.250) = data(1.250-n);
data = datapad;
end

```

```
end
```

```
%Peak Finder and Wavelength Calibration
```

```

l = 1;
%for n = 90.5:200
%dataq = data(n-25:n);
%refq = ref(n-25:n);
%a = nls(dataq,refq);
%concoq() = (((18.975/r)+(19.991/n))^2)/1.3772;
%l=l+1;
%end
%plot(conoq);

```

```

s=1;
x = nls(data,ref)
data = x.'data;
l = (x/2.122)
m = (x/12.1066)
conc = 18.875*x
conc = (((18.975/l)+(3.498/m))^2);
conc = (((((18.975/x)+(19.991/n))^2)/1.3772)^3);
f = ref - data;

```

```

save f f -ascii;
d = diff(ref);
for r = 40:210
if d(r+1) * d(r) < 0
zer(s) = r
s = s+1;
end
end

```

```

end
wavenum = [0.002 0.25953,6002 59230 19,6002 55938 416,6002 524026 7,6002 488563];
c = polyfit(zer,wavenum,1);
x = 1.250;
X = (c(1) * x) + c(2);
plot(X,data);
hold on;

```

Appendix 3 - Linelists

C12 Acetylene(6433 - 6454 cm⁻¹, P = 2Torr, L = 25.1m, T_v = 22°C)

6.4336222e+003	9.9896151e-001	6.4377095e+003	9.9749645e-001	6.4429253e+003	9.9927872e-001
6.4337404e+003	9.9899687e-001	6.4378223e+003	9.9876730e-001	6.4429380e+003	9.9828144e-001
6.4337784e+003	9.9417021e-001	6.4378817e+003	9.9928269e-001	6.4430955e+003	9.9849426e-001
6.4337998e+003	9.9900390e-001	6.4380032e+003	9.9672273e-001	6.4432504e+003	9.8878235e-001
6.4338111e+003	9.9834460e-001	6.4380907e+003	9.9912531e-001	6.4432991e+003	9.9823022e-001
6.4338245e+003	9.9935338e-001	6.4382991e+003	9.9944743e-001	6.4433913e+003	9.9047546e-001
6.4338445e+003	9.9660924e-001	6.4383204e+003	9.9949941e-001	6.4434941e+003	9.9942518e-001
6.4339520e+003	9.9930974e-001	6.4383425e+003	9.9949678e-001	6.4435288e+003	9.8358879e-001
6.4340087e+003	9.9948264e-001	6.4383598e+003	9.9898487e-001	6.4435935e+003	9.9581176e-001
6.4340581e+003	9.9729549e-001	6.4383785e+003	9.9949503e-001	6.4436630e+003	9.7515364e-001
6.4341503e+003	9.9345750e-001	6.4384119e+003	9.9917636e-001	6.4437097e+003	9.9857767e-001
6.4342244e+003	9.9787305e-001	6.4384559e+003	9.9676202e-001	6.4437177e+003	9.9892158e-001
6.4342951e+003	9.9712616e-001	6.4384700e+003	9.9927420e-001	6.4437431e+003	9.9339595e-001
6.4343392e+003	9.9640528e-001	6.4384760e+003	9.9883315e-001	6.4437518e+003	9.9547823e-001
6.4344554e+003	9.8452803e-001	6.4385074e+003	9.9907640e-001	6.4437638e+003	9.9121832e-001
6.4345060e+003	9.9792171e-001	6.4385354e+003	9.9948705e-001	6.4438377e+003	9.9752094e-001
6.4346141e+003	9.8187412e-001	6.4385507e+003	9.9933425e-001	6.4439191e+003	9.9919580e-001
6.4346315e+003	9.9188440e-001	6.4385781e+003	9.9849333e-001	6.4440246e+003	9.9526743e-001
6.4346395e+003	9.9145926e-001	6.4386231e+003	9.9707453e-001	6.4440667e+003	9.9689746e-001
6.4346728e+003	9.9868496e-001	6.4386365e+003	9.9926073e-001	6.4440920e+003	9.9941145e-001
6.4346789e+003	9.9859861e-001	6.4386538e+003	9.9152404e-001	6.4441167e+003	9.9634132e-001
6.4346875e+003	9.9893362e-001	6.4387900e+003	9.9526808e-001	6.4441755e+003	9.9949488e-001
6.4346962e+003	9.9854795e-001	6.4388441e+003	9.9787406e-001	6.4442135e+003	9.9622374e-001
6.4347082e+003	9.9902635e-001	6.4389415e+003	9.9938982e-001	6.4442302e+003	9.9765328e-001
6.4347116e+003	9.9901414e-001	6.4389943e+003	9.9780683e-001	6.4442429e+003	9.9578465e-001
6.4347223e+003	9.9913285e-001	6.4390670e+003	9.9666498e-001	6.4443243e+003	9.9859619e-001
6.4347396e+003	9.9853919e-001	6.4390697e+003	9.9667757e-001	6.4443891e+003	9.9589910e-001
6.4347583e+003	9.9939657e-001	6.4390897e+003	9.8598304e-001	6.4444111e+003	9.9942936e-001
6.4347710e+003	9.9885974e-001	6.4391498e+003	9.9436356e-001	6.4444245e+003	9.9848776e-001
6.4347850e+003	9.9948056e-001	6.4392039e+003	9.5726249e-001	6.4444345e+003	9.9881580e-001
6.4348264e+003	9.6328107e-001	6.4392279e+003	9.9905353e-001	6.4444579e+003	9.8471641e-001
6.4349920e+003	9.9933789e-001	6.4392406e+003	9.9753908e-001	6.4445280e+003	9.7670234e-001
6.4350293e+003	9.9929691e-001	6.4393728e+003	9.9786020e-001	6.4446414e+003	9.9817793e-001
6.4350307e+003	9.9929753e-001	6.4394571e+003	9.5927359e-001	6.4446541e+003	9.9887579e-001
6.4350529e+003	9.9255612e-001	6.4396407e+003	9.9095469e-001	6.4446681e+003	9.9805558e-001
6.4350956e+003	9.9866162e-001	6.4396514e+003	9.9434161e-001	6.4446848e+003	9.9925971e-001
6.4351030e+003	9.9901741e-001	6.4396694e+003	9.7103631e-001	6.4447082e+003	9.9852364e-001
6.4351070e+003	9.9895913e-001	6.4397956e+003	9.9897934e-001	6.4447242e+003	9.9935997e-001
6.4351136e+003	9.9947694e-001	6.4398403e+003	9.9932227e-001	6.4447469e+003	9.9872257e-001
6.4351303e+003	9.9621895e-001	6.4398964e+003	9.9470457e-001	6.4448464e+003	9.9717724e-001
6.4353199e+003	9.9747327e-001	6.4399524e+003	9.9852599e-001	6.4448764e+003	9.9931717e-001
6.4353339e+003	9.9893640e-001	6.4401267e+003	9.7676897e-001	6.4448918e+003	9.9875799e-001
6.4353399e+003	9.9883660e-001	6.4401956e+003	9.9831396e-001	6.4449345e+003	9.9931651e-001
6.4353513e+003	9.9934720e-001	6.4402083e+003	9.9908391e-001	6.4449425e+003	9.9933340e-001
6.4353600e+003	9.9907254e-001	6.4402250e+003	9.9613982e-001	6.4449942e+003	9.9385957e-001
6.4353973e+003	9.9927373e-001	6.4402343e+003	9.9663180e-001	6.4450656e+003	9.9941561e-001
6.4354366e+003	9.9887977e-001	6.4402390e+003	9.9650915e-001	6.4452799e+003	9.9401378e-001
6.4354941e+003	9.9882137e-001	6.4403051e+003	9.9585383e-001	6.4452800e+003	9.9433956e-001
6.4355074e+003	9.9947844e-001	6.4403218e+003	9.9837360e-001	6.4453180e+003	9.6852259e-001
6.4355154e+003	9.9921694e-001	6.4403452e+003	9.7340650e-001	6.4453914e+003	9.8599066e-001
6.4355708e+003	9.9946928e-001	6.4403992e+003	9.9240815e-001	6.4454101e+003	9.9783994e-001
6.4355935e+003	9.9785316e-001	6.4404807e+003	9.9394628e-001	6.4454361e+003	9.1548300e-001
6.4356055e+003	9.9861058e-001	6.4405849e+003	9.8293040e-001	6.4455476e+003	9.6443771e-001
6.4356329e+003	9.9357775e-001	6.4406022e+003	9.9591626e-001	6.4456117e+003	9.9804153e-001
6.4356983e+003	9.9735001e-001	6.4406116e+003	9.9481261e-001	6.4457138e+003	9.9426723e-001
6.4357170e+003	9.9926831e-001	6.4407377e+003	9.9848006e-001	6.4457719e+003	9.8709141e-001
6.4357190e+003	9.9926865e-001	6.4407711e+003	9.9923009e-001	6.4458947e+003	9.8467987e-001
6.4357691e+003	9.9886063e-001	6.4408205e+003	9.9905575e-001	6.4460649e+003	9.9906398e-001
6.4357871e+003	9.9948111e-001	6.4410068e+003	9.9844832e-001	6.4461050e+003	9.0784936e-001
6.4358125e+003	9.9069448e-001	6.4410181e+003	9.9893804e-001	6.4461203e+003	9.6759800e-001
6.4358626e+003	9.9868807e-001	6.4410335e+003	9.9785137e-001	6.4461303e+003	9.5590381e-001
6.4358806e+003	9.9635938e-001	6.4411069e+003	9.9834162e-001	6.4462004e+003	9.9804251e-001
6.4359373e+003	9.7028433e-001	6.4413359e+003	9.9521892e-001	6.4462097e+003	9.9883700e-001
6.4360154e+003	9.9876261e-001	6.4413593e+003	9.9859561e-001	6.4462211e+003	9.9751679e-001
6.4360214e+003	9.9902093e-001	6.4413900e+003	9.8114275e-001	6.4462411e+003	9.9930489e-001
6.4360428e+003	9.8916703e-001	6.4414180e+003	9.9914490e-001	6.4462692e+003	9.9202894e-001
6.4360682e+003	9.9819602e-001	6.4414273e+003	9.9890425e-001	6.4462931e+003	9.9893819e-001
6.4360889e+003	9.9115631e-001	6.4415154e+003	9.9150791e-001	6.4463144e+003	9.9573894e-001
6.4361236e+003	9.9866858e-001	6.4415455e+003	9.9810375e-001	6.4463304e+003	9.9791375e-001
6.4361256e+003	9.9866975e-001	6.4415501e+003	9.9807938e-001	6.4463531e+003	9.8552288e-001
6.4363544e+003	9.9724874e-001	6.4415969e+003	9.9548396e-001	6.4463812e+003	9.9922133e-001
6.4364472e+003	9.9873668e-001	6.4416383e+003	9.9881039e-001	6.4464032e+003	9.9733119e-001
6.4364966e+003	9.9933177e-001	6.4417484e+003	9.9735180e-001	6.4464179e+003	9.9883168e-001
6.4366586e+003	9.9933618e-001	6.4419147e+003	9.9677005e-001	6.4464419e+003	9.9114211e-001
6.4367226e+003	9.8109121e-001	6.4420295e+003	9.9908661e-001	6.4464706e+003	9.9921016e-001
6.4368922e+003	9.8128884e-001	6.4420315e+003	9.9909186e-001	6.4464973e+003	9.9944628e-001
6.4369376e+003	9.9935409e-001	6.4420642e+003	9.2716646e-001	6.4465314e+003	9.9910941e-001
6.4371601e+003	9.9947905e-001	6.4421150e+003	9.9854345e-001	6.4465668e+003	9.9551427e-001
6.4372462e+003	9.0639135e-001	6.4421250e+003	9.9927109e-001	6.4465828e+003	9.9881912e-001
6.4372675e+003	9.9407983e-001	6.4421457e+003	9.9684659e-001	6.4466122e+003	9.9158922e-001
6.4372816e+003	9.8914940e-001	6.4422124e+003	9.4782705e-001	6.4466442e+003	9.9880673e-001
6.4373023e+003	9.9872968e-001	6.4423286e+003	9.8251081e-001	6.4466589e+003	9.9763071e-001
6.4373270e+003	9.8855603e-001	6.4425262e+003	9.7179427e-001	6.4466602e+003	9.9762989e-001
6.4373450e+003	9.9885989e-001	6.4426504e+003	9.9450336e-001	6.4466769e+003	9.9315642e-001
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6.4374097e+003	9.9322108e-001	6.4427111e+003	9.9579867e-001	6.4467257e+003	9.9816435e-001
6.4374338e+003	9.9811600e-001	6.4427886e+003	9.7578444e-001	6.4467390e+003	9.9667951e-001
6.4374358e+003	9.9811639e-001	6.4428125e+003	9.9851296e-001	6.4468044e+003	9.6632134e-001
6.4376033e+003	9.9257666e-001	6.4428445e+003	9.5110969e-001	6.4468672e+003	9.9869818e-001
6.4376594e+003	9.9536237e-001	6.4429093e+003	9.9765464e-001	6.4468819e+003	9.9916652e-001

6.4469072e+003 9.6659573e-001
6.4469426e+003 9.9946614e-001
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6.4469700e+003 9.9939010e-001
6.4469847e+003 9.9634567e-001
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6.4482752e+003 9.9753270e-001
6.4482758e+003 9.9753333e-001
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6.4483786e+003 8.8280655e-001
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6.4493029e+003 9.9449664e-001
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6.4511002e+003 9.9856315e-001
6.4511336e+003 9.9811401e-001
6.4513759e+003 9.9923117e-001
6.4513866e+003 9.9932490e-001
6.4513906e+003 9.9946319e-001
6.4514186e+003 9.5050411e-001
6.4514360e+003 9.8612425e-001
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6.4516520e+003 9.9776078e-001
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6.4521747e+003 9.6973996e-001
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6.4522628e+003 9.9903394e-001
6.4522688e+003 9.9910331e-001
6.4522808e+003 9.9810217e-001
6.4523115e+003 9.9841066e-001
6.4523215e+003 9.9925971e-001
6.4523282e+003 9.9875748e-001
6.4523329e+003 9.9887785e-001
6.4523369e+003 9.9880573e-001
6.4523509e+003 9.9893342e-001
6.4523858e+003 9.9947289e-001
6.4524192e+003 9.9933966e-001
6.4524812e+003 9.8876106e-001
6.4525647e+003 9.9487299e-001
6.4525834e+003 9.9902076e-001
6.4526174e+003 8.1992783e-001
6.4527055e+003 9.9222932e-001
6.4527296e+003 9.9915613e-001
6.4527509e+003 9.9531711e-001
6.4527903e+003 9.9924640e-001
6.4527943e+003 9.9932786e-001
6.4527970e+003 9.9931626e-001
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6.4532156e+003 9.9759676e-001
6.4532703e+003 9.9712695e-001
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6.4534719e+003 9.5018452e-001
6.4535681e+003 9.9500450e-001
6.4536288e+003 9.9792812e-001
6.4536976e+003 9.9942920e-001
6.4537076e+003 9.9920770e-001
6.4537122e+003 9.9931398e-001
6.4537142e+003 9.9931092e-001
6.4537169e+003 9.9933013e-001
6.4537470e+003 9.4719878e-001
6.4537683e+003 9.9475521e-001
6.4537884e+003 9.3419933e-001
6.4538065e+003 9.9916380e-001
6.4538872e+003 9.9949717e-001
6.4539312e+003 9.9929185e-001
6.4539426e+003 9.9944030e-001
6.4539933e+003 9.9893408e-001

C₁₂ Acetylene(6575 - 6454 cm⁻¹, P = 0.15Torr, L = 11.1m, T_v = 24°C)

6.5758279e+003 9.9940089e-001
 6.5758299e+003 9.9938068e-001
 6.5758559e+003 9.9949239e-001
 6.5758569e+003 9.9946938e-001
 6.5758599e+003 9.9939929e-001
 6.5760601e+003 9.9948416e-001
 6.5760611e+003 9.9946237e-001
 6.5760641e+003 9.9937869e-001
 6.5763033e+003 9.6891307e-001
 6.5763203e+003 9.8618649e-001
 6.5763213e+003 9.8640716e-001
 6.5763964e+003 9.9449478e-001
 6.5764164e+003 9.9810998e-001
 6.5764184e+003 9.9811406e-001
 6.5764245e+003 9.9820401e-001
 6.5764805e+003 3.5147488e-001
 6.5765456e+003 9.9909761e-001
 6.5765476e+003 9.9910959e-001
 6.5765566e+003 9.9933641e-001
 6.5765576e+003 9.9933995e-001
 6.5765596e+003 9.9933836e-001
 6.5765916e+003 9.6696790e-001
 6.5766186e+003 9.9925276e-001
 6.5766297e+003 9.9789341e-001
 6.5767117e+003 9.9920200e-001
 6.5767147e+003 9.9920919e-001
 6.5767177e+003 9.9912355e-001
 6.5768078e+003 9.8823780e-001
 6.5769920e+003 9.9947407e-001
 6.5770020e+003 9.9919071e-001
 6.5770050e+003 9.9929025e-001
 6.5770060e+003 9.9930447e-001
 6.5770140e+003 9.9943700e-001
 6.5770200e+003 9.9907587e-001
 6.5770210e+003 9.9907507e-001
 6.5770220e+003 9.9910193e-001
 6.5770240e+003 9.9908530e-001
 6.5770270e+003 9.9908863e-001
 6.5770310e+003 9.9918871e-001
 6.5770360e+003 9.9881627e-001
 6.5770461e+003 9.9907509e-001
 6.5770481e+003 9.9907141e-001
 6.5770501e+003 9.9896452e-001
 6.5770541e+003 9.9889824e-001
 6.5770561e+003 9.9890950e-001
 6.5770571e+003 9.9891464e-001
 6.5770631e+003 9.9905433e-001
 6.5770671e+003 9.9892894e-001
 6.5770731e+003 9.9910584e-001
 6.5770741e+003 9.9914430e-001
 6.5770751e+003 9.9913044e-001
 6.5770771e+003 9.9914119e-001
 6.5770801e+003 9.9918849e-001
 6.5770831e+003 9.9922130e-001
 6.5770901e+003 9.9909711e-001
 6.5771001e+003 9.9945547e-001
 6.5771041e+003 9.9903302e-001
 6.5771071e+003 9.9905171e-001
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 6.5771141e+003 9.9835300e-001
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 6.5772072e+003 9.9914052e-001
 6.5772082e+003 9.9914149e-001
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 6.5772813e+003 9.9921359e-001
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 6.5772993e+003 9.9938489e-001
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 6.5773103e+003 9.9945111e-001
 6.5773113e+003 9.9946451e-001
 6.5773173e+003 9.9919338e-001
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 6.5773363e+003 9.9941492e-001
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 6.5779449e+003 9.9317099e-001
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 6.5780380e+003 9.9941636e-001
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 6.5798358e+003 9.9791132e-001
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6.6051116e+003 9.9926955e-001
6.6051517e+003 5.2106534e-001
6.6051837e+003 9.9725412e-001
6.6051847e+003 9.9756389e-001
6.6051887e+003 9.9801202e-001
6.6051897e+003 9.9828807e-001
6.6051937e+003 9.9882916e-001
6.6051957e+003 9.9926581e-001
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6.6057012e+003 9.9822309e-001
6.6057032e+003 9.9823384e-001
6.6057042e+003 9.9787823e-001
6.6057052e+003 9.9809623e-001
6.6057092e+003 9.9821804e-001
6.6057102e+003 9.9823160e-001
6.6057122e+003 9.9790691e-001
6.6057132e+003 9.9859650e-001

C₁₃ Acetylene(6437 - 6456 cm⁻¹, P = 0.3Torr, L = 25.1m, T_{av} = 25°C)

6.4372249e+003	9.9537199e-001	6.4417266e+003	9.9907468e-001	6.4424820e+003	9.9943497e-001
6.4372599e+003	9.9927733e-001	6.4417312e+003	9.9932246e-001	6.4424894e+003	9.9934951e-001
6.4372857e+003	9.9212424e-001	6.4417340e+003	9.9922959e-001	6.4424940e+003	9.9945995e-001
6.4373070e+003	9.9927759e-001	6.4417349e+003	9.9921285e-001	6.4425004e+003	9.9867647e-001
6.4373337e+003	9.8337820e-001	6.4417414e+003	9.9811966e-001	6.4425014e+003	9.9837087e-001
6.4373734e+003	9.9717311e-001	6.4417423e+003	9.9791405e-001	6.4425124e+003	9.9421747e-001
6.4373881e+003	9.9669495e-001	6.4417543e+003	9.9585659e-001	6.4425152e+003	9.9458626e-001
6.4373992e+003	9.9695512e-001	6.4417654e+003	9.9826222e-001	6.4425161e+003	9.9464121e-001
6.4374038e+003	9.9687496e-001	6.4417672e+003	9.9848032e-001	6.4425235e+003	9.9660084e-001
6.4375062e+003	9.9690371e-001	6.4417700e+003	9.9877083e-001	6.4425244e+003	9.9736381e-001
6.4375145e+003	9.9794080e-001	6.4417792e+003	9.9733806e-001	6.4425281e+003	9.9868404e-001
6.4375302e+003	9.9368833e-001	6.4417811e+003	9.9743392e-001	6.4425290e+003	9.9881073e-001
6.4375421e+003	9.9675545e-001	6.4417829e+003	9.9730091e-001	6.4425309e+003	9.9895091e-001
6.4375578e+003	9.9224188e-001	6.4417838e+003	9.9711275e-001	6.4425318e+003	9.9930118e-001
6.4376021e+003	9.9911257e-001	6.4417866e+003	9.9687438e-001	6.4425337e+003	9.9934714e-001
6.4376067e+003	9.9928503e-001	6.4417986e+003	9.9925399e-001	6.4426167e+003	9.9898970e-001
6.4376196e+003	9.9844366e-001	6.4417995e+003	9.9942764e-001	6.4426849e+003	9.7325503e-001
6.4376999e+003	9.9816665e-001	6.4418410e+003	9.5425379e-001	6.4427107e+003	9.9835193e-001
6.4377469e+003	9.9510893e-001	6.4419378e+003	9.9921081e-001	6.4427504e+003	9.8985438e-001
6.4378142e+003	9.9771531e-001	6.4419738e+003	9.9483233e-001	6.4428030e+003	9.9721361e-001
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6.4379978e+003	9.9902727e-001	6.4419978e+003	9.9933815e-001	6.4428620e+003	9.9910739e-001
6.4380227e+003	9.8979105e-001	6.4420282e+003	9.9886171e-001	6.4428694e+003	9.9928030e-001
6.4380457e+003	9.9909312e-001	6.4420292e+003	9.9870707e-001	6.4428869e+003	9.9576026e-001
6.4380485e+003	9.9906595e-001	6.4420550e+003	9.4562514e-001	6.4429764e+003	9.9122494e-001
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6.4383095e+003	9.9936773e-001	6.4421343e+003	9.9926415e-001	6.4431599e+003	9.9850583e-001
6.4383372e+003	9.8397467e-001	6.4421361e+003	9.9909889e-001	6.4431700e+003	9.9877607e-001
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6.4391986e+003	9.9665010e-001	6.4421657e+003	9.9933368e-001	6.4433563e+003	9.9936868e-001
6.4392180e+003	9.9867955e-001	6.4421675e+003	9.9927884e-001	6.4433582e+003	9.9937096e-001
6.4392696e+003	8.1071637e-001	6.4421684e+003	9.9918697e-001	6.4434449e+003	9.9440969e-001
6.4393176e+003	9.9947870e-001	6.4421712e+003	9.9871604e-001	6.4435094e+003	9.9188119e-001
6.4393452e+003	9.7306846e-001	6.4421721e+003	9.9861380e-001	6.4435233e+003	9.9666339e-001
6.4395666e+003	9.9660136e-001	6.4422035e+003	8.4234231e-001	6.4435380e+003	9.8991386e-001
6.4396966e+003	9.2665405e-001	6.4422330e+003	9.9910580e-001	6.4436819e+003	9.8753820e-001
6.4397215e+003	9.9785197e-001	6.4422339e+003	9.9936713e-001	6.4437253e+003	9.9788940e-001
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6.4398497e+003	9.9292363e-001	6.4422616e+003	9.9909410e-001	6.4437585e+003	9.9222739e-001
6.4399088e+003	9.8881454e-001	6.4422643e+003	9.9112671e-001	6.4438144e+003	9.8508034e-001
6.4399263e+003	9.9830892e-001	6.4422653e+003	9.9116887e-001	6.4439168e+003	9.7267027e-001
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6.4399918e+003	9.8459290e-001	6.4422699e+003	9.9334638e-001	6.4441501e+003	9.9858005e-001
6.4400047e+003	9.8928659e-001	6.4422736e+003	9.9420889e-001	6.4441695e+003	9.8983413e-001
6.4400222e+003	9.6160159e-001	6.4422809e+003	9.9272599e-001	6.4442239e+003	9.9537829e-001
6.4401061e+003	9.9938206e-001	6.4422819e+003	9.9257561e-001	6.4442442e+003	9.9949069e-001
6.4401246e+003	9.9940336e-001	6.4422837e+003	9.9259769e-001	6.4442673e+003	9.9631544e-001
6.4401292e+003	9.9942756e-001	6.4423003e+003	9.9920234e-001	6.4442765e+003	9.9709762e-001
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6.4402187e+003	9.9709782e-001	6.4423400e+003	9.9948463e-001	6.4444296e+003	9.9946204e-001
6.4402712e+003	9.9701725e-001	6.4423446e+003	9.9946817e-001	6.4444453e+003	9.9939412e-001
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6.4411733e+003	9.9489687e-001	6.4423815e+003	9.9909604e-001	6.4445661e+003	9.6731139e-001
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6.4549340e+003 9.9949298e-001
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6.4549709e+003 9.9832550e-001
6.4549912e+003 9.6980645e-001
6.4550115e+003 9.9805617e-001
6.4550290e+003 9.8704011e-001
6.4551628e+003 9.8543622e-001
6.4551812e+003 9.9659175e-001
6.4551867e+003 9.9627172e-001
6.4552043e+003 9.9832690e-001
6.4552292e+003 9.1917164e-001

C₁₃ Acetylene(6570 - 6605 cm⁻¹, P = 1.1Torr, L = 11.1m, T_{av} = 25°C)

6.5702795e+003 9.9306306e-001
 6.5702807e+003 9.9306485e-001
 6.5702935e+003 9.9581377e-001
 6.5702970e+003 9.9620292e-001
 6.5703005e+003 9.9640972e-001
 6.5703017e+003 9.9650860e-001
 6.5703040e+003 9.9655241e-001
 6.5703227e+003 9.9152675e-001
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 6.5703437e+003 9.9729984e-001
 6.5703531e+003 9.9856006e-001
 6.5703647e+003 9.9803008e-001
 6.5703706e+003 9.9873766e-001
 6.5704056e+003 9.7439975e-001
 6.5704313e+003 9.9668399e-001
 6.5704523e+003 9.9241323e-001
 6.5704535e+003 9.9238465e-001
 6.5705212e+003 2.0498097e-001
 6.5705925e+003 9.9742900e-001
 6.5705936e+003 9.9748425e-001
 6.5705995e+003 9.9780685e-001
 6.5706065e+003 9.9763028e-001
 6.5706135e+003 9.9846573e-001
 6.5706228e+003 9.9684730e-001
 6.5706252e+003 9.9688414e-001
 6.5706275e+003 9.9687520e-001
 6.5706392e+003 9.9885802e-001
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 6.5706462e+003 9.9931324e-001
 6.5706497e+003 9.9924113e-001
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 6.5712068e+003 9.9766780e-001
 6.5713703e+003 9.9833411e-001
 6.5713784e+003 9.9866648e-001
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6.6031229e+003 9.9243196e-001
6.6031241e+003 9.9192202e-001
6.6031252e+003 9.9194653e-001
6.6031439e+003 9.9813442e-001
6.6031451e+003 9.9850739e-001
6.6031498e+003 9.9925253e-001
6.6031930e+003 9.9927403e-001
6.6031941e+003 9.9912124e-001
6.6032000e+003 9.9834994e-001
6.6032011e+003 9.9804194e-001
6.6032023e+003 9.9820893e-001
6.6032035e+003 9.9753034e-001
6.6032058e+003 9.9775517e-001
6.6032082e+003 9.9745040e-001
6.6032093e+003 9.9751932e-001
6.6032128e+003 9.9769891e-001
6.6032140e+003 9.9798833e-001
6.6032175e+003 9.9817853e-001
6.6032187e+003 9.9843747e-001
6.6032198e+003 9.9794952e-001
6.6032327e+003 9.9503234e-001
6.6032350e+003 9.9496195e-001
6.6032362e+003 9.9512702e-001
6.6032467e+003 9.9906103e-001
6.6032479e+003 9.9916762e-001
6.6032712e+003 9.9944453e-001
6.6032957e+003 9.9948833e-001
6.6033261e+003 9.9868612e-001

6.6033284e+003 9.9869166e-001
6.6033308e+003 9.9839404e-001
6.6033319e+003 9.9834240e-001
6.6033331e+003 9.9837951e-001
6.6033343e+003 9.9826978e-001
6.6033378e+003 9.9847253e-001
6.6033401e+003 9.9878752e-001
6.6033425e+003 9.9933683e-001
6.6033436e+003 9.9932159e-001
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6.6033530e+003 9.9790513e-001
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6.6033600e+003 9.9810661e-001
6.6033611e+003 9.9808185e-001
6.6033623e+003 9.9837516e-001
6.6033658e+003 9.9814845e-001
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6.6033763e+003 9.9816010e-001
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6.6033810e+003 9.9750515e-001
6.6033857e+003 9.9796819e-001
6.6033868e+003 9.9811699e-001
6.6033892e+003 9.9809079e-001
6.6033915e+003 9.9767836e-001
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6.6033997e+003 9.9821285e-001
6.6034008e+003 9.9816459e-001
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6.6034499e+003 9.9905000e-001
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6.6034546e+003 9.9878803e-001
6.6034581e+003 9.9853479e-001
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6.6034616e+003 9.9892042e-001
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6.6034674e+003 9.9875225e-001
6.6034709e+003 9.9804850e-001
6.6034733e+003 9.9827281e-001
6.6034744e+003 9.9837002e-001
6.6034768e+003 9.9789298e-001
6.6034779e+003 9.9819859e-001
6.6034861e+003 9.9948410e-001
6.6035223e+003 9.9923640e-001
6.6035235e+003 9.9892537e-001
6.6035258e+003 9.9800617e-001
6.6035270e+003 9.9824626e-001
6.6035281e+003 9.9830894e-001

N_{14} Ammonia (6570 - 6605 cm^{-1} , P = 0.47Torr, L = 25.1m, T_{av} = 22°C)

1.7517981e-002	9.9807905e-001	2.2986094e+000	9.9674142e-001	3.7790318e+000	9.9764666e-001
2.2523119e-002	9.9825735e-001	2.3399018e+000	7.8974191e-001	3.8165702e+000	9.9948990e-001
5.1302660e-002	9.9070862e-001	2.3636761e+000	9.5844691e-001	3.8265804e+000	9.9938772e-001
7.1323207e-002	9.9783648e-001	2.3836967e+000	9.0736069e-001	3.8703735e+000	9.9938174e-001
1.1636945e-001	9.4136837e-001	2.4287430e+000	9.9807555e-001	3.8728779e+000	9.9937570e-001
1.7142596e-001	9.9933323e-001	2.4324968e+000	9.9760761e-001	3.8741294e+000	9.9942091e-001
1.9645165e-001	9.9362831e-001	2.4349994e+000	9.9762264e-001	3.8753806e+000	9.9939981e-001
2.3148761e-001	9.9938458e-001	2.4612764e+000	9.8996258e-001	3.8803857e+000	9.9904846e-001
3.1657495e-001	9.7962947e-001	2.4625276e+000	9.8972440e-001	3.8816369e+000	9.9892771e-001
3.5911862e-001	9.9893610e-001	2.4637789e+000	9.8979845e-001	3.8841397e+000	9.9895660e-001
3.7038016e-001	9.9589992e-001	2.5701381e+000	9.8720777e-001	3.8928984e+000	9.9912391e-001
4.3294437e-001	9.9329585e-001	2.5951638e+000	9.9735432e-001	3.9191756e+000	9.9105654e-001
4.3419568e-001	9.9319879e-001	2.6452151e+000	6.8520353e-001	3.9429498e+000	9.9651810e-001
4.3544694e-001	9.9321813e-001	2.7002717e+000	9.9838367e-001	3.9442013e+000	9.9673370e-001
4.5546752e-001	9.9730121e-001	2.7027742e+000	9.9839844e-001	3.9867448e+000	9.9891762e-001
4.6547780e-001	9.9462784e-001	2.7052768e+000	9.9861568e-001	3.9917499e+000	9.9937903e-001
4.7298550e-001	9.9485761e-001	2.7090307e+000	9.9884737e-001	3.9967551e+000	9.9891863e-001
5.1302660e-001	8.7011368e-001	2.7177896e+000	9.9818219e-001	4.0418013e+000	9.9835413e-001
5.3680098e-001	9.7315737e-001	2.7265486e+000	9.9880860e-001	4.0443041e+000	9.9845190e-001
5.4931382e-001	9.6499231e-001	2.7328050e+000	9.9820761e-001	4.0455553e+000	9.9850209e-001
5.7934465e-001	9.9501430e-001	2.7465692e+000	9.9864116e-001	4.0468065e+000	9.9846959e-001
5.8685235e-001	9.9407839e-001	2.7478205e+000	9.9812937e-001	4.1018630e+000	9.1111710e-001
6.2564218e-001	9.9927491e-001	2.7615846e+000	9.9612947e-001	4.2119760e+000	9.0951157e-001
6.2689348e-001	9.9925970e-001	2.7628359e+000	9.9610966e-001	4.2495147e+000	9.9891293e-001
6.3064732e-001	9.9915727e-001	2.7665897e+000	9.9591310e-001	4.2645301e+000	9.9332728e-001
6.3440118e-001	9.9901038e-001	2.7703436e+000	9.9588202e-001	4.2732888e+000	9.9560635e-001
6.3815502e-001	9.9894672e-001	2.7753487e+000	9.9569947e-001	4.2757915e+000	9.9526218e-001
6.4065759e-001	9.9884545e-001	2.7766000e+000	9.9564618e-001	4.2920582e+000	9.9840292e-001
6.4190889e-001	9.9884911e-001	2.8228975e+000	5.6217950e-001	4.2970633e+000	9.9830887e-001
6.4441146e-001	9.9882992e-001	2.8892156e+000	9.9869792e-001	4.4046739e+000	8.6615088e-001
6.4566273e-001	9.9888062e-001	2.8904669e+000	9.9896454e-001	4.4397099e+000	9.9873180e-001
6.6067814e-001	9.9935903e-001	2.8954720e+000	9.9930107e-001	4.4409611e+000	9.9887522e-001
6.6443201e-001	9.9940131e-001	2.9004772e+000	9.9912073e-001	4.4422123e+000	9.9875362e-001
6.6568327e-001	9.9942625e-001	2.9029797e+000	9.9909370e-001	4.4484689e+000	9.9856605e-001
6.6818584e-001	9.9945064e-001	2.9480260e+000	8.1468996e-001	4.4497201e+000	9.9851640e-001
7.1823721e-001	8.3128834e-001	3.0706518e+000	9.9872779e-001	4.4622328e+000	9.9644363e-001
8.1708868e-001	9.9843584e-001	3.0744057e+000	9.9896224e-001	4.4672380e+000	9.9710084e-001
8.2084255e-001	9.9906921e-001	3.0856672e+000	9.9766920e-001	4.4697407e+000	9.9699321e-001
8.4086310e-001	9.9384702e-001	3.0869185e+000	9.9757730e-001	4.4772482e+000	9.9863798e-001
8.4211437e-001	9.9379485e-001	3.0894211e+000	9.9753988e-001	4.4797510e+000	9.9886654e-001
8.7464776e-001	9.7899179e-001	3.1432263e+000	9.8269917e-001	4.5298023e+000	9.7666702e-001
9.9727364e-001	9.9838828e-001	3.1695033e+000	9.9948034e-001	4.5598332e+000	9.9935466e-001
1.0710994e+000	6.2674552e-001	3.1895238e+000	9.9854342e-001	4.5673407e+000	9.9865311e-001
1.1011303e+000	9.6645210e-001	3.1932777e+000	9.9865804e-001	4.5710946e+000	9.9886354e-001
1.1249046e+000	8.6231405e-001	3.1957803e+000	9.9855735e-001	4.5773510e+000	9.9723345e-001
1.1536842e+000	9.8941776e-001	3.1982828e+000	9.9850471e-001	4.5786025e+000	9.9721542e-001
1.1787099e+000	9.8222921e-001	3.2045392e+000	9.9796017e-001	4.5861100e+000	9.9636228e-001
1.2087407e+000	9.9639541e-001	3.2145495e+000	9.9828911e-001	4.6173921e+000	9.9695739e-001
1.2625459e+000	8.3670059e-001	3.2183034e+000	9.9780856e-001	4.6198948e+000	9.9702390e-001
1.2925768e+000	9.8562266e-001	3.2971343e+000	9.9942311e-001	4.6223972e+000	9.9694248e-001
1.3201050e+000	9.2373560e-001	3.3008881e+000	9.9929799e-001	4.6286539e+000	9.9744869e-001
1.3276127e+000	9.2933725e-001	3.3021394e+000	9.9921606e-001	4.6311563e+000	9.9759097e-001
1.3588948e+000	5.5222085e-001	3.3033907e+000	9.9926342e-001	4.6749513e+000	9.9932451e-001
1.4114488e+000	9.9763417e-001	3.3159036e+000	9.9947205e-001	4.7049821e+000	9.8270288e-001
1.4564950e+000	9.7480410e-001	3.3196574e+000	9.9940175e-001	4.7375153e+000	9.9601044e-001
1.4602488e+000	9.7501503e-001	3.3209087e+000	9.9939379e-001	4.7425205e+000	9.9565197e-001
1.4915310e+000	6.5733182e-001	3.3234113e+000	9.9926055e-001	4.7525308e+000	9.9680036e-001
1.5253157e+000	9.8968897e-001	3.3246626e+000	9.9910763e-001	4.8238542e+000	3.5056859e-001
1.5428336e+000	9.7852724e-001	3.3259138e+000	9.9911820e-001	4.8789107e+000	9.9046379e-001
1.6441876e+000	9.8439251e-001	3.3284164e+000	9.9908288e-001	4.8814131e+000	9.9037364e-001
1.6692134e+000	9.9942112e-001	3.3834729e+000	9.3745495e-001	4.8864182e+000	9.9061758e-001
1.6717159e+000	9.9940836e-001	3.4199131e+000	9.9910771e-001	4.9202030e+000	9.1807742e-001
1.6729672e+000	9.9940119e-001	3.4286719e+000	9.9880869e-001	4.9489826e+000	9.8001062e-001
1.6754698e+000	9.9940514e-001	3.4712157e+000	9.9917689e-001	5.0002852e+000	3.9551914e-001
1.7117570e+000	9.9747627e-001	3.4724669e+000	9.9915832e-001	5.0440802e+000	9.8164002e-001
1.7167622e+000	9.9925592e-001	3.4749696e+000	9.9914466e-001	5.0866237e+000	4.3524770e-001
1.7192647e+000	9.9889020e-001	3.4799748e+000	9.9937812e-001	5.1329212e+000	9.8239894e-001
1.8018495e+000	8.5817264e-001	3.4849799e+000	9.9909046e-001	5.1792189e+000	4.3168180e-001
1.8368854e+000	9.9556324e-001	3.4862311e+000	9.9902977e-001	5.2417830e+000	9.9431109e-001
1.8393880e+000	9.9556877e-001	3.5200159e+000	9.8437925e-001	5.2442857e+000	9.9462851e-001
1.8456444e+000	9.9604630e-001	3.5212671e+000	9.8422852e-001	5.2505420e+000	9.9543885e-001
1.8606599e+000	9.9396258e-001	3.5275234e+000	9.8331757e-001	5.2567984e+000	9.9513416e-001
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1.8731727e+000	9.9467674e-001	3.5550518e+000	9.9027019e-001	5.2830756e+000	9.8707256e-001
1.9019522e+000	9.3219751e-001	3.5575542e+000	9.9019862e-001	5.3055985e+000	9.7471068e-001
1.9757780e+000	9.8851034e-001	3.5613082e+000	9.9024189e-001	5.3306242e+000	9.9312190e-001
1.9782806e+000	9.8848793e-001	3.5638106e+000	9.9021680e-001	5.3543987e+000	9.8208761e-001
2.0345884e+000	8.2633089e-001	3.5775748e+000	9.9388227e-001	5.3719165e+000	9.8950108e-001
2.0646192e+000	9.9191231e-001	3.6088571e+000	9.2029845e-001	5.4150348e+000	5.7579462e-001
2.0671218e+000	9.9181029e-001	3.6551546e+000	9.9922169e-001	5.4675889e+000	9.9908895e-001
2.0696243e+000	9.9191737e-001	3.6576570e+000	9.9910214e-001	5.4738452e+000	9.9737073e-001
2.0721269e+000	9.9192669e-001	3.6639136e+000	9.9942970e-001	5.4775991e+000	9.9753982e-001
2.0733782e+000	9.9152317e-001	3.7189702e+000	9.8700837e-001	5.4851067e+000	9.9676305e-001
2.0758807e+000	9.9153808e-001	3.7502522e+000	9.9879599e-001	5.4876094e+000	9.9684025e-001
2.0946500e+000	9.9625604e-001	3.7565085e+000	9.9839492e-001	5.4951169e+000	9.9582294e-001
2.0959013e+000	9.9673538e-001	3.7577597e+000	9.9831670e-001	5.5051272e+000	9.9726477e-001
2.1371937e+000	9.9794666e-001	3.7590112e+000	9.9837170e-001	5.5063788e+000	9.9745708e-001
2.1396963e+000	9.9802269e-001	3.7627649e+000	9.9830516e-001	5.5088812e+000	9.9755725e-001
2.1409476e+000	9.9816557e-001	3.7652676e+000	9.9829315e-001	5.513883e+000	9.9673888e-001
2.1434501e+000	9.9817318e-001	3.7665188e+000	9.9828589e-001	5.5163890e+000	9.9664936e-001
2.1684758e+000	9.8985144e-001	3.7765291e+000	9.9764320e-001	5.5176402e+000	9.9669406e-001
2.2785888e+000	9.8249026e-001	3.7777803e+000	9.9761453e-001	5.5201426e+000	9.9668273e-001

5.5213942e+000 9.9669360e-001
5.5414147e+000 9.9911198e-001
5.5439171e+000 9.9886373e-001
5.5476711e+000 9.9901306e-001
5.5489223e+000 9.9904673e-001
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5.6077327e+000 9.9181628e-001
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5.6815585e+000 4.8801606e-001
5.7503789e+000 9.9847830e-001
5.7541329e+000 9.9817154e-001
5.7616407e+000 9.9905249e-001
5.7691483e+000 9.9754425e-001
5.8129433e+000 9.9349133e-001
5.8141945e+000 9.9331851e-001
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5.9956306e+000 9.9932195e-001
5.9968822e+000 9.9936111e-001
6.0056409e+000 9.9923264e-001
6.0606978e+000 9.9945051e-001
6.0619490e+000 9.9941020e-001
6.0644514e+000 9.9945357e-001
6.1182567e+000 9.9543898e-001
6.1720620e+000 9.7159700e-001
6.1995900e+000 9.9606953e-001
6.2546466e+000 3.3448007e-001
6.3072007e+000 9.9000475e-001
6.3097031e+000 9.9021289e-001
6.3147082e+000 9.9144017e-001
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6.3197133e+000 9.9123035e-001
6.3760214e+000 3.4739513e-001
6.4123085e+000 9.8048527e-001
6.4398366e+000 8.9145019e-001
6.4686162e+000 9.7981174e-001
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2.9997202e+001 9.9808216e-001
2.9999704e+001 9.9820279e-001
3.0000956e+001 9.9801837e-001
3.0023479e+001 9.8528580e-001
3.0024730e+001 9.8481112e-001
3.0025981e+001 9.8492585e-001
3.0093551e+001 9.9074459e-001
3.0103561e+001 9.9305091e-001
3.0106064e+001 9.9310802e-001
3.0107315e+001 9.9294930e-001
3.0123581e+001 9.7837120e-001
3.0149858e+001 9.9708301e-001
3.0152361e+001 9.9761974e-001
3.0153612e+001 9.9757859e-001
3.0212423e+001 9.2080004e-001
3.0281243e+001 9.9943020e-001
3.0303766e+001 9.9279050e-001
3.0305018e+001 9.9255770e-001
3.0306269e+001 9.9256480e-001
3.0307520e+001 9.9192802e-001
3.0308771e+001 9.9234439e-001
3.0312525e+001 9.9293405e-001
3.0313777e+001 9.9304080e-001
3.0320033e+001 9.9308344e-001
3.0321284e+001 9.9309050e-001
3.0326290e+001 9.9415003e-001
3.0327541e+001 9.9420105e-001
3.0328792e+001 9.9398501e-001
3.0390105e+001 9.5613505e-002
3.0422638e+001 8.6284456e-001
3.0425141e+001 8.6302118e-001
3.0458926e+001 9.8420898e-001
3.0473941e+001 9.7671610e-001
3.0571541e+001 9.9111493e-001
3.0621593e+001 9.8076283e-001
3.0676649e+001 9.9405115e-001
3.0677900e+001 9.9382583e-001
3.0680403e+001 9.9358852e-001
3.0681654e+001 9.9350991e-001
3.0687911e+001 9.9281775e-001
3.0689162e+001 9.9259701e-001
3.0699172e+001 9.8962205e-001
3.0701675e+001 9.8954388e-001
3.0704177e+001 9.8961006e-001
3.0705429e+001 9.8943434e-001
3.0706680e+001 9.8944550e-001
3.0707931e+001 9.8940514e-001
3.0765490e+001 7.2853016e-002
3.0809285e+001 9.7640035e-001
3.0851829e+001 2.4993090e-001
3.0904383e+001 9.9642306e-001
3.0905634e+001 9.9694100e-001
3.0910639e+001 9.9784177e-001
3.0913142e+001 9.9823163e-001
3.0916896e+001 9.9891303e-001
3.0918147e+001 9.9898251e-001

3.0920650e+001 9.9869937e-001
3.0921901e+001 9.9890167e-001
3.0978209e+001 9.6823992e-001
3.1005737e+001 9.9882885e-001
3.1010742e+001 9.9890047e-001
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3.1016998e+001 9.9819540e-001
3.1018250e+001 9.9824784e-001
3.1019501e+001 9.9762646e-001
3.1020752e+001 9.9774123e-001
3.1022004e+001 9.9764622e-001
3.1023255e+001 9.9771408e-001
3.1025758e+001 9.9739137e-001
3.1027009e+001 9.9760724e-001
3.1028260e+001 9.9721735e-001
3.1030763e+001 9.9697568e-001
3.1032014e+001 9.9649995e-001
3.1072055e+001 3.9785954e-001
3.1137122e+001 9.9555447e-001
3.1138373e+001 9.9620705e-001

3.1142127e+001 9.9621384e-001
3.1177163e+001 8.4885174e-001
3.1189676e+001 8.9461485e-001
3.1219707e+001 2.0918065e-001
3.1243481e+001 7.8737336e-001
3.1272260e+001 5.8118565e-002
3.1293532e+001 4.2003213e-001
3.1314804e+001 8.4904009e-002
3.1378620e+001 9.9341731e-001
3.1379871e+001 9.9352222e-001
3.1383625e+001 9.9418449e-001
3.1384876e+001 9.9434716e-001
3.1392384e+001 9.9498747e-001
3.1394886e+001 9.9508991e-001
3.1397389e+001 9.9524265e-001
3.1398640e+001 9.9543074e-001
3.1401143e+001 9.9533000e-001
3.1402394e+001 9.9552110e-001
3.1404897e+001 9.9521412e-001
3.1407399e+001 9.9525400e-001
3.1409902e+001 9.9524145e-001

3.1411153e+001 9.9532481e-001
3.1414907e+001 9.9559787e-001
3.1416158e+001 9.9601645e-001
3.1419912e+001 9.9655810e-001
3.1421163e+001 9.9699233e-001
3.1482476e+001 9.9529956e-001
3.1483727e+001 9.9479956e-001
3.1484979e+001 9.9493143e-001
3.1489984e+001 9.9578315e-001
3.1492486e+001 9.9622735e-001
3.1504999e+001 9.9907318e-001
3.1506251e+001 9.9933884e-001
3.1507502e+001 9.9896805e-001
3.1508753e+001 9.9941844e-001
3.1510005e+001 9.9941375e-001
3.1515010e+001 9.9918400e-001
3.1517512e+001 9.9925642e-001
3.1518763e+001 9.9942814e-001
3.1521266e+001 9.9926543e-001
3.1522517e+001 9.9943103e-001

N_{14} Ammonia(6435 - 6455 cm^{-1} , P = 0.47Torr, L = 25.1m, T_{av} = 24°C)

6.3094185e-002	9.5199209e-001	3.0793086e+000	9.9980389e-001	6.4550235e+000	9.9959823e-001
9.1136044e-002	9.9791092e-001	3.1203699e+000	9.9540306e-001	6.5752028e+000	9.9968421e-001
1.0816146e-001	9.9432324e-001	3.1243758e+000	9.9547128e-001	6.5822133e+000	9.9972280e-001
1.2418538e-001	9.9751268e-001	3.1554222e+000	9.0782436e-001	6.5892235e+000	9.9969273e-001
1.4521678e-001	9.8994019e-001	3.1814610e+000	9.9306439e-001	6.6002402e+000	9.9989779e-001
2.0130050e-001	9.9952037e-001	3.1906220e+000	9.9217802e-001	6.6082521e+000	9.9974473e-001
2.0630797e-001	9.9952852e-001	3.2897699e+000	9.9953482e-001	6.6312864e+000	9.9961594e-001
2.2032890e-001	9.9927982e-001	3.3218178e+000	9.9804970e-001	6.6423030e+000	9.9994150e-001
2.2834086e-001	9.9937902e-001	3.3598745e+000	9.9420465e-001	6.6463088e+000	9.9992913e-001
2.7140515e-001	9.8479544e-001	3.4349866e+000	9.9985116e-001	6.7294331e+000	9.6988476e-001
2.8742907e-001	9.9205123e-001	3.4460032e+000	9.9998912e-001	6.8099526e+000	9.9644035e-001
3.0946196e-001	9.7107276e-001	3.4910705e+000	9.7740174e-001	6.9000871e+000	9.9068185e-001
3.6053821e-001	9.9916453e-001	3.5121017e+000	9.9635510e-001	6.9962304e+000	9.9977563e-001
4.2763836e-001	8.6802981e-001	3.5411452e+000	9.9686681e-001	7.0503112e+000	9.9972539e-001
4.7470863e-001	9.9936765e-001	3.5782006e+000	9.9884222e-001	7.0623292e+000	9.9993818e-001
5.2578488e-001	9.4963543e-001	3.5842094e+000	9.9879032e-001	7.0913727e+000	9.9729997e-001
5.6384170e-001	9.9992534e-001	3.6375440e+000	9.9912805e-001	7.1114026e+000	9.9964460e-001
5.7385664e-001	9.9970696e-001	3.6625814e+000	9.9995445e-001	7.1354382e+000	9.9693872e-001
5.7886411e-001	9.9973090e-001	3.6846143e+000	9.9904210e-001	7.2005354e+000	9.9968908e-001
5.9188354e-001	9.9936142e-001	3.6916249e+000	9.9915432e-001	7.2095490e+000	9.9974783e-001
6.0290001e-001	9.9969991e-001	3.7136578e+000	9.9573992e-001	7.2425982e+000	9.9068946e-001
6.1291495e-001	9.9955243e-001	3.7386952e+000	9.9972831e-001	7.2716417e+000	9.9974273e-001
6.6799718e-001	9.9954733e-001	3.7737475e+000	9.8557513e-001	7.2786522e+000	9.9962391e-001
6.8201810e-001	9.9995108e-001	3.8037923e+000	9.9922609e-001	7.3026879e+000	9.9971751e-001
6.9003005e-001	9.9985988e-001	3.8548687e+000	9.7742231e-001	7.3096984e+000	9.9985439e-001
7.4210779e-001	9.9608194e-001	3.8869163e+000	9.9962068e-001	7.3607748e+000	9.9139024e-001
7.6814668e-001	9.9997201e-001	3.9159597e+000	9.9056169e-001	7.4248702e+000	9.997937e-001
7.8216760e-001	9.9957341e-001	4.0030898e+000	9.9997161e-001	7.4959765e+000	9.9162650e-001
8.7649700e-001	9.0571984e-001	4.0111017e+000	9.9999521e-001	7.5700873e+000	9.9510109e-001
9.9166892e-001	9.9513968e-001	4.0531645e+000	9.7089737e-001	7.5831066e+000	9.9641161e-001
1.0397407e+000	9.9703960e-001	4.0772005e+000	9.9571042e-001	7.5981290e+000	9.9433127e-001
1.0968259e+000	9.9599955e-001	4.0892185e+000	9.9486821e-001	7.6201620e+000	9.9939793e-001
1.1789485e+000	9.9980605e-001	4.1192634e+000	9.9872926e-001	7.6271725e+000	9.9935162e-001
1.2440457e+000	9.977753e-001	4.1422977e+000	9.9428644e-001	7.6331813e+000	9.993649e-001
1.2690831e+000	9.9964602e-001	4.1803544e+000	9.9997827e-001	7.6371874e+000	9.9935714e-001
1.2971249e+000	9.9579028e-001	4.1843605e+000	9.9997016e-001	7.6552143e+000	9.9992646e-001
1.3211608e+000	9.9941046e-001	4.2073948e+000	9.9998101e-001	7.6672323e+000	9.9972092e-001
1.3752415e+000	9.7660333e-001	4.2296309e+000	9.9960224e-001	7.7333308e+000	9.9938864e-001
1.4092923e+000	9.9928601e-001	4.2817087e+000	9.9948152e-001	7.7793997e+000	9.9993629e-001
1.4373342e+000	9.9760107e-001	4.2917236e+000	9.9968615e-001	7.7834055e+000	9.9993697e-001
1.4563626e+000	9.9852993e-001	4.3387939e+000	9.0275469e-001	7.8004310e+000	9.9937437e-001
1.4683805e+000	9.9816008e-001	4.3598251e+000	9.7042858e-001	7.8144520e+000	9.9978317e-001
1.4844045e+000	9.9989195e-001	4.3638313e+000	9.7006763e-001	7.8264700e+000	9.9962862e-001
1.5104433e+000	9.8866108e-001	4.4569702e+000	9.7188774e-001	7.8404907e+000	9.9997944e-001
1.5505031e+000	9.9981017e-001	4.5661332e+000	8.5507271e-001	7.8424938e+000	9.9998513e-001
1.5565121e+000	9.9989605e-001	4.7253709e+000	9.9201548e-001	7.8434952e+000	9.9960357e-001
1.5675286e+000	9.9920221e-001	4.7454008e+000	9.9830074e-001	7.8735400e+000	9.9153287e-001
1.5875585e+000	9.9997938e-001	4.7564171e+000	9.9795666e-001	7.8765447e+000	9.9159829e-001
1.6306227e+000	9.4621771e-001	4.7694368e+000	9.9840113e-001	7.8775461e+000	9.9160345e-001
1.6506527e+000	9.8843467e-001	4.7904681e+000	9.9311441e-001	7.8875610e+000	9.9488974e-001
1.6636721e+000	9.8148387e-001	4.8155054e+000	9.9949348e-001	7.9105953e+000	9.8367602e-001
1.6927154e+000	9.9906472e-001	4.8395414e+000	9.9847235e-001	7.9326283e+000	9.9869908e-001
1.7247633e+000	9.8998273e-001	4.8545638e+000	9.9902127e-001	7.9336300e+000	9.9886795e-001
1.7277678e+000	9.9001720e-001	4.8615744e+000	9.9899208e-001	7.9376358e+000	9.9919408e-001
1.7568111e+000	8.6790970e-001	4.8625757e+000	9.9899186e-001	7.9633596e+000	9.9155784e-001
1.8097871e+000	9.9917571e-001	4.8846087e+000	9.9724138e-001	7.9803851e+000	9.9634272e-001
1.8167975e+000	9.9951329e-001	4.9547133e+000	9.9991138e-001	7.9974105e+000	9.9105298e-001
1.8368274e+000	9.9426601e-001	4.9748212e+000	9.9972384e-001	7.9994136e+000	9.9097514e-001
1.8658708e+000	9.9963191e-001	5.0268990e+000	9.95254995e-001	8.0014163e+000	9.9096277e-001
1.8979186e+000	9.9609398e-001	5.1020111e+000	9.9000507e-001	8.0204448e+000	9.9867669e-001
1.9219545e+000	9.9820304e-001	5.1951500e+000	9.9547046e-001	8.0224479e+000	9.9851799e-001
1.9389799e+000	9.9683064e-001	5.2762712e+000	9.9423965e-001	8.0254523e+000	9.9893159e-001
1.9730307e+000	9.9992721e-001	5.2812787e+000	9.9428795e-001	8.0274554e+000	9.9877574e-001
1.9980681e+000	9.9886168e-001	5.3163310e+000	9.9803389e-001	8.0294584e+000	9.9859913e-001
2.0140920e+000	9.9982946e-001	5.3894400e+000	9.8976453e-001	8.0665138e+000	8.8839894e-001
2.0291145e+000	9.9905665e-001	5.4094699e+000	9.9595246e-001	8.1015661e+000	9.8702470e-001
2.1663193e+000	9.9893919e-001	5.4335059e+000	9.7132434e-001	8.1045705e+000	9.8726156e-001
2.2670961e+000	9.8064270e-001	5.4805762e+000	9.9961080e-001	8.1065736e+000	9.8734810e-001
2.3051529e+000	9.9950078e-001	5.4925942e+000	9.9928335e-001	8.1145854e+000	9.8643955e-001
2.3502202e+000	9.1871765e-001	5.5056136e+000	9.9991673e-001	8.1456316e+000	9.9941642e-001
2.3752575e+000	9.9397140e-001	5.5346570e+000	9.8088794e-001	8.1486364e+000	9.9920454e-001
2.4022979e+000	8.9538113e-001	5.5446720e+000	9.8486593e-001	8.1506391e+000	9.9928635e-001
2.4423577e+000	9.9988971e-001	5.5717121e+000	8.6196790e-001	8.1536439e+000	9.9928518e-001
2.4543757e+000	9.9956125e-001	5.6418170e+000	9.8988212e-001	8.1566483e+000	9.9937918e-001
2.4663936e+000	9.9982940e-001	5.7239396e+000	9.9993590e-001	8.1586513e+000	9.9920733e-001
2.4874250e+000	9.9917016e-001	5.7770187e+000	9.6880958e-001	8.1636588e+000	9.9953883e-001
2.4964385e+000	9.9933480e-001	5.8691563e+000	9.9775518e-001	8.1656615e+000	9.9951907e-001
2.5014459e+000	9.9930618e-001	5.8871831e+000	9.9979715e-001	8.1686663e+000	9.9966189e-001
2.5154669e+000	9.9988824e-001	5.9132222e+000	9.9678333e-001	8.1746751e+000	9.9937796e-001
2.5214758e+000	9.9985489e-001	5.9923400e+000	9.9686318e-001	8.1766782e+000	9.9959255e-001
2.5254818e+000	9.9985947e-001	6.0063611e+000	9.985668e-001	8.1816856e+000	9.9949293e-001
2.5385012e+000	9.9956479e-001	6.0263910e+000	9.9419537e-001	8.1846901e+000	9.9955463e-001
2.5415057e+000	9.9957682e-001	6.0564358e+000	9.9991400e-001	8.1866931e+000	9.9954589e-001
2.5535237e+000	9.9918473e-001	6.1065032e+000	9.8964614e-001	8.1886962e+000	9.9948138e-001
2.5735536e+000	9.9980500e-001	6.1235286e+000	9.9559675e-001	8.2137335e+000	9.9180243e-001
2.6005939e+000	9.9646942e-001	6.1385511e+000	9.9183109e-001	8.2377692e+000	9.9925623e-001
2.6787105e+000	9.2076256e-001	6.2447093e+000	9.9895196e-001	8.2387709e+000	9.9927949e-001
2.7047494e+000	9.8966864e-001	6.2907783e+000	9.9931478e-001	8.2407739e+000	9.9939410e-001
2.7087554e+000	9.8957426e-001	6.3017946e+000	9.9977345e-001	8.2417753e+000	9.9958577e-001
2.9761546e+000	9.3024575e-001	6.3298364e+000	9.9242018e-001	8.2447797e+000	9.9974653e-001
3.0332398e+000	9.9985768e-001	6.4109576e+000	9.7651013e-001	8.2467828e+000	9.9960499e-001
3.0662891e+000	9.9923522e-001	6.4520187e+000	9.9959891e-001	8.2487858e+000	9.9966086e-001

8.2517902e+000 9.9977603e-001
8.2527916e+000 9.9968860e-001
8.2547947e+000 9.9965654e-001
8.2557964e+000 9.9955462e-001
8.2588008e+000 9.9957707e-001
8.2608038e+000 9.9976950e-001
8.2628065e+000 9.9980970e-001
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8.2728215e+000 9.9979940e-001
8.2748245e+000 9.9992450e-001
8.2778290e+000 9.9977494e-001
8.2818351e+000 9.9987159e-001
8.2838381e+000 9.9986208e-001
8.2858412e+000 9.9991552e-001
8.2888456e+000 9.9972696e-001
8.2908487e+000 9.9981246e-001
8.2938531e+000 9.9977050e-001
8.2948544e+000 9.9973247e-001
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8.3389203e+000 9.9983594e-001
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8.3459308e+000 9.9984582e-001
8.3479339e+000 9.9960726e-001
8.3509383e+000 9.9972841e-001
8.3529414e+000 9.9971138e-001
8.3559458e+000 9.9979797e-001
8.3589502e+000 9.9964452e-001
8.3619546e+000 9.9981063e-001
8.3639577e+000 9.9974989e-001
8.3669621e+000 9.9976526e-001
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8.3709682e+000 9.9969109e-001
8.3739726e+000 9.9972378e-001
8.3779787e+000 9.9974163e-001
8.3809831e+000 9.9955799e-001
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2.0980209e+001 9.9778802e-001
2.0989223e+001 9.9836086e-001
2.0993228e+001 9.9831917e-001

N_{15} Ammonia(6435 - 6455 cm^{-1} , P = 0.47Torr, L = 25.1m, T_{av} = 21°C)

6.4359164e+003 9.9902943e-001
 6.4359188e+003 9.9903331e-001
 6.4359212e+003 9.9879475e-001
 6.4359276e+003 9.9949398e-001
 6.4359292e+003 9.9936896e-001
 6.4359324e+003 9.9949484e-001
 6.4359348e+003 9.9918357e-001
 6.4359372e+003 9.9900898e-001
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 6.4360526e+003 9.8936900e-001
 6.4360790e+003 9.9893133e-001
 6.4360822e+003 9.9873333e-001
 6.4360846e+003 9.9881929e-001
 6.4360870e+003 9.9875726e-001
 6.4360903e+003 9.9914890e-001
 6.4360911e+003 9.9916075e-001
 6.4361439e+003 9.9907008e-001
 6.4361463e+003 9.9896350e-001
 6.4361487e+003 9.9849095e-001
 6.4361511e+003 9.9865706e-001
 6.4361752e+003 9.8501953e-001
 6.4361960e+003 9.9895361e-001
 6.4361968e+003 9.9921117e-001
 6.4363202e+003 9.9941311e-001
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 6.4364155e+003 9.9863027e-001
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6.4512358e+003 9.9809366e-001
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6.4525868e+003 9.8537798e-001
6.4525876e+003 9.8537225e-001
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6.4538623e+003 6.6885298e-001
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6.4547587e+003 9.9911421e-001
6.4548765e+003 9.9490747e-001
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6.4551457e+003 9.9947229e-001
6.4551529e+003 9.9901607e-001
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6.4551649e+003 9.9822047e-001
6.4551681e+003 9.9831850e-001
6.4552042e+003 6.9444075e-001
6.4552859e+003 9.9913436e-001
6.4552907e+003 9.9944242e-001

N_{15} Ammonia(6435 - 6455 cm^{-1} , P = 1.02Torr, L = 11.1m, $T_v = 24^{\circ}C$)

5.9733501e+003	9.9917265e-001	5.9745368e+003	9.8464531e-001	5.9762077e+003	9.9882826e-001
5.9733549e+003	9.9812068e-001	5.9745912e+003	6.2803275e-001	5.9762246e+003	9.9927314e-001
5.9733609e+003	9.9868611e-001	5.9746503e+003	9.9446569e-001	5.9762271e+003	9.9930768e-001
5.9733682e+003	9.9784434e-001	5.9746588e+003	9.9309082e-001	5.9762343e+003	9.9873083e-001
5.9733742e+003	9.9811338e-001	5.9746636e+003	9.9323982e-001	5.9762355e+003	9.9839087e-001
5.9733778e+003	9.9763224e-001	5.9746902e+003	9.8163302e-001	5.9762379e+003	9.9828637e-001
5.9733790e+003	9.9761515e-001	5.9747095e+003	9.8468101e-001	5.9762415e+003	9.9833810e-001
5.9733827e+003	9.9733480e-001	5.9747469e+003	5.1013113e-001	5.9762427e+003	9.9843812e-001
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5.9734394e+003	5.1526522e-001	5.9747892e+003	9.2825654e-001	5.9763079e+003	7.9082529e-001
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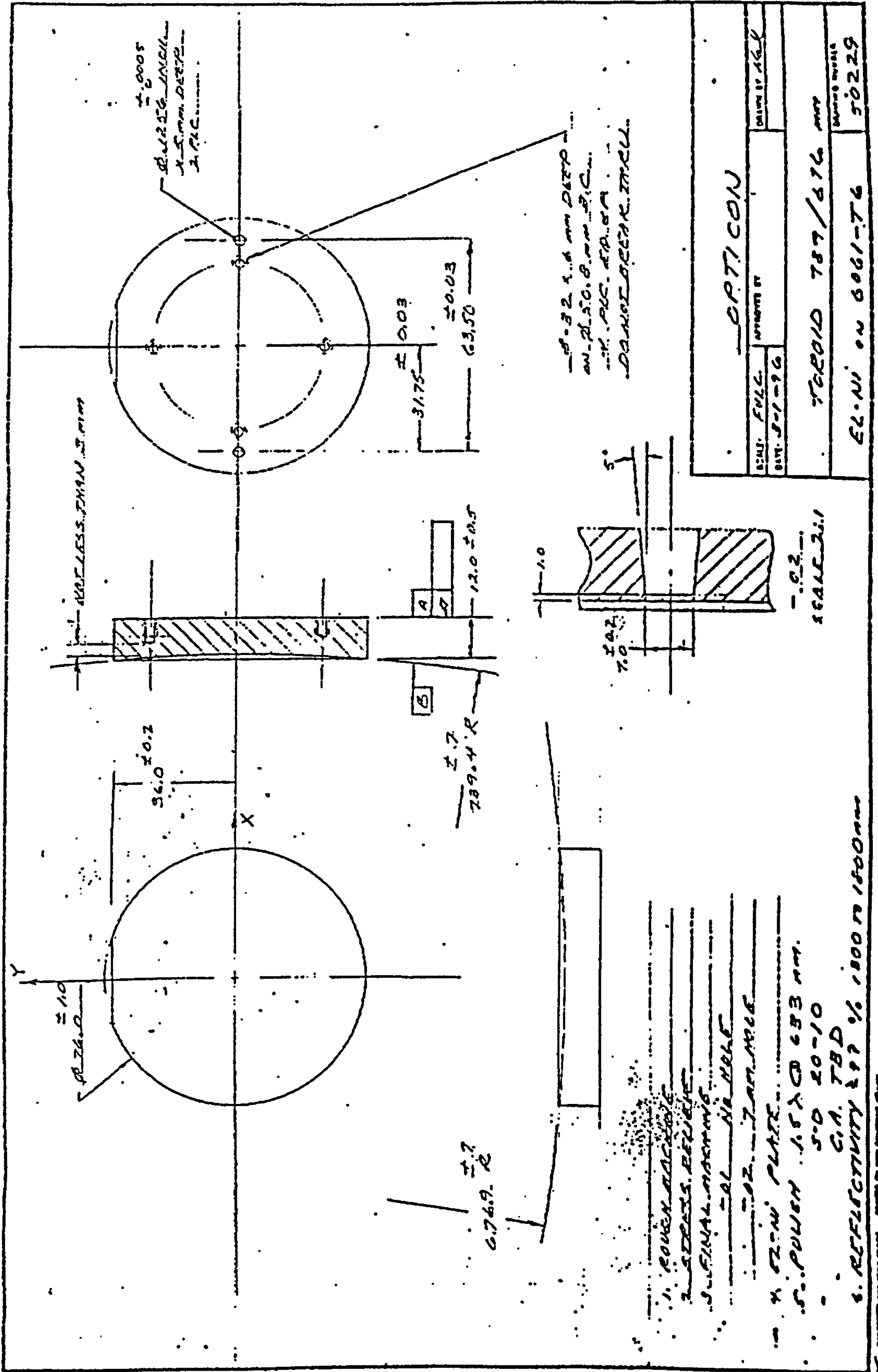
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Appendix 4 - Astigmatic Mirror Design Drawings



1. ROUGH POLISH
2. FINISH POLISH
3. FINAL MACHINING
4. POLISH PLATE
5. POLISH 1.5 \times CD 683 mm.
6. REFLECTIVITY $\geq 97\%$ @ 1000 to 1500 nm