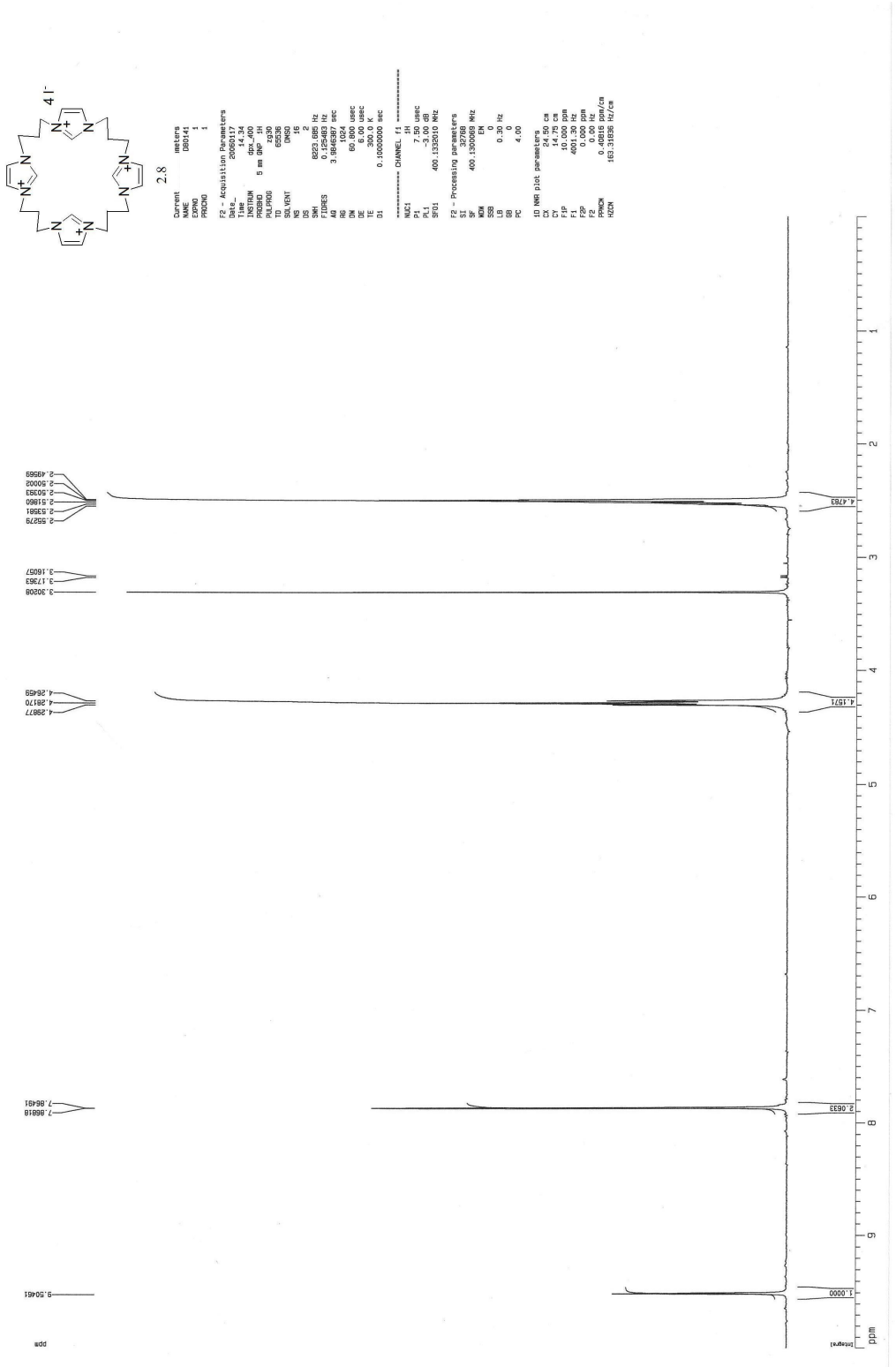
41-
2.8
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NAME DB5141
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
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Time 14.14 h
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PROBHD 5 mm BNP 1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 1024
DW 60.800 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
CHANNEL f1
NUC1 1H
P1 7.50 usec
PL1 -2.00 dB
SFO1 400.1332910 MHz
F2 - Processing parameters
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SF 400.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
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CY 14.75 cm
F1P 10.000 ppm
F1L 4000.53 Hz
F2P 0.000 ppm
F2L 0.00 Hz
PPMCM 0.40816 ppm/cm
HZCM 163.31936 Hz/cm
2.49589
2.49002
2.50393
2.51860
2.53981
2.55279
3.16057
3.17363
3.30208
4.26459
4.28170
4.29877
7.86818
7.86481
9.50461
4.4783
4.1571
2.0633
1.0000
ppm

Crystal Structure data for **1.314**

Table 1. Crystal data and structure refinement for jam0906.

Identification code	jam0906	
Empirical formula	C12 H18 I2 N4	
Formula weight	472.10	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P21/c	
Unit cell dimensions	a = 8.1247(1) Å	α= 90°.
	b = 30.4232(5) Å	β= 92.401(1)°.
	c = 12.9336(2) Å	γ = 90°.
Volume	3194.11(8) $Å^3$	
Z	8	
Density (calculated)	1.963 Mg/m^3	
Absorption coefficient	3.929 mm^{-1}	
F(000)	1792	
Crystal size	0.30 x 0.30 x 0.08 mm^3	
Theta range for data collection	2.98 to 27.53°.	
Index ranges	-10<=h<=10, -39<=k<=39, -16<=l<=16	
Reflections collected	79220	
Independent reflections	7333 [R(int) = 0.0767]	
Completeness to theta = 27.53°	99.5 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.000 and 0.637	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7333 / 0 / 325	
Goodness-of-fit on F^2	1.013	
Final R indices [I>2sigma(I)]	R1 = 0.0296, wR2 = 0.0391	
R indices (all data)	R1 = 0.0620, wR2 = 0.0446	
Largest diff. peak and hole	0.590 and -0.593 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for jam0906. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
I(1)	2342(1)	3916(1)	1264(1)	30(1)
I(2)	3424(1)	7317(1)	3040(1)	30(1)
I(3)	2884(1)	5336(1)	6444(1)	36(1)
I(4)	8804(1)	6545(1)	2112(1)	27(1)
N(1)	9810(3)	6301(1)	5425(2)	23(1)
N(2)	10433(3)	6992(1)	5550(2)	23(1)
N(3)	6101(3)	6549(1)	5183(2)	31(1)
N(4)	6818(3)	7235(1)	5159(2)	30(1)
N(5)	6446(3)	4220(1)	7489(2)	24(1)
N(6)	5664(3)	3774(1)	8668(2)	23(1)
N(7)	8674(3)	4677(1)	9152(2)	25(1)
N(8)	7762(3)	4238(1)	10315(2)	23(1)
C(1)	10061(4)	6667(1)	4897(3)	25(1)
C(2)	10079(4)	6394(1)	6453(3)	35(1)
C(3)	10468(5)	6822(1)	6535(3)	37(1)
C(4)	9091(4)	5891(1)	4992(3)	31(1)
C(5)	7355(4)	5804(1)	5357(3)	35(1)
C(6)	5984(4)	6086(1)	4871(3)	38(1)
C(7)	6632(4)	6872(1)	4595(3)	27(1)
C(8)	5872(5)	6720(2)	6149(3)	53(1)
C(9)	6323(5)	7146(2)	6135(3)	53(1)
C(10)	7528(4)	7649(1)	4800(3)	39(1)
C(11)	9279(4)	7605(1)	4454(3)	34(1)
C(12)	10587(4)	7461(1)	5269(3)	29(1)
C(13)	6763(4)	3835(1)	7947(2)	24(1)
C(14)	5076(4)	4404(1)	7924(3)	29(1)
C(15)	4597(4)	4124(1)	8653(3)	28(1)
C(16)	7547(4)	4448(1)	6793(3)	35(1)
C(17)	8274(4)	4870(1)	7263(3)	31(1)
C(18)	9462(4)	4822(1)	8200(3)	34(1)
C(19)	8793(4)	4272(1)	9541(2)	25(1)

C(20)	7579(4)	4912(1)	9721(3)	31(1)
C(21)	7019(4)	4638(1)	10446(3)	29(1)
C(22)	7402(4)	3830(1)	10884(3)	30(1)
C(23)	7209(4)	3433(1)	10175(3)	30(1)
C(24)	5706(4)	3414(1)	9432(3)	31(1)

Table 3. Bond lengths [Å] and angles [°] for jam0906.

N(1)-C(1)	1.326(4)
N(1)-C(2)	1.368(4)
N(1)-C(4)	1.477(4)
N(2)-C(1)	1.327(4)
N(2)-C(3)	1.374(4)
N(2)-C(12)	1.478(4)
N(3)-C(7)	1.327(4)
N(3)-C(8)	1.374(5)
N(3)-C(6)	1.467(4)
N(4)-C(7)	1.330(4)
N(4)-C(9)	1.369(5)
N(4)-C(10)	1.469(4)
N(5)-C(13)	1.333(4)
N(5)-C(14)	1.385(4)
N(5)-C(16)	1.468(4)
N(6)-C(13)	1.331(4)
N(6)-C(15)	1.374(4)
N(6)-C(24)	1.474(4)
N(7)-C(19)	1.332(4)
N(7)-C(20)	1.378(4)
N(7)-C(18)	1.479(4)
N(8)-C(19)	1.335(4)
N(8)-C(21)	1.372(4)
N(8)-C(22)	1.479(4)
C(1)-H(1)	0.9500
C(2)-C(3)	1.343(5)
C(2)-H(2)	0.9500
C(3)-H(3)	0.9500
C(4)-C(5)	1.530(5)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-C(6)	1.521(5)
C(5)-H(5A)	0.9900
C(5)-H(5B)	0.9900

C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-H(7)	0.9500
C(8)-C(9)	1.345(6)
C(8)-H(8)	0.9500
C(9)-H(9)	0.9500
C(10)-C(11)	1.515(5)
C(10)-H(10A)	0.9900
C(10)-H(10B)	0.9900
C(11)-C(12)	1.529(4)
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900
C(12)-H(12A)	0.9900
C(12)-H(12B)	0.9900
C(13)-H(13)	0.9500
C(14)-C(15)	1.339(5)
C(14)-H(14)	0.9500
C(15)-H(15)	0.9500
C(16)-C(17)	1.528(5)
C(16)-H(16A)	0.9900
C(16)-H(16B)	0.9900
C(17)-C(18)	1.525(4)
C(17)-H(17A)	0.9900
C(17)-H(17B)	0.9900
C(18)-H(18A)	0.9900
C(18)-H(18B)	0.9900
C(19)-H(19)	0.9500
C(20)-C(21)	1.349(5)
C(20)-H(20)	0.9500
C(21)-H(21)	0.9500
C(22)-C(23)	1.519(5)
C(22)-H(22A)	0.9900
C(22)-H(22B)	0.9900
C(23)-C(24)	1.523(5)
C(23)-H(23A)	0.9900
C(23)-H(23B)	0.9900

C(24)-H(24A)	0.9900
C(24)-H(24B)	0.9900

C(1)-N(1)-C(2)	107.8(3)
C(1)-N(1)-C(4)	125.5(3)
C(2)-N(1)-C(4)	126.0(3)
C(1)-N(2)-C(3)	107.8(3)
C(1)-N(2)-C(12)	125.5(3)
C(3)-N(2)-C(12)	126.3(3)
C(7)-N(3)-C(8)	107.5(3)
C(7)-N(3)-C(6)	124.9(3)
C(8)-N(3)-C(6)	127.3(3)
C(7)-N(4)-C(9)	108.0(3)
C(7)-N(4)-C(10)	125.2(3)
C(9)-N(4)-C(10)	126.7(3)
C(13)-N(5)-C(14)	108.4(3)
C(13)-N(5)-C(16)	125.2(3)
C(14)-N(5)-C(16)	125.4(3)
C(13)-N(6)-C(15)	108.8(3)
C(13)-N(6)-C(24)	125.2(3)
C(15)-N(6)-C(24)	125.6(3)
C(19)-N(7)-C(20)	108.5(3)
C(19)-N(7)-C(18)	124.3(3)
C(20)-N(7)-C(18)	126.9(3)
C(19)-N(8)-C(21)	108.4(3)
C(19)-N(8)-C(22)	125.6(3)
C(21)-N(8)-C(22)	125.8(3)
N(1)-C(1)-N(2)	109.4(3)
N(1)-C(1)-H(1)	125.3
N(2)-C(1)-H(1)	125.3
C(3)-C(2)-N(1)	107.8(3)
C(3)-C(2)-H(2)	126.1
N(1)-C(2)-H(2)	126.1
C(2)-C(3)-N(2)	107.2(3)
C(2)-C(3)-H(3)	126.4
N(2)-C(3)-H(3)	126.4

N(1)-C(4)-C(5)	112.6(3)
N(1)-C(4)-H(4A)	109.1
C(5)-C(4)-H(4A)	109.1
N(1)-C(4)-H(4B)	109.1
C(5)-C(4)-H(4B)	109.1
H(4A)-C(4)-H(4B)	107.8
C(6)-C(5)-C(4)	116.3(3)
C(6)-C(5)-H(5A)	108.2
C(4)-C(5)-H(5A)	108.2
C(6)-C(5)-H(5B)	108.2
C(4)-C(5)-H(5B)	108.2
H(5A)-C(5)-H(5B)	107.4
N(3)-C(6)-C(5)	113.0(3)
N(3)-C(6)-H(6A)	109.0
C(5)-C(6)-H(6A)	109.0
N(3)-C(6)-H(6B)	109.0
C(5)-C(6)-H(6B)	109.0
H(6A)-C(6)-H(6B)	107.8
N(3)-C(7)-N(4)	109.5(3)
N(3)-C(7)-H(7)	125.3
N(4)-C(7)-H(7)	125.3
C(9)-C(8)-N(3)	107.8(4)
C(9)-C(8)-H(8)	126.1
N(3)-C(8)-H(8)	126.1
C(8)-C(9)-N(4)	107.2(4)
C(8)-C(9)-H(9)	126.4
N(4)-C(9)-H(9)	126.4
N(4)-C(10)-C(11)	113.8(3)
N(4)-C(10)-H(10A)	108.8
C(11)-C(10)-H(10A)	108.8
N(4)-C(10)-H(10B)	108.8
C(11)-C(10)-H(10B)	108.8
H(10A)-C(10)-H(10B)	107.7
C(10)-C(11)-C(12)	117.1(3)
C(10)-C(11)-H(11A)	108.0
C(12)-C(11)-H(11A)	108.0

C(10)-C(11)-H(11B)	108.0
C(12)-C(11)-H(11B)	108.0
H(11A)-C(11)-H(11B)	107.3
N(2)-C(12)-C(11)	112.6(3)
N(2)-C(12)-H(12A)	109.1
C(11)-C(12)-H(12A)	109.1
N(2)-C(12)-H(12B)	109.1
C(11)-C(12)-H(12B)	109.1
H(12A)-C(12)-H(12B)	107.8
N(6)-C(13)-N(5)	108.2(3)
N(6)-C(13)-H(13)	125.9
N(5)-C(13)-H(13)	125.9
C(15)-C(14)-N(5)	107.1(3)
C(15)-C(14)-H(14)	126.5
N(5)-C(14)-H(14)	126.5
C(14)-C(15)-N(6)	107.5(3)
C(14)-C(15)-H(15)	126.3
N(6)-C(15)-H(15)	126.3
N(5)-C(16)-C(17)	112.8(3)
N(5)-C(16)-H(16A)	109.0
C(17)-C(16)-H(16A)	109.0
N(5)-C(16)-H(16B)	109.0
C(17)-C(16)-H(16B)	109.0
H(16A)-C(16)-H(16B)	107.8
C(18)-C(17)-C(16)	117.2(3)
C(18)-C(17)-H(17A)	108.0
C(16)-C(17)-H(17A)	108.0
C(18)-C(17)-H(17B)	108.0
C(16)-C(17)-H(17B)	108.0
H(17A)-C(17)-H(17B)	107.3
N(7)-C(18)-C(17)	114.1(3)
N(7)-C(18)-H(18A)	108.7
C(17)-C(18)-H(18A)	108.7
N(7)-C(18)-H(18B)	108.7
C(17)-C(18)-H(18B)	108.7
H(18A)-C(18)-H(18B)	107.6

N(7)-C(19)-N(8)	108.6(3)
N(7)-C(19)-H(19)	125.7
N(8)-C(19)-H(19)	125.7
C(21)-C(20)-N(7)	107.0(3)
C(21)-C(20)-H(20)	126.5
N(7)-C(20)-H(20)	126.5
C(20)-C(21)-N(8)	107.5(3)
C(20)-C(21)-H(21)	126.3
N(8)-C(21)-H(21)	126.3
N(8)-C(22)-C(23)	112.6(3)
N(8)-C(22)-H(22A)	109.1
C(23)-C(22)-H(22A)	109.1
N(8)-C(22)-H(22B)	109.1
C(23)-C(22)-H(22B)	109.1
H(22A)-C(22)-H(22B)	107.8
C(22)-C(23)-C(24)	118.1(3)
C(22)-C(23)-H(23A)	107.8
C(24)-C(23)-H(23A)	107.8
C(22)-C(23)-H(23B)	107.8
C(24)-C(23)-H(23B)	107.8
H(23A)-C(23)-H(23B)	107.1
N(6)-C(24)-C(23)	113.0(3)
N(6)-C(24)-H(24A)	109.0
C(23)-C(24)-H(24A)	109.0
N(6)-C(24)-H(24B)	109.0
C(23)-C(24)-H(24B)	109.0
H(24A)-C(24)-H(24B)	107.8

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for jam0906. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
I(1)	37(1)	28(1)	26(1)	2(1)	5(1)	1(1)
I(2)	28(1)	33(1)	30(1)	-5(1)	1(1)	2(1)
I(3)	34(1)	41(1)	32(1)	11(1)	1(1)	5(1)
I(4)	26(1)	35(1)	20(1)	-3(1)	1(1)	0(1)
N(1)	26(2)	24(2)	20(2)	3(1)	-1(1)	4(1)
N(2)	22(2)	25(2)	22(2)	-2(1)	0(1)	0(1)
N(3)	22(2)	40(2)	30(2)	7(2)	4(1)	2(1)
N(4)	26(2)	36(2)	27(2)	-8(1)	-2(1)	10(1)
N(5)	24(2)	28(2)	18(2)	2(1)	-2(1)	-5(1)
N(6)	22(2)	19(2)	27(2)	-2(1)	1(1)	-4(1)
N(7)	28(2)	24(2)	22(2)	2(1)	-3(1)	-7(1)
N(8)	28(2)	22(2)	20(2)	3(1)	-1(1)	-3(1)
C(1)	26(2)	29(2)	19(2)	2(2)	-1(2)	6(2)
C(2)	51(2)	35(2)	20(2)	6(2)	-5(2)	-8(2)
C(3)	55(3)	39(3)	16(2)	-1(2)	-5(2)	-8(2)
C(4)	39(2)	22(2)	31(2)	-3(2)	-5(2)	0(2)
C(5)	43(2)	24(2)	36(2)	7(2)	-7(2)	-9(2)
C(6)	34(2)	37(2)	43(2)	11(2)	-4(2)	-12(2)
C(7)	23(2)	36(2)	21(2)	0(2)	0(2)	3(2)
C(8)	60(3)	74(4)	27(2)	10(2)	22(2)	23(3)
C(9)	68(3)	62(3)	29(2)	-6(2)	5(2)	30(3)
C(10)	42(2)	23(2)	52(3)	-6(2)	-14(2)	6(2)
C(11)	42(2)	19(2)	39(2)	1(2)	-5(2)	-2(2)
C(12)	34(2)	20(2)	34(2)	-3(2)	1(2)	-4(2)
C(13)	23(2)	25(2)	25(2)	-2(2)	1(2)	-2(2)
C(14)	25(2)	27(2)	36(2)	-1(2)	-3(2)	2(2)
C(15)	17(2)	28(2)	39(2)	-5(2)	3(2)	-1(2)
C(16)	41(2)	39(2)	25(2)	4(2)	3(2)	-5(2)
C(17)	37(2)	31(2)	25(2)	10(2)	4(2)	-7(2)
C(18)	37(2)	34(2)	32(2)	7(2)	5(2)	-13(2)
C(19)	25(2)	25(2)	26(2)	-1(2)	-2(2)	-1(2)

C(20)	40(2)	23(2)	31(2)	-4(2)	-4(2)	1(2)
C(21)	35(2)	27(2)	25(2)	-6(2)	0(2)	0(2)
C(22)	37(2)	30(2)	22(2)	10(2)	2(2)	-3(2)
C(23)	34(2)	22(2)	35(2)	11(2)	8(2)	2(2)
C(24)	37(2)	23(2)	33(2)	-1(2)	9(2)	-6(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for jam0906.

	x	y	z	U(eq)
H(1)	9986	6693	4164	30
H(2)	10005	6192	7009	42
H(3)	10720	6978	7158	44
H(4A)	9813	5641	5197	37
H(4B)	9052	5910	4227	37
H(5A)	7083	5492	5218	42
H(5B)	7367	5846	6116	42
H(6A)	6024	6067	4108	46
H(6B)	4908	5968	5071	46
H(7)	6846	6847	3880	32
H(8)	5467	6566	6725	64
H(9)	6302	7346	6698	64
H(10A)	7508	7868	5367	47
H(10B)	6828	7764	4215	47
H(11A)	9619	7893	4175	40
H(11B)	9277	7391	3877	40
H(12A)	11693	7512	5000	35
H(12B)	10488	7643	5898	35
H(13)	7623	3639	7786	29
H(14)	4572	4676	7740	35
H(15)	3685	4162	9081	33
H(16A)	6925	4521	6141	42
H(16B)	8456	4248	6622	42
H(17A)	8855	5027	6716	37
H(17B)	7350	5060	7464	37
H(18A)	10326	4607	8032	41
H(18B)	10009	5108	8336	41
H(19)	9493	4046	9308	30
H(20)	7276	5211	9621	38
H(21)	6250	4708	10954	35

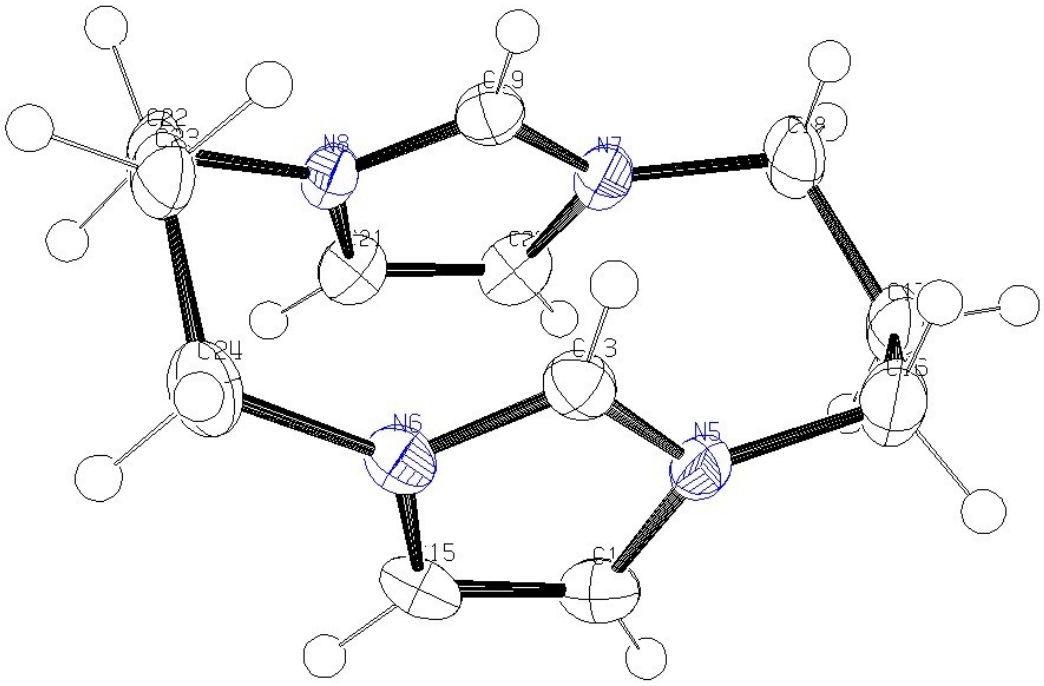
H(22A)	6376	3870	11258	36
H(22B)	8307	3773	11403	36
H(23A)	7197	3167	10616	36
H(23B)	8203	3415	9760	36
H(24A)	5701	3130	9061	37
H(24B)	4698	3428	9835	37

Table 6. Torsion angles [°] for jam0906.

C(2)-N(1)-C(1)-N(2)	-1.8(4)
C(4)-N(1)-C(1)-N(2)	168.6(3)
C(3)-N(2)-C(1)-N(1)	1.9(4)
C(12)-N(2)-C(1)-N(1)	-171.7(3)
C(1)-N(1)-C(2)-C(3)	1.0(4)
C(4)-N(1)-C(2)-C(3)	-169.3(3)
N(1)-C(2)-C(3)-N(2)	0.1(4)
C(1)-N(2)-C(3)-C(2)	-1.2(4)
C(12)-N(2)-C(3)-C(2)	172.3(3)
C(1)-N(1)-C(4)-C(5)	-109.1(4)
C(2)-N(1)-C(4)-C(5)	59.6(4)
N(1)-C(4)-C(5)-C(6)	74.6(4)
C(7)-N(3)-C(6)-C(5)	103.9(4)
C(8)-N(3)-C(6)-C(5)	-68.1(5)
C(4)-C(5)-C(6)-N(3)	-68.4(4)
C(8)-N(3)-C(7)-N(4)	2.7(4)
C(6)-N(3)-C(7)-N(4)	-170.7(3)
C(9)-N(4)-C(7)-N(3)	-2.5(4)
C(10)-N(4)-C(7)-N(3)	174.1(3)
C(7)-N(3)-C(8)-C(9)	-1.9(4)
C(6)-N(3)-C(8)-C(9)	171.3(3)
N(3)-C(8)-C(9)-N(4)	0.4(5)
C(7)-N(4)-C(9)-C(8)	1.3(4)
C(10)-N(4)-C(9)-C(8)	-175.3(3)
C(7)-N(4)-C(10)-C(11)	-59.3(4)
C(9)-N(4)-C(10)-C(11)	116.7(4)
N(4)-C(10)-C(11)-C(12)	-62.4(4)
C(1)-N(2)-C(12)-C(11)	39.4(4)
C(3)-N(2)-C(12)-C(11)	-132.9(3)
C(10)-C(11)-C(12)-N(2)	72.1(4)
C(15)-N(6)-C(13)-N(5)	1.4(3)
C(24)-N(6)-C(13)-N(5)	-172.3(3)
C(14)-N(5)-C(13)-N(6)	-1.3(3)
C(16)-N(5)-C(13)-N(6)	167.9(3)

C(13)-N(5)-C(14)-C(15)	0.7(4)
C(16)-N(5)-C(14)-C(15)	-168.4(3)
N(5)-C(14)-C(15)-N(6)	0.2(4)
C(13)-N(6)-C(15)-C(14)	-1.0(4)
C(24)-N(6)-C(15)-C(14)	172.7(3)
C(13)-N(5)-C(16)-C(17)	-111.4(4)
C(14)-N(5)-C(16)-C(17)	56.0(4)
N(5)-C(16)-C(17)-C(18)	66.4(4)
C(19)-N(7)-C(18)-C(17)	104.8(4)
C(20)-N(7)-C(18)-C(17)	-68.0(4)
C(16)-C(17)-C(18)-N(7)	-69.9(4)
C(20)-N(7)-C(19)-N(8)	2.3(3)
C(18)-N(7)-C(19)-N(8)	-171.7(3)
C(21)-N(8)-C(19)-N(7)	-2.5(3)
C(22)-N(8)-C(19)-N(7)	173.7(3)
C(19)-N(7)-C(20)-C(21)	-1.2(4)
C(18)-N(7)-C(20)-C(21)	172.6(3)
N(7)-C(20)-C(21)-N(8)	-0.3(4)
C(19)-N(8)-C(21)-C(20)	1.7(4)
C(22)-N(8)-C(21)-C(20)	-174.5(3)
C(19)-N(8)-C(22)-C(23)	-41.7(4)
C(21)-N(8)-C(22)-C(23)	133.9(3)
N(8)-C(22)-C(23)-C(24)	-68.9(4)
C(13)-N(6)-C(24)-C(23)	63.8(4)
C(15)-N(6)-C(24)-C(23)	-108.8(3)
C(22)-C(23)-C(24)-N(6)	62.9(4)

Symmetry transformations used to generate equivalent atoms:


C19
N8
N7
C21
C24
C3
N6
N5
C15
C1

Crystal Structure data for **2.8**

Table 1. Crystal data and structure refinement for jam1006.

Identification code	jam1006	
Empirical formula	C25.30 H41.20 I4 N8 O1.30	
Formula weight	985.86	
Temperature	150(2) K	
Wavelength	0.71070 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 7.6944(4) Å	α= 76.506(3)°.
	b = 9.5717(6) Å	β= 83.023(4)°.
	c = 12.3057(8) Å	γ = 80.749(4)°.
Volume	866.48(9) $Å^3$	
Z	1	
Density (calculated)	1.889 Mg/m^3	
Absorption coefficient	3.628 mm^{-1}	
F(000)	471	
Crystal size	0.25 x 0.20 x 0.04 mm^3	
Theta range for data collection	2.69 to 27.21°.	
Index ranges	-9<=h<=9, -12<=k<=12, -15<=l<=15	
Reflections collected	16447	
Independent reflections	3822 [R(int) = 0.0554]	
Completeness to theta = 27.21°	98.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.000 and 0.430	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3822 / 2 / 177	
Goodness-of-fit on F^2	1.063	
Final R indices [I>2sigma(I)]	R1 = 0.0393, wR2 = 0.0682	
R indices (all data)	R1 = 0.0742, wR2 = 0.0757	
Largest diff. peak and hole	0.818 and -0.729 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for jam1006. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
I(1)	2849(1)	1770(1)	9708(1)	39(1)
I(2)	146(1)	-7429(1)	14122(1)	35(1)
N(1)	1302(5)	-3018(5)	11603(4)	31(1)
N(2)	2034(5)	-3078(4)	9874(4)	29(1)
N(3)	4497(5)	-824(5)	13222(4)	34(1)
N(4)	5347(6)	1214(5)	13239(4)	33(1)
C(1)	1707(7)	-2231(6)	10596(5)	31(1)
C(2)	1807(7)	-4470(6)	10440(5)	37(1)
C(3)	1340(7)	-4430(6)	11511(5)	37(1)
C(4)	584(7)	-2442(6)	12604(5)	39(1)
C(5)	1637(7)	-1294(6)	12756(5)	34(1)
C(6)	3530(7)	-1885(6)	12953(5)	40(1)
C(7)	4159(7)	606(6)	12873(5)	36(1)
C(8)	6483(7)	129(6)	13838(5)	37(1)
C(9)	5951(7)	-1145(6)	13819(5)	35(1)
C(10)	2413(7)	-2571(6)	8663(5)	36(1)
C(11)	4144(7)	-3380(6)	8242(5)	36(1)
C(12)	4575(7)	-2775(6)	6984(5)	39(1)
O(1S)	7147(19)	-4718(14)	15095(10)	42(3)
C(1S)	6310(30)	-4610(20)	14442(18)	50
O(2S)	6174(19)	-4759(15)	15039(13)	42(4)
C(2S)	7430(30)	-4540(30)	14660(20)	51

Table 3. Bond lengths [Å] and angles [°] for jam1006.

N(1)-C(1)	1.321(7)
N(1)-C(3)	1.377(6)
N(1)-C(4)	1.477(7)
N(2)-C(1)	1.314(6)
N(2)-C(2)	1.380(6)
N(2)-C(10)	1.463(7)
N(3)-C(7)	1.328(6)
N(3)-C(9)	1.367(7)
N(3)-C(6)	1.468(6)
N(4)-C(7)	1.331(6)
N(4)-C(8)	1.376(7)
N(4)-C(12)#1	1.464(7)
C(1)-H(1)	0.9500
C(2)-C(3)	1.331(8)
C(2)-H(2)	0.9500
C(3)-H(3)	0.9500
C(4)-C(5)	1.522(7)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-C(6)	1.503(7)
C(5)-H(5A)	0.9900
C(5)-H(5B)	0.9900
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-H(7)	0.9500
C(8)-C(9)	1.353(7)
C(8)-H(8)	0.9500
C(9)-H(9)	0.9500
C(10)-C(11)	1.526(7)
C(10)-H(10A)	0.9900
C(10)-H(10B)	0.9900
C(11)-C(12)	1.537(8)
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900

C(12)-N(4)#1	1.464(7)
C(12)-H(12A)	0.9900
C(12)-H(12B)	0.9900
O(1S)-C(2S)	0.56(3)
O(1S)-O(2S)	0.768(15)
O(1S)-C(1S)	1.066(15)
C(1S)-O(2S)	0.71(2)
C(1S)-C(2S)	0.95(3)
O(2S)-C(2S)	1.056(17)

C(1)-N(1)-C(3)	108.3(5)
C(1)-N(1)-C(4)	125.6(5)
C(3)-N(1)-C(4)	125.1(5)
C(1)-N(2)-C(2)	108.3(5)
C(1)-N(2)-C(10)	124.6(4)
C(2)-N(2)-C(10)	126.9(4)
C(7)-N(3)-C(9)	108.8(4)
C(7)-N(3)-C(6)	125.4(4)
C(9)-N(3)-C(6)	125.7(5)
C(7)-N(4)-C(8)	108.3(4)
C(7)-N(4)-C(12)#1	125.0(5)
C(8)-N(4)-C(12)#1	126.6(4)
N(2)-C(1)-N(1)	109.0(5)
N(2)-C(1)-H(1)	125.5
N(1)-C(1)-H(1)	125.5
C(3)-C(2)-N(2)	107.2(5)
C(3)-C(2)-H(2)	126.4
N(2)-C(2)-H(2)	126.4
C(2)-C(3)-N(1)	107.1(5)
C(2)-C(3)-H(3)	126.4
N(1)-C(3)-H(3)	126.4
N(1)-C(4)-C(5)	111.1(4)
N(1)-C(4)-H(4A)	109.4
C(5)-C(4)-H(4A)	109.4
N(1)-C(4)-H(4B)	109.4
C(5)-C(4)-H(4B)	109.4

H(4A)-C(4)-H(4B)	108.0
C(6)-C(5)-C(4)	112.7(5)
C(6)-C(5)-H(5A)	109.1
C(4)-C(5)-H(5A)	109.1
C(6)-C(5)-H(5B)	109.1
C(4)-C(5)-H(5B)	109.1
H(5A)-C(5)-H(5B)	107.8
N(3)-C(6)-C(5)	112.8(4)
N(3)-C(6)-H(6A)	109.0
C(5)-C(6)-H(6A)	109.0
N(3)-C(6)-H(6B)	109.0
C(5)-C(6)-H(6B)	109.0
H(6A)-C(6)-H(6B)	107.8
N(3)-C(7)-N(4)	108.7(5)
N(3)-C(7)-H(7)	125.7
N(4)-C(7)-H(7)	125.7
C(9)-C(8)-N(4)	107.0(5)
C(9)-C(8)-H(8)	126.5
N(4)-C(8)-H(8)	126.5
C(8)-C(9)-N(3)	107.2(5)
C(8)-C(9)-H(9)	126.4
N(3)-C(9)-H(9)	126.4
N(2)-C(10)-C(11)	111.1(4)
N(2)-C(10)-H(10A)	109.4
C(11)-C(10)-H(10A)	109.4
N(2)-C(10)-H(10B)	109.4
C(11)-C(10)-H(10B)	109.4
H(10A)-C(10)-H(10B)	108.0
C(10)-C(11)-C(12)	110.4(5)
C(10)-C(11)-H(11A)	109.6
C(12)-C(11)-H(11A)	109.6
C(10)-C(11)-H(11B)	109.6
C(12)-C(11)-H(11B)	109.6
H(11A)-C(11)-H(11B)	108.1
N(4)#1-C(12)-C(11)	110.7(4)
N(4)#1-C(12)-H(12A)	109.5

C(11)-C(12)-H(12A)	109.5
N(4)#1-C(12)-H(12B)	109.5
C(11)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	108.1
C(2S)-O(1S)-O(2S)	105(3)
C(2S)-O(1S)-C(1S)	63(3)
O(2S)-O(1S)-C(1S)	42.0(15)
O(2S)-C(1S)-C(2S)	77(2)
O(2S)-C(1S)-O(1S)	46.1(15)
C(2S)-C(1S)-O(1S)	31.5(16)
C(1S)-O(2S)-O(1S)	92(2)
C(1S)-O(2S)-C(2S)	61(2)
O(1S)-O(2S)-C(2S)	30.7(18)
O(1S)-C(2S)-C(1S)	86(3)
O(1S)-C(2S)-O(2S)	45(2)
C(1S)-C(2S)-O(2S)	41.3(14)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y,-z+2

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for jam1006. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
I(1)	38(1)	29(1)	49(1)	-11(1)	-7(1)	3(1)
I(2)	37(1)	36(1)	34(1)	-6(1)	-4(1)	-7(1)
N(1)	34(3)	30(3)	28(3)	-5(2)	-2(2)	-5(2)
N(2)	26(2)	28(2)	33(3)	-9(2)	-5(2)	2(2)
N(3)	30(3)	38(3)	34(3)	-13(2)	-2(2)	-2(2)
N(4)	32(2)	35(3)	35(3)	-12(2)	-2(2)	-5(2)
C(1)	35(3)	26(3)	32(3)	-8(3)	-2(2)	0(2)
C(2)	40(3)	19(3)	51(4)	-8(3)	-8(3)	1(2)
C(3)	40(3)	25(3)	44(4)	1(3)	-9(3)	-7(2)
C(4)	37(3)	45(4)	36(4)	-10(3)	2(3)	-14(3)
C(5)	34(3)	41(3)	28(3)	-11(3)	1(2)	-6(2)
C(6)	38(3)	37(3)	46(4)	-10(3)	-8(3)	-6(3)
C(7)	34(3)	33(3)	41(4)	-11(3)	-7(3)	-2(2)
C(8)	26(3)	46(4)	41(4)	-16(3)	-5(3)	-2(3)
C(9)	28(3)	40(3)	38(4)	-14(3)	0(3)	3(2)
C(10)	36(3)	38(3)	34(3)	-13(3)	-6(3)	7(2)
C(11)	32(3)	34(3)	44(4)	-17(3)	4(3)	-3(2)
C(12)	33(3)	35(3)	53(4)	-21(3)	4(3)	-7(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for jam1006.

	x	y	z	U(eq)
H(1)	1754	-1217	10423	37
H(2)	1956	-5302	10124	44
H(3)	1080	-5225	12101	45
H(4A)	622	-3245	13278	46
H(4B)	-666	-2013	12526	46
H(5A)	1603	-497	12077	41
H(5B)	1072	-882	13400	41
H(6A)	3564	-2751	13578	48
H(6B)	4130	-2195	12273	48
H(7)	3224	1110	12435	43
H(8)	7457	251	14197	44
H(9)	6487	-2086	14157	43
H(10A)	2491	-1519	8491	43
H(10B)	1434	-2724	8270	43
H(11A)	4043	-4425	8372	43
H(11B)	5113	-3274	8664	43
H(12A)	5726	-3279	6730	46
H(12B)	3658	-2958	6556	46

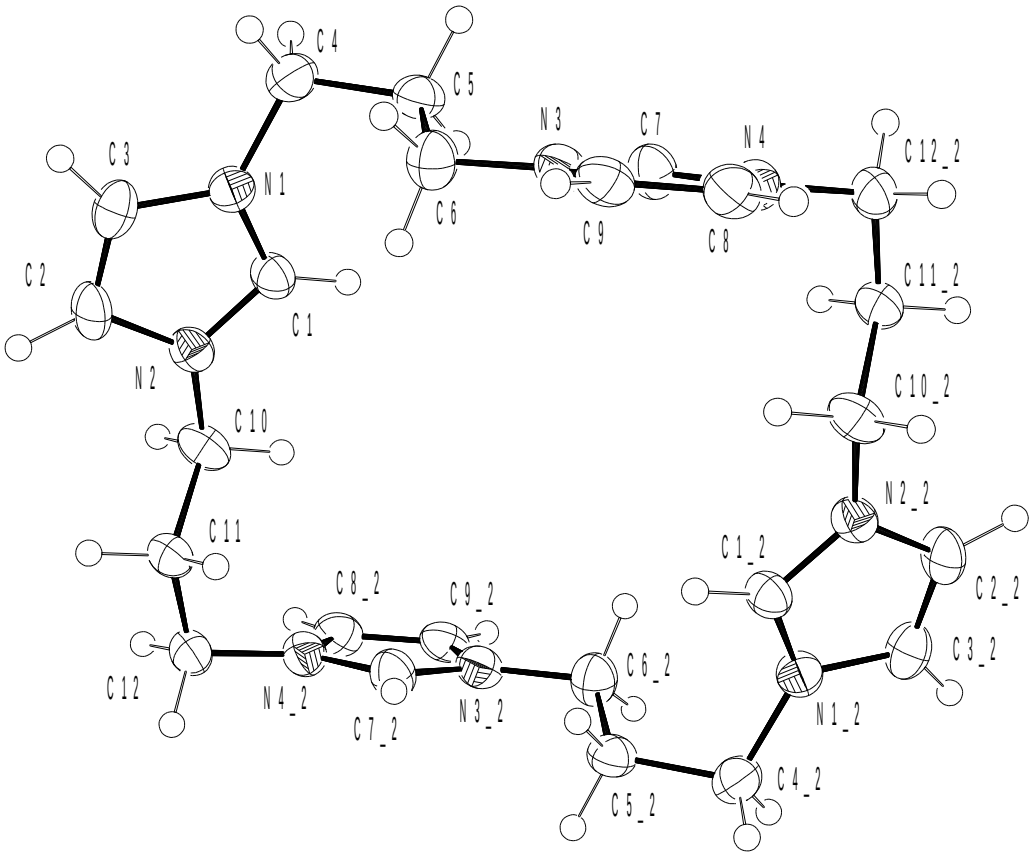
Table 6. Torsion angles [°] for jam1006.

C(2)-N(2)-C(1)-N(1)	0.8(6)
C(10)-N(2)-C(1)-N(1)	175.7(4)
C(3)-N(1)-C(1)-N(2)	-1.3(6)
C(4)-N(1)-C(1)-N(2)	-169.9(4)
C(1)-N(2)-C(2)-C(3)	0.0(6)
C(10)-N(2)-C(2)-C(3)	-174.8(5)
N(2)-C(2)-C(3)-N(1)	-0.7(6)
C(1)-N(1)-C(3)-C(2)	1.2(6)
C(4)-N(1)-C(3)-C(2)	170.0(5)
C(1)-N(1)-C(4)-C(5)	-47.8(7)
C(3)-N(1)-C(4)-C(5)	145.4(5)
N(1)-C(4)-C(5)-C(6)	-62.7(6)
C(7)-N(3)-C(6)-C(5)	-29.0(8)
C(9)-N(3)-C(6)-C(5)	154.9(5)
C(4)-C(5)-C(6)-N(3)	-173.7(5)
C(9)-N(3)-C(7)-N(4)	-0.6(6)
C(6)-N(3)-C(7)-N(4)	-177.2(5)
C(8)-N(4)-C(7)-N(3)	0.2(6)
C(12)#1-N(4)-C(7)-N(3)	179.3(5)
C(7)-N(4)-C(8)-C(9)	0.2(6)
C(12)#1-N(4)-C(8)-C(9)	-178.9(5)
N(4)-C(8)-C(9)-N(3)	-0.6(6)
C(7)-N(3)-C(9)-C(8)	0.7(6)
C(6)-N(3)-C(9)-C(8)	177.3(5)
C(1)-N(2)-C(10)-C(11)	126.1(5)
C(2)-N(2)-C(10)-C(11)	-59.9(7)
N(2)-C(10)-C(11)-C(12)	-176.7(4)
C(10)-C(11)-C(12)-N(4)#1	56.7(6)
C(2S)-O(1S)-C(1S)-O(2S)	-174(5)
O(2S)-O(1S)-C(1S)-C(2S)	174(5)
C(2S)-C(1S)-O(2S)-O(1S)	-3(3)
O(1S)-C(1S)-O(2S)-C(2S)	3(3)
C(2S)-O(1S)-O(2S)-C(1S)	6(5)
C(1S)-O(1S)-O(2S)-C(2S)	-6(5)

O(2S)-O(1S)-C(2S)-C(1S)	-4(4)
C(1S)-O(1S)-C(2S)-O(2S)	4(4)
O(2S)-C(1S)-C(2S)-O(1S)	5(4)
O(1S)-C(1S)-C(2S)-O(2S)	-5(4)
C(1S)-O(2S)-C(2S)-O(1S)	-173(6)
O(1S)-O(2S)-C(2S)-C(1S)	173(6)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y,-z+2


C4
C5
N3
C7
N4
C12_2
C3
N1
C6
C9
C8
C2
C11_2
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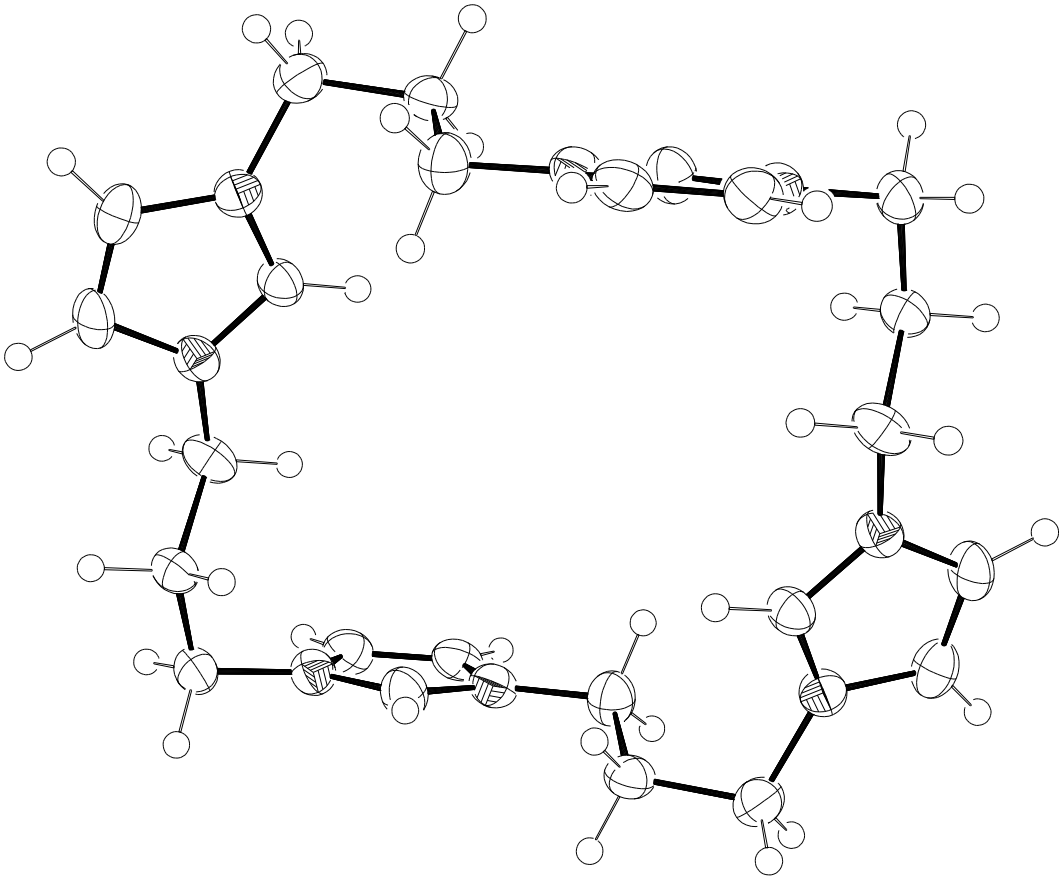
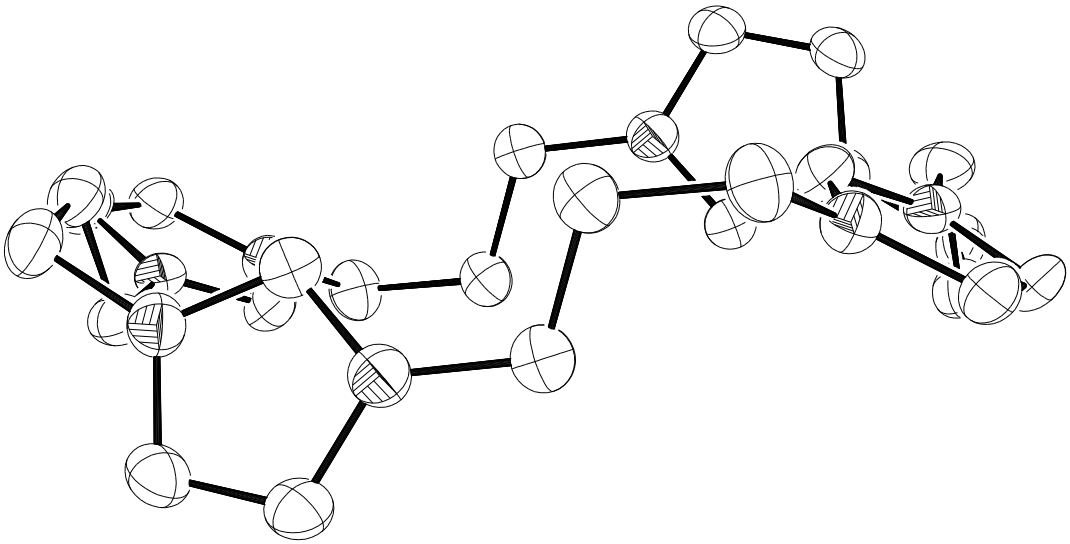
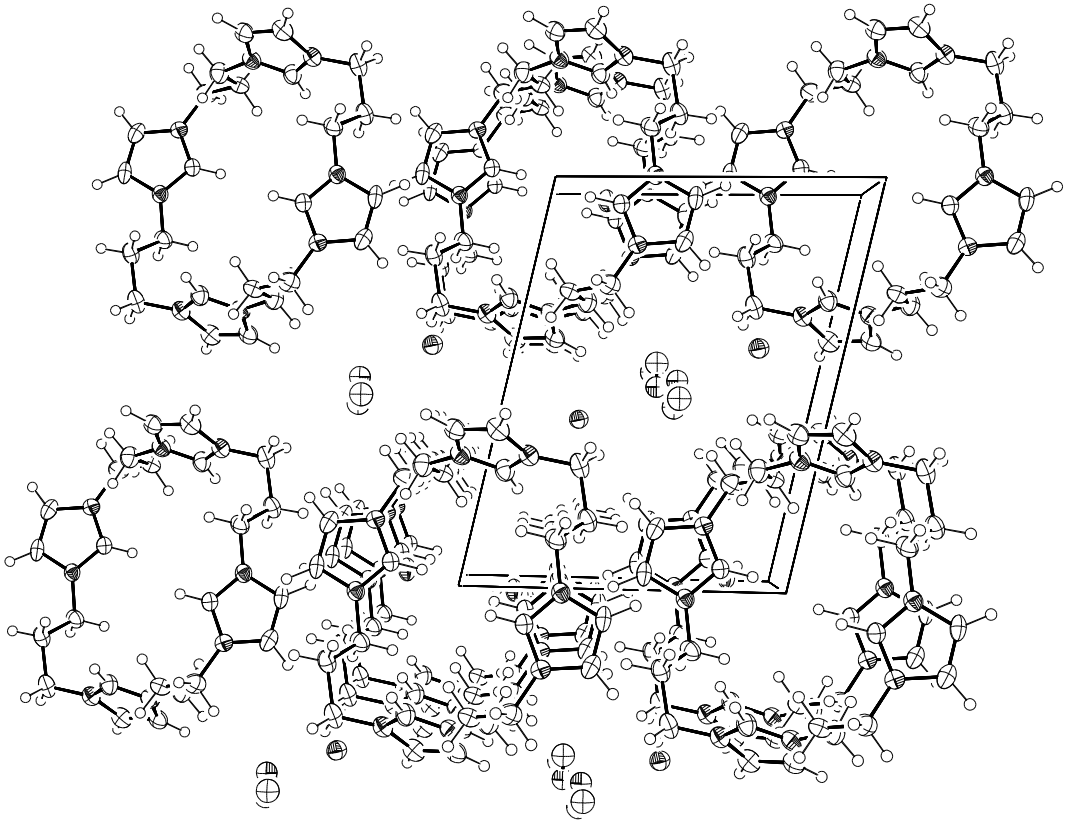
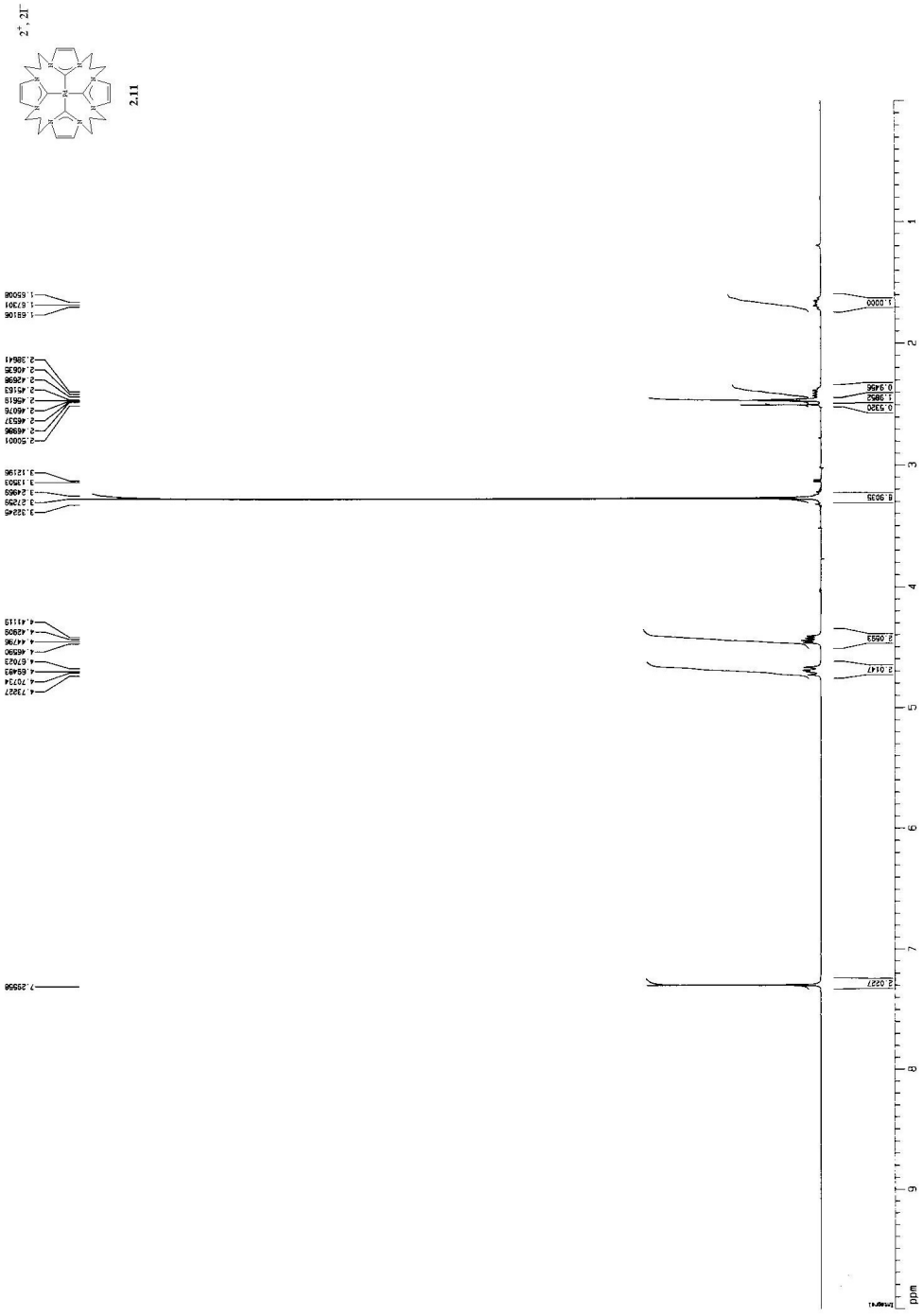
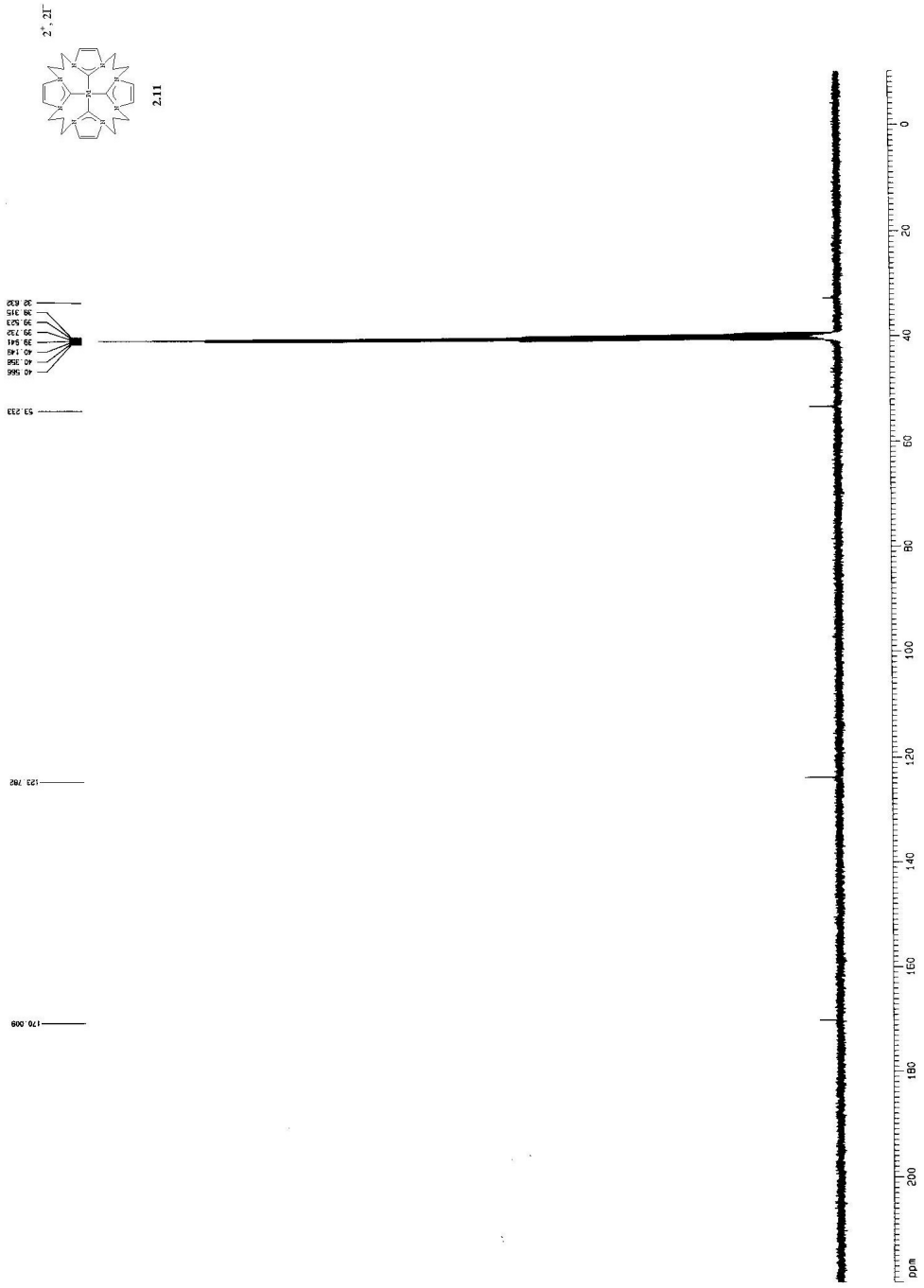
N2
C10_2
C10
N2_2
C11
C1_2
C8_2
C9_2
C2_2
C6_2
C3_2
C12
N4_2
C7_2
N3_2
N1_2
C4_2
C5_2

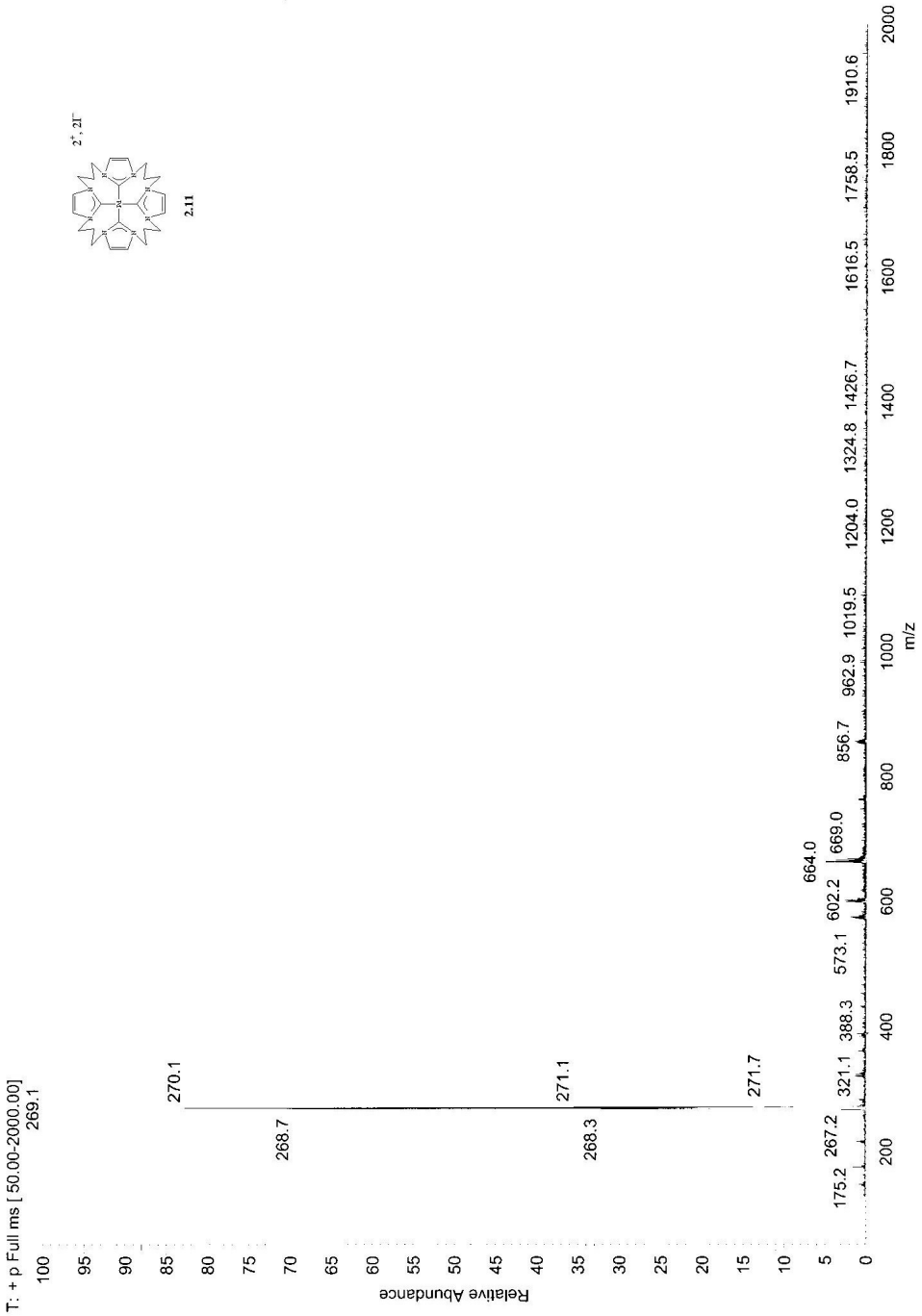







2+, 2I-
2.11
ppm


2.11
ppm


2+, 2I-
2.11
T: + p Full ms [50.00-2000.00]
Relative Abundance
m/z
269.1
270.1
268.7
271.1
268.3
271.7
175.2
267.2
321.1
388.3
573.1
602.2
664.0
669.0
856.7
962.9
1019.5
1204.0
1324.8
1426.7
1616.5
1758.5
1910.6

Crystal Structure Data for **2.11**

```
CIF_1.1

CIF produced by WinGX routine CIF_UPDATE
Created on 2006-08-04 at 15:05:50
Using CIFtbx version 2.6.2 16 Jun 1998

Dictionary name : cif_core.dic
Dictionary vers : 2.3
Request file    : h:\wingx\files\archive.dat
CIF files read  : stuartp2 struct


data_stuartp2

_audit_creation_date                    2006-08-04T15:05:50-00:00
_audit_creation_method                  'WinGX routine CIF_UPDATE'
_audit_conform_dict_name                cif_core.dic
_audit_conform_dict_version             2.3
_audit_conform_dict_location
ftp://ftp.iucr.org/pub/cif_core.dic
_publ_requested_category                FM

#------------------------------------------------------------------
---------#
#                   CHEMICAL INFORMATION
#
#------------------------------------------------------------------
---------#

_chemical_name_systematic
;
 ?
;
_chemical_formula_moiety                'C26.75 H38 I2 N8 O1 Pd1
S0.25'
_chemical_formula_sum                   'C26.75 H38 I2 N8 O1 Pd1
S0.25'
_chemical_formula_weight                855.60
_chemical_compound_source               'synthesis as described'

#------------------------------------------------------------------
---------#
```

```
#                    UNIT CELL INFORMATION
#
#--------------------------------------------------------------------
---------#

_symmetry_cell_setting                  triclinic
_symmetry_space_group_name_H-M          'P -1'
_symmetry_space_group_name_Hall         '-P 1'
_symmetry_Int_Tables_number             2
loop_
    _symmetry_equiv_pos_as_xyz
'x, y, z'
'-x, -y, -z'

_cell_length_a                          9.6438(2)
_cell_length_b                          11.1519(3)
_cell_length_c                          16.9242(4)
_cell_angle_alpha                       92.8040(10)
_cell_angle_beta                        103.9330(10)
_cell_angle_gamma                       115.2880(10)
_cell_volume                            1573.23(7)
_cell_formula_units_Z                   2
_cell_measurement_temperature           120(2)
_cell_measurement_reflns_used           6989
_cell_measurement_theta_min             2.91
_cell_measurement_theta_max             27.48
_cell_measurement_wavelength            0.71073

#--------------------------------------------------------------------
---------#
#                    CRYSTAL INFORMATION
#
#--------------------------------------------------------------------
---------#

_exptl_crystal_description              slab
_exptl_crystal_colour                   colourless
_exptl_crystal_size_max                 0.18
_exptl_crystal_size_mid                 0.06
_exptl_crystal_size_min                 0.03
_exptl_crystal_density_diffrn           1.806
_exptl_crystal_density_method           'not measured'
_exptl_crystal_F_000                    837
_exptl_special_details
;
```

```
 ?
;

#------------------------------------------------------------------
---------#
#                   ABSORPTION CORRECTION
#
#------------------------------------------------------------------
---------#

_exptl_absorpt_coefficient_mu           2.62
_exptl_absorpt_correction_type
_exptl_absorpt_process_details          'SADABS V2.10 (Sheldrick,
G.M., 2003)'

#------------------------------------------------------------------
---------#
#                   DATA COLLECTION
#
#------------------------------------------------------------------
---------#
_diffrn_ambient_temperature             120(2)
_diffrn_radiation_wavelength            0.71073
_diffrn_radiation_type                  MoK\a
_diffrn_radiation_source                'Bruker-Nonius FR591
rotating anode'
_diffrn_radiation_monochromator         '10cm confocal mirrors'
_diffrn_measurement_device_type         'Bruker-Nonius 95mm CCD
camera on \k-goniostat'
_diffrn_measurement_method              '\f & \w scans'
_diffrn_detector_area_resol_mean        9.091
_diffrn_reflns_av_R_equivalents         0.0357
_diffrn_reflns_av_unetI/netI            0.0305
_diffrn_reflns_number                   31692
_diffrn_reflns_limit_h_min              -12
_diffrn_reflns_limit_h_max              12
_diffrn_reflns_limit_k_min              -14
_diffrn_reflns_limit_k_max              14
_diffrn_reflns_limit_l_min              -22
_diffrn_reflns_limit_l_max              21
_diffrn_reflns_theta_min                2.96
_diffrn_reflns_theta_max                27.56
_diffrn_reflns_theta_full               27.56
_diffrn_measured_fraction_theta_full    0.994
_diffrn_measured_fraction_theta_max     0.994
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_reflns_number_total                    7226
_reflns_number_gt                       6721
_reflns_threshold_expression            >2sigma(I)

#------------------------------------------------------------------
---------#
#                   COMPUTER PROGRAMS USED
#
#------------------------------------------------------------------
---------#
_computing_data_collection              'COLLECT (Hooft, R.W.W.,
1998)'
_computing_cell_refinement
'DENZO (Otwinowski & Minor, 1997) & COLLECT (Hooft, R.W.W., 1998)'
#Although determined using DirAx, the cell is refined in the HKL
#package during data reduction
_computing_data_reduction
'DENZO (Otwinowski & Minor, 1997) & COLLECT (Hooft, R.W.W., 1998)'
_computing_structure_solution           'SHELXS-86 (Sheldrick,
1986)'
_computing_structure_refinement         'SHELXL-97 (Sheldrick,
1997)'
_computing_molecular_graphics           'Ortep-3 for Windows
(Farrugia, 1997)'
_computing_publication_material
                                    'WinGX publication routines
(Farrugia, 1999)'

#------------------------------------------------------------------
---------#
#                   REFINEMENT INFORMATION
#
#------------------------------------------------------------------
---------#

_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor
wR and
 goodness of fit S are based on F^2^, conventional R-factors R are
based
 on F, with F set to zero for negative F^2^. The threshold
expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc.
and is
```

```
 not relevant to the choice of reflections for refinement.  R-
factors based
 on F^2^ are statistically about twice as large as those based on F,
and R-
 factors based on ALL data will be even larger.
;
_refine_ls_structure_factor_coef        Fsqd
_refine_ls_matrix_type                  full
_refine_ls_weighting_scheme             calc
_refine_ls_weighting_details
         'calc w=1/[\s^2^(Fo^2^)+(0.0065P)^2^+2.2962P] where
P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary            direct
_atom_sites_solution_secondary          difmap
_atom_sites_solution_hydrogens          geom
_refine_ls_hydrogen_treatment           mixed
_refine_ls_extinction_method            none
_refine_ls_number_reflns                7226
_refine_ls_number_parameters            360
_refine_ls_number_restraints            0
_refine_ls_R_factor_all                 0.0274
_refine_ls_R_factor_gt                  0.0239
_refine_ls_wR_factor_ref                0.0513
_refine_ls_wR_factor_gt                 0.0499
_refine_ls_goodness_of_fit_ref          1.086
_refine_ls_restrained_S_all             1.086
_refine_ls_shift/su_max                 0.005
_refine_ls_shift/su_mean                0
_refine_diff_density_max                0.539
_refine_diff_density_min                -0.91
_refine_diff_density_rms                0.091

#-------------------------------------------------------------------
---------#
#                   ATOMIC TYPES, COORDINATES AND THERMAL PARAMETERS
#
#-------------------------------------------------------------------
---------#

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    _atom_type_scat_dispersion_imag
    _atom_type_scat_source
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C C 0.0033 0.0016 'International Tables Vol C Tables 4.2.6.8 and
6.1.1.4'
H H 0 0 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
I I -0.4742 1.8119 'International Tables Vol C Tables 4.2.6.8 and
6.1.1.4'
N N 0.0061 0.0033 'International Tables Vol C Tables 4.2.6.8 and
6.1.1.4'
Pd Pd -0.9988 1.0072 'International Tables Vol C Tables 4.2.6.8 and
6.1.1.4'
O O 0.0106 0.006 'International Tables Vol C Tables 4.2.6.8 and
6.1.1.4'
S S 0.1246 0.1234 'International Tables Vol C Tables 4.2.6.8 and
6.1.1.4'

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    _atom_site_fract_x
    _atom_site_fract_y
    _atom_site_fract_z
    _atom_site_U_iso_or_equiv
    _atom_site_adp_type
    _atom_site_occupancy
    _atom_site_symmetry_multiplicity
    _atom_site_calc_flag
    _atom_site_refinement_flags
    _atom_site_disorder_assembly
    _atom_site_disorder_group
N7 N 0.5749(2) 0.9825(2) 0.86529(13) 0.0166(4) Uani 1 1 d . . .
C17 C 0.4430(3) 1.1372(3) 0.84903(17) 0.0210(5) Uani 1 1 d . . .
H17A H 0.4868 1.2335 0.8495 0.025 Uiso 1 1 calc R . .
H17B H 0.4435 1.123 0.9052 0.025 Uiso 1 1 calc R . .
C18 C 0.5552(3) 1.0890(3) 0.82411(16) 0.0202(5) Uani 1 1 d . . .
H18A H 0.5154 1.0568 0.7649 0.024 Uiso 1 1 calc R . .
H18B H 0.6599 1.1656 0.8352 0.024 Uiso 1 1 calc R . .
C19 C 0.4709(3) 0.8498(2) 0.84502(15) 0.0150(5) Uani 1 1 d . . .
C16 C 0.2685(3) 1.0708(3) 0.79473(17) 0.0208(5) Uani 1 1 d . . .
H16A H 0.2287 1.1375 0.7934 0.025 Uiso 1 1 calc R . .
H16B H 0.2664 1.0448 0.7388 0.025 Uiso 1 1 calc R . .
C21 C 0.6824(3) 0.8856(3) 0.95314(15) 0.0193(5) Uani 1 1 d . . .
H21 H 0.7499 0.8693 0.9957 0.023 Uiso 1 1 calc R . .
C20 C 0.7059(3) 1.0057(3) 0.93170(16) 0.0209(5) Uani 1 1 d . . .
H20 H 0.7931 1.0885 0.9565 0.025 Uiso 1 1 calc R . .
O1SA O 0.9796(4) 0.7635(4) 0.3494(2) 0.0270(8) Uiso 0.75 1 d P . .
C3S C 1.1796(3) 0.8997(3) 0.29062(18) 0.0297(7) Uani 1 1 d . . .
```

```
C1S C 1.0587(4) 0.9964(3) 0.38228(19) 0.0306(7) Uani 1 1 d . . .
C2S C 1.0688(5) 0.8802(4) 0.3420(2) 0.0221(8) Uiso 0.75 1 d P . .
O2 O 0.7778(10) 1.3543(8) 0.9560(5) 0.0315(19) Uiso 0.25 1 d P . .
S1 S 0.9977(6) 0.8390(5) 0.3190(3) 0.0530(10) Uiso 0.25 1 d P . .
O1SB O 0.9867(13) 0.7383(12) 0.3673(7) 0.039(3) Uiso 0.25 1 d P . .
H1SC H 1.0507 1.0654 0.3507 0.055(11) Uiso 1 1 d . . .
H1SA H 0.9482 0.9561 0.398 0.043(10) Uiso 1 1 d . . .
H3SA H 1.2749 0.8885 0.3229 0.053(11) Uiso 1 1 d . . .
H3SC H 1.2485 0.9925 0.2855 0.061(12) Uiso 1 1 d . . .
H1SB H 1.1447 1.0424 0.428 0.048(10) Uiso 1 1 d . . .
H3SB H 1.1246 0.8351 0.2418 0.049(11) Uiso 1 1 d . . .
I1 I 0.436836(19) 0.204681(16) 0.609414(10) 0.01875(5) Uani 1 1 d .
. .
I2 I 0.11980(2) 0.328793(16) 0.927257(10) 0.02096(5) Uani 1 1 d . .
.
Pd1 Pd 0.26577(2) 0.757821(17) 0.751272(11) 0.01237(5) Uani 1 1 d .
. .
C7 C 0.0619(3) 0.6666(2) 0.65579(15) 0.0145(5) Uani 1 1 d . . .
C12 C -0.0122(3) 0.6125(2) 0.85638(16) 0.0191(5) Uani 1 1 d . . .
H12A H -0.0454 0.5989 0.9063 0.023 Uiso 1 1 calc R . .
H12B H 0.0807 0.596 0.8638 0.023 Uiso 1 1 calc R . .
N2 N 0.4380(2) 0.7363(2) 0.62681(12) 0.0156(4) Uani 1 1 d . . .
N6 N 0.1583(2) 0.9523(2) 0.82049(13) 0.0165(4) Uani 1 1 d . . .
C8 C -0.1234(3) 0.6236(3) 0.53344(16) 0.0212(5) Uani 1 1 d . . .
H8 H -0.172 0.632 0.4808 0.025 Uiso 1 1 calc R . .
N1 N 0.4135(2) 0.5835(2) 0.70166(12) 0.0153(4) Uani 1 1 d . . .
N5 N 0.0356(2) 0.7537(2) 0.84665(12) 0.0153(4) Uani 1 1 d . . .
C4 C 0.4271(3) 0.8493(2) 0.58951(15) 0.0168(5) Uani 1 1 d . . .
H4A H 0.5116 0.8862 0.563 0.02 Uiso 1 1 calc R . .
H4B H 0.4473 0.9191 0.6334 0.02 Uiso 1 1 calc R . .
N3 N 0.0305(2) 0.7086(2) 0.58304(13) 0.0164(4) Uani 1 1 d . . .
C9 C -0.1882(3) 0.5273(3) 0.57623(16) 0.0206(5) Uani 1 1 d . . .
H9 H -0.291 0.456 0.559 0.025 Uiso 1 1 calc R . .
C10 C -0.0977(3) 0.4652(2) 0.71327(16) 0.0193(5) Uani 1 1 d . . .
H10A H 0.0011 0.4594 0.7363 0.023 Uiso 1 1 calc R . .
H10B H -0.1791 0.3756 0.6858 0.023 Uiso 1 1 calc R . .
N8 N 0.5379(2) 0.7905(2) 0.89955(12) 0.0156(4) Uani 1 1 d . . .
C6 C 0.1406(3) 0.8295(3) 0.55926(16) 0.0184(5) Uani 1 1 d . . .
H6A H 0.1946 0.901 0.6071 0.022 Uiso 1 1 calc R . .
H6B H 0.0779 0.8575 0.5174 0.022 Uiso 1 1 calc R . .
N4 N -0.0734(2) 0.5533(2) 0.65108(13) 0.0171(4) Uani 1 1 d . . .
C23 C 0.4928(3) 0.5541(3) 0.85006(16) 0.0188(5) Uani 1 1 d . . .
H23A H 0.5993 0.6029 0.844 0.023 Uiso 1 1 calc R . .
H23B H 0.4913 0.4793 0.8774 0.023 Uiso 1 1 calc R . .
C24 C 0.3740(3) 0.4966(3) 0.76383(16) 0.0189(5) Uani 1 1 d . . .
```

```
H24A H 0.3683 0.4106 0.745 0.023 Uiso 1 1 calc R . .
H24B H 0.2687 0.4801 0.7675 0.023 Uiso 1 1 calc R . .
C1 C 0.3780(3) 0.6866(2) 0.68893(15) 0.0151(5) Uani 1 1 d . . .
C15 C 0.0479(3) 0.9549(3) 0.85884(15) 0.0185(5) Uani 1 1 d . . .
H15 H 0.03 1.0284 0.871 0.022 Uiso 1 1 calc R . .
C2 C 0.4954(3) 0.5688(3) 0.64826(16) 0.0196(5) Uani 1 1 d . . .
H2 H 0.5325 0.5047 0.6454 0.024 Uiso 1 1 calc R . .
C13 C 0.1501(3) 0.8279(2) 0.81176(14) 0.0148(5) Uani 1 1 d . . .
C3 C 0.5115(3) 0.6651(3) 0.60104(16) 0.0186(5) Uani 1 1 d . . .
H3 H 0.5619 0.6806 0.5595 0.022 Uiso 1 1 calc R . .
C11 C -0.1474(3) 0.5088(3) 0.78432(17) 0.0215(6) Uani 1 1 d . . .
H11A H -0.2206 0.5455 0.7621 0.026 Uiso 1 1 calc R . .
H11B H -0.2062 0.4293 0.8058 0.026 Uiso 1 1 calc R . .
C5 C 0.2676(3) 0.8157(3) 0.52599(15) 0.0181(5) Uani 1 1 d . . .
H5A H 0.2889 0.8737 0.485 0.022 Uiso 1 1 calc R . .
H5B H 0.2232 0.7237 0.4978 0.022 Uiso 1 1 calc R . .
C22 C 0.4657(3) 0.6476(2) 0.90681(16) 0.0189(5) Uani 1 1 d . . .
H22A H 0.3512 0.6158 0.8959 0.023 Uiso 1 1 calc R . .
H22B H 0.5074 0.6401 0.9635 0.023 Uiso 1 1 calc R . .
C14 C -0.0287(3) 0.8304(3) 0.87520(15) 0.0187(5) Uani 1 1 d . . .
H14 H -0.1096 0.8015 0.9009 0.022 Uiso 1 1 calc R . .

loop_
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    _atom_site_aniso_U_11
    _atom_site_aniso_U_22
    _atom_site_aniso_U_33
    _atom_site_aniso_U_23
    _atom_site_aniso_U_13
    _atom_site_aniso_U_12
N7 0.0169(10) 0.0135(10) 0.0161(10) 0.0006(8) 0.0055(8) 0.0036(8)
C17 0.0241(13) 0.0131(12) 0.0237(14) 0.0006(10) 0.0101(11)
0.0052(11)
C18 0.0241(13) 0.0144(12) 0.0205(13) 0.0040(10) 0.0096(11)
0.0055(11)
C19 0.0165(12) 0.0137(12) 0.0145(12) 0.0021(9) 0.0076(10) 0.0050(10)
C16 0.0257(14) 0.0141(12) 0.0248(14) 0.0070(10) 0.0119(11)
0.0081(11)
C21 0.0173(12) 0.0228(14) 0.0128(12) -0.0011(10) 0.0019(10)
0.0063(11)
C20 0.0166(12) 0.0217(13) 0.0173(13) -0.0018(10) 0.0028(10)
0.0038(11)
C3S 0.0242(14) 0.0353(17) 0.0265(16) 0.0024(13) 0.0055(12)
0.0120(13)
C1S 0.0343(16) 0.0295(16) 0.0303(16) -0.0011(13) 0.0012(13)
```
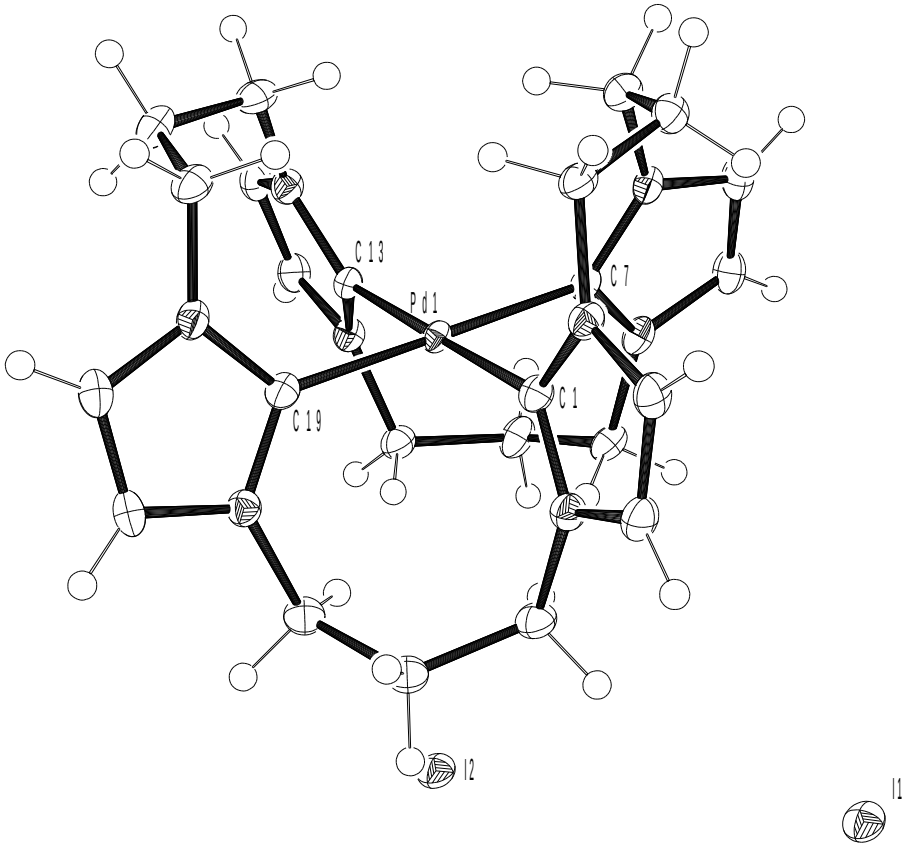

```
0.0215(14)
I1 0.02122(9) 0.01331(8) 0.02084(9) 0.00499(6) 0.00885(7) 0.00538(7)
I2 0.02695(9) 0.01557(9) 0.02092(9) 0.00640(7) 0.00958(7) 0.00841(7)
Pd1 0.01250(9) 0.01001(9) 0.01376(9) 0.00119(7) 0.00519(7)
0.00369(7)
C7 0.0146(11) 0.0123(11) 0.0163(12) 0.0000(9) 0.0052(9) 0.0058(9)
C12 0.0222(13) 0.0158(12) 0.0197(13) 0.0040(10) 0.0084(10)
0.0076(11)
N2 0.0167(10) 0.0142(10) 0.0165(10) 0.0014(8) 0.0061(8) 0.0070(8)
N6 0.0178(10) 0.0161(11) 0.0150(10) 0.0015(8) 0.0059(8) 0.0067(9)
C8 0.0179(12) 0.0214(14) 0.0183(13) -0.0030(10) 0.0002(10)
0.0069(11)
N1 0.0163(10) 0.0146(10) 0.0153(10) 0.0019(8) 0.0047(8) 0.0072(8)
N5 0.0155(10) 0.0159(10) 0.0152(10) 0.0019(8) 0.0068(8) 0.0067(8)
C4 0.0188(12) 0.0121(12) 0.0193(13) 0.0031(10) 0.0071(10) 0.0060(10)
N3 0.0165(10) 0.0134(10) 0.0162(10) -0.0010(8) 0.0040(8) 0.0048(8)
C9 0.0139(12) 0.0200(13) 0.0207(13) -0.0034(11) 0.0025(10)
0.0033(10)
C10 0.0198(12) 0.0121(12) 0.0246(14) 0.0025(10) 0.0083(11)
0.0051(10)
N8 0.0165(10) 0.0153(10) 0.0145(10) 0.0021(8) 0.0052(8) 0.0065(8)
C6 0.0195(12) 0.0166(12) 0.0201(13) 0.0031(10) 0.0068(10) 0.0084(10)
N4 0.0149(10) 0.0128(10) 0.0199(11) -0.0007(8) 0.0048(8) 0.0035(8)
C23 0.0192(12) 0.0182(13) 0.0208(13) 0.0064(10) 0.0078(10)
0.0087(10)
C24 0.0210(13) 0.0139(12) 0.0227(13) 0.0058(10) 0.0081(10)
0.0075(10)
C1 0.0129(11) 0.0122(11) 0.0170(12) 0.0009(9) 0.0042(9) 0.0032(9)
C15 0.0194(12) 0.0214(13) 0.0153(12) -0.0011(10) 0.0041(10)
0.0107(11)
C2 0.0211(13) 0.0193(13) 0.0199(13) -0.0003(10) 0.0066(10)
0.0106(11)
C13 0.0160(12) 0.0132(12) 0.0124(11) 0.0001(9) 0.0025(9) 0.0053(10)
C3 0.0208(13) 0.0218(13) 0.0166(12) 0.0010(10) 0.0076(10) 0.0118(11)
C11 0.0189(13) 0.0152(13) 0.0270(14) 0.0007(11) 0.0090(11)
0.0038(10)
C5 0.0209(13) 0.0172(12) 0.0176(13) 0.0028(10) 0.0079(10) 0.0088(10)
C22 0.0200(13) 0.0175(13) 0.0193(13) 0.0074(10) 0.0084(10)
0.0065(10)
C14 0.0175(12) 0.0217(13) 0.0166(12) -0.0005(10) 0.0064(10)
0.0081(11)


#------------------------------------------------------------------
---------#
```

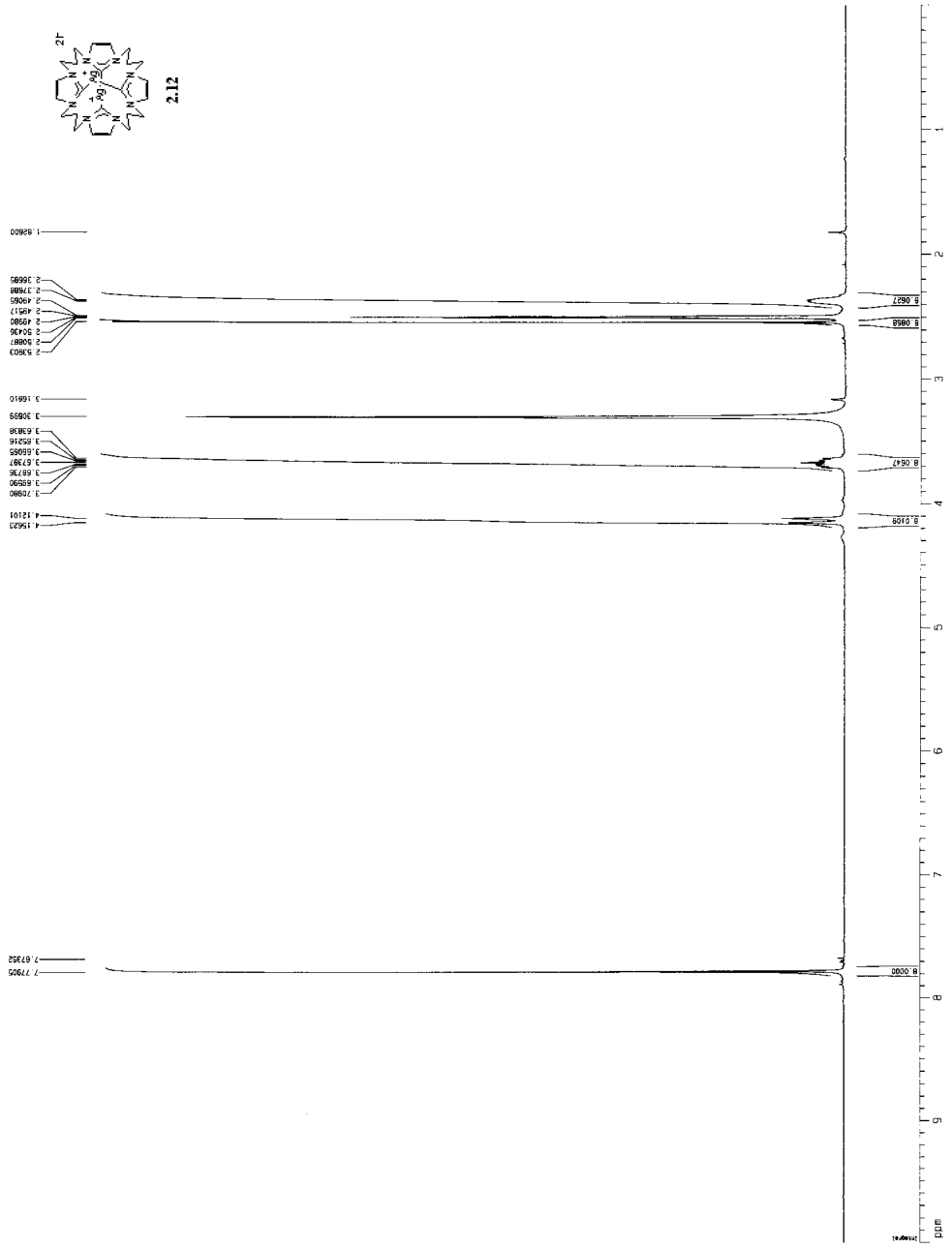
```
#                   MOLECULAR GEOMETRY
#
#------------------------------------------------------------------
---------#

_geom_special_details
;
 All esds (except the esd in the dihedral angle between two l.s.
planes)
 are estimated using the full covariance matrix.  The cell esds are
taken
 into account individually in the estimation of esds in distances,
angles
 and torsion angles; correlations between esds in cell parameters
are only
 used when they are defined by crystal symmetry.  An approximate
(isotropic)
 treatment of cell esds is used for estimating esds involving l.s.
planes.
;
loop_
    _geom_bond_atom_site_label_1
    _geom_bond_atom_site_label_2
    _geom_bond_distance
    _geom_bond_site_symmetry_2
    _geom_bond_publ_flag
N7 C19 1.353(3) . ?
N7 C20 1.391(3) . ?
N7 C18 1.463(3) . ?
C17 C18 1.528(4) . ?
C17 C16 1.533(4) . ?
C19 N8 1.358(3) . ?
C19 Pd1 2.020(2) . ?
C16 N6 1.464(3) . ?
C21 C20 1.344(4) . ?
C21 N8 1.387(3) . ?
O1SA S1 0.982(5) . ?
O1SA C2S 1.249(5) . ?
C3S C2S 1.488(5) . ?
C3S S1 1.787(6) . ?
C1S C2S 1.486(5) . ?
C1S S1 1.790(6) . ?
C2S S1 0.646(5) . ?
C2S O1SB 1.573(12) . ?
S1 O1SB 1.403(12) . ?
```

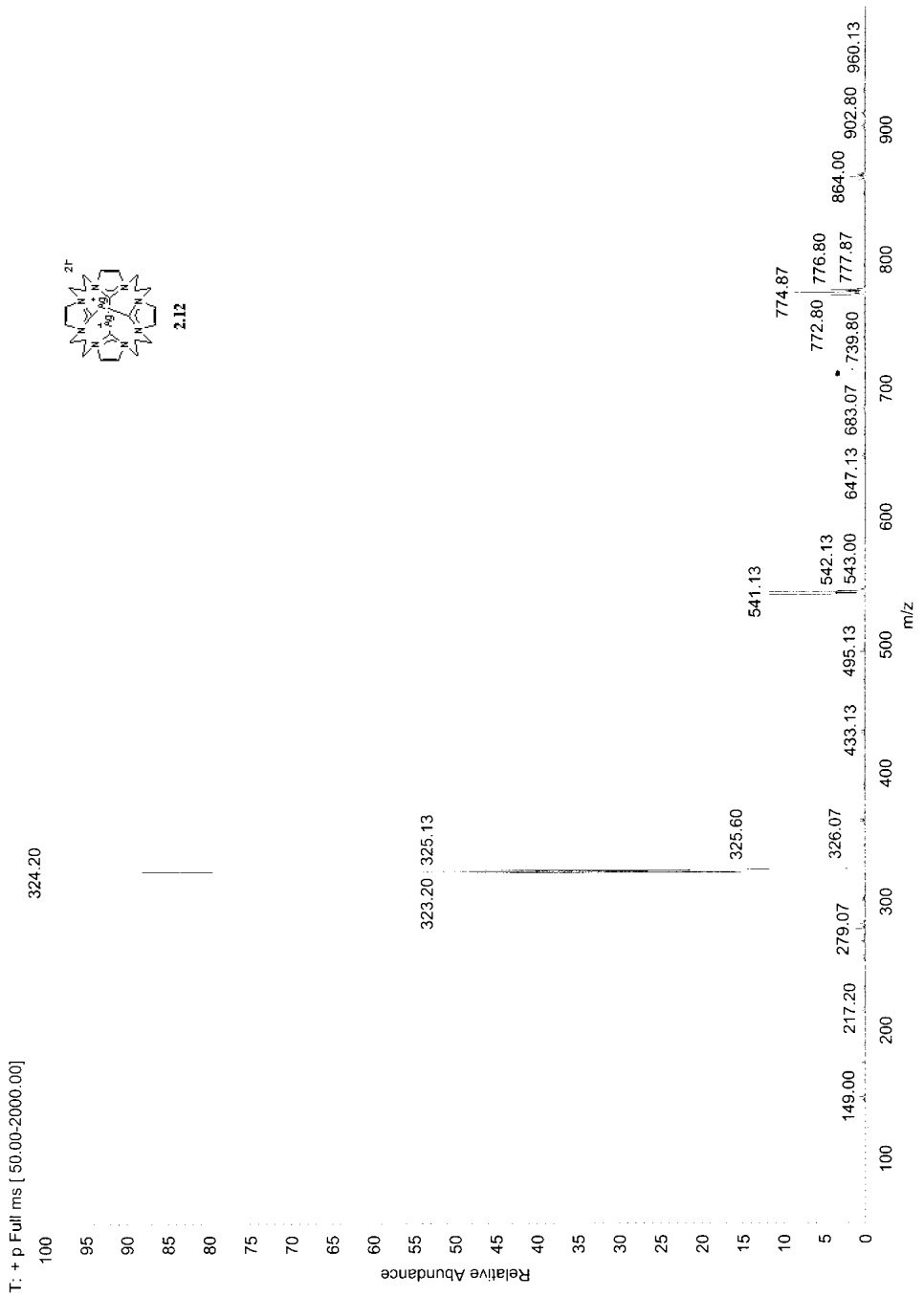
```
Pd1 C7 2.028(2) . ?
Pd1 C1 2.029(2) . ?
Pd1 C13 2.030(2) . ?
C7 N3 1.351(3) . ?
C7 N4 1.355(3) . ?
C12 N5 1.470(3) . ?
C12 C11 1.527(4) . ?
N2 C1 1.350(3) . ?
N2 C3 1.385(3) . ?
N2 C4 1.468(3) . ?
N6 C13 1.355(3) . ?
N6 C15 1.383(3) . ?
C8 C9 1.338(4) . ?
C8 N3 1.388(3) . ?
N1 C1 1.346(3) . ?
N1 C2 1.381(3) . ?
N1 C24 1.472(3) . ?
N5 C13 1.354(3) . ?
N5 C14 1.381(3) . ?
C4 C5 1.528(3) . ?
N3 C6 1.465(3) . ?
C9 N4 1.388(3) . ?
C10 N4 1.467(3) . ?
C10 C11 1.527(4) . ?
N8 C22 1.466(3) . ?
C6 C5 1.525(3) . ?
C23 C24 1.526(4) . ?
C23 C22 1.528(4) . ?
C15 C14 1.345(4) . ?
C2 C3 1.348(4) . ?

loop_
    _geom_angle_atom_site_label_1
    _geom_angle_atom_site_label_2
    _geom_angle_atom_site_label_3
    _geom_angle
    _geom_angle_site_symmetry_1
    _geom_angle_site_symmetry_3
    _geom_angle_publ_flag
C19 N7 C20 110.7(2) . . ?
C19 N7 C18 125.6(2) . . ?
C20 N7 C18 123.7(2) . . ?
C18 C17 C16 116.7(2) . . ?
N7 C18 C17 115.9(2) . . ?
N7 C19 N8 104.9(2) . . ?
```

```
N7 C19 Pd1 127.86(18) . . ?
N8 C19 Pd1 127.22(17) . . ?
N6 C16 C17 115.9(2) . . ?
C20 C21 N8 106.8(2) . . ?
C21 C20 N7 106.9(2) . . ?
S1 O1SA C2S 30.8(3) . . ?
C2S C3S S1 20.23(19) . . ?
C2S C1S S1 20.13(18) . . ?
S1 C2S O1SA 51.1(5) . . ?
S1 C2S C1S 107.6(6) . . ?
O1SA C2S C1S 119.6(3) . . ?
S1 C2S C3S 107.0(7) . . ?
O1SA C2S C3S 119.2(3) . . ?
C1S C2S C3S 121.2(3) . . ?
S1 C2S O1SB 63.0(7) . . ?
O1SA C2S O1SB 12.0(5) . . ?
C1S C2S O1SB 119.3(5) . . ?
C3S C2S O1SB 118.4(5) . . ?
C2S S1 O1SA 98.1(7) . . ?
C2S S1 O1SB 92.7(8) . . ?
O1SA S1 O1SB 5.4(6) . . ?
C2S S1 C3S 52.8(5) . . ?
O1SA S1 C3S 114.1(4) . . ?
O1SB S1 C3S 110.5(5) . . ?
C2S S1 C1S 52.3(5) . . ?
O1SA S1 C1S 114.1(4) . . ?
O1SB S1 C1S 110.9(5) . . ?
C3S S1 C1S 92.8(3) . . ?
S1 O1SB C2S 24.2(3) . . ?
C19 Pd1 C7 179.06(10) . . ?
C19 Pd1 C1 89.24(10) . . ?
C7 Pd1 C1 90.08(9) . . ?
C19 Pd1 C13 91.78(9) . . ?
C7 Pd1 C13 88.90(9) . . ?
C1 Pd1 C13 178.97(9) . . ?
N3 C7 N4 104.8(2) . . ?
N3 C7 Pd1 126.89(17) . . ?
N4 C7 Pd1 128.28(18) . . ?
N5 C12 C11 115.0(2) . . ?
C1 N2 C3 110.9(2) . . ?
C1 N2 C4 125.7(2) . . ?
C3 N2 C4 123.3(2) . . ?
C13 N6 C15 110.7(2) . . ?
C13 N6 C16 126.1(2) . . ?
C15 N6 C16 123.2(2) . . ?
```

```
C9 C8 N3 106.4(2) . . ?
C1 N1 C2 110.8(2) . . ?
C1 N1 C24 126.1(2) . . ?
C2 N1 C24 123.1(2) . . ?
C13 N5 C14 110.6(2) . . ?
C13 N5 C12 125.8(2) . . ?
C14 N5 C12 123.5(2) . . ?
N2 C4 C5 115.7(2) . . ?
C7 N3 C8 111.1(2) . . ?
C7 N3 C6 125.3(2) . . ?
C8 N3 C6 123.6(2) . . ?
C8 C9 N4 107.3(2) . . ?
N4 C10 C11 115.6(2) . . ?
C19 N8 C21 110.8(2) . . ?
C19 N8 C22 126.0(2) . . ?
C21 N8 C22 123.0(2) . . ?
N3 C6 C5 116.0(2) . . ?
C7 N4 C9 110.4(2) . . ?
C7 N4 C10 126.3(2) . . ?
C9 N4 C10 123.3(2) . . ?
C24 C23 C22 116.9(2) . . ?
N1 C24 C23 114.6(2) . . ?
N1 C1 N2 105.0(2) . . ?
N1 C1 Pd1 128.06(18) . . ?
N2 C1 Pd1 126.89(18) . . ?
C14 C15 N6 106.7(2) . . ?
C3 C2 N1 107.0(2) . . ?
N5 C13 N6 104.9(2) . . ?
N5 C13 Pd1 125.97(17) . . ?
N6 C13 Pd1 128.92(18) . . ?
C2 C3 N2 106.2(2) . . ?
C12 C11 C10 116.4(2) . . ?
C6 C5 C4 116.4(2) . . ?
N8 C22 C23 116.3(2) . . ?
C15 C14 N5 107.1(2) . . ?
```


C13
C7
Pd1
C1
C19
I2
I1


2F
2Ag
2.12
ppm


2.12
T: + p Full ms [50.00-2000.00]
Relative Abundance
m/z
324.20
323.20
325.13
325.60
326.07
279.07
217.20
149.00
433.13
495.13
541.13
542.13
543.00
647.13
683.07
739.80
772.80
774.87
776.80
777.87
864.00
902.80
960.13

Crystal Structure Data for **2.12**

```
CIF_1.1

CIF produced by WinGX routine CIF_UPDATE
Created on 2006-08-04 at 15:30:06
Using CIFtbx version 2.6.2 16 Jun 1998

Dictionary name : cif_core.dic
Dictionary vers : 2.3
Request file    : h:\wingx\files\archive.dat
CIF files read  : stuartp3 struct


data_stuartp3

_audit_creation_date                    2006-08-04T15:30:06-00:00
_audit_creation_method                  'WinGX routine CIF_UPDATE'
_audit_conform_dict_name                cif_core.dic
_audit_conform_dict_version             2.3
_audit_conform_dict_location
ftp://ftp.iucr.org/pub/cif_core.dic
_publ_requested_category                FM

#------------------------------------------------------------------
---------#
#                   CHEMICAL INFORMATION
#
#------------------------------------------------------------------
---------#

_chemical_name_systematic
;
 ?
;
_chemical_formula_moiety                'C28 H44 Ag4 I4 N8 O2 S2'
_chemical_formula_sum                   'C28 H44 Ag4 I4 N8 O2 S2'
_chemical_formula_weight                1527.91
_chemical_compound_source               'synthesis as described'

#------------------------------------------------------------------
---------#
#                   UNIT CELL INFORMATION
#
```

```
#------------------------------------------------------------------
---------#

_symmetry_cell_setting                  triclinic
_symmetry_space_group_name_H-M          'P -1'
_symmetry_space_group_name_Hall         '-P 1'
_symmetry_Int_Tables_number             2
loop_
    _symmetry_equiv_pos_as_xyz
'x, y, z'
'-x, -y, -z'

_cell_length_a                          12.3163(2)
_cell_length_b                          12.8750(3)
_cell_length_c                          15.2227(3)
_cell_angle_alpha                       114.8260(10)
_cell_angle_beta                        96.1580(10)
_cell_angle_gamma                       97.8440(10)
_cell_volume                            2133.66(7)
_cell_formula_units_Z                   2
_cell_measurement_temperature           120(2)
_cell_measurement_reflns_used           9621
_cell_measurement_theta_min             2.91
_cell_measurement_theta_max             27.48
_cell_measurement_wavelength            0.71073

#------------------------------------------------------------------
---------#
#                   CRYSTAL INFORMATION
#
#------------------------------------------------------------------
---------#

_exptl_crystal_description              slab
_exptl_crystal_colour                   colourless
_exptl_crystal_size_max                 0.24
_exptl_crystal_size_mid                 0.14
_exptl_crystal_size_min                 0.06
_exptl_crystal_density_diffrn           2.378
_exptl_crystal_density_method           'not measured'
_exptl_crystal_F_000                    1432
_exptl_special_details
;
 ?
;
```

```
#------------------------------------------------------------------
---------#
#                   ABSORPTION CORRECTION
#
#------------------------------------------------------------------
---------#

_exptl_absorpt_coefficient_mu           4.836
_exptl_absorpt_correction_type          none
_exptl_absorpt_process_details          'SADABS V2.10 (Sheldrick,
G.M., 2003)'

#------------------------------------------------------------------
---------#
#                   DATA COLLECTION
#
#------------------------------------------------------------------
---------#

_diffrn_ambient_temperature             120(2)
_diffrn_radiation_wavelength            0.71073
_diffrn_radiation_type                  MoK\a
_diffrn_radiation_source                'Bruker-Nonius FR591
rotating anode'
_diffrn_radiation_monochromator         '10cm confocal mirrors'
_diffrn_measurement_device_type         'Bruker-Nonius 95mm CCD
camera on \k-goniostat'
_diffrn_measurement_method              '\f & \w scans'
_diffrn_detector_area_resol_mean        9.091
_diffrn_reflns_av_R_equivalents         0.0325
_diffrn_reflns_av_unetI/netI            0.0274
_diffrn_reflns_number                   44761
_diffrn_reflns_limit_h_min              -15
_diffrn_reflns_limit_h_max              16
_diffrn_reflns_limit_k_min              -16
_diffrn_reflns_limit_k_max              16
_diffrn_reflns_limit_l_min              -19
_diffrn_reflns_limit_l_max              19
_diffrn_reflns_theta_min                2.92
_diffrn_reflns_theta_max                27.6
_diffrn_reflns_theta_full               27.6
_diffrn_measured_fraction_theta_full
                                        0.99
_diffrn_measured_fraction_theta_max
```

```
                                        0.99
_reflns_number_total                    9793
_reflns_number_gt                       9179
_reflns_threshold_expression            >2sigma(I)

#------------------------------------------------------------------
---------#
#                   COMPUTER PROGRAMS USED
#
#------------------------------------------------------------------
---------#

_computing_structure_solution           'SHELXS-86 (Sheldrick,
1986)'
_computing_structure_refinement         'SHELXL-97 (Sheldrick,
1997)'
_computing_molecular_graphics           'Ortep-3 for Windows
(Farrugia, 1997)'
_computing_publication_material
                                    'WinGX publication routines
(Farrugia, 1999)'

#------------------------------------------------------------------
---------#
#                   REFINEMENT INFORMATION
#
#------------------------------------------------------------------
---------#

_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor
wR and
 goodness of fit S are based on F^2^, conventional R-factors R are
based
 on F, with F set to zero for negative F^2^. The threshold
expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc.
and is
 not relevant to the choice of reflections for refinement.  R-
factors based
 on F^2^ are statistically about twice as large as those based on F,
and R-
 factors based on ALL data will be even larger.
;
```

```
_refine_ls_structure_factor_coef        Fsqd
_refine_ls_matrix_type                  full
_refine_ls_weighting_scheme             calc
_refine_ls_weighting_details
        'calc w=1/[\s^2^(Fo^2^)+(0.0231P)^2^+29.4817P] where
P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary            direct
_atom_sites_solution_secondary          difmap
_atom_sites_solution_hydrogens          geom
_refine_ls_hydrogen_treatment           mixed
_refine_ls_extinction_method            none
_refine_ls_number_reflns                9793
_refine_ls_number_parameters            434
_refine_ls_number_restraints            0
_refine_ls_R_factor_all                 0.0443
_refine_ls_R_factor_gt                  0.0413
_refine_ls_wR_factor_ref                0.0931
_refine_ls_wR_factor_gt                 0.0913
_refine_ls_goodness_of_fit_ref          1.032
_refine_ls_restrained_S_all             1.032
_refine_ls_shift/su_max                 0.026
_refine_ls_shift/su_mean                0
_refine_diff_density_max                7.551
_refine_diff_density_min                -5.304
_refine_diff_density_rms                0.188

#---------------------------------------------------------------------
---------#
#                   ATOMIC TYPES, COORDINATES AND THERMAL PARAMETERS
#
#---------------------------------------------------------------------
---------#

loop_
    _atom_type_symbol
    _atom_type_description
    _atom_type_scat_dispersion_real
    _atom_type_scat_dispersion_imag
    _atom_type_scat_source
C C 0.0033 0.0016 'International Tables Vol C Tables 4.2.6.8 and
6.1.1.4'
H H 0 0 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
Ag Ag -0.8971 1.1015 'International Tables Vol C Tables 4.2.6.8 and
6.1.1.4'
I I -0.4742 1.8119 'International Tables Vol C Tables 4.2.6.8 and
```

```
6.1.1.4'
N N 0.0061 0.0033 'International Tables Vol C Tables 4.2.6.8 and
6.1.1.4'
S S 0.1246 0.1234 'International Tables Vol C Tables 4.2.6.8 and
6.1.1.4'
O O 0.0106 0.006 'International Tables Vol C Tables 4.2.6.8 and
6.1.1.4'

loop_
    _atom_site_label
    _atom_site_type_symbol
    _atom_site_fract_x
    _atom_site_fract_y
    _atom_site_fract_z
    _atom_site_U_iso_or_equiv
    _atom_site_adp_type
    _atom_site_occupancy
    _atom_site_symmetry_multiplicity
    _atom_site_calc_flag
    _atom_site_refinement_flags
    _atom_site_disorder_assembly
    _atom_site_disorder_group
C1 C 0.5993(4) -0.0754(5) 0.2929(4) 0.0174(10) Uani 1 1 d . . .
C2 C 0.5656(5) -0.1057(5) 0.1345(4) 0.0223(12) Uani 1 1 d . . .
H2 H 0.5527 -0.0907 0.0787 0.027 Uiso 1 1 calc R . .
C3 C 0.5677(5) -0.2103(6) 0.1338(4) 0.0238(12) Uani 1 1 d . . .
H3 H 0.5565 -0.2835 0.0776 0.029 Uiso 1 1 calc R . .
C4 C 0.6084(5) -0.2823(5) 0.2604(4) 0.0216(11) Uani 1 1 d . . .
H4A H 0.5407 -0.345 0.2345 0.026 Uiso 1 1 calc R . .
H4B H 0.6226 -0.2498 0.3331 0.026 Uiso 1 1 calc R . .
C5 C 0.7073(5) -0.3335(5) 0.2210(4) 0.0247(12) Uani 1 1 d . . .
H5A H 0.7088 -0.4047 0.2307 0.03 Uiso 1 1 calc R . .
H5B H 0.6963 -0.3572 0.1493 0.03 Uiso 1 1 calc R . .
C6 C 0.8196(5) -0.2503(6) 0.2694(4) 0.0240(12) Uani 1 1 d . . .
H6A H 0.8781 -0.2849 0.2337 0.029 Uiso 1 1 calc R . .
H6B H 0.8169 -0.1758 0.2655 0.029 Uiso 1 1 calc R . .
C7 C 0.8578(4) -0.1224(5) 0.4517(4) 0.0168(10) Uani 1 1 d . . .
C8 C 0.8586(5) -0.3148(5) 0.4014(5) 0.0239(12) Uani 1 1 d . . .
H8 H 0.8556 -0.3948 0.3595 0.029 Uiso 1 1 calc R . .
C9 C 0.8739(5) -0.2641(5) 0.5001(4) 0.0210(11) Uani 1 1 d . . .
H9 H 0.8836 -0.3016 0.5416 0.025 Uiso 1 1 calc R . .
C10 C 0.8796(5) -0.0646(6) 0.6327(4) 0.0232(12) Uani 1 1 d . . .
H10A H 0.8803 0.0147 0.6379 0.028 Uiso 1 1 calc R . .
H10B H 0.95 -0.0623 0.6723 0.028 Uiso 1 1 calc R . .
C11 C 0.7811(5) -0.1003(6) 0.6737(5) 0.0266(13) Uani 1 1 d . . .
```

```
H11A H 0.776 -0.1831 0.6606 0.032 Uiso 1 1 calc R . .
H11B H 0.7958 -0.0528 0.746 0.032 Uiso 1 1 calc R . .
C12 C 0.6700(5) -0.0866(5) 0.6316(4) 0.0208(11) Uani 1 1 d . . .
H12A H 0.6575 -0.1281 0.5588 0.025 Uiso 1 1 calc R . .
H12B H 0.6096 -0.1226 0.6541 0.025 Uiso 1 1 calc R . .
C13 C 0.6508(4) 0.0878(5) 0.6014(4) 0.0160(10) Uani 1 1 d . . .
C14 C 0.6842(5) 0.1182(5) 0.7602(4) 0.0210(11) Uani 1 1 d . . .
H14 H 0.6962 0.1034 0.8162 0.025 Uiso 1 1 calc R . .
C15 C 0.6816(5) 0.2224(5) 0.7601(4) 0.0222(12) Uani 1 1 d . . .
H15 H 0.6915 0.2956 0.8162 0.027 Uiso 1 1 calc R . .
C16 C 0.6610(5) 0.2956(5) 0.6306(4) 0.0227(12) Uani 1 1 d . . .
H16A H 0.5994 0.3362 0.6533 0.027 Uiso 1 1 calc R . .
H16B H 0.6481 0.2611 0.5578 0.027 Uiso 1 1 calc R . .
C17 C 0.7712(5) 0.3832(5) 0.6725(5) 0.0259(13) Uani 1 1 d . . .
H17A H 0.7851 0.4138 0.7451 0.031 Uiso 1 1 calc R . .
H17B H 0.7646 0.4497 0.6571 0.031 Uiso 1 1 calc R . .
C18 C 0.8718(5) 0.3342(5) 0.6341(4) 0.0237(12) Uani 1 1 d . . .
H18A H 0.9412 0.3909 0.6744 0.028 Uiso 1 1 calc R . .
H18B H 0.8732 0.2609 0.6406 0.028 Uiso 1 1 calc R . .
C19 C 0.8501(4) 0.2042(5) 0.4537(4) 0.0170(10) Uani 1 1 d . . .
C20 C 0.8758(5) 0.3982(5) 0.5004(4) 0.0226(12) Uani 1 1 d . . .
H20 H 0.8896 0.4799 0.5413 0.027 Uiso 1 1 calc R . .
C21 C 0.8603(5) 0.3451(5) 0.4018(4) 0.0209(11) Uani 1 1 d . . .
H21 H 0.8605 0.382 0.3592 0.025 Uiso 1 1 calc R . .
C22 C 0.8132(5) 0.1380(5) 0.2704(4) 0.0225(12) Uani 1 1 d . . .
H22A H 0.8108 0.0591 0.267 0.027 Uiso 1 1 calc R . .
H22B H 0.8702 0.1514 0.2331 0.027 Uiso 1 1 calc R . .
C23 C 0.6997(5) 0.1442(6) 0.2243(4) 0.0249(12) Uani 1 1 d . . .
H23A H 0.6886 0.0968 0.1521 0.03 Uiso 1 1 calc R . .
H23B H 0.6999 0.2262 0.2369 0.03 Uiso 1 1 calc R . .
C24 C 0.6012(5) 0.1014(5) 0.2623(4) 0.0207(11) Uani 1 1 d . . .
H24A H 0.6145 0.1437 0.3351 0.025 Uiso 1 1 calc R . .
H24B H 0.5324 0.1189 0.2364 0.025 Uiso 1 1 calc R . .
C25 C 0.6708(11) 0.3118(10) 0.0564(7) 0.073(3) Uani 1 1 d . . .
C26 C 0.8942(12) 0.3977(13) 0.0648(9) 0.082(4) Uani 1 1 d . . .
C27 C 0.4152(10) 0.3895(10) 0.3319(9) 0.079(4) Uani 1 1 d . . .
H27A H 0.3635 0.3138 0.3033 0.119 Uiso 1 1 calc R . .
H27B H 0.4159 0.4209 0.2834 0.119 Uiso 1 1 calc R . .
H27C H 0.3911 0.4438 0.3904 0.119 Uiso 1 1 calc R . .
C28 C 0.6227(10) 0.5074(10) 0.4000(10) 0.083(4) Uani 1 1 d . . .
H28A H 0.5854 0.5662 0.4433 0.125 Uiso 1 1 calc R . .
H28B H 0.6312 0.5204 0.3419 0.125 Uiso 1 1 calc R . .
H28C H 0.6964 0.5136 0.4355 0.125 Uiso 1 1 calc R . .
Ag1 Ag 0.61759(3) 0.00471(3) 0.44696(3) 0.01541(9) Uani 1 1 d . . .
Ag2 Ag 0.85119(3) 0.04067(3) 0.45364(3) 0.01745(9) Uani 1 1 d . . .
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I2 I 0.32339(3) 0.06765(4) 0.08200(3) 0.02451(9) Uani 1 1 d . . .
I3 I 0.08896(3) 0.29910(3) 0.23508(3) 0.01988(8) Uani 1 1 d . . .
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N2 N 0.5895(4) -0.1900(4) 0.2316(3) 0.0195(9) Uani 1 1 d . . .
N3 N 0.8479(4) -0.2274(4) 0.3725(3) 0.0189(9) Uani 1 1 d . . .
N4 N 0.8729(4) -0.1468(4) 0.5299(3) 0.0171(9) Uani 1 1 d . . .
N5 N 0.6660(4) 0.0367(4) 0.6621(3) 0.0172(9) Uani 1 1 d . . .
N6 N 0.6616(4) 0.2022(4) 0.6622(3) 0.0169(9) Uani 1 1 d . . .
N7 N 0.8677(4) 0.3106(4) 0.5310(3) 0.0187(9) Uani 1 1 d . . .
N8 N 0.8440(4) 0.2264(4) 0.3740(3) 0.0179(9) Uani 1 1 d . . .
S1 S 0.81075(19) 0.3343(2) 0.12821(17) 0.0345(5) Uiso 0.8 1 d P . .
S2 S 0.5451(3) 0.3722(3) 0.3641(3) 0.0876(10) Uani 1 1 d . . .
O1 O 0.8084(6) 0.4314(5) 0.2250(4) 0.0506(16) Uani 1 1 d . . .
O2 O 0.5495(5) 0.3469(5) 0.4462(4) 0.0466(13) Uani 1 1 d . . .
I4 I 0.34913(4) 0.45671(3) 0.08409(3) 0.03707(12) Uani 1 1 d . . .
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C3 0.027(3) 0.028(3) 0.011(3) 0.005(2) 0.002(2) 0.004(2)
C4 0.025(3) 0.020(3) 0.018(3) 0.008(2) 0.002(2) 0.000(2)
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C7 0.013(2) 0.018(3) 0.017(3) 0.005(2) 0.0039(19) 0.0011(19)
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C9 0.020(3) 0.018(3) 0.024(3) 0.008(2) 0.002(2) 0.004(2)
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C16 0.028(3) 0.018(3) 0.025(3) 0.009(2) 0.015(2) 0.008(2)
C17 0.038(3) 0.014(3) 0.023(3) 0.004(2) 0.020(3) 0.002(2)
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0.00149(14)
Ag2 0.01715(19) 0.01540(19) 0.0181(2) 0.00568(16) 0.00646(15)
0.00092(14)
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-0.00730(15)
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0.00815(16)
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S2 0.087(2) 0.078(2) 0.107(3) 0.053(2) 0.0125(19) 0.0068(17)
O1 0.080(4) 0.044(3) 0.022(2) 0.006(2) -0.002(3) 0.033(3)
O2 0.056(4) 0.041(3) 0.049(3) 0.026(3) 0.006(3) 0.013(3)
I4 0.0578(3) 0.01598(18) 0.0255(2) -0.00125(16) 0.0242(2) -0.00846
(18)


#------------------------------------------------------------------
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#                   MOLECULAR GEOMETRY
#
#------------------------------------------------------------------
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planes)
 are estimated using the full covariance matrix.  The cell esds are
taken
 into account individually in the estimation of esds in distances,
angles
 and torsion angles; correlations between esds in cell parameters
are only
 used when they are defined by crystal symmetry.  An approximate
(isotropic)
 treatment of cell esds is used for estimating esds involving l.s.
planes.
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C5 C6 1.523(9) . ?
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C7 N3 1.360(7) . ?
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C12 N5 1.465(7) . ?
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C13 N5 1.352(7) . ?
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C24 N1 1.464(7) . ?
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C25 S1 1.856(11) . ?
C26 S3 1.764(16) . ?
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Ag4 I1 3.0209(7) . ?
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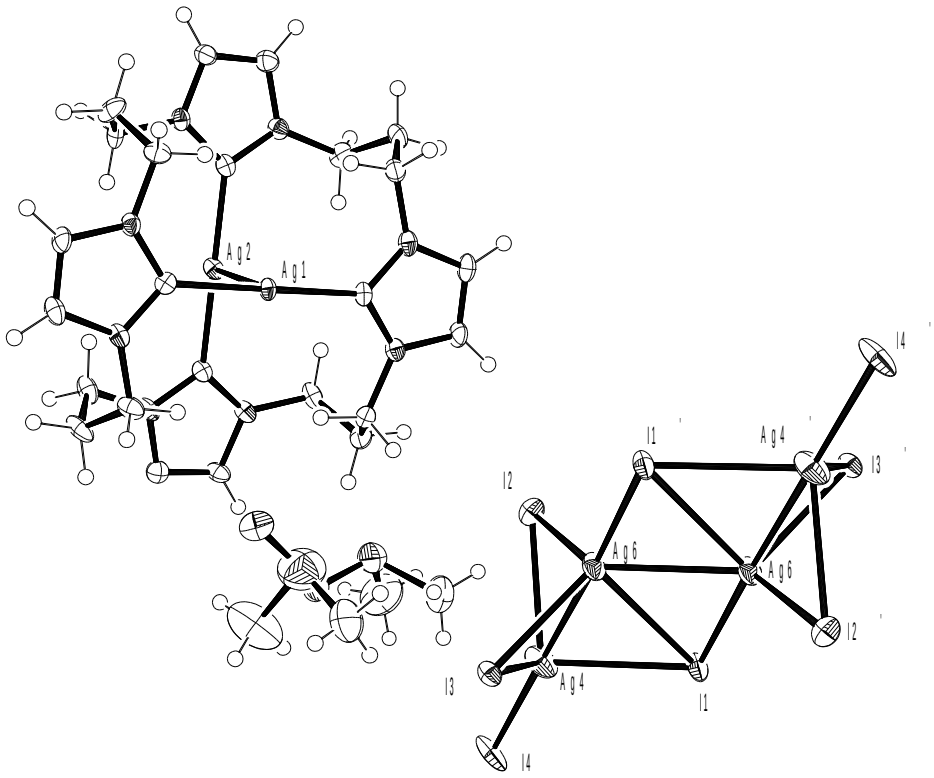
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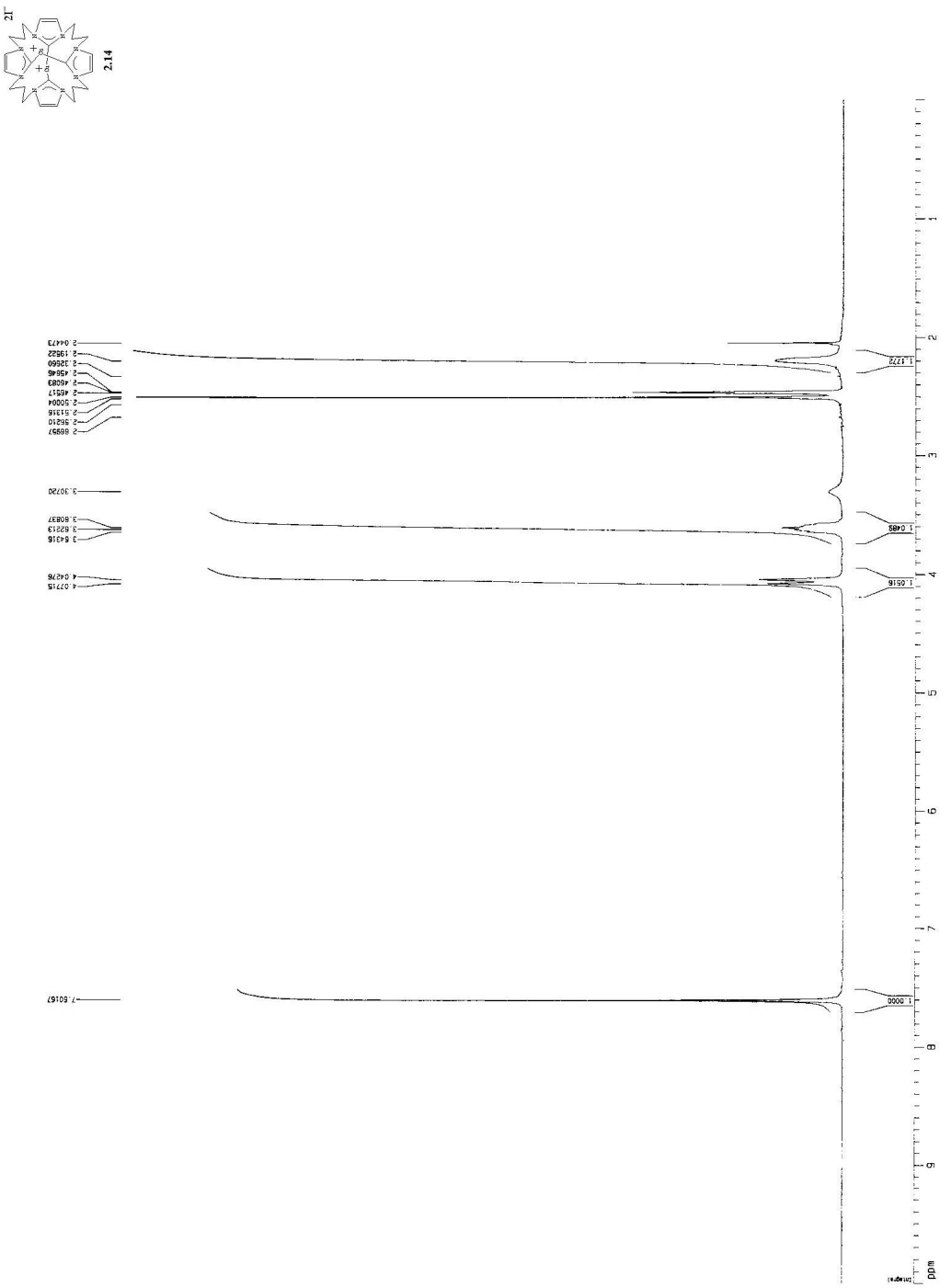
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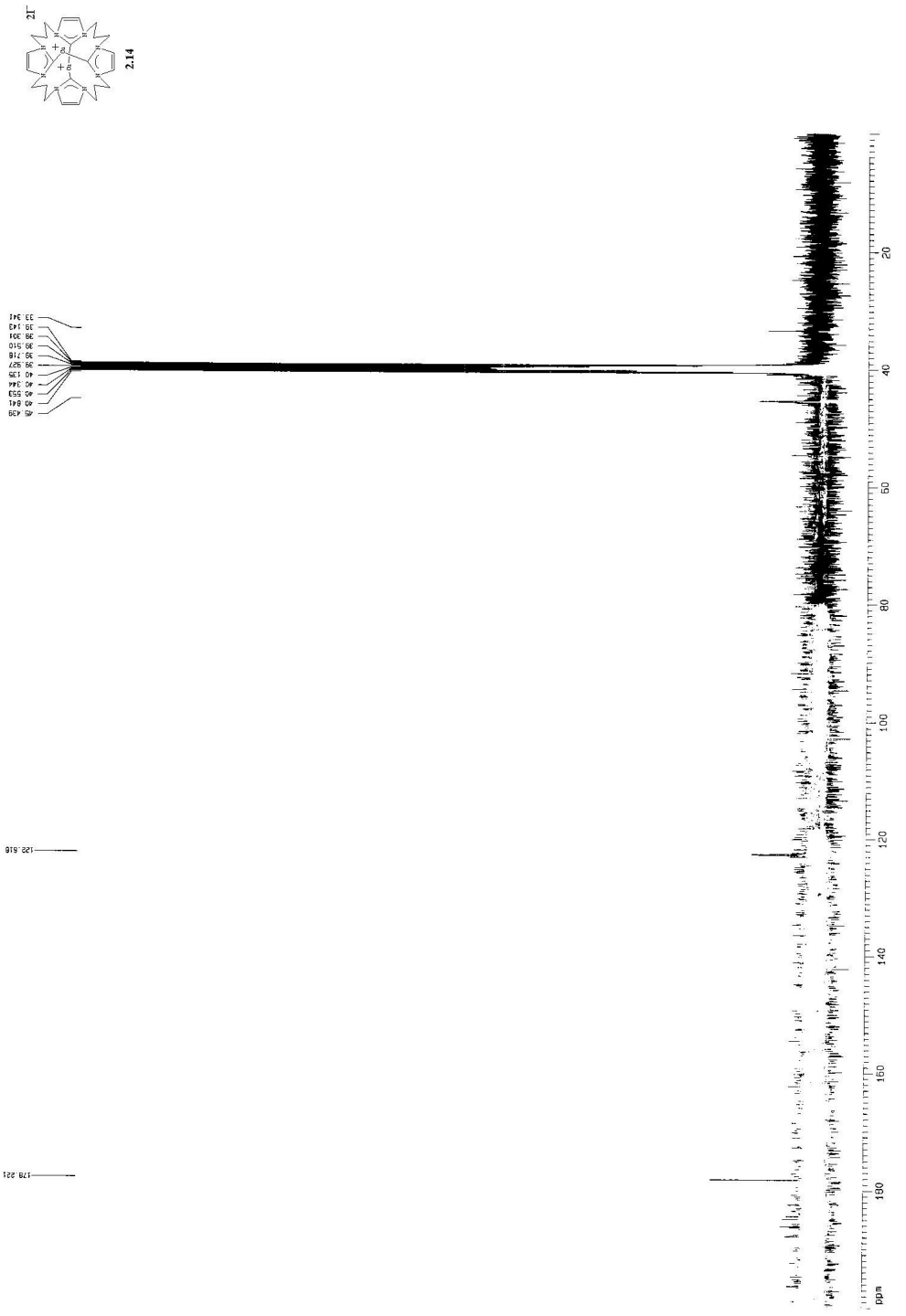
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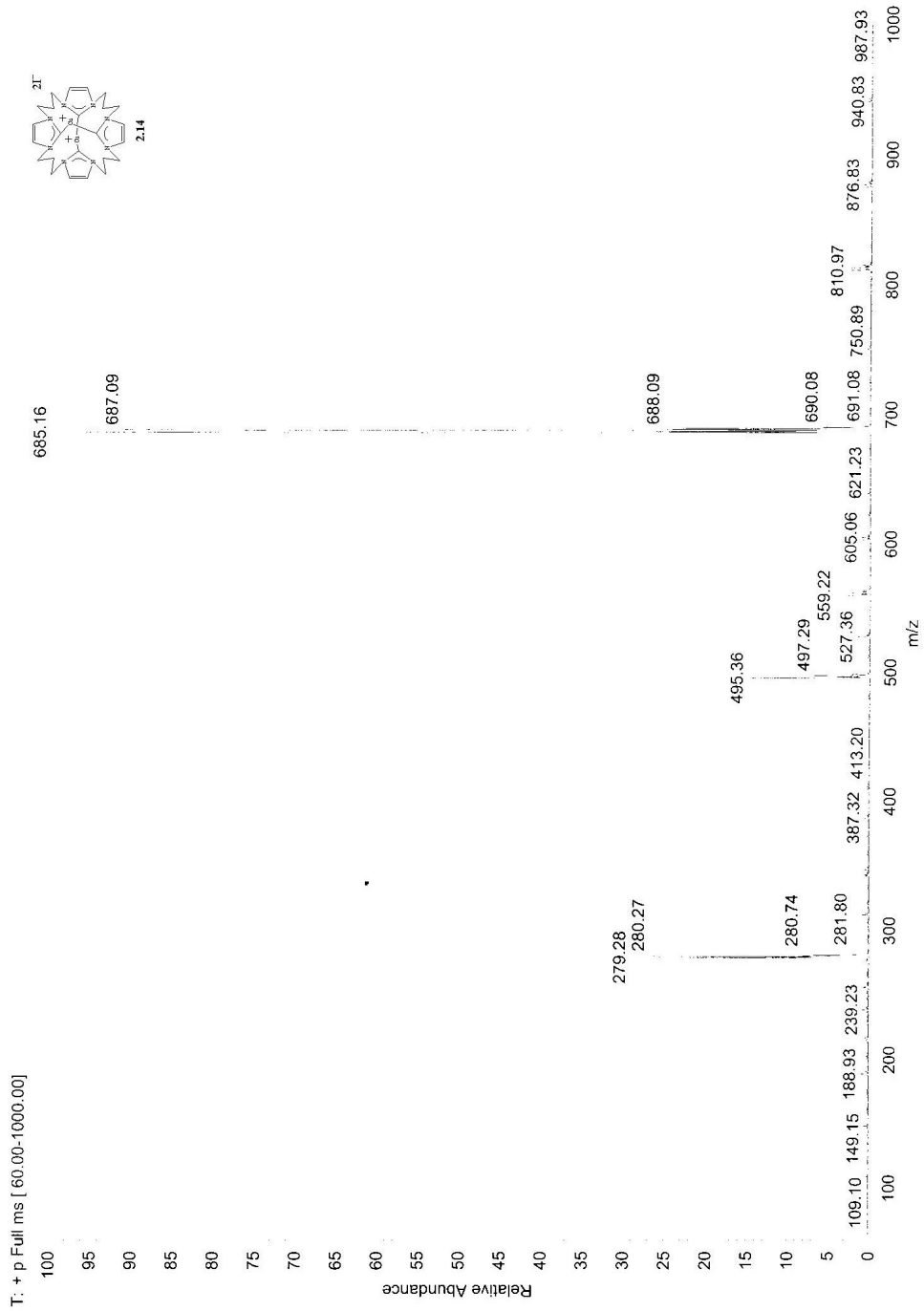
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Crystal Structure Data for **2.14**

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Created on 2006-11-16 at 12:07:02
Using CIFtbx version 2.6.2 16 Jun 1998

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Dictionary vers : 2.3
Request file : h:\wingx\files\archive.dat
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_cell_measurement_theta_max 0
_cell_measurement_wavelength 0.71073

#---#
CRYSTAL INFORMATION
#---#

```
_exptl_crystal_description          rhombus
_exptl_crystal_colour               'Light Green'
_exptl_crystal_size_max             0.12
_exptl_crystal_size_mid             0.1
_exptl_crystal_size_min             0.05
_exptl_crystal_density_diffrn       1.782
_exptl_crystal_density_method       'not measured'
_exptl_crystal_F_000                3672
_exptl_special_details
;
 ?
;

#---------------------------------------------------------------------------#
#                ABSORPTION CORRECTION                                      #
#---------------------------------------------------------------------------#

_exptl_absorpt_coefficient_mu       3.133
_exptl_absorpt_correction_type      none

#---------------------------------------------------------------------------#
#                DATA COLLECTION                                            #
#---------------------------------------------------------------------------#

_diffrn_ambient_temperature         123(2)
_diffrn_radiation_wavelength        0.71073
_diffrn_radiation_type              MoK\a
_diffrn_radiation_monochromator     graphite
_diffrn_radiation_probe             x-ray
_diffrn_reflns_av_R_equivalents     0.0520
_diffrn_reflns_av_unetI/netI        0.0454
_diffrn_reflns_number               39619
_diffrn_reflns_limit_h_min          -33
_diffrn_reflns_limit_h_max          33
_diffrn_reflns_limit_k_min          -16
```

```
_diffrn_reflns_limit_k_max            16
_diffrn_reflns_limit_l_min            -30
_diffrn_reflns_limit_l_max            30
_diffrn_reflns_theta_min              3
_diffrn_reflns_theta_max              27.58
_diffrn_reflns_theta_full             27.58
_diffrn_measured_fraction_theta_full
                                0.989
_diffrn_measured_fraction_theta_max
                                0.989
_reflns_number_total                  7943
_reflns_number_gt                     6549
_reflns_threshold_expression          >2sigma(I)

#---------------------------------------------------------------------------#
#                COMPUTER PROGRAMS USED                                     #
#---------------------------------------------------------------------------#

_computing_structure_solution         'SHELXS-86 (Sheldrick, 1986)'
_computing_structure_refinement       'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics         'Ortep-3 for Windows (Farrugia, 1997)'
_computing_publication_material
                        'WinGX publication routines (Farrugia, 1999)'

#---------------------------------------------------------------------------#
#                REFINEMENT INFORMATION                                     #
#---------------------------------------------------------------------------#

_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
```

```
 factors based on ALL data will be even larger.
;
_refine_ls_structure_factor_coef        Fsqd
_refine_ls_matrix_type                  full
_refine_ls_weighting_scheme             calc
_refine_ls_weighting_details
      'calc w=1/[\s^2^(Fo^2^)+(0.0212P)^2^+401.3428P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary            direct
_atom_sites_solution_secondary          difmap
_atom_sites_solution_hydrogens          geom
_refine_ls_hydrogen_treatment           mixed
_refine_ls_extinction_method            none
_refine_ls_number_reflns                7943
_refine_ls_number_parameters            369
_refine_ls_number_restraints            0
_refine_ls_R_factor_all                 0.107
_refine_ls_R_factor_gt                  0.0886
_refine_ls_wR_factor_ref                0.1895
_refine_ls_wR_factor_gt                 0.1817
_refine_ls_goodness_of_fit_ref          1.121
_refine_ls_restrained_S_all             1.121
_refine_ls_shift/su_max                 0.099
_refine_ls_shift/su_mean                0.019
_refine_diff_density_max                1.89
_refine_diff_density_min                -1.433
_refine_diff_density_rms                0.203

#---------------------------------------------------------------------------#
#               ATOMIC TYPES, COORDINATES AND THERMAL PARAMETERS        #
#---------------------------------------------------------------------------#

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   _atom_type_description
   _atom_type_scat_dispersion_real
   _atom_type_scat_dispersion_imag
```

```
   _atom_type_scat_source
C C 0.0033 0.0016 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
H H 0 0 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
Cu Cu 0.3201 1.2651 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
I I -0.4742 1.8119 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
N N 0.0061 0.0033 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
S S 0.1246 0.1234 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
O O 0.0106 0.006 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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   _atom_site_fract_y
   _atom_site_fract_z
   _atom_site_U_iso_or_equiv
   _atom_site_adp_type
   _atom_site_occupancy
   _atom_site_symmetry_multiplicity
   _atom_site_calc_flag
   _atom_site_refinement_flags
   _atom_site_disorder_assembly
   _atom_site_disorder_group
I1 I 0.12196(3) 0.62964(6) 0.05778(3) 0.03125(18) Uani 1 1 d . . .
I2 I 0.20543(3) 0.86747(6) 0.58896(3) 0.03465(19) Uani 1 1 d . . .
Cu2 Cu 0.08129(5) 0.13177(10) 0.26770(5) 0.0230(3) Uani 1 1 d . . .
Cu1 Cu 0.16831(5) 0.13311(10) 0.24119(6) 0.0254(3) Uani 1 1 d . . .
N2 N 0.1427(3) 0.0478(7) 0.3951(4) 0.0225(17) Uani 1 1 d . . .
N3 N 0.2033(3) -0.0831(7) 0.2894(4) 0.0236(17) Uani 1 1 d . . .
N1 N 0.1407(4) 0.2169(6) 0.3972(4) 0.0235(17) Uani 1 1 d . . .
N8 N 0.1994(3) 0.3509(6) 0.2915(4) 0.0232(17) Uani 1 1 d . . .
C2 C 0.1682(4) 0.1851(8) 0.4588(5) 0.025(2) Uani 1 1 d . . .
H2 H 0.183 0.2299 0.4946 0.03 Uiso 1 1 calc R . .
N6 N 0.0083(3) 0.2112(7) 0.1419(4) 0.0249(18) Uani 1 1 d . . .
C11 C 0.0545(4) -0.1137(9) 0.1192(5) 0.030(2) Uani 1 1 d . . .
H11A H 0.0287 -0.1058 0.0746 0.036 Uiso 1 1 calc R . .
```

H11B H 0.0609 -0.1904 0.1283 0.036 Uiso 1 1 calc R . .
N4 N 0.1529(3) -0.0874(6) 0.1920(4) 0.0204(16) Uani 1 1 d . . .
C5 C 0.2002(4) -0.1041(8) 0.3934(5) 0.029(2) Uani 1 1 d . . .
H5A H 0.1982 -0.1815 0.3857 0.035 Uiso 1 1 calc R . .
H5B H 0.2225 -0.0927 0.4388 0.035 Uiso 1 1 calc R . .
N7 N 0.1482(3) 0.3510(7) 0.1922(4) 0.0244(18) Uani 1 1 d . . .
C23 C 0.1959(4) 0.3745(8) 0.3940(5) 0.027(2) Uani 1 1 d . . .
H23A H 0.1931 0.4517 0.3854 0.032 Uiso 1 1 calc R . .
H23B H 0.2184 0.3646 0.4395 0.032 Uiso 1 1 calc R . .
C3 C 0.1699(4) 0.0795(8) 0.4586(4) 0.025(2) Uani 1 1 d . . .
H3 H 0.1863 0.0344 0.494 0.03 Uiso 1 1 calc R . .
C1 C 0.1241(3) 0.1311(7) 0.3569(4) 0.0157(16) Uani 1 1 d . . .
C21 C 0.1964(4) 0.4543(8) 0.2716(5) 0.025(2) Uani 1 1 d . . .
H21 H 0.2138 0.5138 0.297 0.03 Uiso 1 1 calc R . .
C17 C 0.0514(4) 0.3721(9) 0.1179(5) 0.032(2) Uani 1 1 d . . .
H17A H 0.0268 0.3624 0.0729 0.039 Uiso 1 1 calc R . .
H17B H 0.0559 0.4493 0.1262 0.039 Uiso 1 1 calc R . .
N5 N 0.0101(4) 0.0432(7) 0.1433(4) 0.0285(19) Uani 1 1 d . . .
C14 C -0.0266(4) 0.0724(10) 0.0833(5) 0.033(2) Uani 1 1 d . . .
H14 H -0.0478 0.0259 0.0501 0.039 Uiso 1 1 calc R . .
C10 C 0.1107(4) -0.0604(9) 0.1297(5) 0.029(2) Uani 1 1 d . . .
H10A H 0.1239 -0.0841 0.0971 0.035 Uiso 1 1 calc R . .
H10B H 0.1056 0.0175 0.1261 0.035 Uiso 1 1 calc R . .
C20 C 0.1643(4) 0.4559(8) 0.2101(5) 0.025(2) Uani 1 1 d . . .
H20 H 0.1544 0.5165 0.1835 0.03 Uiso 1 1 calc R . .
C22 C 0.2265(5) 0.3230(8) 0.3576(5) 0.028(2) Uani 1 1 d . . .
H22A H 0.2662 0.3469 0.3745 0.034 Uiso 1 1 calc R . .
H22B H 0.2261 0.245 0.3623 0.034 Uiso 1 1 calc R . .
C7 C 0.1734(4) -0.0189(8) 0.2403(5) 0.029(2) Uani 1 1 d . . .
C6 C 0.2312(4) -0.0524(9) 0.3562(5) 0.027(2) Uani 1 1 d . . .
H6A H 0.2305 0.0257 0.3602 0.033 Uiso 1 1 calc R . .
H6B H 0.271 -0.0759 0.3732 0.033 Uiso 1 1 calc R . .
C16 C 0.0218(5) 0.3238(8) 0.1580(5) 0.028(2) Uani 1 1 d . . .
H16A H 0.0467 0.3301 0.2031 0.033 Uiso 1 1 calc R . .
H16B H -0.0133 0.3636 0.1507 0.033 Uiso 1 1 calc R . .
C9 C 0.1715(5) -0.1898(8) 0.2089(6) 0.030(2) Uani 1 1 d . . .

H9 H 0.1632 -0.2501 0.1821 0.036 Uiso 1 1 calc R . .
C19 C 0.1703(4) 0.2849(8) 0.2424(5) 0.022(2) Uani 1 1 d . . .
C12 C 0.0267(4) -0.0669(8) 0.1604(5) 0.030(2) Uani 1 1 d . . .
H12A H -0.0069 -0.1094 0.1557 0.036 Uiso 1 1 calc R . .
H12B H 0.0533 -0.0703 0.205 0.036 Uiso 1 1 calc R . .
C15 C -0.0264(4) 0.1781(9) 0.0810(6) 0.033(3) Uani 1 1 d . . .
H15 H -0.0456 0.2217 0.0456 0.04 Uiso 1 1 calc R . .
C13 C 0.0313(4) 0.1257(9) 0.1791(5) 0.030(2) Uani 1 1 d . . .
C24 C 0.1361(4) 0.3296(8) 0.3776(5) 0.026(2) Uani 1 1 d . . .
H24A H 0.1168 0.3707 0.3997 0.031 Uiso 1 1 calc R . .
H24B H 0.1137 0.3354 0.3319 0.031 Uiso 1 1 calc R . .
C18 C 0.1081(5) 0.3250(9) 0.1300(5) 0.030(2) Uani 1 1 d . . .
H18A H 0.1046 0.247 0.1254 0.036 Uiso 1 1 calc R . .
H18B H 0.1218 0.3521 0.0985 0.036 Uiso 1 1 calc R . .
C8 C 0.2038(5) -0.1869(9) 0.2710(6) 0.035(3) Uani 1 1 d . . .
H8 H 0.223 -0.2448 0.2968 0.042 Uiso 1 1 calc R . .
C4 C 0.1414(4) -0.0627(8) 0.3773(5) 0.027(2) Uani 1 1 d . . .
H4A H 0.1193 -0.0701 0.3315 0.032 Uiso 1 1 calc R . .
H4B H 0.1227 -0.1053 0.3994 0.032 Uiso 1 1 calc R . .
S1 S 0.0799(2) -0.3723(4) 0.4058(2) 0.0264(10) Uiso 0.5 1 d P . .
S1A S 0.0835(3) -0.3066(5) 0.4638(3) 0.0423(14) Uiso 0.5 1 d P . .
C1S C 0.1496(5) -0.3715(10) 0.4625(6) 0.038(3) Uiso 1 1 d . . .
O1SB O 0.0692(7) -0.2677(13) 0.3725(8) 0.039(4) Uiso 0.5 1 d P . .
O1SA O 0.0648(8) -0.2337(15) 0.4068(9) 0.046(4) Uiso 0.5 1 d P . .
C2S C 0.0411(10) -0.416(2) 0.4393(11) 0.036(5) Uiso 0.5 1 d P . .
C2SA C 0.0466(11) -0.362(2) 0.4605(12) 0.041(5) Uiso 0.5 1 d P . .
S2 S 0.0200(4) 0.5742(8) 0.2513(5) 0.081(3) Uiso 0.5 1 d P . .
C4S C -0.0419(11) 0.639(2) 0.2649(13) 0.044(6) Uiso 0.5 1 d P . .
C3SA C 0.0651(14) 0.679(3) 0.2638(15) 0.056(7) Uiso 0.5 1 d P . .
O2SA O 0.0449(12) 0.494(2) 0.2952(13) 0.092(8) Uiso 0.5 1 d P . .

loop_
_atom_site_aniso_label
_atom_site_aniso_U_11
_atom_site_aniso_U_22
_atom_site_aniso_U_33

_atom_site_aniso_U_23
_atom_site_aniso_U_13
_atom_site_aniso_U_12
I1 0.0354(4) 0.0277(3) 0.0321(4) 0.0078(3) 0.0148(3) 0.0026(3)
I2 0.0455(4) 0.0244(3) 0.0332(4) 0.0040(3) 0.0147(3) -0.0006(3)
Cu2 0.0175(5) 0.0257(6) 0.0235(6) 0.0047(5) 0.0056(5) 0.0000(5)
Cu1 0.0204(6) 0.0226(6) 0.0315(6) 0.0035(5) 0.0085(5) -0.0007(5)
N2 0.016(4) 0.028(4) 0.025(4) 0.006(3) 0.011(4) 0.001(3)
N3 0.019(4) 0.030(4) 0.026(4) -0.001(3) 0.013(4) -0.002(3)
N1 0.025(5) 0.023(4) 0.021(4) 0.001(3) 0.008(4) 0.000(3)
N8 0.021(4) 0.022(4) 0.024(4) -0.001(3) 0.006(3) -0.006(3)
C2 0.020(5) 0.031(5) 0.027(5) 0.005(4) 0.012(4) 0.001(4)
N6 0.013(4) 0.037(5) 0.023(4) 0.006(4) 0.006(3) 0.001(3)
C11 0.027(5) 0.030(5) 0.033(5) -0.004(4) 0.013(5) -0.002(4)
N4 0.011(4) 0.027(4) 0.024(4) 0.006(3) 0.007(3) 0.000(3)
C5 0.030(6) 0.029(5) 0.029(5) 0.008(4) 0.012(5) 0.011(4)
N7 0.022(4) 0.026(4) 0.028(4) 0.008(3) 0.013(4) 0.001(3)
C23 0.021(5) 0.027(5) 0.023(5) -0.001(4) -0.001(4) -0.007(4)
C3 0.026(5) 0.037(6) 0.015(4) 0.005(4) 0.013(4) 0.005(4)
C1 0.015(4) 0.020(4) 0.014(4) -0.005(3) 0.008(3) -0.007(3)
C21 0.015(5) 0.024(5) 0.033(6) 0.003(4) 0.007(4) 0.001(4)
C17 0.026(5) 0.039(6) 0.028(5) 0.016(5) 0.007(4) 0.003(5)
N5 0.017(4) 0.039(5) 0.030(5) 0.001(4) 0.009(4) 0.001(4)
C14 0.020(5) 0.051(7) 0.023(5) -0.001(5) 0.005(4) -0.003(5)
C10 0.028(6) 0.034(6) 0.029(6) 0.002(4) 0.014(5) -0.002(4)
C20 0.025(5) 0.027(5) 0.025(5) 0.007(4) 0.013(4) 0.001(4)
C22 0.028(6) 0.027(5) 0.028(5) 0.005(4) 0.010(5) -0.006(4)
C7 0.024(5) 0.028(5) 0.042(6) 0.004(5) 0.021(5) -0.001(4)
C6 0.014(5) 0.041(6) 0.022(5) -0.001(4) 0.002(4) 0.004(4)
C16 0.027(5) 0.025(5) 0.032(6) 0.003(4) 0.012(5) 0.000(4)
C9 0.032(6) 0.020(5) 0.044(7) 0.005(4) 0.021(5) 0.006(4)
C19 0.020(5) 0.029(5) 0.020(5) 0.002(4) 0.010(4) 0.003(4)
C12 0.018(5) 0.031(5) 0.036(6) 0.001(5) 0.004(5) -0.002(4)
C15 0.012(5) 0.044(6) 0.038(6) 0.015(5) 0.004(5) 0.001(4)
C13 0.020(5) 0.030(5) 0.042(6) 0.010(5) 0.016(5) 0.000(4)
C24 0.017(5) 0.027(5) 0.034(6) -0.002(4) 0.010(4) -0.003(4)

C18 0.035(6) 0.031(5) 0.029(5) 0.001(4) 0.018(5) -0.005(4)
C8 0.044(7) 0.034(6) 0.038(6) 0.004(5) 0.029(6) 0.008(5)
C4 0.028(5) 0.023(5) 0.030(5) 0.009(4) 0.012(5) -0.002(4)

#---#
MOLECULAR GEOMETRY
#---#

_geom_special_details
;
All esds (except the esd in the dihedral angle between two l.s. planes)
are estimated using the full covariance matrix. The cell esds are taken
into account individually in the estimation of esds in distances, angles
and torsion angles; correlations between esds in cell parameters are only
used when they are defined by crystal symmetry. An approximate (isotropic)
treatment of cell esds is used for estimating esds involving l.s. planes.
;
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Cu2 C1 1.919(8) . ?
Cu2 C13 1.948(11) . ?
Cu2 Cu1 2.5531(16) . ?
Cu1 C19 1.915(10) . ?
Cu1 C7 1.923(11) . ?
N2 C1 1.336(12) . ?
N2 C3 1.414(13) . ?
N2 C4 1.451(13) . ?
N3 C7 1.361(14) . ?
N3 C8 1.380(14) . ?
N3 C6 1.477(13) . ?
N1 C2 1.379(13) . ?

N1 C1 1.382(11) . ?
N1 C24 1.484(13) . ?
N8 C19 1.370(13) . ?
N8 C21 1.375(12) . ?
N8 C22 1.454(13) . ?
C2 C3 1.333(14) . ?
N6 C13 1.362(13) . ?
N6 C15 1.403(14) . ?
N6 C16 1.476(13) . ?
C11 C12 1.521(15) . ?
C11 C10 1.530(14) . ?
N4 C7 1.344(14) . ?
N4 C9 1.380(12) . ?
N4 C10 1.465(13) . ?
C5 C4 1.506(14) . ?
C5 C6 1.534(14) . ?
N7 C19 1.360(12) . ?
N7 C20 1.400(13) . ?
N7 C18 1.442(13) . ?
C23 C22 1.511(15) . ?
C23 C24 1.547(13) . ?
C21 C20 1.337(15) . ?
C17 C18 1.500(15) . ?
C17 C16 1.542(14) . ?
N5 C13 1.307(14) . ?
N5 C14 1.387(14) . ?
N5 C12 1.462(14) . ?
C14 C15 1.334(16) . ?
C9 C8 1.349(17) . ?
S1 O1SB 1.498(17) . ?
S1 S1A 1.550(8) . ?
S1 C2S 1.59(2) . ?
S1 C1S 1.761(13) . ?
S1 O1SA 1.794(19) . ?
S1 C2SA 1.80(3) . ?
S1A C2SA 1.17(3) . ?

S1A O1SA 1.523(19) . ?
S1A C2S 1.72(3) . ?
S1A C1S 1.904(14) . ?
S1A O1SB 2.057(18) . ?
O1SB O1SA 0.95(2) . ?
C2S C2SA 0.82(3) . ?
S2 S2 1.011(19) 2 ?
S2 C4S 1.14(3) 2 ?
S2 O2SA 1.40(3) . ?
S2 C3SA 1.71(3) . ?
S2 O2SA 1.89(3) 2 ?
S2 C4S 1.93(3) . ?
C4S C3SA 0.86(4) 2 ?
C4S S2 1.14(3) 2 ?
C3SA C4S 0.86(4) 2 ?
O2SA S2 1.89(3) 2 ?

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_geom_angle_atom_site_label_3
_geom_angle
_geom_angle_site_symmetry_1
_geom_angle_site_symmetry_3
_geom_angle_publ_flag
C1 Cu2 C13 173.9(4) . . ?
C1 Cu2 Cu1 94.0(2) . . ?
C13 Cu2 Cu1 91.6(3) . . ?
C19 Cu1 C7 174.7(4) . . ?
C19 Cu1 Cu2 91.3(3) . . ?
C7 Cu1 Cu2 93.8(3) . . ?
C1 N2 C3 111.6(8) . . ?
C1 N2 C4 127.3(8) . . ?
C3 N2 C4 120.9(8) . . ?
C7 N3 C8 112.2(9) . . ?
C7 N3 C6 127.0(9) . . ?

C8 N3 C6 120.8(9) . . ?
C2 N1 C1 111.6(8) . . ?
C2 N1 C24 122.8(8) . . ?
C1 N1 C24 125.3(8) . . ?
C19 N8 C21 111.1(8) . . ?
C19 N8 C22 127.2(8) . . ?
C21 N8 C22 121.5(8) . . ?
C3 C2 N1 106.9(10) . . ?
C13 N6 C15 110.2(9) . . ?
C13 N6 C16 127.0(9) . . ?
C15 N6 C16 122.4(9) . . ?
C12 C11 C10 112.5(9) . . ?
C7 N4 C9 112.8(9) . . ?
C7 N4 C10 124.4(9) . . ?
C9 N4 C10 122.4(9) . . ?
C4 C5 C6 114.2(8) . . ?
C19 N7 C20 110.6(8) . . ?
C19 N7 C18 127.5(9) . . ?
C20 N7 C18 121.5(8) . . ?
C22 C23 C24 113.4(8) . . ?
C2 C3 N2 106.5(9) . . ?
N2 C1 N1 103.4(7) . . ?
N2 C1 Cu2 128.4(7) . . ?
N1 C1 Cu2 128.1(7) . . ?
C20 C21 N8 107.4(9) . . ?
C18 C17 C16 114.2(9) . . ?
C13 N5 C14 112.0(10) . . ?
C13 N5 C12 125.3(9) . . ?
C14 N5 C12 122.3(10) . . ?
C15 C14 N5 107.0(11) . . ?
N4 C10 C11 110.2(8) . . ?
C21 C20 N7 106.8(9) . . ?
N8 C22 C23 110.1(9) . . ?
N4 C7 N3 102.7(9) . . ?
N4 C7 Cu1 130.0(8) . . ?
N3 C7 Cu1 127.1(8) . . ?

N3 C6 C5 109.2(8) . . ?
N6 C16 C17 110.6(9) . . ?
C8 C9 N4 106.0(10) . . ?
N7 C19 N8 104.1(8) . . ?
N7 C19 Cu1 126.9(7) . . ?
N8 C19 Cu1 128.5(7) . . ?
N5 C12 C11 111.1(9) . . ?
C14 C15 N6 105.6(10) . . ?
N5 C13 N6 105.0(9) . . ?
N5 C13 Cu2 129.6(8) . . ?
N6 C13 Cu2 125.3(8) . . ?
N1 C24 C23 108.9(8) . . ?
N7 C18 C17 111.1(9) . . ?
C9 C8 N3 106.2(10) . . ?
N2 C4 C5 110.8(9) . . ?
O1SB S1 S1A 84.9(8) . . ?
O1SB S1 C2S 122.3(11) . . ?
S1A S1 C2S 66.2(10) . . ?
O1SB S1 C1S 107.8(8) . . ?
S1A S1 C1S 69.9(5) . . ?
C2S S1 C1S 107.1(10) . . ?
O1SB S1 O1SA 31.9(8) . . ?
S1A S1 O1SA 53.6(7) . . ?
C2S S1 O1SA 98.7(11) . . ?
C1S S1 O1SA 98.5(8) . . ?
O1SB S1 C2SA 105.0(11) . . ?
S1A S1 C2SA 39.9(9) . . ?
C2S S1 C2SA 27.0(10) . . ?
C1S S1 C2SA 96.2(9) . . ?
O1SA S1 C2SA 75.9(11) . . ?
C2SA S1A O1SA 109.7(15) . . ?
C2SA S1A S1 81.6(14) . . ?
O1SA S1A S1 71.4(8) . . ?
C2SA S1A C2S 24.6(14) . . ?
O1SA S1A C2S 104.9(11) . . ?
S1 S1A C2S 58.1(9) . . ?

C2SA S1A C1S 117.2(14) . . ?
O1SA S1A C1S 103.1(9) . . ?
S1 S1A C1S 60.3(5) . . ?
C2S S1A C1S 96.3(10) . . ?
C2SA S1A O1SB 105.5(14) . . ?
O1SA S1A O1SB 25.5(8) . . ?
S1 S1A O1SB 46.5(5) . . ?
C2S S1A O1SB 91.2(10) . . ?
C1S S1A O1SB 83.4(6) . . ?
S1 C1S S1A 49.8(4) . . ?
O1SA O1SB S1 91.5(17) . . ?
O1SA O1SB S1A 43.9(14) . . ?
S1 O1SB S1A 48.6(6) . . ?
O1SB O1SA S1A 110.6(19) . . ?
O1SB O1SA S1 56.6(14) . . ?
S1A O1SA S1 55.0(7) . . ?
C2SA C2S S1 91(3) . . ?
C2SA C2S S1A 36(2) . . ?
S1 C2S S1A 55.7(8) . . ?
C2S C2SA S1A 119(3) . . ?
C2S C2SA S1 62(2) . . ?
S1A C2SA S1 58.6(11) . . ?
S2 S2 C4S 127.6(16) 2 2 ?
S2 S2 O2SA 102.0(16) 2 . ?
C4S S2 O2SA 128(2) 2 . ?
S2 S2 C3SA 128.5(11) 2 . ?
C4S S2 C3SA 26.7(16) 2 . ?
O2SA S2 C3SA 110.3(17) . . ?
S2 S2 O2SA 46.4(11) 2 2 ?
C4S S2 O2SA 129.9(19) 2 2 ?
O2SA S2 O2SA 95(2) . 2 ?
C3SA S2 O2SA 154.6(16) . 2 ?
S2 S2 C4S 27.9(9) 2 . ?
C4S S2 C4S 107(2) 2 . ?
O2SA S2 C4S 111.1(15) . . ?
C3SA S2 C4S 101.4(13) . . ?

O2SA S2 C4S 73.4(12) 2 . ?

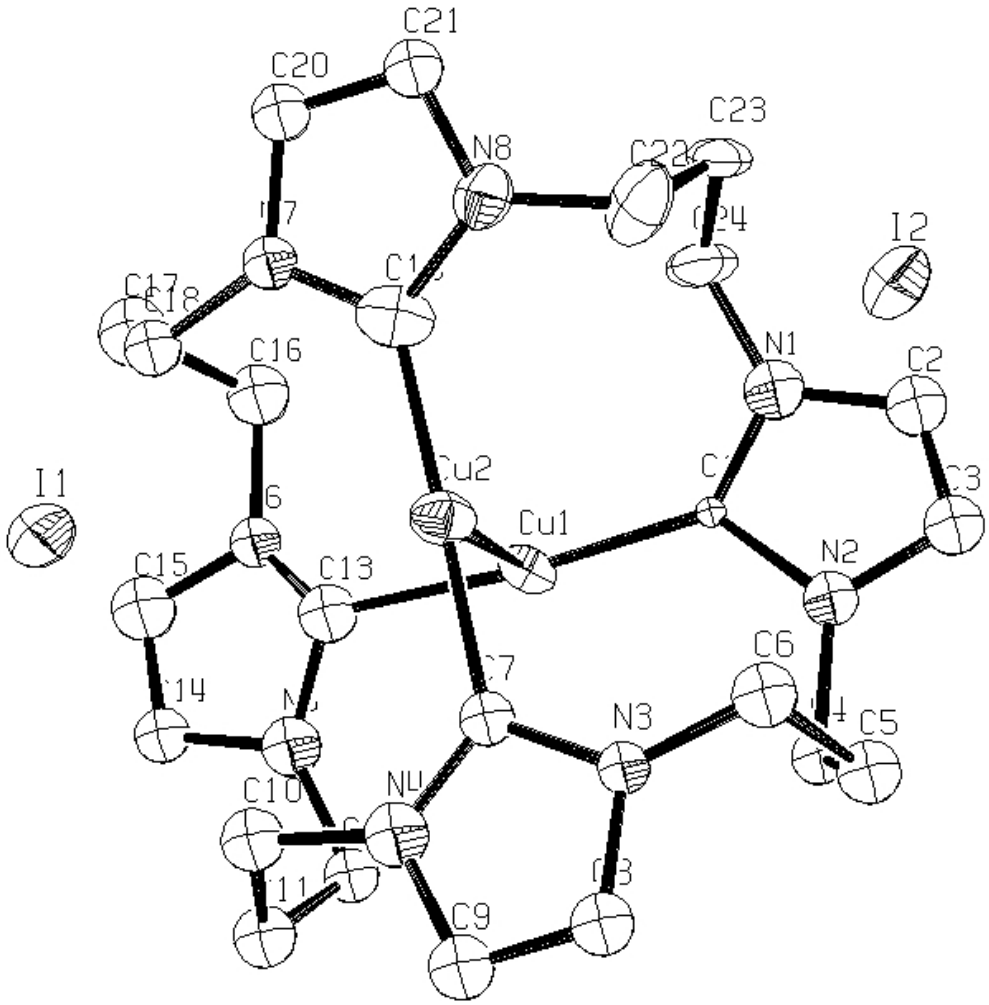
C3SA C4S S2 117(4) 2 2 ?

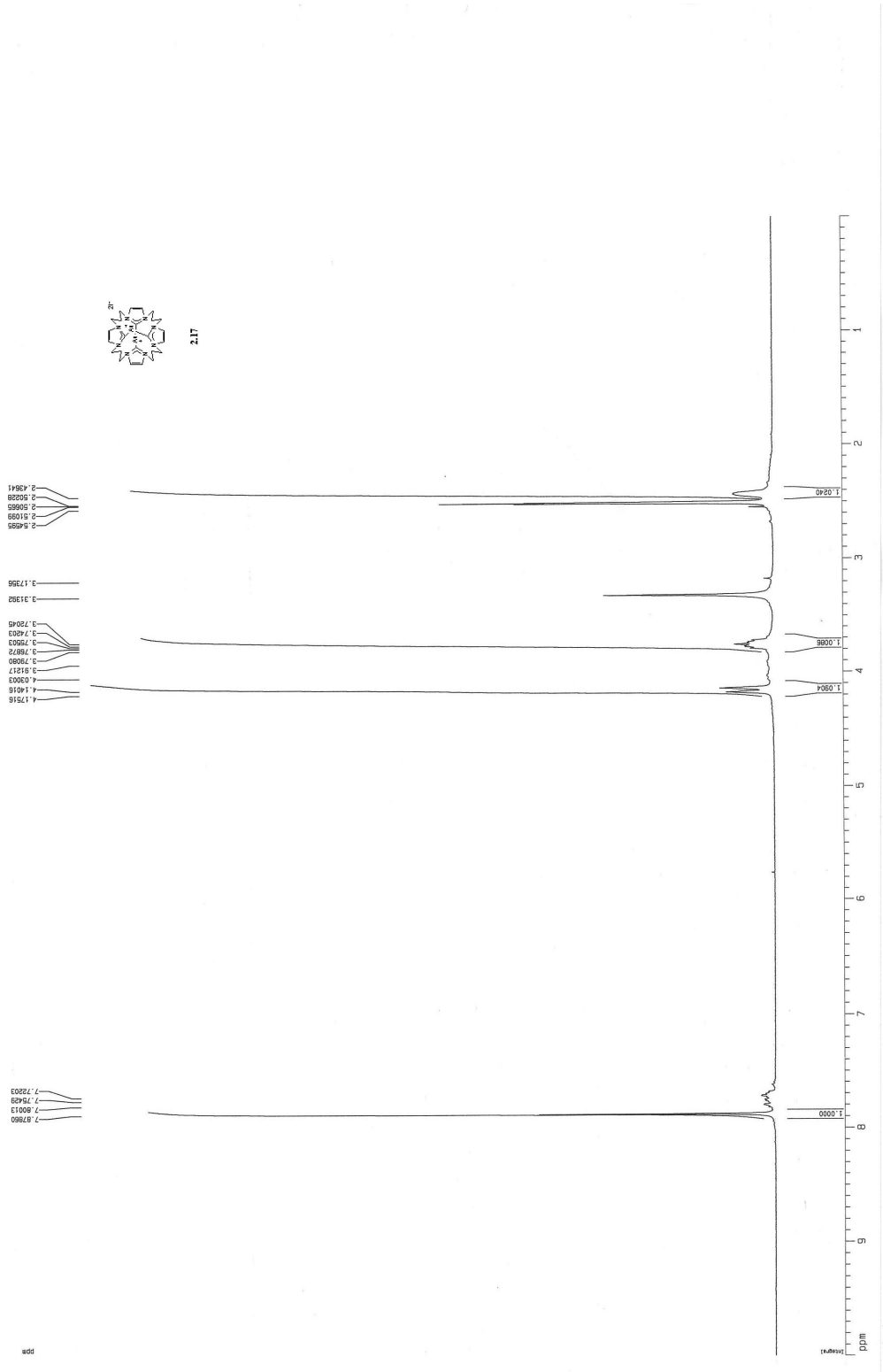
C3SA C4S S2 119(3) 2 . ?

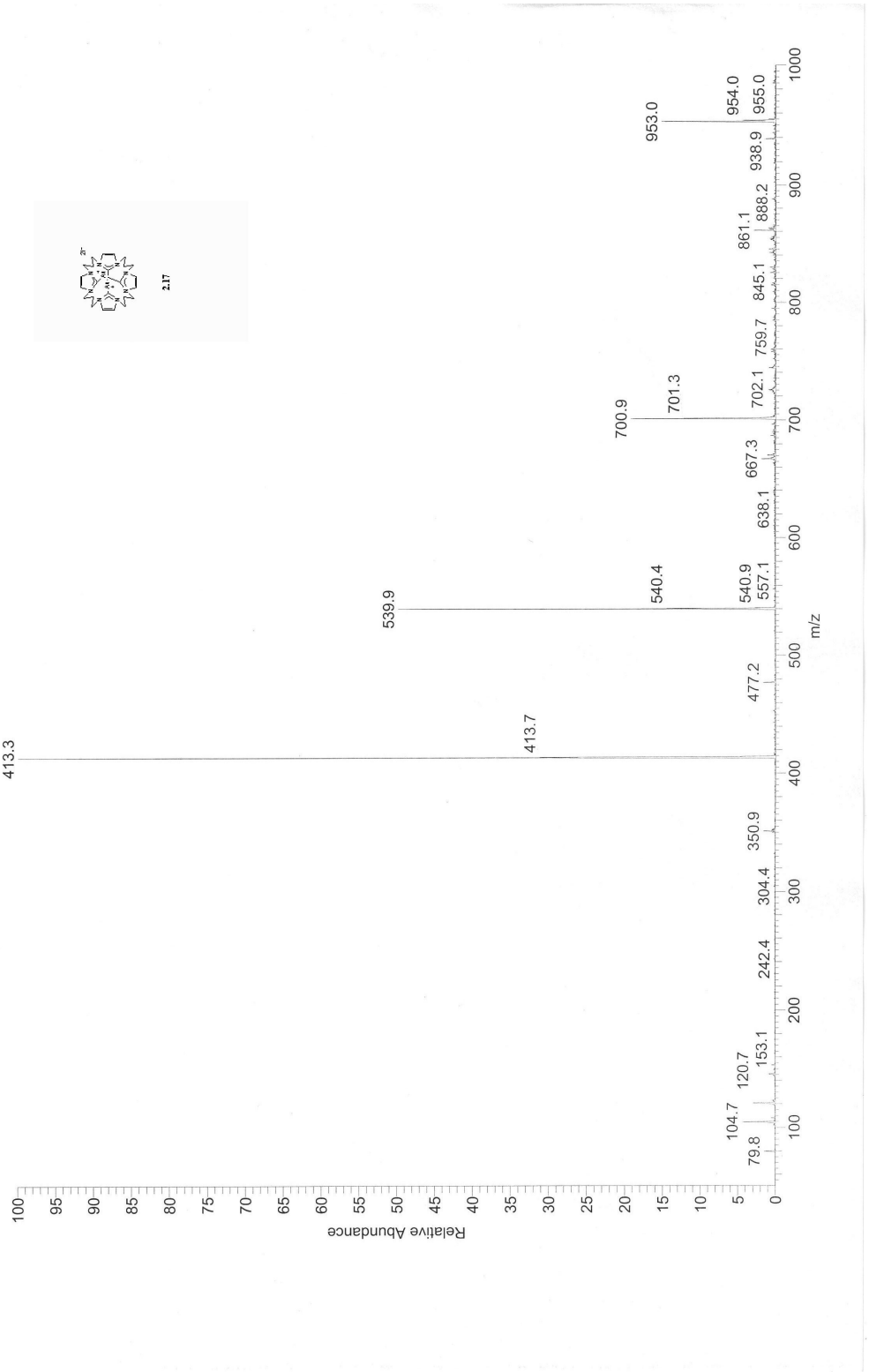
S2 C4S S2 24.5(9) 2 . ?

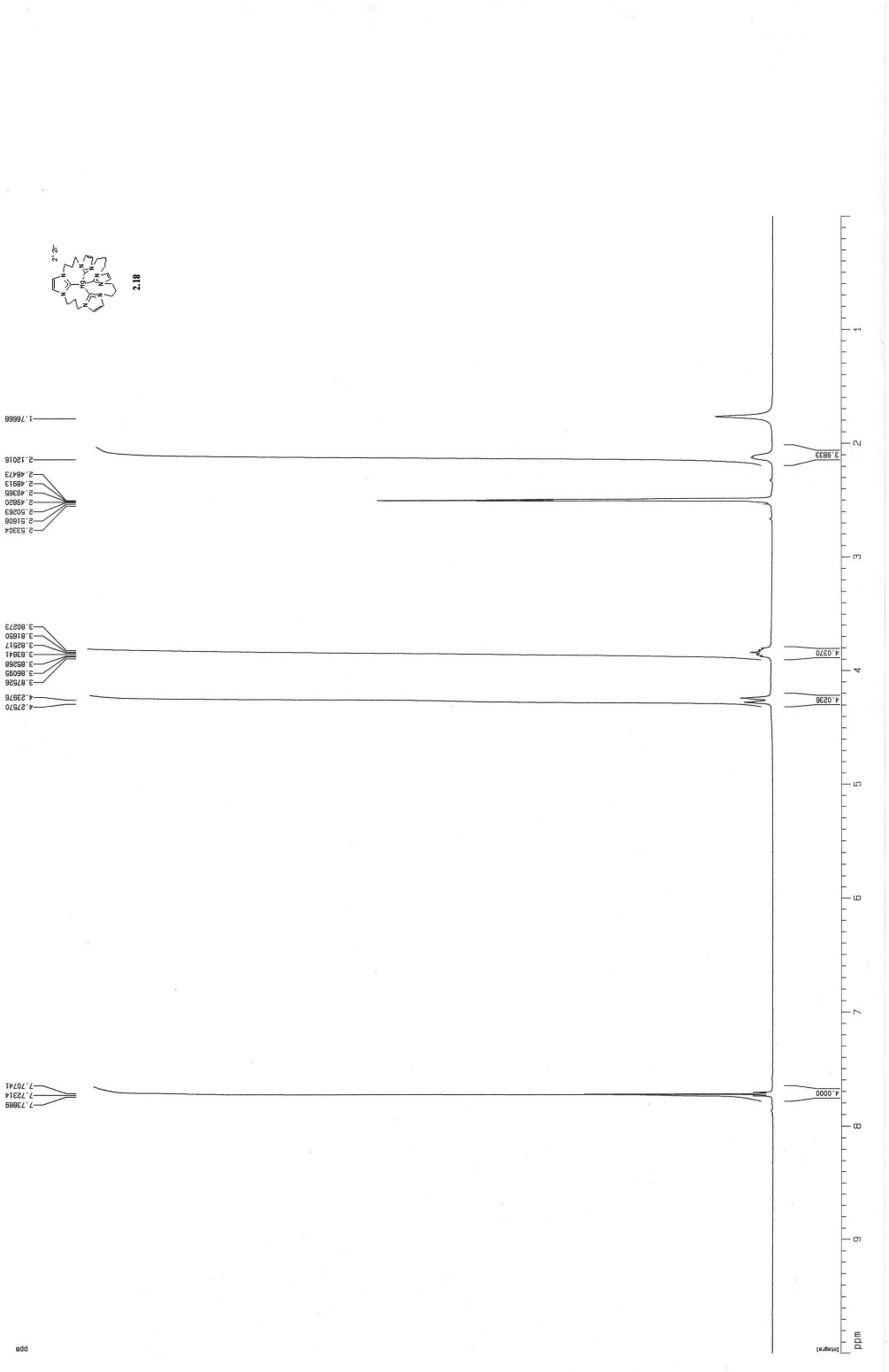
C4S C3SA S2 37(2) 2 . ?

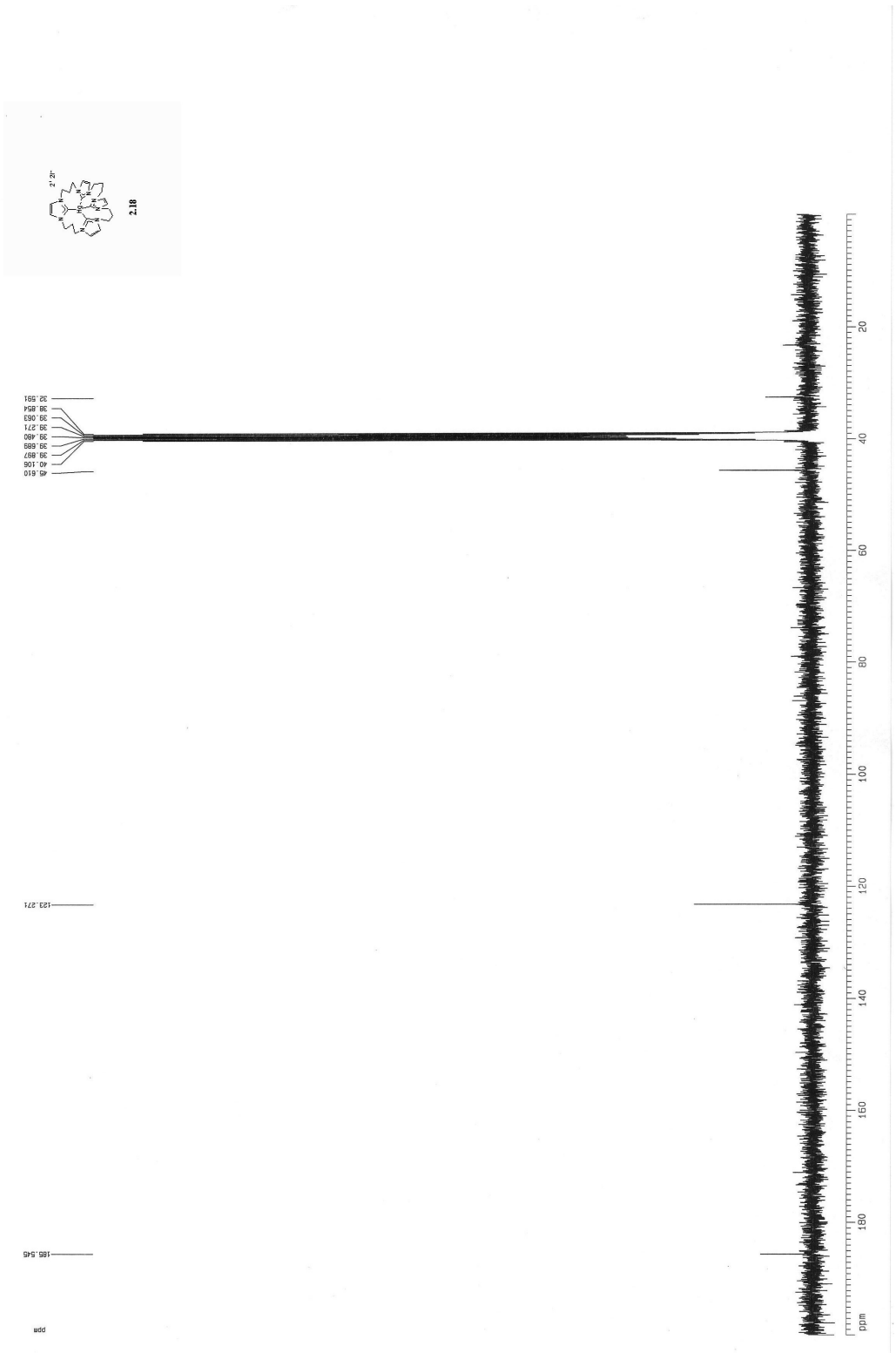
S2 O2SA S2 31.6(9) . 2 ?

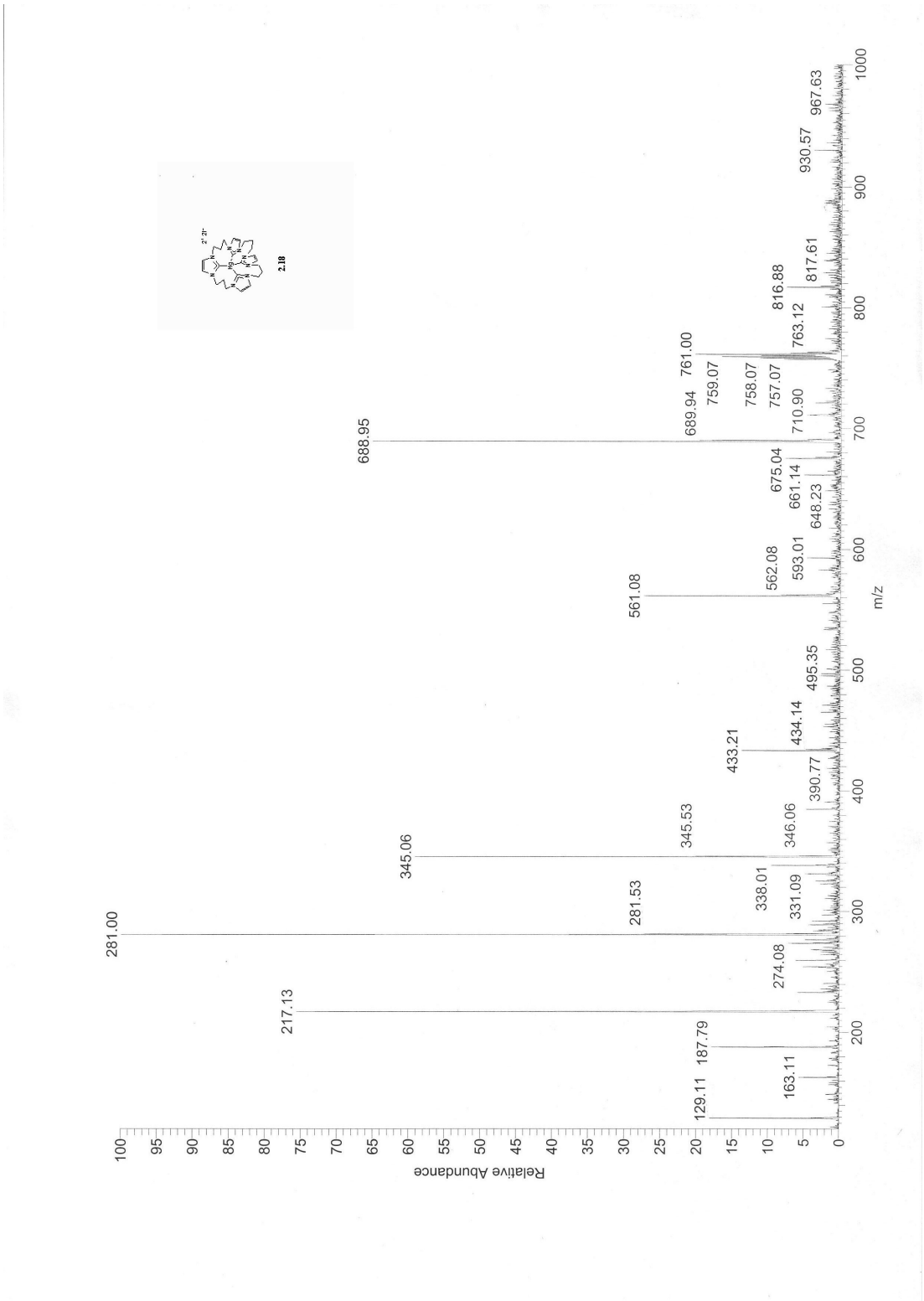


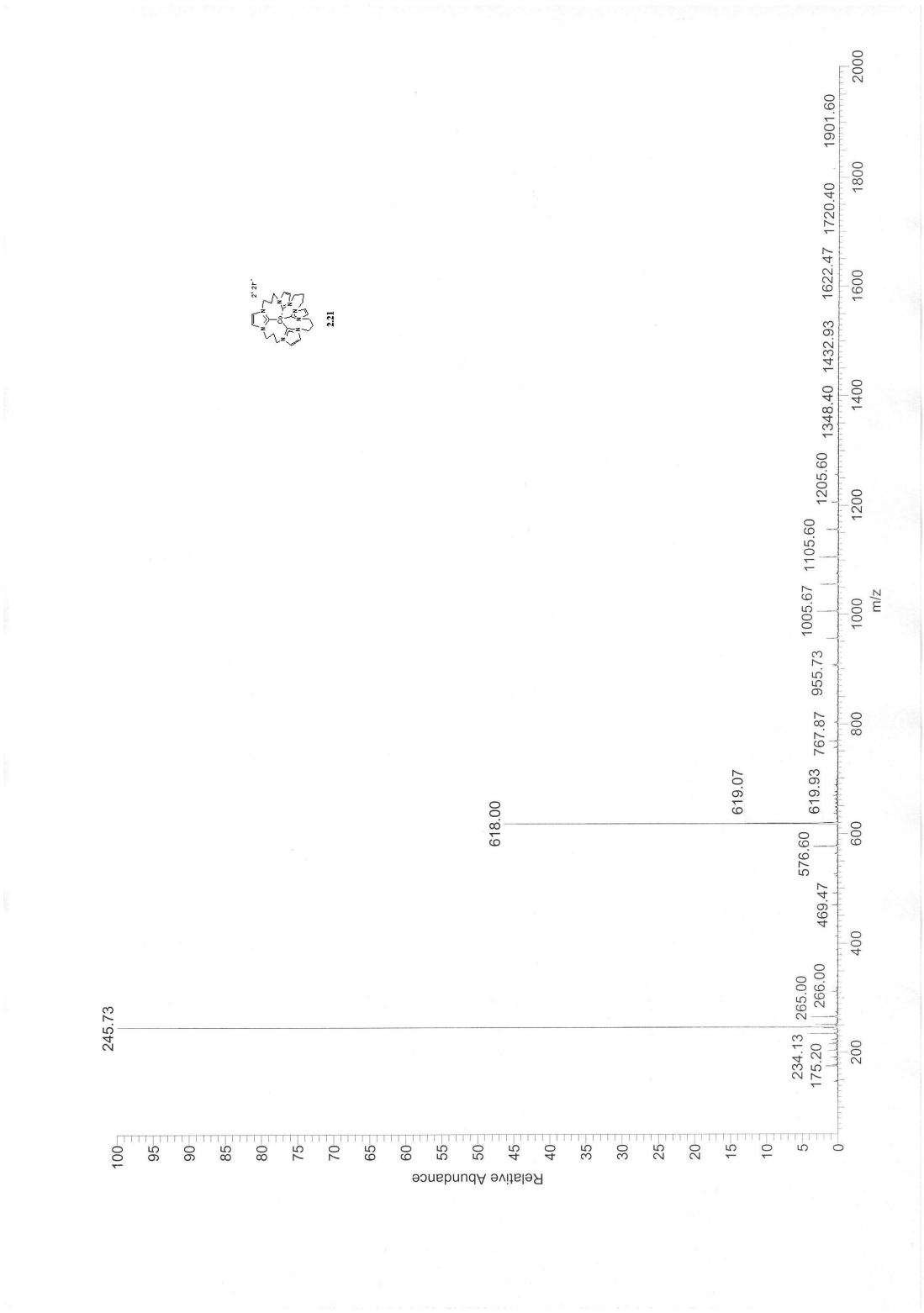

2.17
2F
2.43641
2.50228
2.50665
2.51099
2.54595
3.17356
3.13282
3.72045
3.74203
3.75503
3.76872
3.79080
3.91317
4.03003
4.14016
4.17516
7.72203
7.75429
7.80013
7.87860
1.02410
1.00686
1.09064
1.00000
ppm


2.17
Relative Abundance
m/z
413.3
413.7
539.9
540.4
700.9
701.3
953.0
954.0


2.18
1.76668
2.12016
2.48473
2.48913
2.49365
2.49820
2.50263
2.51608
2.53304
3.80273
3.81650
3.82517
3.83841
3.85368
3.86095
3.87526
4.23976
4.27570
7.70741
7.72314
7.73869
3.9833
4.0370
4.0236
4.0000
ppm
integral


2.18
32.591
38.854
39.063
39.271
39.480
39.688
39.897
40.106
45.610
123.271
185.545
ppm
20
40
60
80
100
120
140
160
180
ppm


2.18
Relative Abundance
m/z
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
200
300
400
500
600
700
800
900
1000
281.00
217.13
345.06
688.95
561.08
129.11
187.79
163.11
274.08
281.53
338.01
331.09
345.53
346.06
433.21
390.77
434.14
495.35
562.08
593.01
648.23
661.14
675.04
689.94
710.90
757.07
758.07
759.07
761.00
763.12
816.88
817.61
930.57
967.63


2.21
Relative Abundance
m/z
245.73
234.13
175.20
265.00
266.00
469.47
576.60
618.00
619.07
619.93
767.87
955.73
1005.67
1105.60
1205.60
1348.40
1432.93
1622.47
1720.40
1901.60

Crystal Structure Data for **2.21**

```
CIF_1.1

CIF produced by WinGX routine CIF_UPDATE
Created on 2006-11-29 at 12:54:44
Using CIFtbx version 2.6.2 16 Jun 1998

Dictionary name : cif_core.dic
Dictionary vers : 2.3
Request file    : h:\wingx\files\archive.dat
CIF files read  : stuartp8 dreduc import struct


data_stuartp8

_audit_creation_date                2006-11-29T12:54:44-00:00
_audit_creation_method              'WinGX routine CIF_UPDATE'
_audit_conform_dict_name            cif_core.dic
_audit_conform_dict_version         2.3
_audit_conform_dict_location        ftp://ftp.iucr.org/pub/cif_core.dic
_publ_requested_category            FM

#------------------------------------------------------------------------#
#                CHEMICAL INFORMATION                                    #
#------------------------------------------------------------------------#

_chemical_name_systematic
;
 ?
;
_chemical_name_common               'Cobalt Macrocycle'
_chemical_formula_moiety            'C24.5 H32 Co1 I2 N8 O0.5'
_chemical_formula_sum               'C24.5 H32 Co1 I2 N8 O0.5'
_chemical_formula_weight            759.32
```

```
_chemical_compound_source              'synthesis as described'

#------------------------------------------------------------------------------#
#                UNIT CELL INFORMATION                                         #
#------------------------------------------------------------------------------#

_symmetry_cell_setting                 orthorhombic
_symmetry_space_group_name_H-M         Pnmm
_symmetry_space_group_name_Hall        '-P 2c 2bc'
_symmetry_Int_Tables_number            59
loop_
   _symmetry_equiv_pos_as_xyz
'x, y, z'
'-x, -y, z+1/2'
'x, -y+1/2, -z+1/2'
'-x, y+1/2, -z'
'-x, -y, -z'
'x, y, -z-1/2'
'-x, y-1/2, z-1/2'
'x, -y-1/2, z'

_cell_length_a                         6.79870(10)
_cell_length_b                         11.0938(3)
_cell_length_c                         19.2961(5)
_cell_angle_alpha                      90
_cell_angle_beta                       90
_cell_angle_gamma                      90
_cell_volume                           1455.38(6)
_cell_formula_units_Z                  2
_cell_measurement_temperature          123(2)
_cell_measurement_reflns_used          5077
_cell_measurement_theta_min            3
_cell_measurement_theta_max            27.485
_cell_measurement_wavelength           0.71073

#------------------------------------------------------------------------------#
```

```
#                CRYSTAL INFORMATION                                #
#-------------------------------------------------------------------------#

_exptl_crystal_description            prism
_exptl_crystal_colour                 Blue
_exptl_crystal_size_max               0.4
_exptl_crystal_size_mid               0.2
_exptl_crystal_size_min               0.15
_exptl_crystal_density_diffrn         1.733
_exptl_crystal_density_method         'not measured'
_exptl_crystal_F_000                  744
_exptl_special_details
;
 ?
;

#-------------------------------------------------------------------------#
#                ABSORPTION CORRECTION                                #
#-------------------------------------------------------------------------#

_exptl_absorpt_coefficient_mu         2.741
_exptl_absorpt_correction_type        none

#-------------------------------------------------------------------------#
#                DATA COLLECTION                                      #
#-------------------------------------------------------------------------#

_diffrn_source                        'Enraf Nonius FR590'
_diffrn_ambient_temperature           123(2)
_diffrn_radiation_wavelength          0.71073
_diffrn_radiation_type                MoK\a
_diffrn_radiation_monochromator       graphite
_diffrn_radiation_probe               x-ray
_diffrn_detector                      'CCD plate'
_diffrn_detector_area_resol_mean      9
_diffrn_orient_matrix_type
```

'by Bruker AXS Collect from scalepack cell'

_diffrn_orient_matrix_ub_11 -0.113441

_diffrn_orient_matrix_ub_12 -0.418651E-1

_diffrn_orient_matrix_ub_13 -0.225575E-1

_diffrn_orient_matrix_ub_21 -0.850638E-1

_diffrn_orient_matrix_ub_22 0.718382E-1

_diffrn_orient_matrix_ub_23 0.90358E-2

_diffrn_orient_matrix_ub_31 0.391126E-1

_diffrn_orient_matrix_ub_32 0.348121E-1

_diffrn_orient_matrix_ub_33 -0.457738E-1

_diffrn_measurement_device '95mm CCD camera on \k-goniostat'

_diffrn_measurement_device_type KappaCCD

_diffrn_measurement_method 'CCD rotation images, thick slices'

_diffrn_reflns_av_R_equivalents 0.0292

_diffrn_reflns_av_unetI/netI 0.0211

_diffrn_reflns_number 9778

_diffrn_reflns_limit_h_min -8

_diffrn_reflns_limit_h_max 8

_diffrn_reflns_limit_k_min -13

_diffrn_reflns_limit_k_max 14

_diffrn_reflns_limit_l_min -24

_diffrn_reflns_limit_l_max 24

_diffrn_reflns_theta_min 3.18

_diffrn_reflns_theta_max 27.46

_diffrn_reflns_theta_full 27.46

_diffrn_measured_fraction_theta_full

0.994

_diffrn_measured_fraction_theta_max

0.994

_reflns_number_total 1790

_reflns_number_gt 1551

_reflns_threshold_expression >2sigma(I)

#---#

COMPUTER PROGRAMS USED

#---#

```
_computing_data_collection          'Collect (Bruker AXS BV, 1997-2004)'
_computing_cell_refinement
                              'HKL Scalepack (Otwinowski & Minor 1997)'
_computing_data_reduction
                        'HKL Denzo and Scalepack (Otwinowski & Minor 1997)'
_computing_structure_solution       'SHELXS-86 (Sheldrick, 1986)'
_computing_structure_refinement      'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics       'Ortep-3 for Windows (Farrugia, 1997)'
_computing_publication_material
                           'WinGX publication routines (Farrugia, 1999)'

#------------------------------------------------------------------------------#
#               REFINEMENT INFORMATION                                         #
#------------------------------------------------------------------------------#

_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
;
_refine_ls_structure_factor_coef     Fsqd
_refine_ls_matrix_type               full
_refine_ls_weighting_scheme          calc
_refine_ls_weighting_details
      'calc w=1/[\s^2^(Fo^2^)+(0.0358P)^2^+2.3637P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary         direct
_atom_sites_solution_secondary       difmap
_atom_sites_solution_hydrogens       geom
_refine_ls_hydrogen_treatment        mixed
_refine_ls_extinction_method         none
```

```
_refine_ls_number_reflns              1790
_refine_ls_number_parameters           91
_refine_ls_number_restraints           0
_refine_ls_R_factor_all               0.0323
_refine_ls_R_factor_gt                0.0244
_refine_ls_wR_factor_ref               0.0642
_refine_ls_wR_factor_gt                0.0606
_refine_ls_goodness_of_fit_ref          0.986
_refine_ls_restrained_S_all            0.986
_refine_ls_shift/su_max                0.008
_refine_ls_shift/su_mean               0.001
_refine_diff_density_max               1.724
_refine_diff_density_min               -0.506
_refine_diff_density_rms               0.101

#---------------------------------------------------------------------------#
#                ATOMIC TYPES, COORDINATES AND THERMAL PARAMETERS          #
#---------------------------------------------------------------------------#

loop_
   _atom_type_symbol
   _atom_type_description
   _atom_type_scat_dispersion_real
   _atom_type_scat_dispersion_imag
   _atom_type_scat_source
C C 0.003 0.002 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
H H 0 0 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
Br Br -0.29 2.46 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
Co Co 0.349 0.972 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
I I -0.474 1.812 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
N N 0.006 0.003 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
O O 0.011 0.006 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

loop_
   _atom_site_label
   _atom_site_type_symbol
```

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   _atom_site_fract_y
   _atom_site_fract_z
   _atom_site_U_iso_or_equiv
   _atom_site_adp_type
   _atom_site_occupancy
   _atom_site_symmetry_multiplicity
   _atom_site_calc_flag
   _atom_site_refinement_flags
   _atom_site_disorder_assembly
   _atom_site_disorder_group
C7 C 0.7068(4) 0.1898(3) 0.57053(14) 0.0256(6) Uani 1 1 d . . .
H7 H 0.7716 0.1387 0.5383 0.031 Uiso 1 1 calc R . .
O1 O 0.5266(13) 0.75 0.75 0.036(2) Uiso 0.5 4 d SP . .
C1S C 0.4685(17) 0.75 0.6846(6) 0.009(2) Uiso 0.25 2 d SP . .
I1 I 0.82995(4) 0.75 0.548397(12) 0.02136(10) Uani 1 2 d S . .
Co1 Co 0.35100(9) 0.25 0.75 0.01356(15) Uani 1 4 d S . .
N1 N 0.1231(3) 0.02963(19) 0.69470(11) 0.0173(4) Uani 1 1 d . . .
C3 C 0.1702(4) 0.0557(2) 0.62184(13) 0.0204(5) Uani 1 1 d . . .
H3A H 0.1873 0.1437 0.6159 0.024 Uiso 1 1 calc R . .
H3B H 0.0593 0.0298 0.5921 0.024 Uiso 1 1 calc R . .
C1 C 0.1853(5) 0.0949(3) 0.75 0.0159(7) Uani 1 2 d S . .
N2 N 0.5943(3) 0.1537(2) 0.62652(11) 0.0204(5) Uani 1 1 d . . .
C6 C 0.5216(5) 0.25 0.66196(18) 0.0181(7) Uani 1 2 d S . .
C4 C 0.3573(4) -0.0087(3) 0.59882(14) 0.0249(6) Uani 1 1 d . . .
H4A H 0.3791 0.008 0.549 0.03 Uiso 1 1 calc R . .
H4B H 0.3375 -0.0967 0.604 0.03 Uiso 1 1 calc R . .
C2 C 0.0264(4) -0.0739(2) 0.71498(14) 0.0222(5) Uani 1 1 d . . .
H2 H -0.0293 -0.1334 0.6855 0.027 Uiso 1 1 calc R . .
C5 C 0.5417(4) 0.0275(2) 0.63893(14) 0.0235(6) Uani 1 1 d . . .
H5A H 0.5189 0.0152 0.6891 0.028 Uiso 1 1 calc R . .
H5B H 0.6525 -0.0249 0.6248 0.028 Uiso 1 1 calc R . .

loop_
   _atom_site_aniso_label
   _atom_site_aniso_U_11
```

```
   _atom_site_aniso_U_22
   _atom_site_aniso_U_33
   _atom_site_aniso_U_23
   _atom_site_aniso_U_13
   _atom_site_aniso_U_12
C7 0.0167(11) 0.0449(16) 0.0152(12) -0.0017(12) 0.0028(10) 0.0034(12)
I1 0.02612(15) 0.02229(14) 0.01566(14) 0 0.00236(9) 0
Co1 0.0156(3) 0.0136(3) 0.0114(3) 0 0 0
N1 0.0188(10) 0.0159(10) 0.0171(10) 0.0003(9) -0.0010(8) -0.0003(8)
C3 0.0246(13) 0.0237(13) 0.0128(11) 0.0004(10) -0.0038(10) -0.0024(11)
C1 0.0153(15) 0.0180(16) 0.0144(16) 0 0 0.0044(14)
N2 0.0162(10) 0.0284(12) 0.0168(11) -0.0018(9) 0.0008(8) 0.0029(9)
C6 0.0156(16) 0.0230(18) 0.0156(16) 0 0.0002(13) 0
C4 0.0321(15) 0.0241(14) 0.0185(13) -0.0059(11) 0.0010(11) 0.0007(12)
C2 0.0235(12) 0.0159(12) 0.0271(13) -0.0007(10) -0.0020(11) -0.0039(11)
C5 0.0241(13) 0.0251(14) 0.0215(13) -0.0042(11) 0.0026(11) 0.0075(11)



#---------------------------------------------------------------------------#
#                MOLECULAR GEOMETRY                                         #
#---------------------------------------------------------------------------#


_geom_special_details
;
 All esds (except the esd in the dihedral angle between two l.s. planes)
 are estimated using the full covariance matrix.  The cell esds are taken
 into account individually in the estimation of esds in distances, angles
 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry.  An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
;
loop_
   _geom_bond_atom_site_label_1
   _geom_bond_atom_site_label_2
   _geom_bond_distance
   _geom_bond_site_symmetry_2
```

```
   _geom_bond_publ_flag
C7 C7 1.337(6) 8_565 ?
C7 N2 1.383(3) . ?
O1 C1S 1.323(12) . ?
O1 C1S 1.323(12) 6_557 ?
Co1 C1 2.056(4) . ?
Co1 C1 2.056(4) 3_556 ?
Co1 C6 2.057(4) 6_557 ?
Co1 C6 2.057(4) . ?
N1 C1 1.357(3) . ?
N1 C2 1.380(3) . ?
N1 C3 1.471(3) . ?
C3 C4 1.525(4) . ?
C1 N1 1.357(3) 6_557 ?
N2 C6 1.361(3) . ?
N2 C5 1.464(3) . ?
C6 N2 1.361(3) 8_565 ?
C4 C5 1.527(4) . ?
C2 C2 1.351(5) 6_557 ?

loop_
   _geom_angle_atom_site_label_1
   _geom_angle_atom_site_label_2
   _geom_angle_atom_site_label_3
   _geom_angle
   _geom_angle_site_symmetry_1
   _geom_angle_site_symmetry_3
   _geom_angle_publ_flag
C7 C7 N2 106.81(16) 8_565 . ?
C1S O1 C1S 145.3(13) . 6_557 ?
C1 Co1 C1 113.57(19) . 3_556 ?
C1 Co1 C6 107.99(6) . 6_557 ?
C1 Co1 C6 107.99(6) 3_556 6_557 ?
C1 Co1 C6 107.99(6) . . ?
C1 Co1 C6 107.99(6) 3_556 . ?
C6 Co1 C6 111.3(2) 6_557 . ?
```

C1 N1 C2 111.7(2) . . ?

C1 N1 C3 125.4(2) . . ?

C2 N1 C3 122.6(2) . . ?

N1 C3 C4 111.6(2) . . ?

N1 C1 N1 103.7(3) . 6_557 ?

N1 C1 Co1 128.10(15) . . ?

N1 C1 Co1 128.10(15) 6_557 . ?

C6 N2 C7 111.5(2) . . ?

C6 N2 C5 125.4(2) . . ?

C7 N2 C5 122.6(2) . . ?

N2 C6 N2 103.4(3) 8_565 . ?

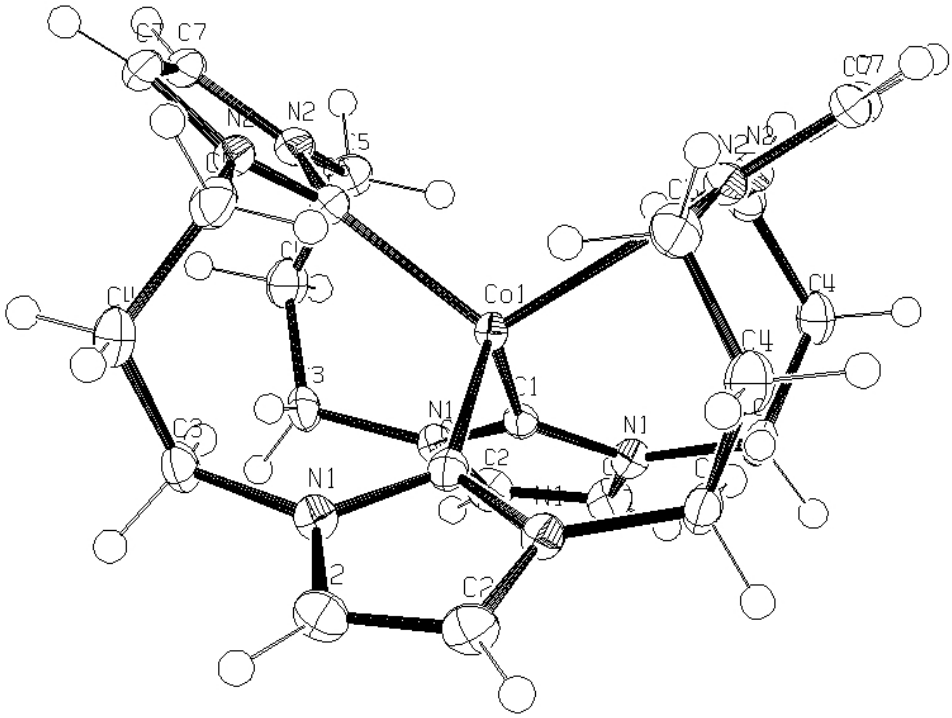
N2 C6 Co1 128.27(15) 8_565 . ?

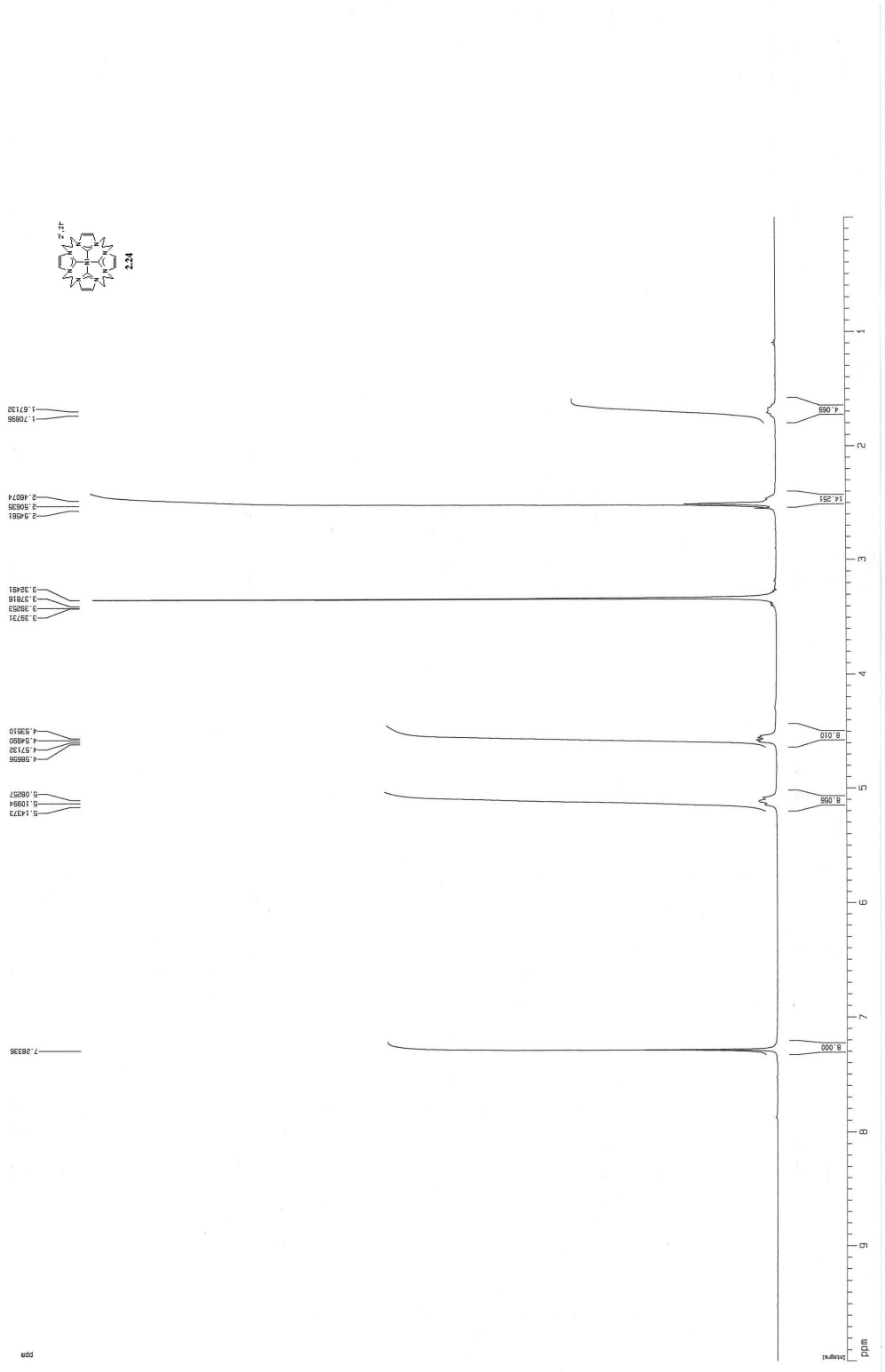
N2 C6 Co1 128.27(15) . . ?

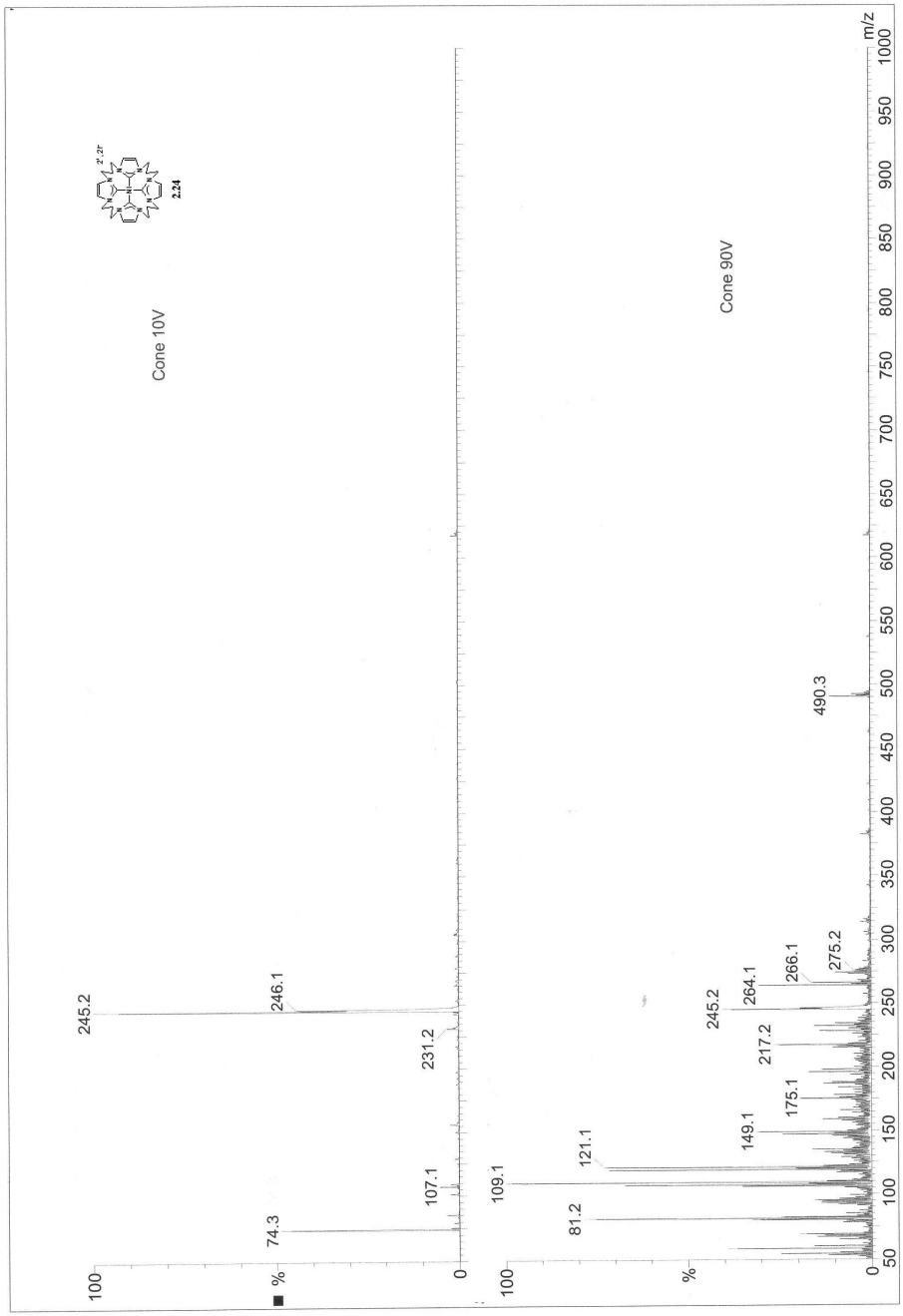
C3 C4 C5 114.4(2) . . ?

C2 C2 N1 106.47(14) 6_557 . ?

N2 C5 C4 111.7(2) . . ?




2.24
1.67132
1.70856
2.46074
2.50635
2.54561
3.32481
3.37816
3.39253
3.39731
4.53510
4.54990
4.57132
4.58656
5.08257
5.10994
5.14373
7.28336
4.069
14.251
8.010
8.056
8.000
ppm
Integral


2.24
Cone 10V
Cone 90V
245.2
246.1
231.2
107.1
74.3
109.1
121.1
81.2
149.1
175.1
217.2
245.2
264.1
266.1
275.2
490.3
100
%
0
m/z
50
100
150
200
250
300
350
400
450
500
550
600
650
700
750
800
850
900
950
1000

Crystal Structure Data for **2.24**

CIF_1.1

CIF produced by WinGX routine CIF_UPDATE
Created on 2007-10-01 at 11:49:28
Using CIFtbx version 2.6.2 16 Jun 1998

Dictionary name : cif_core.dic
Dictionary vers : 2.3
Request file : h:\wingx\files\archive.dat
CIF files read : stuartp9 dreduc import struct

data_stuartp9

_audit_creation_date	2007-10-01T11:49:28-00:00
_audit_creation_method	'WinGX routine CIF_UPDATE'
_audit_conform_dict_name	cif_core.dic
_audit_conform_dict_version	2.3
_audit_conform_dict_location	ftp://ftp.iucr.org/pub/cif_core.dic
_publ_requested_category	FM

#---#
CHEMICAL INFORMATION
#---#

_chemical_name_systematic
;
?
;

_chemical_name_common	NiTetI2
_chemical_formula_moiety	'C24 H32 I2 N8 Ni1'
_chemical_formula_sum	'C24 H32 I2 N8 Ni'
_chemical_formula_weight	745.08
_chemical_compound_source	'synthesis as described'

```
#------------------------------------------------------------------------------#
#                UNIT CELL INFORMATION                                         #
#------------------------------------------------------------------------------#

_symmetry_cell_setting              orthorhombic
_symmetry_space_group_name_H-M      I4/mmm
_symmetry_space_group_name_Hall     '-I 4 2'
_symmetry_Int_Tables_number         139
loop_
   _symmetry_equiv_pos_as_xyz
'x, y, z'
'x, -y, -z'
'-x, -y, z'
'-x, y, -z'
'y, x, -z'
'y, -x, z'
'-y, x, z'
'-y, -x, -z'
'x+1/2, y+1/2, z+1/2'
'x+1/2, -y+1/2, -z+1/2'
'-x+1/2, -y+1/2, z+1/2'
'-x+1/2, y+1/2, -z+1/2'
'y+1/2, x+1/2, -z+1/2'
'y+1/2, -x+1/2, z+1/2'
'-y+1/2, x+1/2, z+1/2'
'-y+1/2, -x+1/2, -z+1/2'
'-x, -y, -z'
'-x, y, z'
'x, y, -z'
'x, -y, z'
'-y, -x, z'
'-y, x, -z'
'y, -x, -z'
'y, x, z'
'-x+1/2, -y+1/2, -z+1/2'
```

'-x+1/2, y+1/2, z+1/2'
'x+1/2, y+1/2, -z+1/2'
'x+1/2, -y+1/2, z+1/2'
'-y+1/2, -x+1/2, z+1/2'
'-y+1/2, x+1/2, -z+1/2'
'y+1/2, -x+1/2, -z+1/2'
'y+1/2, x+1/2, z+1/2'

_cell_length_a 8.9459(3)
_cell_length_b 8.9419(3)
_cell_length_c 18.6755(6)
_cell_angle_alpha 90
_cell_angle_beta 90
_cell_angle_gamma 90
_cell_volume 1493.92(9)
_cell_formula_units_Z 2
_cell_measurement_temperature 123(2)
_cell_measurement_reflns_used 5812
_cell_measurement_theta_min 3
_cell_measurement_theta_max 27.485
_cell_measurement_wavelength 0.71073

#---#
CRYSTAL INFORMATION
#---#

_exptl_crystal_description cube
_exptl_crystal_colour colourless
_exptl_crystal_size_max 0.4
_exptl_crystal_size_mid 0.4
_exptl_crystal_size_min 0.4
_exptl_crystal_density_diffrn 1.656
_exptl_crystal_density_method 'not measured'
_exptl_crystal_F_000 732
_exptl_special_details
;

```
 ?
;

#---------------------------------------------------------------------------#
#                ABSORPTION CORRECTION                                      #
#---------------------------------------------------------------------------#

_exptl_absorpt_coefficient_mu          2.742
_exptl_absorpt_correction_type         none

#---------------------------------------------------------------------------#
#                DATA COLLECTION                                            #
#---------------------------------------------------------------------------#

_diffrn_source                         'Enraf Nonius FR590'
_diffrn_ambient_temperature            123(2)
_diffrn_radiation_wavelength           0.71073
_diffrn_radiation_type                 MoK\a
_diffrn_radiation_monochromator        graphite
_diffrn_radiation_probe                x-ray
_diffrn_detector                       'CCD plate'
_diffrn_detector_area_resol_mean       9
_diffrn_orient_matrix_type
                              'by Bruker AXS Collect from scalepack cell'
_diffrn_orient_matrix_ub_11            -0.51881E-1
_diffrn_orient_matrix_ub_12            -0.955972E-1
_diffrn_orient_matrix_ub_13            -0.124283E-1
_diffrn_orient_matrix_ub_21            0.503478E-1
_diffrn_orient_matrix_ub_22            -0.2375E-3
_diffrn_orient_matrix_ub_23            -0.478071E-1
_diffrn_orient_matrix_ub_31            0.852579E-1
_diffrn_orient_matrix_ub_32            -0.580324E-1
_diffrn_orient_matrix_ub_33            0.206689E-1
_diffrn_measurement_device             '95mm CCD camera on \k-goniostat'
_diffrn_measurement_device_type        KappaCCD
_diffrn_measurement_method             'CCD rotation images, thick slices'
```

```
_diffrn_reflns_av_R_equivalents        0.0268
_diffrn_reflns_av_unetI/netI           0.0119
_diffrn_reflns_number                  6131
_diffrn_reflns_limit_h_min             -11
_diffrn_reflns_limit_h_max             11
_diffrn_reflns_limit_k_min             -11
_diffrn_reflns_limit_k_max             11
_diffrn_reflns_limit_l_min             -24
_diffrn_reflns_limit_l_max             24
_diffrn_reflns_theta_min               3.22
_diffrn_reflns_theta_max               27.49
_diffrn_reflns_theta_full              27.49
_diffrn_measured_fraction_theta_full
                                0.996
_diffrn_measured_fraction_theta_max
                                0.996
_reflns_number_total                   542
_reflns_number_gt                      505
_reflns_threshold_expression           >2sigma(I)

#---------------------------------------------------------------------------#
#                 COMPUTER PROGRAMS USED                                    #
#---------------------------------------------------------------------------#

_computing_data_collection             'Collect (Bruker AXS BV, 1997-2004)'
_computing_cell_refinement
                                'HKL Scalepack (Otwinowski & Minor 1997)'
_computing_data_reduction
                          'HKL Denzo and Scalepack (Otwinowski & Minor 1997)'
_computing_structure_solution          'DIRDIF99 (Beurskens et al, 1999)'
_computing_structure_refinement        'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics          'Ortep-3 for Windows (Farrugia, 1997)'
_computing_publication_material
                          'WinGX publication routines (Farrugia, 1999)'

#---------------------------------------------------------------------------#
```

```
#                REFINEMENT INFORMATION                          #
#--------------------------------------------------------------------------#

_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
;
_refine_ls_structure_factor_coef       Fsqd
_refine_ls_matrix_type                 full
_refine_ls_weighting_scheme            calc
_refine_ls_weighting_details
       'calc w=1/[\s^2^(Fo^2^)+(0.0493P)^2^+3.1094P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary           direct
_atom_sites_solution_secondary         difmap
_atom_sites_solution_hydrogens         geom
_refine_ls_hydrogen_treatment          mixed
_refine_ls_extinction_method           none
_refine_ls_number_reflns               542
_refine_ls_number_parameters           45
_refine_ls_number_restraints           0
_refine_ls_R_factor_all                0.0315
_refine_ls_R_factor_gt                 0.0290
_refine_ls_wR_factor_ref               0.0824
_refine_ls_wR_factor_gt                0.0810
_refine_ls_goodness_of_fit_ref         1.166
_refine_ls_restrained_S_all            1.166
_refine_ls_shift/su_max                0.000
_refine_ls_shift/su_mean               0.000
_refine_diff_density_max               0.622
_refine_diff_density_min               -0.336
```

```
_refine_diff_density_rms        0.096

#---------------------------------------------------------------------------#
#        ATOMIC TYPES, COORDINATES AND THERMAL PARAMETERS        #
#---------------------------------------------------------------------------#

loop_
   _atom_type_symbol
   _atom_type_description
   _atom_type_scat_dispersion_real
   _atom_type_scat_dispersion_imag
   _atom_type_scat_source
C C 0.003 0.002 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
H H 0 0 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
I I -0.474 1.812 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
N N 0.006 0.003 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
Ni Ni 0.339 1.112 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

loop_
   _atom_site_label
   _atom_site_type_symbol
   _atom_site_fract_x
   _atom_site_fract_y
   _atom_site_fract_z
   _atom_site_U_iso_or_equiv
   _atom_site_adp_type
   _atom_site_occupancy
   _atom_site_symmetry_multiplicity
   _atom_site_calc_flag
   _atom_site_refinement_flags
   _atom_site_disorder_assembly
   _atom_site_disorder_group
Ni1 Ni 0 0 0 0.0197(3) Uani 1 16 d S . .
C1 C 0.1796(8) 0.1135(7) 0 0.0234(13) Uani 0.5 2 d SP . .
C2 C 0.3888(6) 0.2385(7) 0.0355(3) 0.0363(12) Uani 0.5 1 d P . .
N1 N 0.2595(6) 0.1611(6) 0.0556(2) 0.0451(14) Uani 0.5 1 d P . .
```

C3 C 0.1887(6) 0.1887(6) 0.1320(4) 0.0289(14) Uani 0.5 2 d SP . .
C4 C 0.2770(13) 0.0819(9) 0.1378(4) 0.071(3) Uani 0.5 1 d P . .
I1A I 0.5 0.5 0.20078(2) 0.0369(2) Uani 1 8 d S . .

loop_
_atom_site_aniso_label
_atom_site_aniso_U_11
_atom_site_aniso_U_22
_atom_site_aniso_U_33
_atom_site_aniso_U_23
_atom_site_aniso_U_13
_atom_site_aniso_U_12
Ni1 0.0205(4) 0.0205(4) 0.0180(6) 0 0 0
C1 0.027(3) 0.021(3) 0.022(3) 0 0 0.005(2)
C2 0.031(2) 0.041(3) 0.037(3) 0.001(2) 0.000(2) -0.012(2)
N1 0.045(3) 0.070(3) 0.0203(18) 0.004(2) -0.0032(18) -0.035(2)
C3 0.034(2) 0.034(2) 0.018(3) -0.002(2) -0.002(2) 0.002(3)
C4 0.122(8) 0.050(5) 0.041(3) -0.018(3) 0.045(4) -0.034(5)
I1A 0.0453(3) 0.0453(3) 0.0199(3) 0 0 0

#---#
MOLECULAR GEOMETRY
#---#

_geom_special_details
;
All esds (except the esd in the dihedral angle between two l.s. planes)
are estimated using the full covariance matrix. The cell esds are taken
into account individually in the estimation of esds in distances, angles
and torsion angles; correlations between esds in cell parameters are only
used when they are defined by crystal symmetry. An approximate (isotropic)
treatment of cell esds is used for estimating esds involving l.s. planes.
;
loop_
_geom_bond_atom_site_label_1

```
   _geom_bond_atom_site_label_2
   _geom_bond_distance
   _geom_bond_site_symmetry_2
   _geom_bond_publ_flag
Ni1 C1 1.900(7) 5 ?
Ni1 C1 1.900(7) 21 ?
Ni1 C1 1.900(7) 22 ?
Ni1 C1 1.900(7) 6 ?
Ni1 C1 1.900(7) . ?
Ni1 C1 1.900(7) 17 ?
Ni1 C1 1.900(7) 2 ?
Ni1 C1 1.900(7) 18 ?
C1 C1 0.836(12) 5 ?
C1 N1 1.331(6) . ?
C1 N1 1.331(6) 19 ?
C1 N1 1.677(7) 24 ?
C1 N1 1.677(7) 5 ?
C1 C1 2.030(13) 2 ?
C2 C2 1.327(11) 19 ?
C2 N1 1.399(7) . ?
C2 C2 1.900(13) 24 ?
C2 C2 1.990(11) 18_655 ?
N1 N1 1.245(11) 24 ?
N1 C3 1.579(8) . ?
N1 C1 1.677(7) 5 ?
N1 C4 1.698(10) . ?
C3 C4 1.244(11) . ?
C3 C4 1.245(11) 24 ?
C3 N1 1.579(8) 24 ?
C4 C4 1.465(16) 20 ?

loop_
   _geom_angle_atom_site_label_1
   _geom_angle_atom_site_label_2
   _geom_angle_atom_site_label_3
   _geom_angle
```

_geom_angle_site_symmetry_1
_geom_angle_site_symmetry_3
_geom_angle_publ_flag
C1 Ni1 C1 180.0(4) 5 21 ?
C1 Ni1 C1 64.6(4) 5 22 ?
C1 Ni1 C1 115.4(4) 21 22 ?
C1 Ni1 C1 115.4(4) 5 6 ?
C1 Ni1 C1 64.6(4) 21 6 ?
C1 Ni1 C1 180.0(3) 22 6 ?
C1 Ni1 C1 25.4(4) 5 . ?
C1 Ni1 C1 154.6(4) 21 . ?
C1 Ni1 C1 90 22 . ?
C1 Ni1 C1 90 6 . ?
C1 Ni1 C1 154.6(4) 5 17 ?
C1 Ni1 C1 25.4(4) 21 17 ?
C1 Ni1 C1 90 22 17 ?
C1 Ni1 C1 90 6 17 ?
C1 Ni1 C1 180.0(4) . 17 ?
C1 Ni1 C1 90 5 2 ?
C1 Ni1 C1 90 21 2 ?
C1 Ni1 C1 154.6(4) 22 2 ?
C1 Ni1 C1 25.4(4) 6 2 ?
C1 Ni1 C1 64.6(4) . 2 ?
C1 Ni1 C1 115.4(4) 17 2 ?
C1 Ni1 C1 90 5 18 ?
C1 Ni1 C1 90 21 18 ?
C1 Ni1 C1 25.4(4) 22 18 ?
C1 Ni1 C1 154.6(4) 6 18 ?
C1 Ni1 C1 115.4(4) . 18 ?
C1 Ni1 C1 64.6(4) 17 18 ?
C1 Ni1 C1 180 2 18 ?
C1 C1 N1 98.8(4) 5 . ?
C1 C1 N1 98.8(4) 5 19 ?
N1 C1 N1 102.6(6) . 19 ?
C1 C1 N1 51.7(3) 5 24 ?
N1 C1 N1 47.2(5) . 24 ?

N1 C1 N1 106.7(6) 19 24 ?
C1 C1 N1 51.7(3) 5 5 ?
N1 C1 N1 106.7(6) . 5 ?
N1 C1 N1 47.2(5) 19 5 ?
N1 C1 N1 76.6(4) 24 5 ?
C1 C1 Ni1 77.27(19) 5 . ?
N1 C1 Ni1 128.7(3) . . ?
N1 C1 Ni1 128.7(3) 19 . ?
N1 C1 Ni1 109.4(3) 24 . ?
N1 C1 Ni1 109.4(3) 5 . ?
C1 C1 C1 135 5 2 ?
N1 C1 C1 108.7(4) . 2 ?
N1 C1 C1 108.7(4) 19 2 ?
N1 C1 C1 141.1(2) 24 2 ?
N1 C1 C1 141.1(2) 5 2 ?
Ni1 C1 C1 57.72(19) . 2 ?
C2 C2 N1 105.5(3) 19 . ?
C2 C2 C2 90 19 24 ?
N1 C2 C2 76.4(3) . 24 ?
C2 C2 C2 90.0000(10) 19 18_655 ?
N1 C2 C2 145.7(3) . 18_655 ?
C2 C2 C2 135.013(2) 24 18_655 ?
N1 N1 C1 81.2(4) 24 . ?
N1 N1 C2 103.6(3) 24 . ?
C1 N1 C2 113.1(5) . . ?
N1 N1 C3 66.8(2) 24 . ?
C1 N1 C3 122.6(5) . . ?
C2 N1 C3 119.7(5) . . ?
N1 N1 C1 51.6(3) 24 5 ?
C1 N1 C1 29.5(5) . 5 ?
C2 N1 C1 115.4(4) . 5 ?
C3 N1 C1 103.4(4) . 5 ?
N1 N1 C4 111.1(4) 24 . ?
C1 N1 C4 128.5(5) . . ?
C2 N1 C4 111.9(5) . . ?
C3 N1 C4 44.5(4) . . ?

C1 N1 C4 132.3(5) 5 . ?
C4 C3 C4 165.2(10) . 24 ?
C4 C3 N1 119.1(6) . 24 ?
C4 C3 N1 72.8(4) 24 24 ?
C4 C3 N1 72.8(4) . . ?
C4 C3 N1 119.1(6) 24 . ?
N1 C3 N1 46.4(4) 24 . ?
C3 C4 C4 140.1(6) . 20 ?
C3 C4 N1 62.7(6) . . ?
C4 C4 N1 114.6(3) 20 . ?