# Appendix 1

Crystallographic Data for 2-DMAP Disalt **4.16**

Table 1. Crystal data and structure refinement for jam0508.

| | | |
|---|---|---|
| Identification code | jam0508 | |
| Empirical formula | C12 H16 F6 N2 O6 S2 | |
| Formula weight | 462.39 | |
| Temperature | 123(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Monoclinic | |
| Space group | $P2_1/n$ | |
| Unit cell dimensions | a = 8.2712(3) Å | α= 90°. |
| | b = 22.5001(7) Å | β= 112.117(5)°. |
| | c = 10.3639(5) Å | γ = 90°. |
| Volume | 1786.83(12) $Å^3$ | |
| Z | 4 | |
| Density (calculated) | 1.719 $Mg/m^3$ | |
| Absorption coefficient | 0.393 $mm^{-1}$ | |
| F(000) | 944 | |
| Crystal size | 0.20 x 0.18 x 0.04 $mm^3$ | |
| Theta range for data collection | 2.71 to 30.00°. | |
| Index ranges | -11<=h<=11, -31<=k<=27, -14<=l<=12 | |
| Reflections collected | 15663 | |
| Independent reflections | 4934 [R(int) = 0.0281] | |
| Completeness to theta = 26.00° | 98.4 % | |
| Absorption correction | None | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 4934 / 0 / 255 | |
| Goodness-of-fit on $F^2$ | 0.952 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0320, wR2 = 0.0699 | |
| R indices (all data) | R1 = 0.0563, wR2 = 0.0736 | |
| Largest diff. peak and hole | 0.400 and -0.346 $e.Å^{-3}$ | |

Table 2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for jam0508. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| S(1) | 10811(1) | 2115(1) | 477(1) | 17(1) |
| S(2) | 14245(1) | 487(1) | 6912(1) | 20(1) |
| F(1) | 10751(2) | 1750(1) | -1924(1) | 56(1) |
| F(2) | 8206(1) | 1850(1) | -1837(1) | 40(1) |
| F(3) | 9820(1) | 1106(1) | -839(1) | 49(1) |
| F(4) | 14486(1) | 1490(1) | 8261(1) | 46(1) |
| F(5) | 16486(1) | 854(1) | 9322(1) | 44(1) |
| F(6) | 13878(1) | 729(1) | 9261(1) | 46(1) |
| O(1) | 12507(1) | 1852(1) | 1161(1) | 29(1) |
| O(2) | 9601(1) | 2022(1) | 1163(1) | 25(1) |
| O(3) | 10816(1) | 2706(1) | -40(1) | 26(1) |
| O(4) | 15299(1) | 751(1) | 6236(1) | 29(1) |
| O(5) | 14738(1) | -111(1) | 7419(1) | 26(1) |
| O(6) | 12387(1) | 577(1) | 6237(1) | 26(1) |
| N(1) | 8502(1) | 1453(1) | 3710(1) | 15(1) |
| N(2) | 11242(1) | 1155(1) | 3484(1) | 16(1) |
| C(1) | 9382(2) | 2052(1) | 4201(2) | 19(1) |
| C(2) | 11278(2) | 1967(1) | 5108(2) | 22(1) |
| C(3) | 12223(2) | 1681(1) | 4280(2) | 20(1) |
| C(4) | 9525(2) | 1052(1) | 3163(2) | 15(1) |
| C(5) | 8718(2) | 570(1) | 2395(2) | 20(1) |
| C(6) | 9665(2) | 180(1) | 1915(2) | 25(1) |
| C(7) | 11413(2) | 287(1) | 2239(2) | 26(1) |
| C(8) | 12166(2) | 774(1) | 3020(2) | 22(1) |
| C(9) | 6730(2) | 1579(1) | 2588(2) | 20(1) |
| C(10) | 8230(2) | 1132(1) | 4908(2) | 20(1) |
| C(11) | 9843(2) | 1683(1) | -1110(2) | 29(1) |
| C(12) | 14796(2) | 913(1) | 8513(2) | 30(1) |

Table 3. Bond lengths [Å] and angles [°] for jam0508.

| | |
|---|---|
| S(1)-O(3) | 1.4354(10) |
| S(1)-O(1) | 1.4387(10) |
| S(1)-O(2) | 1.4445(9) |
| S(1)-C(11) | 1.8154(18) |
| S(2)-O(4) | 1.4366(10) |
| S(2)-O(6) | 1.4429(10) |
| S(2)-O(5) | 1.4461(10) |
| S(2)-C(12) | 1.8203(19) |
| F(1)-C(11) | 1.3325(18) |
| F(2)-C(11) | 1.3321(19) |
| F(3)-C(11) | 1.3310(19) |
| F(4)-C(12) | 1.3300(19) |
| F(5)-C(12) | 1.3385(19) |
| F(6)-C(12) | 1.3384(18) |
| N(1)-C(4) | 1.4869(16) |
| N(1)-C(9) | 1.5159(18) |
| N(1)-C(10) | 1.5236(18) |
| N(1)-C(1) | 1.5254(17) |
| N(2)-C(8) | 1.3508(17) |
| N(2)-C(4) | 1.3517(16) |
| N(2)-C(3) | 1.4948(18) |
| C(1)-C(2) | 1.507(2) |
| C(1)-H(1A) | 0.9900 |
| C(1)-H(1B) | 0.9900 |
| C(2)-C(3) | 1.5061(19) |
| C(2)-H(2A) | 0.9900 |
| C(2)-H(2B) | 0.9900 |
| C(3)-H(3A) | 0.9900 |
| C(3)-H(3B) | 0.9900 |
| C(4)-C(5) | 1.363(2) |
| C(5)-C(6) | 1.3868(19) |
| C(5)-H(5) | 0.9500 |
| C(6)-C(7) | 1.377(2) |
| C(6)-H(6) | 0.9500 |
| C(7)-C(8) | 1.365(2) |
| C(7)-H(7) | 0.9500 |

| | |
|---|---|
| C(8)-H(8) | 0.9500 |
| C(9)-H(9A) | 0.9800 |
| C(9)-H(9B) | 0.9800 |
| C(9)-H(9C) | 0.9800 |
| C(10)-H(10A) | 0.9800 |
| C(10)-H(10B) | 0.9800 |
| C(10)-H(10C) | 0.9800 |

| | |
|---|---|
| O(3)-S(1)-O(1) | 115.17(6) |
| O(3)-S(1)-O(2) | 114.65(6) |
| O(1)-S(1)-O(2) | 115.22(6) |
| O(3)-S(1)-C(11) | 102.73(8) |
| O(1)-S(1)-C(11) | 104.03(7) |
| O(2)-S(1)-C(11) | 102.57(7) |
| O(4)-S(2)-O(6) | 115.86(7) |
| O(4)-S(2)-O(5) | 114.96(6) |
| O(6)-S(2)-O(5) | 114.24(6) |
| O(4)-S(2)-C(12) | 103.72(7) |
| O(6)-S(2)-C(12) | 102.93(7) |
| O(5)-S(2)-C(12) | 102.66(8) |
| C(4)-N(1)-C(9) | 110.25(11) |
| C(4)-N(1)-C(10) | 107.65(10) |
| C(9)-N(1)-C(10) | 108.50(10) |
| C(4)-N(1)-C(1) | 113.20(9) |
| C(9)-N(1)-C(1) | 106.96(10) |
| C(10)-N(1)-C(1) | 110.20(11) |
| C(8)-N(2)-C(4) | 119.14(12) |
| C(8)-N(2)-C(3) | 115.84(11) |
| C(4)-N(2)-C(3) | 124.97(11) |
| C(2)-C(1)-N(1) | 110.48(11) |
| C(2)-C(1)-H(1A) | 109.6 |
| N(1)-C(1)-H(1A) | 109.6 |
| C(2)-C(1)-H(1B) | 109.6 |
| N(1)-C(1)-H(1B) | 109.6 |
| H(1A)-C(1)-H(1B) | 108.1 |
| C(3)-C(2)-C(1) | 109.91(13) |
| C(3)-C(2)-H(2A) | 109.7 |
| C(1)-C(2)-H(2A) | 109.7 |

| | |
|---|---|
| C(3)-C(2)-H(2B) | 109.7 |
| C(1)-C(2)-H(2B) | 109.7 |
| H(2A)-C(2)-H(2B) | 108.2 |
| N(2)-C(3)-C(2) | 111.24(10) |
| N(2)-C(3)-H(3A) | 109.4 |
| C(2)-C(3)-H(3A) | 109.4 |
| N(2)-C(3)-H(3B) | 109.4 |
| C(2)-C(3)-H(3B) | 109.4 |
| H(3A)-C(3)-H(3B) | 108.0 |
| N(2)-C(4)-C(5) | 121.20(12) |
| N(2)-C(4)-N(1) | 119.93(12) |
| C(5)-C(4)-N(1) | 118.80(11) |
| C(4)-C(5)-C(6) | 119.64(12) |
| C(4)-C(5)-H(5) | 120.2 |
| C(6)-C(5)-H(5) | 120.2 |
| C(7)-C(6)-C(5) | 119.01(14) |
| C(7)-C(6)-H(6) | 120.5 |
| C(5)-C(6)-H(6) | 120.5 |
| C(8)-C(7)-C(6) | 119.18(13) |
| C(8)-C(7)-H(7) | 120.4 |
| C(6)-C(7)-H(7) | 120.4 |
| N(2)-C(8)-C(7) | 121.82(13) |
| N(2)-C(8)-H(8) | 119.1 |
| C(7)-C(8)-H(8) | 119.1 |
| N(1)-C(9)-H(9A) | 109.5 |
| N(1)-C(9)-H(9B) | 109.5 |
| H(9A)-C(9)-H(9B) | 109.5 |
| N(1)-C(9)-H(9C) | 109.5 |
| H(9A)-C(9)-H(9C) | 109.5 |
| H(9B)-C(9)-H(9C) | 109.5 |
| N(1)-C(10)-H(10A) | 109.5 |
| N(1)-C(10)-H(10B) | 109.5 |
| H(10A)-C(10)-H(10B) | 109.5 |
| N(1)-C(10)-H(10C) | 109.5 |
| H(10A)-C(10)-H(10C) | 109.5 |
| H(10B)-C(10)-H(10C) | 109.5 |
| F(3)-C(11)-F(2) | 107.60(13) |
| F(3)-C(11)-F(1) | 107.44(13) |

| | |
|---|---|
| F(2)-C(11)-F(1) | 107.76(15) |
| F(3)-C(11)-S(1) | 111.58(13) |
| F(2)-C(11)-S(1) | 111.64(10) |
| F(1)-C(11)-S(1) | 110.62(11) |
| F(4)-C(12)-F(6) | 107.86(13) |
| F(4)-C(12)-F(5) | 107.37(13) |
| F(6)-C(12)-F(5) | 107.05(15) |
| F(4)-C(12)-S(2) | 111.81(13) |
| F(6)-C(12)-S(2) | 111.25(11) |
| F(5)-C(12)-S(2) | 111.27(11) |

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Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for jam0508. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| S(1) | 18(1) | 20(1) | 16(1) | 2(1) | 8(1) | 2(1) |
| S(2) | 17(1) | 22(1) | 22(1) | 3(1) | 6(1) | 0(1) |
| F(1) | 60(1) | 87(1) | 33(1) | -21(1) | 32(1) | -12(1) |
| F(2) | 33(1) | 46(1) | 29(1) | -4(1) | -3(1) | -4(1) |
| F(3) | 50(1) | 22(1) | 69(1) | -15(1) | 17(1) | -1(1) |
| F(4) | 50(1) | 27(1) | 57(1) | -10(1) | 15(1) | 1(1) |
| F(5) | 29(1) | 58(1) | 33(1) | -5(1) | -3(1) | -4(1) |
| F(6) | 48(1) | 65(1) | 34(1) | -8(1) | 24(1) | -8(1) |
| O(1) | 21(1) | 36(1) | 29(1) | 6(1) | 9(1) | 8(1) |
| O(2) | 24(1) | 33(1) | 22(1) | 7(1) | 14(1) | 4(1) |
| O(3) | 28(1) | 19(1) | 31(1) | 6(1) | 12(1) | -2(1) |
| O(4) | 25(1) | 33(1) | 30(1) | 8(1) | 13(1) | -3(1) |
| O(5) | 21(1) | 21(1) | 38(1) | 6(1) | 13(1) | 2(1) |
| O(6) | 16(1) | 31(1) | 27(1) | 6(1) | 4(1) | 1(1) |
| N(1) | 16(1) | 15(1) | 17(1) | 0(1) | 7(1) | 1(1) |
| N(2) | 15(1) | 19(1) | 15(1) | 3(1) | 6(1) | 0(1) |
| C(1) | 23(1) | 14(1) | 23(1) | -4(1) | 11(1) | -3(1) |
| C(2) | 23(1) | 21(1) | 21(1) | -5(1) | 8(1) | -6(1) |
| C(3) | 18(1) | 20(1) | 18(1) | -1(1) | 5(1) | -6(1) |
| C(4) | 16(1) | 16(1) | 17(1) | 4(1) | 9(1) | 2(1) |
| C(5) | 19(1) | 18(1) | 25(1) | 0(1) | 10(1) | -1(1) |
| C(6) | 26(1) | 19(1) | 30(1) | -5(1) | 12(1) | 0(1) |
| C(7) | 23(1) | 27(1) | 30(1) | -1(1) | 14(1) | 7(1) |
| C(8) | 17(1) | 28(1) | 23(1) | 4(1) | 10(1) | 5(1) |
| C(9) | 17(1) | 21(1) | 19(1) | 1(1) | 4(1) | 4(1) |
| C(10) | 22(1) | 21(1) | 18(1) | 3(1) | 11(1) | 0(1) |
| C(11) | 30(1) | 31(1) | 26(1) | -6(1) | 11(1) | -2(1) |
| C(12) | 25(1) | 32(1) | 29(1) | -1(1) | 7(1) | -1(1) |

Table 5. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters ($Å^2$x $10^3$) for jam0508.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(1A) | 9285 | 2298 | 3383 | 23 |
| H(1B) | 8784 | 2263 | 4732 | 23 |
| H(2A) | 11816 | 2356 | 5467 | 26 |
| H(2B) | 11379 | 1712 | 5914 | 26 |
| H(3A) | 12372 | 1975 | 3622 | 23 |
| H(3B) | 13398 | 1553 | 4918 | 23 |
| H(5) | 7513 | 501 | 2189 | 24 |
| H(6) | 9116 | -156 | 1370 | 30 |
| H(7) | 12088 | 26 | 1923 | 31 |
| H(8) | 13372 | 848 | 3243 | 26 |
| H(9A) | 6881 | 1719 | 1745 | 30 |
| H(9B) | 6135 | 1886 | 2916 | 30 |
| H(9C) | 6028 | 1215 | 2379 | 30 |
| H(10A) | 7561 | 767 | 4560 | 29 |
| H(10B) | 7587 | 1391 | 5308 | 29 |
| H(10C) | 9366 | 1032 | 5625 | 29 |

Table 6. Torsion angles [°] for jam0508.

| | |
|---|---|
| C(4)-N(1)-C(1)-C(2) | -47.20(16) |
| C(9)-N(1)-C(1)-C(2) | -168.83(12) |
| C(10)-N(1)-C(1)-C(2) | 73.42(13) |
| N(1)-C(1)-C(2)-C(3) | 62.86(15) |
| C(8)-N(2)-C(3)-C(2) | -162.63(13) |
| C(4)-N(2)-C(3)-C(2) | 20.06(19) |
| C(1)-C(2)-C(3)-N(2) | -47.82(16) |
| C(8)-N(2)-C(4)-C(5) | 0.7(2) |
| C(3)-N(2)-C(4)-C(5) | 177.91(14) |
| C(8)-N(2)-C(4)-N(1) | 177.64(13) |
| C(3)-N(2)-C(4)-N(1) | -5.1(2) |
| C(9)-N(1)-C(4)-N(2) | 138.29(13) |
| C(10)-N(1)-C(4)-N(2) | -103.53(14) |
| C(1)-N(1)-C(4)-N(2) | 18.53(18) |
| C(9)-N(1)-C(4)-C(5) | -44.68(16) |
| C(10)-N(1)-C(4)-C(5) | 73.50(16) |
| C(1)-N(1)-C(4)-C(5) | -164.44(13) |
| N(2)-C(4)-C(5)-C(6) | -0.9(2) |
| N(1)-C(4)-C(5)-C(6) | -177.86(13) |
| C(4)-C(5)-C(6)-C(7) | 0.6(2) |
| C(5)-C(6)-C(7)-C(8) | -0.2(2) |
| C(4)-N(2)-C(8)-C(7) | -0.2(2) |
| C(3)-N(2)-C(8)-C(7) | -177.71(14) |
| C(6)-C(7)-C(8)-N(2) | 0.0(2) |
| O(3)-S(1)-C(11)-F(3) | 176.39(10) |
| O(1)-S(1)-C(11)-F(3) | 56.00(11) |
| O(2)-S(1)-C(11)-F(3) | -64.38(11) |
| O(3)-S(1)-C(11)-F(2) | -63.17(12) |
| O(1)-S(1)-C(11)-F(2) | 176.45(11) |
| O(2)-S(1)-C(11)-F(2) | 56.07(12) |
| O(3)-S(1)-C(11)-F(1) | 56.83(13) |
| O(1)-S(1)-C(11)-F(1) | -63.55(13) |
| O(2)-S(1)-C(11)-F(1) | 176.07(11) |
| O(4)-S(2)-C(12)-F(4) | 57.86(12) |
| O(6)-S(2)-C(12)-F(4) | -63.25(11) |
| O(5)-S(2)-C(12)-F(4) | 177.86(10) |

| | |
|---|---|
| O(4)-S(2)-C(12)-F(6) | 178.52(11) |
| O(6)-S(2)-C(12)-F(6) | 57.42(13) |
| O(5)-S(2)-C(12)-F(6) | -61.48(12) |
| O(4)-S(2)-C(12)-F(5) | -62.20(13) |
| O(6)-S(2)-C(12)-F(5) | 176.69(11) |
| O(5)-S(2)-C(12)-F(5) | 57.80(12) |

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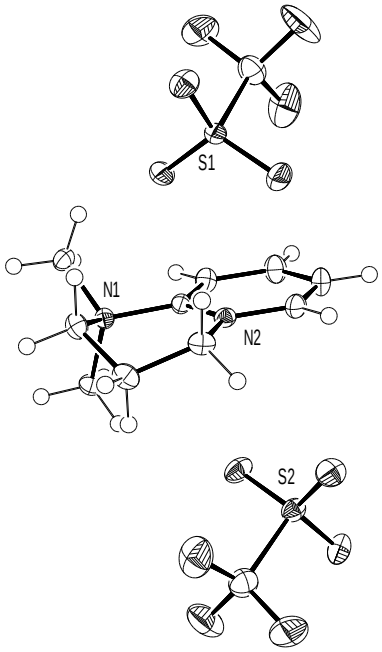
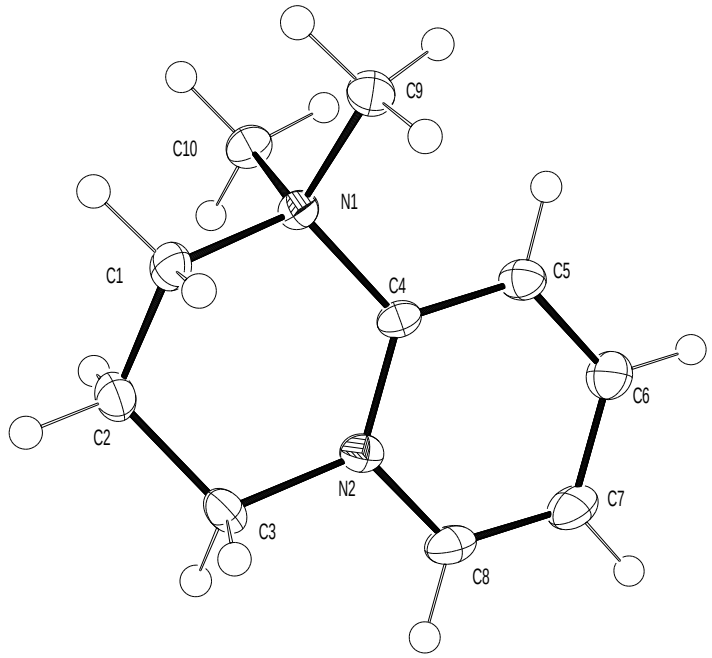
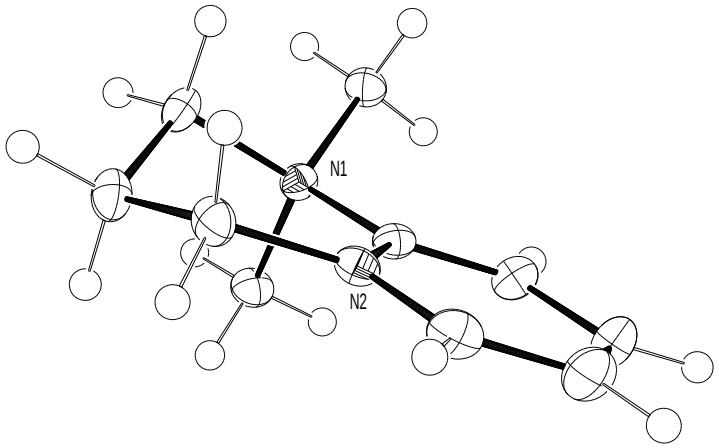
Symmetry transformations used to generate equivalent atoms:




C11

Above – contents of the asymmetric unit.


C9
C10
N1
C1
C4
C5
C6
C2
N2
C7
C3
C8


N1
N2

# Appendix 2

Crystallographic Data for 2-Dimethylamino-pyrimidine Disalt **4.30**

Table 1. Crystal data and structure refinement for jam0408.

| | | |
|---|---|---|
| Identification code | jam0408 | |
| Empirical formula | C11 H15 F6 N3 O6 S2 | |
| Formula weight | 463.38 | |
| Temperature | 123(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Monoclinic | |
| Space group | P21/c | |
| Unit cell dimensions | a = 9.3365(8) Å | α= 90°. |
| | b = 14.1848(12) Å | β= 95.355(8)°. |
| | c = 13.2684(10) Å | γ = 90°. |
| Volume | 1749.5(2) $Å^3$ | |
| Z | 4 | |
| Density (calculated) | 1.759 $Mg/m^3$ | |
| Absorption coefficient | 0.403 $mm^{-1}$ | |
| F(000) | 944 | |
| Crystal size | 0.35 x 0.30 x 0.12 $mm^3$ | |
| Theta range for data collection | 2.87 to 30.72°. | |
| Index ranges | -13<=h<=13, -19<=k<=20, -18<=l<=17 | |
| Reflections collected | 16299 | |
| Independent reflections | 4957 [R(int) = 0.0249] | |
| Completeness to theta = 26.00° | 99.6 % | |
| Absorption correction | None | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 4957 / 0 / 255 | |
| Goodness-of-fit on $F^2$ | 1.113 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0389, wR2 = 0.1072 | |
| R indices (all data) | R1 = 0.0474, wR2 = 0.1110 | |
| Largest diff. peak and hole | 0.648 and -0.515 $e.Å^{-3}$ | |

Table 2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for jam0408. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| S(1) | 8282(1) | 1362(1) | 1098(1) | 17(1) |
| S(2) | 2220(1) | 1274(1) | 5195(1) | 20(1) |
| F(1) | 5734(1) | 886(1) | 274(1) | 31(1) |
| F(2) | 6948(1) | -265(1) | 948(1) | 39(1) |
| F(3) | 5996(1) | 750(1) | 1883(1) | 50(1) |
| F(4) | 2389(1) | 3103(1) | 5449(1) | 45(1) |
| F(5) | 4362(1) | 2375(1) | 5840(1) | 38(1) |
| F(6) | 2708(2) | 2307(1) | 6833(1) | 46(1) |
| O(1) | 8851(1) | 1132(1) | 163(1) | 31(1) |
| O(2) | 9129(1) | 1049(1) | 1997(1) | 23(1) |
| O(3) | 7729(1) | 2307(1) | 1160(1) | 26(1) |
| O(4) | 2563(1) | 1440(1) | 4175(1) | 29(1) |
| O(5) | 2968(2) | 509(1) | 5710(1) | 42(1) |
| O(6) | 705(1) | 1342(1) | 5308(1) | 34(1) |
| N(1) | 7575(1) | 769(1) | -2471(1) | 15(1) |
| N(2) | 8370(1) | 2240(1) | -1636(1) | 18(1) |
| N(3) | 5948(1) | 1801(1) | -1820(1) | 21(1) |
| C(1) | 9047(2) | 754(1) | -2864(1) | 20(1) |
| C(2) | 10172(2) | 1063(1) | -2043(1) | 23(1) |
| C(3) | 9914(2) | 2071(1) | -1761(1) | 25(1) |
| C(4) | 7286(2) | 1658(1) | -1948(1) | 15(1) |
| C(5) | 5605(2) | 2613(1) | -1382(1) | 26(1) |
| C(6) | 6633(2) | 3270(1) | -1070(1) | 27(1) |
| C(7) | 8029(2) | 3060(1) | -1195(1) | 24(1) |
| C(8) | 7439(2) | -48(1) | -1752(1) | 20(1) |
| C(9) | 6449(2) | 648(1) | -3360(1) | 22(1) |
| C(10) | 6664(2) | 643(1) | 1054(1) | 22(1) |
| C(11) | 2947(2) | 2315(1) | 5867(1) | 24(1) |

Table 3. Bond lengths [Å] and angles [°] for jam0408.

| | |
|---|---|
| S(1)-O(1) | 1.4330(12) |
| S(1)-O(2) | 1.4382(11) |
| S(1)-O(3) | 1.4410(11) |
| S(1)-C(10) | 1.8190(16) |
| S(2)-O(5) | 1.4290(13) |
| S(2)-O(6) | 1.4388(13) |
| S(2)-O(4) | 1.4394(12) |
| S(2)-C(11) | 1.8232(16) |
| F(1)-C(10) | 1.3319(17) |
| F(2)-C(10) | 1.3256(18) |
| F(3)-C(10) | 1.3232(19) |
| F(4)-C(11) | 1.3313(19) |
| F(5)-C(11) | 1.328(2) |
| F(6)-C(11) | 1.3218(19) |
| N(1)-C(4) | 1.4769(17) |
| N(1)-C(1) | 1.5142(19) |
| N(1)-C(8) | 1.5144(17) |
| N(1)-C(9) | 1.5144(18) |
| N(2)-C(4) | 1.3405(18) |
| N(2)-C(7) | 1.3536(19) |
| N(2)-C(3) | 1.486(2) |
| N(3)-C(4) | 1.2932(19) |
| N(3)-C(5) | 1.3421(19) |
| C(1)-C(2) | 1.507(2) |
| C(1)-H(1A) | 0.9900 |
| C(1)-H(1B) | 0.9900 |
| C(2)-C(3) | 1.503(2) |
| C(2)-H(2A) | 0.9900 |
| C(2)-H(2B) | 0.9900 |
| C(3)-H(3A) | 0.9900 |
| C(3)-H(3B) | 0.9900 |
| C(5)-C(6) | 1.373(3) |
| C(5)-H(5) | 0.9500 |
| C(6)-C(7) | 1.361(3) |
| C(6)-H(6) | 0.9500 |
| C(7)-H(7) | 0.9500 |

| | |
|---|---|
| C(8)-H(8A) | 0.9800 |
| C(8)-H(8B) | 0.9800 |
| C(8)-H(8C) | 0.9800 |
| C(9)-H(9A) | 0.9800 |
| C(9)-H(9B) | 0.9800 |
| C(9)-H(9C) | 0.9800 |

| | |
|---|---|
| O(1)-S(1)-O(2) | 115.37(8) |
| O(1)-S(1)-O(3) | 115.12(8) |
| O(2)-S(1)-O(3) | 114.34(7) |
| O(1)-S(1)-C(10) | 102.58(7) |
| O(2)-S(1)-C(10) | 104.19(7) |
| O(3)-S(1)-C(10) | 102.84(7) |
| O(5)-S(2)-O(6) | 116.27(9) |
| O(5)-S(2)-O(4) | 115.59(9) |
| O(6)-S(2)-O(4) | 113.22(8) |
| O(5)-S(2)-C(11) | 103.78(8) |
| O(6)-S(2)-C(11) | 102.69(8) |
| O(4)-S(2)-C(11) | 102.77(7) |
| C(4)-N(1)-C(1) | 112.68(11) |
| C(4)-N(1)-C(8) | 109.06(11) |
| C(1)-N(1)-C(8) | 109.80(11) |
| C(4)-N(1)-C(9) | 108.58(11) |
| C(1)-N(1)-C(9) | 108.49(11) |
| C(8)-N(1)-C(9) | 108.14(11) |
| C(4)-N(2)-C(7) | 117.48(13) |
| C(4)-N(2)-C(3) | 125.33(12) |
| C(7)-N(2)-C(3) | 117.17(13) |
| C(4)-N(3)-C(5) | 117.63(14) |
| C(2)-C(1)-N(1) | 109.91(12) |
| C(2)-C(1)-H(1A) | 109.7 |
| N(1)-C(1)-H(1A) | 109.7 |
| C(2)-C(1)-H(1B) | 109.7 |
| N(1)-C(1)-H(1B) | 109.7 |
| H(1A)-C(1)-H(1B) | 108.2 |
| C(3)-C(2)-C(1) | 109.87(12) |
| C(3)-C(2)-H(2A) | 109.7 |
| C(1)-C(2)-H(2A) | 109.7 |

| | |
|---|---|
| C(3)-C(2)-H(2B) | 109.7 |
| C(1)-C(2)-H(2B) | 109.7 |
| H(2A)-C(2)-H(2B) | 108.2 |
| N(2)-C(3)-C(2) | 111.16(12) |
| N(2)-C(3)-H(3A) | 109.4 |
| C(2)-C(3)-H(3A) | 109.4 |
| N(2)-C(3)-H(3B) | 109.4 |
| C(2)-C(3)-H(3B) | 109.4 |
| H(3A)-C(3)-H(3B) | 108.0 |
| N(3)-C(4)-N(2) | 125.00(13) |
| N(3)-C(4)-N(1) | 114.64(12) |
| N(2)-C(4)-N(1) | 120.35(12) |
| N(3)-C(5)-C(6) | 121.62(16) |
| N(3)-C(5)-H(5) | 119.2 |
| C(6)-C(5)-H(5) | 119.2 |
| C(7)-C(6)-C(5) | 117.74(14) |
| C(7)-C(6)-H(6) | 121.1 |
| C(5)-C(6)-H(6) | 121.1 |
| N(2)-C(7)-C(6) | 120.44(14) |
| N(2)-C(7)-H(7) | 119.8 |
| C(6)-C(7)-H(7) | 119.8 |
| N(1)-C(8)-H(8A) | 109.5 |
| N(1)-C(8)-H(8B) | 109.5 |
| H(8A)-C(8)-H(8B) | 109.5 |
| N(1)-C(8)-H(8C) | 109.5 |
| H(8A)-C(8)-H(8C) | 109.5 |
| H(8B)-C(8)-H(8C) | 109.5 |
| N(1)-C(9)-H(9A) | 109.5 |
| N(1)-C(9)-H(9B) | 109.5 |
| H(9A)-C(9)-H(9B) | 109.5 |
| N(1)-C(9)-H(9C) | 109.5 |
| H(9A)-C(9)-H(9C) | 109.5 |
| H(9B)-C(9)-H(9C) | 109.5 |
| F(3)-C(10)-F(2) | 108.25(14) |
| F(3)-C(10)-F(1) | 107.03(14) |
| F(2)-C(10)-F(1) | 106.99(12) |
| F(3)-C(10)-S(1) | 111.29(10) |
| F(2)-C(10)-S(1) | 111.97(11) |

| | |
|---|---|
| F(1)-C(10)-S(1) | 111.07(11) |
| F(6)-C(11)-F(5) | 106.54(14) |
| F(6)-C(11)-F(4) | 108.42(14) |
| F(5)-C(11)-F(4) | 106.66(14) |
| F(6)-C(11)-S(2) | 112.26(12) |
| F(5)-C(11)-S(2) | 111.36(11) |
| F(4)-C(11)-S(2) | 111.31(11) |

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Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for jam0408. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| S(1) | 19(1) | 14(1) | 17(1) | -1(1) | 0(1) | -1(1) |
| S(2) | 19(1) | 19(1) | 22(1) | 1(1) | 2(1) | -2(1) |
| F(1) | 25(1) | 27(1) | 38(1) | -3(1) | -14(1) | 2(1) |
| F(2) | 37(1) | 16(1) | 59(1) | 4(1) | -18(1) | -6(1) |
| F(3) | 39(1) | 80(1) | 33(1) | -14(1) | 16(1) | -30(1) |
| F(4) | 59(1) | 20(1) | 53(1) | -5(1) | -14(1) | 9(1) |
| F(5) | 27(1) | 38(1) | 49(1) | -1(1) | 0(1) | -11(1) |
| F(6) | 54(1) | 60(1) | 24(1) | -12(1) | 7(1) | -15(1) |
| O(1) | 34(1) | 38(1) | 24(1) | -7(1) | 10(1) | -7(1) |
| O(2) | 25(1) | 18(1) | 25(1) | -1(1) | -8(1) | 2(1) |
| O(3) | 31(1) | 14(1) | 30(1) | -1(1) | -7(1) | 3(1) |
| O(4) | 36(1) | 27(1) | 26(1) | -4(1) | 10(1) | -3(1) |
| O(5) | 49(1) | 22(1) | 53(1) | 10(1) | -17(1) | 2(1) |
| O(6) | 20(1) | 51(1) | 31(1) | -2(1) | 5(1) | -7(1) |
| N(1) | 17(1) | 13(1) | 16(1) | -1(1) | 3(1) | 1(1) |
| N(2) | 21(1) | 14(1) | 17(1) | 2(1) | -3(1) | -1(1) |
| N(3) | 21(1) | 21(1) | 21(1) | -2(1) | 5(1) | 3(1) |
| C(1) | 19(1) | 20(1) | 24(1) | 3(1) | 9(1) | 4(1) |
| C(2) | 15(1) | 21(1) | 33(1) | 8(1) | 3(1) | 3(1) |
| C(3) | 15(1) | 21(1) | 37(1) | 5(1) | -4(1) | -2(1) |
| C(4) | 19(1) | 13(1) | 13(1) | -1(1) | 2(1) | 0(1) |
| C(5) | 31(1) | 26(1) | 21(1) | 1(1) | 9(1) | 10(1) |
| C(6) | 46(1) | 18(1) | 16(1) | -2(1) | 4(1) | 8(1) |
| C(7) | 40(1) | 14(1) | 17(1) | -1(1) | -6(1) | -1(1) |
| C(8) | 26(1) | 14(1) | 22(1) | 3(1) | 6(1) | -2(1) |
| C(9) | 24(1) | 21(1) | 20(1) | -5(1) | -3(1) | -1(1) |
| C(10) | 22(1) | 22(1) | 21(1) | 0(1) | -2(1) | -3(1) |
| C(11) | 25(1) | 23(1) | 24(1) | -1(1) | 0(1) | 2(1) |

Table 5. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters ($Å^2$x $10^3$) for jam0408.

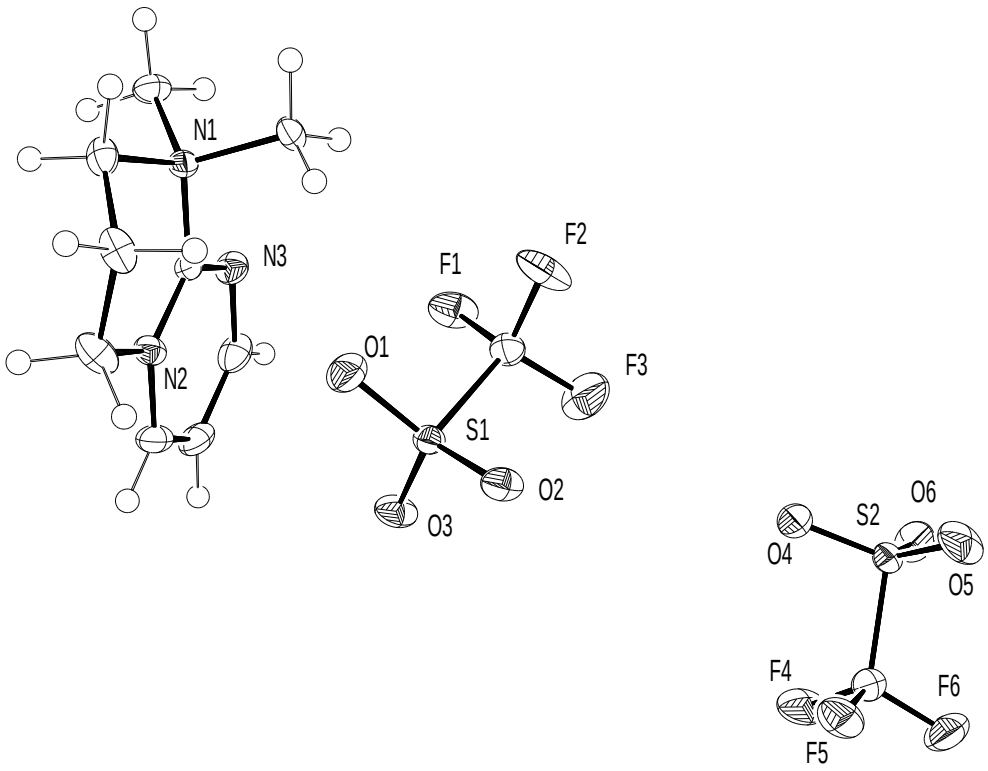
| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(1A) | 9266 | 108 | -3089 | 24 |
| H(1B) | 9054 | 1181 | -3454 | 24 |
| H(2A) | 10133 | 654 | -1441 | 28 |
| H(2B) | 11140 | 1001 | -2284 | 28 |
| H(3A) | 10498 | 2223 | -1120 | 30 |
| H(3B) | 10222 | 2493 | -2294 | 30 |
| H(5) | 4627 | 2736 | -1284 | 31 |
| H(6) | 6379 | 3850 | -778 | 32 |
| H(7) | 8768 | 3492 | -970 | 29 |
| H(8A) | 8187 | 2 | -1185 | 30 |
| H(8B) | 7552 | -643 | -2111 | 30 |
| H(8C) | 6490 | -31 | -1493 | 30 |
| H(9A) | 5496 | 596 | -3111 | 33 |
| H(9B) | 6653 | 75 | -3733 | 33 |
| H(9C) | 6468 | 1195 | -3809 | 33 |

Table 6. Torsion angles [°] for jam0408.

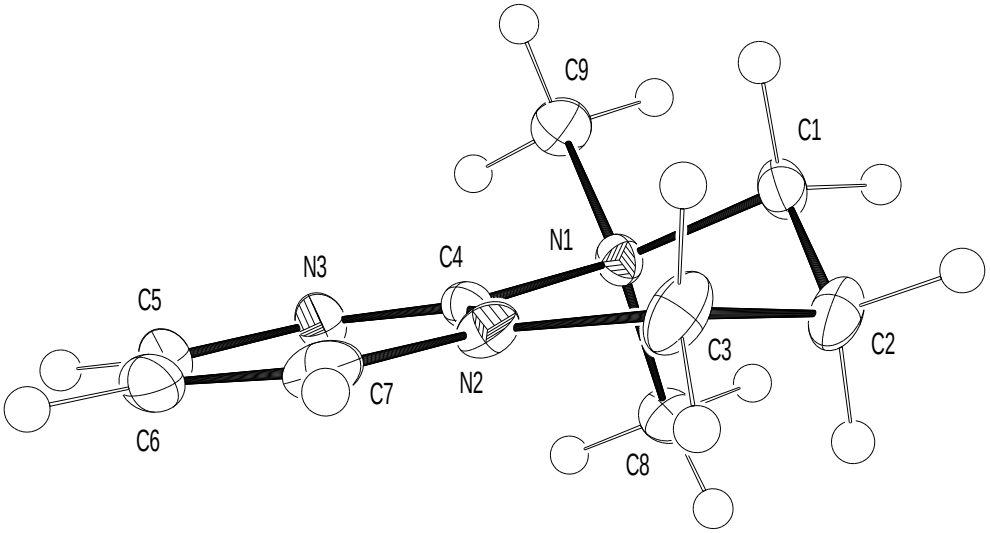
| | |
|---|---|
| C(4)-N(1)-C(1)-C(2) | 48.51(15) |
| C(8)-N(1)-C(1)-C(2) | -73.26(14) |
| C(9)-N(1)-C(1)-C(2) | 168.76(11) |
| N(1)-C(1)-C(2)-C(3) | -64.06(15) |
| C(4)-N(2)-C(3)-C(2) | -14.2(2) |
| C(7)-N(2)-C(3)-C(2) | 167.49(13) |
| C(1)-C(2)-C(3)-N(2) | 45.60(17) |
| C(5)-N(3)-C(4)-N(2) | 3.0(2) |
| C(5)-N(3)-C(4)-N(1) | -177.61(12) |
| C(7)-N(2)-C(4)-N(3) | -3.1(2) |
| C(3)-N(2)-C(4)-N(3) | 178.65(13) |
| C(7)-N(2)-C(4)-N(1) | 177.60(12) |
| C(3)-N(2)-C(4)-N(1) | -0.7(2) |
| C(1)-N(1)-C(4)-N(3) | 163.88(12) |
| C(8)-N(1)-C(4)-N(3) | -73.93(15) |
| C(9)-N(1)-C(4)-N(3) | 43.68(16) |
| C(1)-N(1)-C(4)-N(2) | -16.74(17) |
| C(8)-N(1)-C(4)-N(2) | 105.45(14) |
| C(9)-N(1)-C(4)-N(2) | -136.93(13) |
| C(4)-N(3)-C(5)-C(6) | -0.5(2) |
| N(3)-C(5)-C(6)-C(7) | -1.8(2) |
| C(4)-N(2)-C(7)-C(6) | 0.5(2) |
| C(3)-N(2)-C(7)-C(6) | 178.93(13) |
| C(5)-C(6)-C(7)-N(2) | 1.7(2) |
| O(1)-S(1)-C(10)-F(3) | 178.63(12) |
| O(2)-S(1)-C(10)-F(3) | 58.02(13) |
| O(3)-S(1)-C(10)-F(3) | -61.56(13) |
| O(1)-S(1)-C(10)-F(2) | 57.32(13) |
| O(2)-S(1)-C(10)-F(2) | -63.29(12) |
| O(3)-S(1)-C(10)-F(2) | 177.13(11) |
| O(1)-S(1)-C(10)-F(1) | -62.23(12) |
| O(2)-S(1)-C(10)-F(1) | 177.16(10) |
| O(3)-S(1)-C(10)-F(1) | 57.58(12) |
| O(5)-S(2)-C(11)-F(6) | -60.63(15) |
| O(6)-S(2)-C(11)-F(6) | 60.87(14) |
| O(4)-S(2)-C(11)-F(6) | 178.60(12) |

| | |
|---|---|
| O(5)-S(2)-C(11)-F(5) | 58.74(14) |
| O(6)-S(2)-C(11)-F(5) | -179.77(11) |
| O(4)-S(2)-C(11)-F(5) | -62.03(13) |
| O(5)-S(2)-C(11)-F(4) | 177.63(13) |
| O(6)-S(2)-C(11)-F(4) | -60.88(14) |
| O(4)-S(2)-C(11)-F(4) | 56.86(14) |

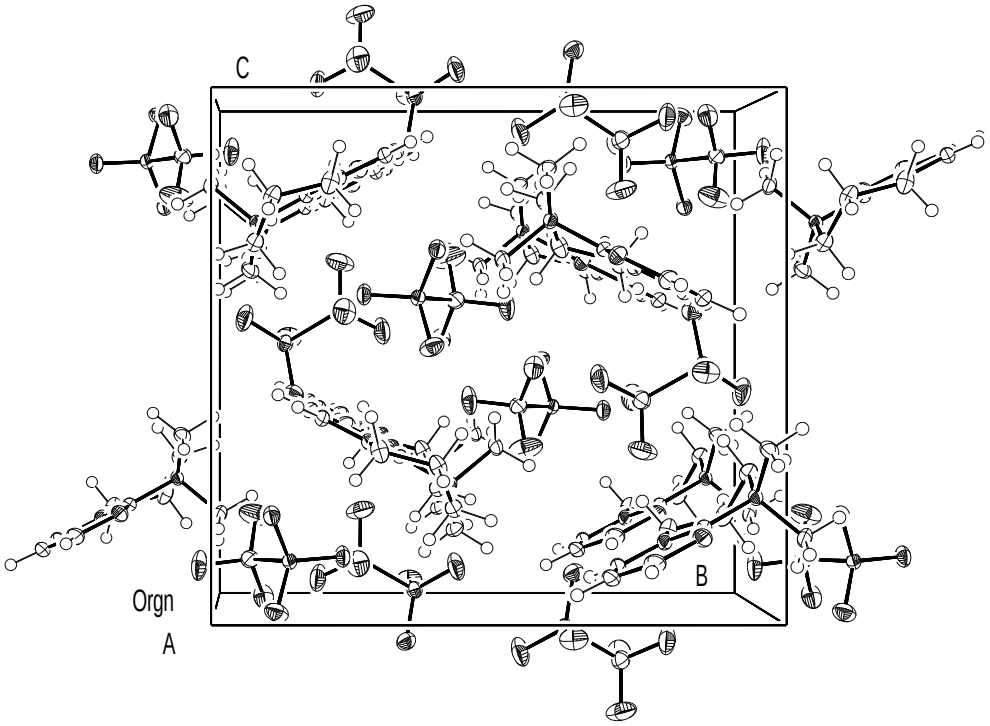
Symmetry transformations used to generate equivalent atoms:




Above – View of the asymmetric unit.




Above – view of the cation




Above – packing diagram of unit cell.

# Appendix 3

Crystallographic Data for 1-(Dimethylammoniumpropyl)-2-hydroxypyridinium Trifluoromethanesulfonate **5.28**

Table 1. Crystal data and structure refinement for jam0808.

| | | |
|---|---|---|
| Identification code | jam0808 | |
| Empirical formula | C12 H18 F6 N2 O7 S2 | |
| Formula weight | 480.40 | |
| Temperature | 123(2) K | |
| Wavelength | 1.54180 Å | |
| Crystal system | Triclinic | |
| Space group | P-1 | |
| Unit cell dimensions | a = 8.3711(8) Å | α= 66.405(9)°. |
| | b = 10.5740(10) Å | β= 71.879(9)°. |
| | c = 12.9188(13) Å | γ = 85.019(8)°. |
| Volume | 995.01(17) $Å^3$ | |
| Z | 2 | |
| Density (calculated) | 1.603 $Mg/m^3$ | |
| Absorption coefficient | 3.315 $mm^{-1}$ | |
| F(000) | 492 | |
| Crystal size | 1 x 0.1 x 0.07 $mm^3$ | |
| Theta range for data collection | 3.92 to 62.50°. | |
| Index ranges | -8<=h<=9, -10<=k<=12, -14<=l<=14 | |
| Reflections collected | 7146 | |
| Independent reflections | 3073 [R(int) = 0.0454] | |
| Completeness to theta = 62.50° | 97.0 % | |
| Absorption correction | Analytical | |
| Max. and min. transmission | 0.785 and 0.255 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 3073 / 0 / 270 | |
| Goodness-of-fit on $F^2$ | 1.117 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0668, wR2 = 0.2089 | |
| R indices (all data) | R1 = 0.0728, wR2 = 0.2175 | |
| Extinction coefficient | 0.0028(11) | |
| Largest diff. peak and hole | 0.533 and -0.565 $e.Å^{-3}$ | |

Table 2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for jam0808. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| S(1) | 7679(1) | 7350(1) | 12689(1) | 36(1) |
| F(1) | 5547(3) | 8427(3) | 14035(2) | 68(1) |
| F(2) | 7637(4) | 7539(3) | 14650(2) | 81(1) |
| F(3) | 5684(4) | 6229(3) | 14854(2) | 81(1) |
| S(2) | 12725(1) | 2077(1) | 11624(1) | 37(1) |
| F(4) | 11015(3) | 2264(2) | 13636(2) | 59(1) |
| F(5) | 12751(3) | 630(3) | 13803(2) | 69(1) |
| F(6) | 10444(3) | 409(2) | 13489(2) | 54(1) |
| O(1) | 11468(3) | 5478(2) | 10527(2) | 40(1) |
| O(2) | 8564(3) | 8666(2) | 12050(2) | 48(1) |
| O(3) | 6351(3) | 7137(3) | 12287(2) | 54(1) |
| O(4) | 8764(3) | 6213(2) | 12945(2) | 44(1) |
| O(5) | 11394(3) | 2837(2) | 11172(2) | 43(1) |
| O(6) | 13206(3) | 913(2) | 11314(2) | 48(1) |
| O(7) | 14055(3) | 2950(3) | 11495(2) | 48(1) |
| N(1) | 11974(3) | 7528(3) | 8969(2) | 34(1) |
| N(2) | 12277(4) | 6811(3) | 12183(2) | 35(1) |
| C(1) | 12006(4) | 6143(3) | 9358(3) | 35(1) |
| C(2) | 12545(4) | 5522(3) | 8549(3) | 37(1) |
| C(3) | 13044(5) | 6329(4) | 7358(3) | 42(1) |
| C(4) | 13046(5) | 7760(4) | 6965(3) | 42(1) |
| C(5) | 12505(5) | 8332(4) | 7792(3) | 38(1) |
| C(6) | 11247(4) | 8213(3) | 9806(3) | 35(1) |
| C(7) | 12578(4) | 8710(3) | 10164(3) | 37(1) |
| C(8) | 13435(4) | 7570(3) | 10935(3) | 36(1) |
| C(9) | 12043(5) | 7628(4) | 12924(3) | 41(1) |
| C(10) | 12885(5) | 5425(3) | 12765(3) | 45(1) |
| C(11) | 6574(6) | 7388(4) | 14132(3) | 50(1) |
| C(12) | 11680(5) | 1311(4) | 13224(3) | 45(1) |

Table 3. Bond lengths [Å] and angles [°] for jam0808.

| | |
|---|---|
| S(1)-O(2) | 1.431(3) |
| S(1)-O(3) | 1.433(3) |
| S(1)-O(4) | 1.435(3) |
| S(1)-C(11) | 1.820(4) |
| F(1)-C(11) | 1.325(5) |
| F(2)-C(11) | 1.322(5) |
| F(3)-C(11) | 1.318(4) |
| S(2)-O(6) | 1.431(3) |
| S(2)-O(7) | 1.434(2) |
| S(2)-O(5) | 1.450(3) |
| S(2)-C(12) | 1.831(4) |
| F(4)-C(12) | 1.325(4) |
| F(5)-C(12) | 1.325(4) |
| F(6)-C(12) | 1.337(4) |
| O(1)-C(1) | 1.326(4) |
| O(1)-H(1) | 0.8400 |
| N(1)-C(1) | 1.345(4) |
| N(1)-C(5) | 1.358(4) |
| N(1)-C(6) | 1.485(4) |
| N(2)-C(10) | 1.483(4) |
| N(2)-C(9) | 1.489(5) |
| N(2)-C(8) | 1.520(4) |
| N(2)-H(1N) | 0.81(4) |
| C(1)-C(2) | 1.390(5) |
| C(2)-C(3) | 1.372(5) |
| C(2)-H(2) | 0.9500 |
| C(3)-C(4) | 1.390(5) |
| C(3)-H(3) | 0.9500 |
| C(4)-C(5) | 1.371(5) |
| C(4)-H(4) | 0.9500 |
| C(5)-H(5) | 0.9500 |
| C(6)-C(7) | 1.531(4) |
| C(6)-H(6A) | 0.9900 |
| C(6)-H(6B) | 0.9900 |
| C(7)-C(8) | 1.515(5) |
| C(7)-H(7A) | 0.9900 |

| | |
|---|---|
| C(7)-H(7B) | 0.9900 |
| C(8)-H(8A) | 0.9900 |
| C(8)-H(8B) | 0.9900 |
| C(9)-H(9A) | 0.9800 |
| C(9)-H(9B) | 0.9800 |
| C(9)-H(9C) | 0.9800 |
| C(10)-H(10A) | 0.9800 |
| C(10)-H(10B) | 0.9800 |
| C(10)-H(10C) | 0.9800 |

| | |
|---|---|
| O(2)-S(1)-O(3) | 116.22(17) |
| O(2)-S(1)-O(4) | 113.45(15) |
| O(3)-S(1)-O(4) | 114.49(16) |
| O(2)-S(1)-C(11) | 103.00(16) |
| O(3)-S(1)-C(11) | 103.45(18) |
| O(4)-S(1)-C(11) | 104.06(18) |
| O(6)-S(2)-O(7) | 117.04(17) |
| O(6)-S(2)-O(5) | 113.59(15) |
| O(7)-S(2)-O(5) | 113.46(15) |
| O(6)-S(2)-C(12) | 104.12(16) |
| O(7)-S(2)-C(12) | 104.03(17) |
| O(5)-S(2)-C(12) | 102.39(17) |
| C(1)-O(1)-H(1) | 109.5 |
| C(1)-N(1)-C(5) | 120.7(3) |
| C(1)-N(1)-C(6) | 121.0(3) |
| C(5)-N(1)-C(6) | 118.2(3) |
| C(10)-N(2)-C(9) | 110.7(3) |
| C(10)-N(2)-C(8) | 111.4(3) |
| C(9)-N(2)-C(8) | 111.9(3) |
| C(10)-N(2)-H(1N) | 112(3) |
| C(9)-N(2)-H(1N) | 103(3) |
| C(8)-N(2)-H(1N) | 107(3) |
| O(1)-C(1)-N(1) | 114.8(3) |
| O(1)-C(1)-C(2) | 125.3(3) |
| N(1)-C(1)-C(2) | 119.9(3) |
| C(3)-C(2)-C(1) | 119.6(3) |
| C(3)-C(2)-H(2) | 120.2 |
| C(1)-C(2)-H(2) | 120.2 |

| | |
|---|---|
| C(2)-C(3)-C(4) | 120.1(3) |
| C(2)-C(3)-H(3) | 119.9 |
| C(4)-C(3)-H(3) | 119.9 |
| C(5)-C(4)-C(3) | 118.5(3) |
| C(5)-C(4)-H(4) | 120.8 |
| C(3)-C(4)-H(4) | 120.8 |
| N(1)-C(5)-C(4) | 121.2(3) |
| N(1)-C(5)-H(5) | 119.4 |
| C(4)-C(5)-H(5) | 119.4 |
| N(1)-C(6)-C(7) | 113.1(3) |
| N(1)-C(6)-H(6A) | 109.0 |
| C(7)-C(6)-H(6A) | 109.0 |
| N(1)-C(6)-H(6B) | 109.0 |
| C(7)-C(6)-H(6B) | 109.0 |
| H(6A)-C(6)-H(6B) | 107.8 |
| C(8)-C(7)-C(6) | 114.9(3) |
| C(8)-C(7)-H(7A) | 108.5 |
| C(6)-C(7)-H(7A) | 108.5 |
| C(8)-C(7)-H(7B) | 108.5 |
| C(6)-C(7)-H(7B) | 108.5 |
| H(7A)-C(7)-H(7B) | 107.5 |
| C(7)-C(8)-N(2) | 112.8(3) |
| C(7)-C(8)-H(8A) | 109.0 |
| N(2)-C(8)-H(8A) | 109.0 |
| C(7)-C(8)-H(8B) | 109.0 |
| N(2)-C(8)-H(8B) | 109.0 |
| H(8A)-C(8)-H(8B) | 107.8 |
| N(2)-C(9)-H(9A) | 109.5 |
| N(2)-C(9)-H(9B) | 109.5 |
| H(9A)-C(9)-H(9B) | 109.5 |
| N(2)-C(9)-H(9C) | 109.5 |
| H(9A)-C(9)-H(9C) | 109.5 |
| H(9B)-C(9)-H(9C) | 109.5 |
| N(2)-C(10)-H(10A) | 109.5 |
| N(2)-C(10)-H(10B) | 109.5 |
| H(10A)-C(10)-H(10B) | 109.5 |
| N(2)-C(10)-H(10C) | 109.5 |
| H(10A)-C(10)-H(10C) | 109.5 |

| | |
|---|---|
| H(10B)-C(10)-H(10C) | 109.5 |
| F(3)-C(11)-F(2) | 106.8(3) |
| F(3)-C(11)-F(1) | 108.4(4) |
| F(2)-C(11)-F(1) | 107.5(3) |
| F(3)-C(11)-S(1) | 111.4(3) |
| F(2)-C(11)-S(1) | 111.3(3) |
| F(1)-C(11)-S(1) | 111.2(3) |
| F(5)-C(12)-F(4) | 108.2(3) |
| F(5)-C(12)-F(6) | 107.2(3) |
| F(4)-C(12)-F(6) | 107.8(3) |
| F(5)-C(12)-S(2) | 111.0(3) |
| F(4)-C(12)-S(2) | 111.5(3) |
| F(6)-C(12)-S(2) | 110.9(2) |

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Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for jam0808. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| S(1) | 41(1) | 31(1) | 37(1) | -11(1) | -16(1) | -6(1) |
| F(1) | 79(2) | 59(2) | 57(2) | -25(1) | -6(1) | 7(1) |
| F(2) | 110(2) | 98(2) | 57(2) | -38(2) | -41(2) | -4(2) |
| F(3) | 99(2) | 59(2) | 51(2) | -5(1) | 8(1) | -24(2) |
| S(2) | 43(1) | 29(1) | 41(1) | -9(1) | -16(1) | -8(1) |
| F(4) | 74(2) | 55(2) | 53(1) | -28(1) | -15(1) | -5(1) |
| F(5) | 75(2) | 73(2) | 53(1) | -6(1) | -37(1) | 3(1) |
| F(6) | 64(2) | 40(1) | 48(1) | -6(1) | -11(1) | -18(1) |
| O(1) | 53(2) | 27(1) | 36(1) | -5(1) | -16(1) | -8(1) |
| O(2) | 54(2) | 33(1) | 46(2) | -7(1) | -10(1) | -6(1) |
| O(3) | 51(2) | 66(2) | 59(2) | -31(1) | -29(1) | 2(1) |
| O(4) | 45(2) | 30(1) | 58(2) | -14(1) | -21(1) | -2(1) |
| O(5) | 48(1) | 32(1) | 45(1) | -5(1) | -21(1) | -8(1) |
| O(6) | 58(2) | 35(1) | 53(2) | -19(1) | -13(1) | -6(1) |
| O(7) | 48(2) | 39(2) | 62(2) | -17(1) | -24(1) | -8(1) |
| N(1) | 38(2) | 30(2) | 35(2) | -9(1) | -16(1) | -7(1) |
| N(2) | 39(2) | 28(2) | 40(2) | -8(1) | -19(1) | -5(1) |
| C(1) | 36(2) | 29(2) | 39(2) | -8(2) | -16(2) | -6(1) |
| C(2) | 42(2) | 29(2) | 42(2) | -12(2) | -15(2) | -9(2) |
| C(3) | 48(2) | 39(2) | 43(2) | -17(2) | -18(2) | -4(2) |
| C(4) | 48(2) | 38(2) | 36(2) | -8(2) | -13(2) | -9(2) |
| C(5) | 43(2) | 34(2) | 37(2) | -8(2) | -17(2) | -7(2) |
| C(6) | 42(2) | 29(2) | 35(2) | -10(2) | -15(2) | -3(2) |
| C(7) | 47(2) | 27(2) | 36(2) | -7(1) | -17(2) | -7(2) |
| C(8) | 33(2) | 32(2) | 42(2) | -10(2) | -14(2) | -10(1) |
| C(9) | 46(2) | 38(2) | 42(2) | -13(2) | -17(2) | -5(2) |
| C(10) | 56(2) | 29(2) | 53(2) | -10(2) | -28(2) | 2(2) |
| C(11) | 68(3) | 41(2) | 38(2) | -11(2) | -14(2) | -9(2) |
| C(12) | 51(2) | 40(2) | 46(2) | -15(2) | -20(2) | -2(2) |

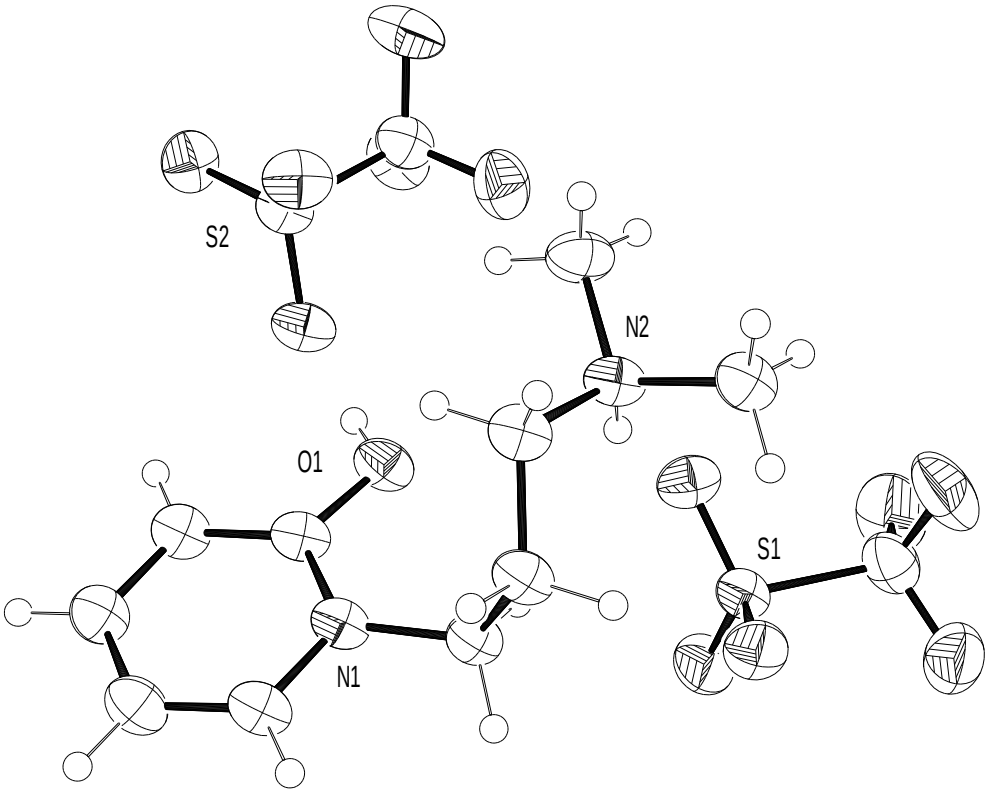
Table 5. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters ($Å^2$x $10^3$) for jam0808.

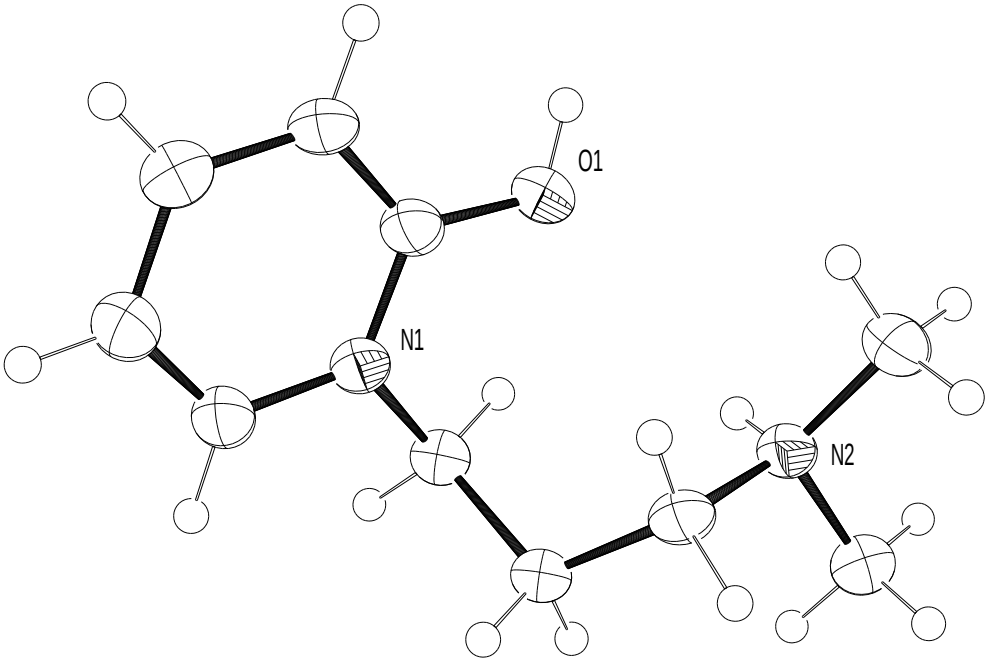
| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(1) | 11441 | 4623 | 10702 | 60 |
| H(2) | 12567 | 4544 | 8820 | 45 |
| H(3) | 13388 | 5909 | 6801 | 50 |
| H(4) | 13413 | 8328 | 6143 | 51 |
| H(5) | 12501 | 9309 | 7536 | 46 |
| H(6A) | 10439 | 7557 | 10528 | 42 |
| H(6B) | 10618 | 9014 | 9434 | 42 |
| H(7A) | 13447 | 9287 | 9435 | 44 |
| H(7B) | 12038 | 9304 | 10592 | 44 |
| H(8A) | 14424 | 7972 | 10979 | 43 |
| H(8B) | 13846 | 6900 | 10562 | 43 |
| H(9A) | 11240 | 7131 | 13704 | 62 |
| H(9B) | 11611 | 8529 | 12536 | 62 |
| H(9C) | 13125 | 7762 | 13019 | 62 |
| H(10A) | 13986 | 5525 | 12849 | 68 |
| H(10B) | 12989 | 4890 | 12280 | 68 |
| H(10C) | 12084 | 4944 | 13549 | 68 |
| H(1N) | 11350(50) | 6770(40) | 12130(30) | 36(10) |

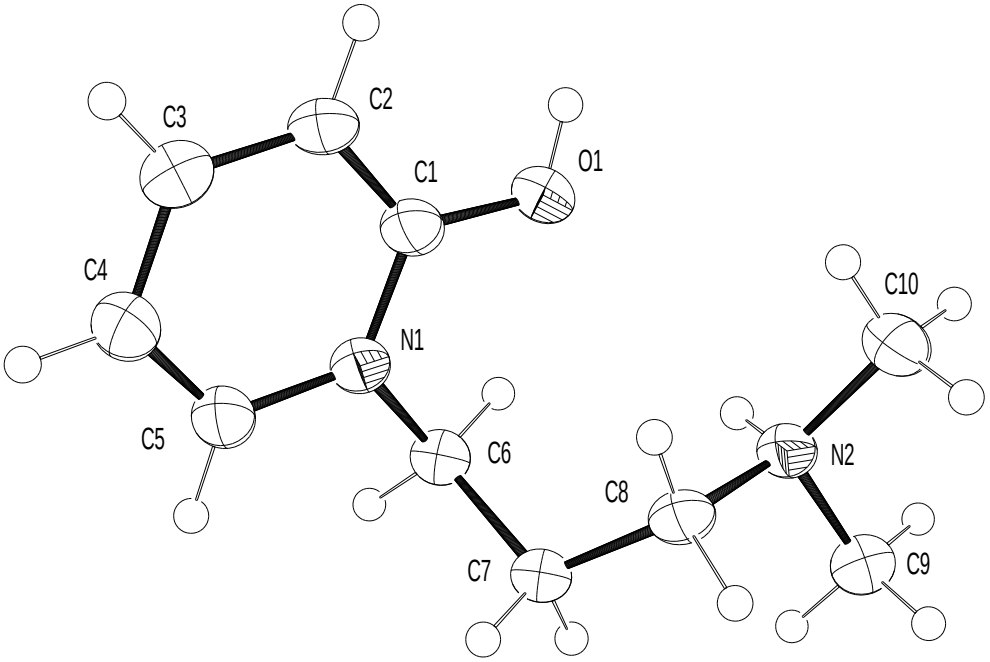
Table 6. Torsion angles [°] for jam0808.

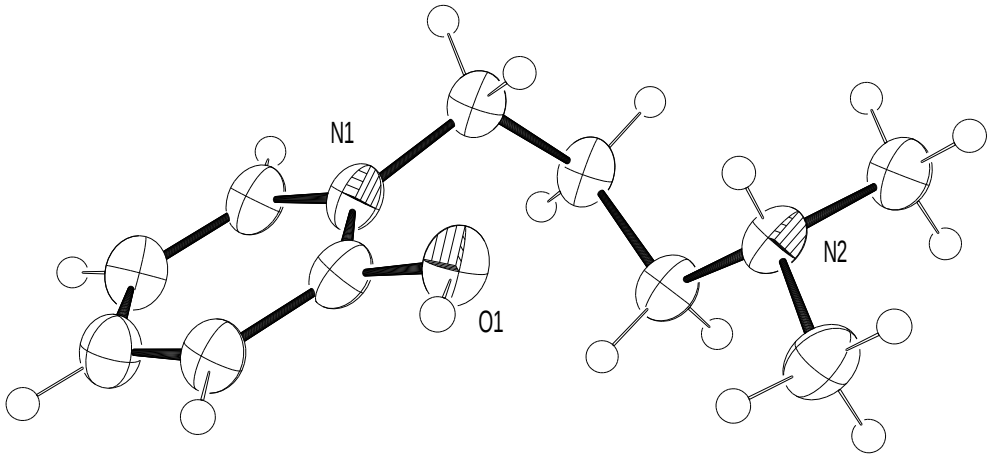
| | |
|---|---|
| C(5)-N(1)-C(1)-O(1) | 179.5(3) |
| C(6)-N(1)-C(1)-O(1) | -4.9(4) |
| C(5)-N(1)-C(1)-C(2) | -1.7(5) |
| C(6)-N(1)-C(1)-C(2) | 173.9(3) |
| O(1)-C(1)-C(2)-C(3) | 178.8(3) |
| N(1)-C(1)-C(2)-C(3) | 0.1(5) |
| C(1)-C(2)-C(3)-C(4) | 1.5(5) |
| C(2)-C(3)-C(4)-C(5) | -1.5(5) |
| C(1)-N(1)-C(5)-C(4) | 1.7(5) |
| C(6)-N(1)-C(5)-C(4) | -174.0(3) |
| C(3)-C(4)-C(5)-N(1) | -0.1(5) |
| C(1)-N(1)-C(6)-C(7) | 97.7(3) |
| C(5)-N(1)-C(6)-C(7) | -86.5(4) |
| N(1)-C(6)-C(7)-C(8) | -68.9(4) |
| C(6)-C(7)-C(8)-N(2) | -70.6(4) |
| C(10)-N(2)-C(8)-C(7) | 158.8(3) |
| C(9)-N(2)-C(8)-C(7) | -76.7(3) |
| O(2)-S(1)-C(11)-F(3) | -179.5(3) |
| O(3)-S(1)-C(11)-F(3) | 59.0(3) |
| O(4)-S(1)-C(11)-F(3) | -60.9(3) |
| O(2)-S(1)-C(11)-F(2) | -60.4(3) |
| O(3)-S(1)-C(11)-F(2) | 178.1(3) |
| O(4)-S(1)-C(11)-F(2) | 58.2(3) |
| O(2)-S(1)-C(11)-F(1) | 59.4(3) |
| O(3)-S(1)-C(11)-F(1) | -62.0(3) |
| O(4)-S(1)-C(11)-F(1) | 178.0(2) |
| O(6)-S(2)-C(12)-F(5) | 65.2(3) |
| O(7)-S(2)-C(12)-F(5) | -57.9(3) |
| O(5)-S(2)-C(12)-F(5) | -176.3(2) |
| O(6)-S(2)-C(12)-F(4) | -174.1(2) |
| O(7)-S(2)-C(12)-F(4) | 62.8(3) |
| O(5)-S(2)-C(12)-F(4) | -55.6(3) |
| O(6)-S(2)-C(12)-F(6) | -53.9(3) |
| O(7)-S(2)-C(12)-F(6) | -177.0(3) |
| O(5)-S(2)-C(12)-F(6) | 64.6(3) |

Symmetry transformations used to generate equivalent atoms:




O1
N1
N2


C3
C2
C1
O1
C4
C10
N1
C5
C6
N2
C8
C7
C9


N1
O1
N2

# Appendix 4

Crystallographic Data for 2,4,6-*Tris*-(dimethyl-amino)pyridine Trisalt **6.47**

Table 1. Crystal data and structure refinement for jam0309.

| | | |
|---|---|---|
| Identification code | jam0309 | |
| Empirical formula | C19 H29 F9 N4 O9 S3 | |
| Formula weight | 724.64 | |
| Temperature | 123(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Monoclinic | |
| Space group | $P2_1/n$ | |
| Unit cell dimensions | a = 10.6076(3) Å | α= 90°. |
| | b = 14.4559(4) Å | β= 93.899(3)°. |
| | c = 18.4307(6) Å | γ = 90°. |
| Volume | 2819.67(14) $Å^3$ | |
| Z | 4 | |
| Density (calculated) | 1.707 $Mg/m^3$ | |
| Absorption coefficient | 0.378 $mm^{-1}$ | |
| F(000) | 1488 | |
| Crystal size | 0.15 x 0.15 x 0.03 $mm^3$ | |
| Theta range for data collection | 2.57 to 26.00°. | |
| Index ranges | -13<=h<=13, -17<=k<=17, -22<=l<=22 | |
| Reflections collected | 21967 | |
| Independent reflections | 21967 [R(int) = 0.0000] | |
| Completeness to theta = 26.00° | 99.8 % | |
| Absorption correction | Semi-empirical from equivalents | |
| Max. and min. transmission | 1.00000 and 0.99713 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 21967 / 0 / 404 | |
| Goodness-of-fit on $F^2$ | 0.833 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0490, wR2 = 0.0958 | |
| R indices (all data) | R1 = 0.1024, wR2 = 0.1039 | |
| Largest diff. peak and hole | 0.359 and -0.379 $e.Å^{-3}$ | |

Table 2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for jam0309. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| S(1) | 7512(1) | 897(1) | -327(1) | 19(1) |
| S(2) | 12451(1) | 4287(1) | 1913(1) | 18(1) |
| S(3) | 7620(1) | 2223(1) | 3785(1) | 23(1) |
| F(1) | 5952(1) | 1768(1) | -1248(1) | 38(1) |
| F(2) | 7853(1) | 2264(1) | -1240(1) | 49(1) |
| F(3) | 6701(1) | 2612(1) | -363(1) | 51(1) |
| F(4) | 12120(2) | 4283(1) | 500(1) | 74(1) |
| F(5) | 13237(1) | 5415(1) | 936(1) | 39(1) |
| F(6) | 11226(1) | 5425(1) | 1008(1) | 43(1) |
| F(7) | 5412(1) | 3024(1) | 3720(1) | 44(1) |
| F(8) | 6630(1) | 3431(1) | 4629(1) | 55(1) |
| F(9) | 5724(1) | 2100(1) | 4620(1) | 56(1) |
| O(1) | 6412(1) | 644(1) | 44(1) | 27(1) |
| O(2) | 8562(1) | 1210(1) | 148(1) | 29(1) |
| O(3) | 7829(1) | 269(1) | -890(1) | 24(1) |
| O(4) | 13623(1) | 3798(1) | 1856(1) | 35(1) |
| O(5) | 12514(1) | 5039(1) | 2417(1) | 21(1) |
| O(6) | 11335(1) | 3717(1) | 1924(1) | 34(1) |
| O(7) | 7998(1) | 2968(1) | 3337(1) | 44(1) |
| O(8) | 8487(1) | 1969(1) | 4384(1) | 42(1) |
| O(9) | 7088(1) | 1444(1) | 3382(1) | 37(1) |
| N(1) | 7423(2) | 4039(1) | 1044(1) | 16(1) |
| N(2) | 7340(2) | 1272(1) | 1653(1) | 13(1) |
| N(3) | 4993(1) | 1330(1) | 1546(1) | 15(1) |
| N(4) | 9676(1) | 1280(1) | 1837(1) | 13(1) |
| C(1) | 7401(2) | 3165(1) | 1274(1) | 14(1) |
| C(2) | 6257(2) | 2684(1) | 1332(1) | 16(1) |
| C(3) | 6254(2) | 1777(1) | 1494(1) | 11(1) |
| C(4) | 8452(2) | 1756(1) | 1628(1) | 12(1) |
| C(5) | 8514(2) | 2662(1) | 1467(1) | 14(1) |
| C(6) | 6252(2) | 4518(1) | 802(1) | 21(1) |
| C(7) | 8635(2) | 4484(1) | 910(1) | 21(1) |
| C(8) | 5004(2) | 281(1) | 1554(1) | 17(1) |

| | | | | |
|---|---|---|---|---|
| C(9) | 6085(2) | -54(1) | 2048(1) | 16(1) |
| C(10) | 7289(2) | 227(1) | 1730(1) | 16(1) |
| C(11) | 8468(2) | -99(1) | 2160(1) | 18(1) |
| C(12) | 9598(2) | 229(1) | 1796(1) | 18(1) |
| C(13) | 4105(2) | 1607(1) | 892(1) | 22(1) |
| C(14) | 4456(2) | 1685(1) | 2236(1) | 23(1) |
| C(15) | 10692(2) | 1557(1) | 1340(1) | 19(1) |
| C(16) | 10121(2) | 1578(1) | 2604(1) | 20(1) |
| C(17) | 6989(2) | 1932(2) | -818(1) | 28(1) |
| C(18) | 12255(2) | 4874(2) | 1038(1) | 31(1) |
| C(19) | 6287(2) | 2708(2) | 4218(1) | 28(1) |

Table 3. Bond lengths [Å] and angles [°] for jam0309.

| | |
|---|---|
| S(1)-O(3) | 1.4351(14) |
| S(1)-O(1) | 1.4386(14) |
| S(1)-O(2) | 1.4428(14) |
| S(1)-C(17) | 1.816(2) |
| S(2)-O(5) | 1.4286(13) |
| S(2)-O(4) | 1.4397(14) |
| S(2)-O(6) | 1.4438(14) |
| S(2)-C(18) | 1.821(2) |
| S(3)-O(7) | 1.4306(16) |
| S(3)-O(8) | 1.4361(16) |
| S(3)-O(9) | 1.4434(15) |
| S(3)-C(19) | 1.812(2) |
| F(1)-C(17) | 1.332(2) |
| F(2)-C(17) | 1.331(2) |
| F(3)-C(17) | 1.342(2) |
| F(4)-C(18) | 1.310(3) |
| F(5)-C(18) | 1.326(2) |
| F(6)-C(18) | 1.349(3) |
| F(7)-C(19) | 1.340(2) |
| F(8)-C(19) | 1.327(3) |
| F(9)-C(19) | 1.319(2) |
| N(1)-C(1) | 1.333(2) |
| N(1)-C(6) | 1.465(2) |
| N(1)-C(7) | 1.473(2) |
| N(2)-C(4) | 1.375(2) |
| N(2)-C(3) | 1.378(2) |
| N(2)-C(10) | 1.519(2) |
| N(3)-C(3) | 1.494(2) |
| N(3)-C(14) | 1.517(3) |
| N(3)-C(8) | 1.517(2) |
| N(3)-C(13) | 1.532(2) |
| N(4)-C(4) | 1.497(2) |
| N(4)-C(15) | 1.515(2) |
| N(4)-C(16) | 1.522(2) |
| N(4)-C(12) | 1.524(2) |
| C(1)-C(2) | 1.409(3) |

| | |
|---|---|
| C(1)-C(5) | 1.411(3) |
| C(2)-C(3) | 1.345(3) |
| C(2)-H(2) | 0.9500 |
| C(4)-C(5) | 1.346(2) |
| C(5)-H(5) | 0.9500 |
| C(6)-H(6A) | 0.9800 |
| C(6)-H(6B) | 0.9800 |
| C(6)-H(6C) | 0.9800 |
| C(7)-H(7A) | 0.9800 |
| C(7)-H(7B) | 0.9800 |
| C(7)-H(7C) | 0.9800 |
| C(8)-C(9) | 1.495(3) |
| C(8)-H(8A) | 0.9900 |
| C(8)-H(8B) | 0.9900 |
| C(9)-C(10) | 1.496(3) |
| C(9)-H(9A) | 0.9900 |
| C(9)-H(9B) | 0.9900 |
| C(10)-C(11) | 1.510(3) |
| C(10)-H(10) | 1.0000 |
| C(11)-C(12) | 1.490(3) |
| 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(13A) | 0.9800 |
| C(13)-H(13B) | 0.9800 |
| C(13)-H(13C) | 0.9800 |
| C(14)-H(14A) | 0.9800 |
| C(14)-H(14B) | 0.9800 |
| C(14)-H(14C) | 0.9800 |
| C(15)-H(15A) | 0.9800 |
| C(15)-H(15B) | 0.9800 |
| C(15)-H(15C) | 0.9800 |
| C(16)-H(16A) | 0.9800 |
| C(16)-H(16B) | 0.9800 |
| C(16)-H(16C) | 0.9800 |

| | |
|---|---|
| O(3)-S(1)-O(1) | 114.84(9) |

| | |
|---|---|
| O(3)-S(1)-O(2) | 115.37(9) |
| O(1)-S(1)-O(2) | 114.14(9) |
| O(3)-S(1)-C(17) | 103.81(10) |
| O(1)-S(1)-C(17) | 102.50(10) |
| O(2)-S(1)-C(17) | 103.94(10) |
| O(5)-S(2)-O(4) | 114.75(9) |
| O(5)-S(2)-O(6) | 115.28(9) |
| O(4)-S(2)-O(6) | 115.69(9) |
| O(5)-S(2)-C(18) | 102.59(9) |
| O(4)-S(2)-C(18) | 102.21(11) |
| O(6)-S(2)-C(18) | 103.58(10) |
| O(7)-S(3)-O(8) | 116.54(10) |
| O(7)-S(3)-O(9) | 113.95(11) |
| O(8)-S(3)-O(9) | 113.83(10) |
| O(7)-S(3)-C(19) | 102.73(10) |
| O(8)-S(3)-C(19) | 103.78(11) |
| O(9)-S(3)-C(19) | 103.79(10) |
| C(1)-N(1)-C(6) | 120.80(16) |
| C(1)-N(1)-C(7) | 120.05(16) |
| C(6)-N(1)-C(7) | 118.30(15) |
| C(4)-N(2)-C(3) | 115.46(15) |
| C(4)-N(2)-C(10) | 123.07(15) |
| C(3)-N(2)-C(10) | 120.78(16) |
| C(3)-N(3)-C(14) | 107.23(14) |
| C(3)-N(3)-C(8) | 115.18(14) |
| C(14)-N(3)-C(8) | 109.48(15) |
| C(3)-N(3)-C(13) | 110.02(15) |
| C(14)-N(3)-C(13) | 109.03(15) |
| C(8)-N(3)-C(13) | 105.80(14) |
| C(4)-N(4)-C(15) | 111.33(15) |
| C(4)-N(4)-C(16) | 108.53(14) |
| C(15)-N(4)-C(16) | 107.40(14) |
| C(4)-N(4)-C(12) | 113.70(14) |
| C(15)-N(4)-C(12) | 105.80(15) |
| C(16)-N(4)-C(12) | 109.89(15) |
| N(1)-C(1)-C(2) | 121.70(18) |
| N(1)-C(1)-C(5) | 122.40(18) |
| C(2)-C(1)-C(5) | 115.89(17) |

| | |
|---|---|
| C(3)-C(2)-C(1) | 120.88(18) |
| C(3)-C(2)-H(2) | 119.6 |
| C(1)-C(2)-H(2) | 119.6 |
| C(2)-C(3)-N(2) | 123.28(18) |
| C(2)-C(3)-N(3) | 116.80(16) |
| N(2)-C(3)-N(3) | 119.81(16) |
| C(5)-C(4)-N(2) | 123.91(17) |
| C(5)-C(4)-N(4) | 116.70(17) |
| N(2)-C(4)-N(4) | 119.27(16) |
| C(4)-C(5)-C(1) | 120.29(18) |
| C(4)-C(5)-H(5) | 119.9 |
| C(1)-C(5)-H(5) | 119.9 |
| N(1)-C(6)-H(6A) | 109.5 |
| N(1)-C(6)-H(6B) | 109.5 |
| H(6A)-C(6)-H(6B) | 109.5 |
| N(1)-C(6)-H(6C) | 109.5 |
| H(6A)-C(6)-H(6C) | 109.5 |
| H(6B)-C(6)-H(6C) | 109.5 |
| N(1)-C(7)-H(7A) | 109.5 |
| N(1)-C(7)-H(7B) | 109.5 |
| H(7A)-C(7)-H(7B) | 109.5 |
| N(1)-C(7)-H(7C) | 109.5 |
| H(7A)-C(7)-H(7C) | 109.5 |
| H(7B)-C(7)-H(7C) | 109.5 |
| C(9)-C(8)-N(3) | 109.57(15) |
| C(9)-C(8)-H(8A) | 109.8 |
| N(3)-C(8)-H(8A) | 109.8 |
| C(9)-C(8)-H(8B) | 109.8 |
| N(3)-C(8)-H(8B) | 109.8 |
| H(8A)-C(8)-H(8B) | 108.2 |
| C(8)-C(9)-C(10) | 108.27(17) |
| C(8)-C(9)-H(9A) | 110.0 |
| C(10)-C(9)-H(9A) | 110.0 |
| C(8)-C(9)-H(9B) | 110.0 |
| C(10)-C(9)-H(9B) | 110.0 |
| H(9A)-C(9)-H(9B) | 108.4 |
| C(9)-C(10)-C(11) | 114.11(17) |
| C(9)-C(10)-N(2) | 110.06(16) |

| | |
|---|---|
| C(11)-C(10)-N(2) | 109.02(15) |
| C(9)-C(10)-H(10) | 107.8 |
| C(11)-C(10)-H(10) | 107.8 |
| N(2)-C(10)-H(10) | 107.8 |
| C(12)-C(11)-C(10) | 109.12(17) |
| C(12)-C(11)-H(11A) | 109.9 |
| C(10)-C(11)-H(11A) | 109.9 |
| C(12)-C(11)-H(11B) | 109.9 |
| C(10)-C(11)-H(11B) | 109.9 |
| H(11A)-C(11)-H(11B) | 108.3 |
| C(11)-C(12)-N(4) | 109.76(16) |
| C(11)-C(12)-H(12A) | 109.7 |
| N(4)-C(12)-H(12A) | 109.7 |
| C(11)-C(12)-H(12B) | 109.7 |
| N(4)-C(12)-H(12B) | 109.7 |
| H(12A)-C(12)-H(12B) | 108.2 |
| N(3)-C(13)-H(13A) | 109.5 |
| N(3)-C(13)-H(13B) | 109.5 |
| H(13A)-C(13)-H(13B) | 109.5 |
| N(3)-C(13)-H(13C) | 109.5 |
| H(13A)-C(13)-H(13C) | 109.5 |
| H(13B)-C(13)-H(13C) | 109.5 |
| N(3)-C(14)-H(14A) | 109.5 |
| N(3)-C(14)-H(14B) | 109.5 |
| H(14A)-C(14)-H(14B) | 109.5 |
| N(3)-C(14)-H(14C) | 109.5 |
| H(14A)-C(14)-H(14C) | 109.5 |
| H(14B)-C(14)-H(14C) | 109.5 |
| N(4)-C(15)-H(15A) | 109.5 |
| N(4)-C(15)-H(15B) | 109.5 |
| H(15A)-C(15)-H(15B) | 109.5 |
| N(4)-C(15)-H(15C) | 109.5 |
| H(15A)-C(15)-H(15C) | 109.5 |
| H(15B)-C(15)-H(15C) | 109.5 |
| N(4)-C(16)-H(16A) | 109.5 |
| N(4)-C(16)-H(16B) | 109.5 |
| H(16A)-C(16)-H(16B) | 109.5 |
| N(4)-C(16)-H(16C) | 109.5 |

| | |
|---|---|
| H(16A)-C(16)-H(16C) | 109.5 |
| H(16B)-C(16)-H(16C) | 109.5 |
| F(2)-C(17)-F(1) | 106.92(19) |
| F(2)-C(17)-F(3) | 107.19(19) |
| F(1)-C(17)-F(3) | 106.74(18) |
| F(2)-C(17)-S(1) | 112.93(16) |
| F(1)-C(17)-S(1) | 111.24(15) |
| F(3)-C(17)-S(1) | 111.48(16) |
| F(4)-C(18)-F(5) | 108.9(2) |
| F(4)-C(18)-F(6) | 107.89(19) |
| F(5)-C(18)-F(6) | 106.69(18) |
| F(4)-C(18)-S(2) | 111.44(16) |
| F(5)-C(18)-S(2) | 110.92(16) |
| F(6)-C(18)-S(2) | 110.85(17) |
| F(9)-C(19)-F(8) | 108.7(2) |
| F(9)-C(19)-F(7) | 107.03(18) |
| F(8)-C(19)-F(7) | 105.85(19) |
| F(9)-C(19)-S(3) | 112.75(16) |
| F(8)-C(19)-S(3) | 111.35(16) |
| F(7)-C(19)-S(3) | 110.85(16) |

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Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for jam0309. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| S(1) | 19(1) | 21(1) | 18(1) | -2(1) | -1(1) | 2(1) |
| S(2) | 22(1) | 15(1) | 19(1) | 0(1) | 0(1) | 1(1) |
| S(3) | 24(1) | 17(1) | 29(1) | 1(1) | 8(1) | 1(1) |
| F(1) | 32(1) | 41(1) | 39(1) | 8(1) | -13(1) | 4(1) |
| F(2) | 43(1) | 47(1) | 58(1) | 26(1) | 13(1) | -6(1) |
| F(3) | 78(1) | 24(1) | 49(1) | -6(1) | 4(1) | 14(1) |
| F(4) | 157(2) | 44(1) | 18(1) | -6(1) | -7(1) | -7(1) |
| F(5) | 39(1) | 41(1) | 38(1) | 16(1) | 14(1) | -1(1) |
| F(6) | 29(1) | 46(1) | 52(1) | 26(1) | -11(1) | 0(1) |
| F(7) | 31(1) | 46(1) | 55(1) | 11(1) | 6(1) | 12(1) |
| F(8) | 63(1) | 50(1) | 55(1) | -32(1) | 14(1) | 4(1) |
| F(9) | 52(1) | 52(1) | 68(1) | 28(1) | 39(1) | 10(1) |
| O(1) | 24(1) | 35(1) | 24(1) | 4(1) | 9(1) | -2(1) |
| O(2) | 24(1) | 41(1) | 23(1) | -7(1) | -7(1) | -5(1) |
| O(3) | 27(1) | 26(1) | 19(1) | -10(1) | 3(1) | 6(1) |
| O(4) | 35(1) | 31(1) | 39(1) | 6(1) | 12(1) | 19(1) |
| O(5) | 26(1) | 15(1) | 21(1) | -8(1) | 2(1) | 0(1) |
| O(6) | 37(1) | 29(1) | 35(1) | 9(1) | -9(1) | -20(1) |
| O(7) | 46(1) | 22(1) | 68(1) | 19(1) | 33(1) | 10(1) |
| O(8) | 35(1) | 41(1) | 47(1) | -1(1) | -12(1) | 9(1) |
| O(9) | 42(1) | 25(1) | 42(1) | -13(1) | 1(1) | -3(1) |
| N(1) | 14(1) | 11(1) | 23(1) | 4(1) | 2(1) | -1(1) |
| N(2) | 16(1) | 10(1) | 13(1) | -1(1) | 2(1) | 2(1) |
| N(3) | 14(1) | 13(1) | 19(1) | -2(1) | 4(1) | -3(1) |
| N(4) | 11(1) | 12(1) | 16(1) | -2(1) | 1(1) | 2(1) |
| C(1) | 21(1) | 11(1) | 12(1) | -3(1) | 2(1) | -1(1) |
| C(2) | 14(1) | 13(1) | 20(1) | -2(1) | -1(1) | 2(1) |
| C(3) | 7(1) | 15(1) | 13(1) | -3(1) | 2(1) | 0(1) |
| C(4) | 7(1) | 17(1) | 11(1) | -2(1) | 0(1) | 0(1) |
| C(5) | 11(1) | 12(1) | 18(1) | -3(1) | 0(1) | -2(1) |
| C(6) | 19(1) | 13(1) | 30(1) | 0(1) | 1(1) | 1(1) |
| C(7) | 19(1) | 14(1) | 29(1) | 2(1) | 1(1) | -4(1) |
| C(8) | 17(1) | 10(1) | 26(1) | 1(1) | 4(1) | -4(1) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C(9) | 17(1) | 12(1) | 21(1) | 1(1) | 2(1) | 0(1) |
| C(10) | 24(1) | 7(1) | 19(1) | 0(1) | -1(1) | -2(1) |
| C(11) | 18(1) | 10(1) | 24(1) | 3(1) | -2(1) | 2(1) |
| C(12) | 24(1) | 9(1) | 22(1) | -1(1) | -3(1) | 1(1) |
| C(13) | 14(1) | 23(1) | 29(1) | 0(1) | -6(1) | 4(1) |
| C(14) | 21(1) | 20(1) | 29(1) | -1(1) | 7(1) | 1(1) |
| C(15) | 9(1) | 23(1) | 24(1) | 0(1) | 4(1) | -1(1) |
| C(16) | 16(1) | 26(1) | 16(1) | -4(1) | -5(1) | 3(1) |
| C(17) | 33(2) | 26(1) | 26(1) | -1(1) | 6(1) | 3(1) |
| C(18) | 43(2) | 24(1) | 25(2) | 0(1) | 2(1) | -8(1) |
| C(19) | 27(1) | 27(1) | 31(1) | 3(1) | 7(1) | 1(1) |

Table 5. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters ($Å^2$x $10^3$) for jam0309.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(2) | 5478 | 3005 | 1257 | 19 |
| H(5) | 9310 | 2965 | 1482 | 16 |
| H(6A) | 5882 | 4224 | 358 | 31 |
| H(6B) | 6438 | 5168 | 702 | 31 |
| H(6C) | 5654 | 4482 | 1183 | 31 |
| H(7A) | 9223 | 4413 | 1340 | 31 |
| H(7B) | 8495 | 5143 | 809 | 31 |
| H(7C) | 8993 | 4190 | 491 | 31 |
| H(8A) | 4199 | 47 | 1725 | 21 |
| H(8B) | 5090 | 44 | 1056 | 21 |
| H(9A) | 6049 | -735 | 2095 | 20 |
| H(9B) | 6041 | 221 | 2537 | 20 |
| H(10) | 7289 | -47 | 1232 | 20 |
| H(11A) | 8486 | 148 | 2661 | 21 |
| H(11B) | 8472 | -783 | 2187 | 21 |
| H(12A) | 9537 | 30 | 1281 | 22 |
| H(12B) | 10371 | -46 | 2038 | 22 |
| H(13A) | 3869 | 2259 | 935 | 33 |
| H(13B) | 3343 | 1222 | 879 | 33 |
| H(13C) | 4534 | 1516 | 443 | 33 |
| H(14A) | 5053 | 1555 | 2653 | 35 |
| H(14B) | 3651 | 1376 | 2305 | 35 |
| H(14C) | 4318 | 2354 | 2196 | 35 |
| H(15A) | 10360 | 1506 | 832 | 28 |
| H(15B) | 11424 | 1148 | 1423 | 28 |
| H(15C) | 10949 | 2198 | 1442 | 28 |
| H(16A) | 10228 | 2251 | 2618 | 30 |
| H(16B) | 10929 | 1278 | 2746 | 30 |
| H(16C) | 9491 | 1395 | 2942 | 30 |

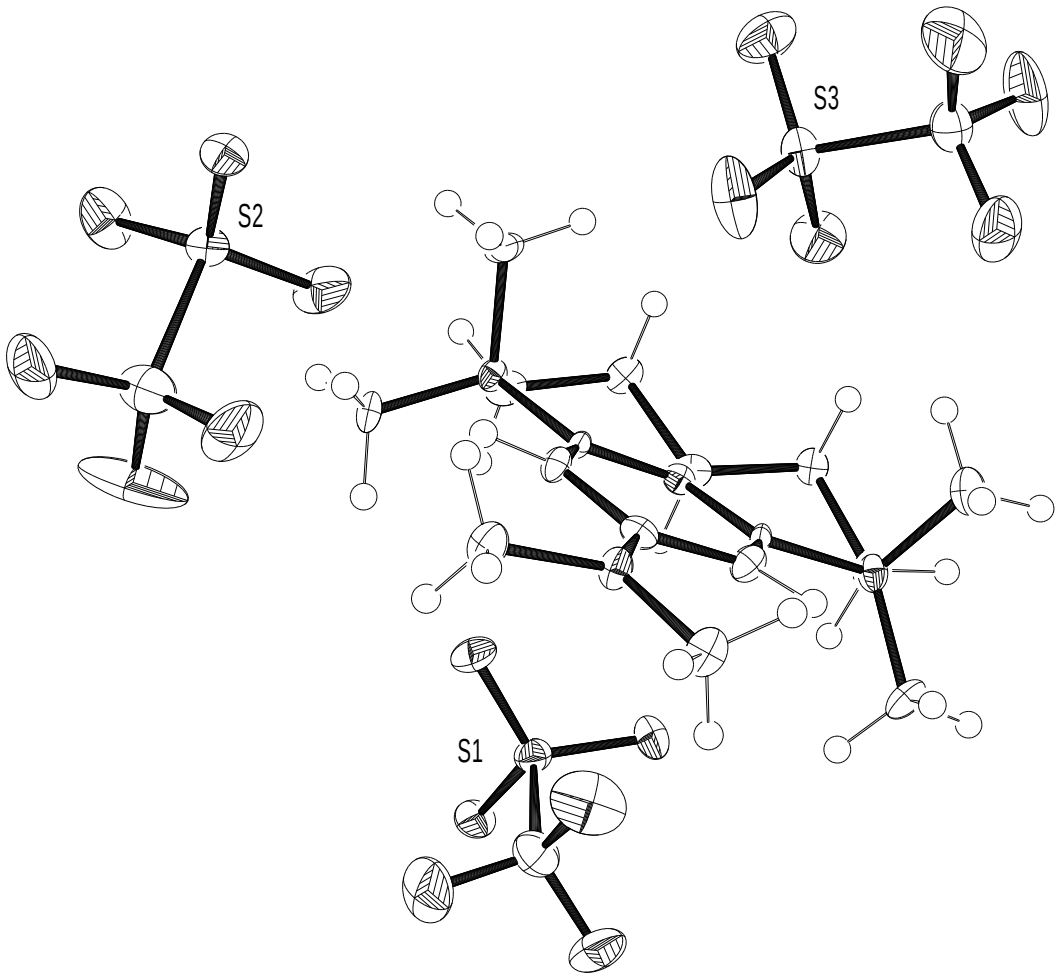
Table 6. Torsion angles [°] for jam0309.

| | |
|---|---|
| C(6)-N(1)-C(1)-C(2) | -3.7(3) |
| C(7)-N(1)-C(1)-C(2) | -172.96(18) |
| C(6)-N(1)-C(1)-C(5) | 175.79(18) |
| C(7)-N(1)-C(1)-C(5) | 6.5(3) |
| N(1)-C(1)-C(2)-C(3) | 173.3(2) |
| C(5)-C(1)-C(2)-C(3) | -6.2(3) |
| C(1)-C(2)-C(3)-N(2) | 3.8(3) |
| C(1)-C(2)-C(3)-N(3) | 179.82(18) |
| C(4)-N(2)-C(3)-C(2) | -0.8(3) |
| C(10)-N(2)-C(3)-C(2) | -171.54(19) |
| C(4)-N(2)-C(3)-N(3) | -176.63(17) |
| C(10)-N(2)-C(3)-N(3) | 12.6(3) |
| C(14)-N(3)-C(3)-C(2) | -71.5(2) |
| C(8)-N(3)-C(3)-C(2) | 166.39(18) |
| C(13)-N(3)-C(3)-C(2) | 46.9(2) |
| C(14)-N(3)-C(3)-N(2) | 104.63(19) |
| C(8)-N(3)-C(3)-N(2) | -17.5(2) |
| C(13)-N(3)-C(3)-N(2) | -136.92(18) |
| C(3)-N(2)-C(4)-C(5) | 0.5(3) |
| C(10)-N(2)-C(4)-C(5) | 171.02(19) |
| C(3)-N(2)-C(4)-N(4) | 176.29(16) |
| C(10)-N(2)-C(4)-N(4) | -13.2(3) |
| C(15)-N(4)-C(4)-C(5) | -43.7(2) |
| C(16)-N(4)-C(4)-C(5) | 74.3(2) |
| C(12)-N(4)-C(4)-C(5) | -163.07(18) |
| C(15)-N(4)-C(4)-N(2) | 140.20(17) |
| C(16)-N(4)-C(4)-N(2) | -101.80(19) |
| C(12)-N(4)-C(4)-N(2) | 20.8(2) |
| N(2)-C(4)-C(5)-C(1) | -3.2(3) |
| N(4)-C(4)-C(5)-C(1) | -179.16(16) |
| N(1)-C(1)-C(5)-C(4) | -173.59(19) |
| C(2)-C(1)-C(5)-C(4) | 5.9(3) |
| C(3)-N(3)-C(8)-C(9) | 43.7(2) |
| C(14)-N(3)-C(8)-C(9) | -77.23(19) |
| C(13)-N(3)-C(8)-C(9) | 165.42(16) |
| N(3)-C(8)-C(9)-C(10) | -65.3(2) |

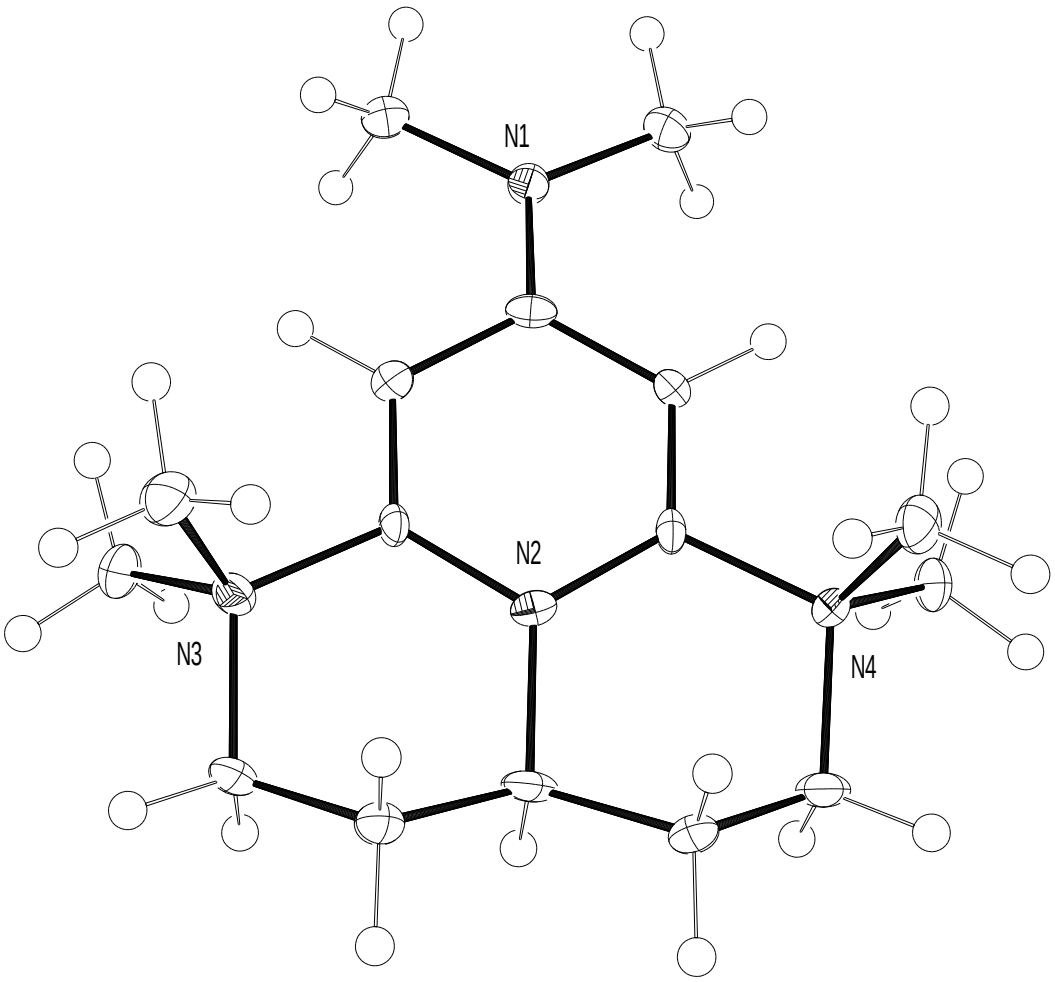
| | |
|---|---|
| C(8)-C(9)-C(10)-C(11) | -177.78(16) |
| C(8)-C(9)-C(10)-N(2) | 59.3(2) |
| C(4)-N(2)-C(10)-C(9) | 156.30(18) |
| C(3)-N(2)-C(10)-C(9) | -33.6(2) |
| C(4)-N(2)-C(10)-C(11) | 30.4(3) |
| C(3)-N(2)-C(10)-C(11) | -159.50(17) |
| C(9)-C(10)-C(11)-C(12) | -179.43(17) |
| N(2)-C(10)-C(11)-C(12) | -55.9(2) |
| C(10)-C(11)-C(12)-N(4) | 66.4(2) |
| C(4)-N(4)-C(12)-C(11) | -47.3(2) |
| C(15)-N(4)-C(12)-C(11) | -169.77(16) |
| C(16)-N(4)-C(12)-C(11) | 74.57(19) |
| O(3)-S(1)-C(17)-F(2) | -56.70(19) |
| O(1)-S(1)-C(17)-F(2) | -176.54(16) |
| O(2)-S(1)-C(17)-F(2) | 64.34(18) |
| O(3)-S(1)-C(17)-F(1) | 63.53(17) |
| O(1)-S(1)-C(17)-F(1) | -56.31(17) |
| O(2)-S(1)-C(17)-F(1) | -175.43(15) |
| O(3)-S(1)-C(17)-F(3) | -177.46(15) |
| O(1)-S(1)-C(17)-F(3) | 62.70(18) |
| O(2)-S(1)-C(17)-F(3) | -56.42(18) |
| O(5)-S(2)-C(18)-F(4) | -177.13(17) |
| O(4)-S(2)-C(18)-F(4) | 63.7(2) |
| O(6)-S(2)-C(18)-F(4) | -56.9(2) |
| O(5)-S(2)-C(18)-F(5) | 61.39(18) |
| O(4)-S(2)-C(18)-F(5) | -57.77(18) |
| O(6)-S(2)-C(18)-F(5) | -178.33(16) |
| O(5)-S(2)-C(18)-F(6) | -56.95(17) |
| O(4)-S(2)-C(18)-F(6) | -176.11(15) |
| O(6)-S(2)-C(18)-F(6) | 63.32(17) |
| O(7)-S(3)-C(19)-F(9) | 175.05(17) |
| O(8)-S(3)-C(19)-F(9) | -63.14(19) |
| O(9)-S(3)-C(19)-F(9) | 56.10(19) |
| O(7)-S(3)-C(19)-F(8) | -62.48(19) |
| O(8)-S(3)-C(19)-F(8) | 59.32(19) |
| O(9)-S(3)-C(19)-F(8) | 178.56(16) |
| O(7)-S(3)-C(19)-F(7) | 55.08(18) |
| O(8)-S(3)-C(19)-F(7) | 176.88(15) |

| | |
|---|---|
| O(9)-S(3)-C(19)-F(7) | -63.88(18) |

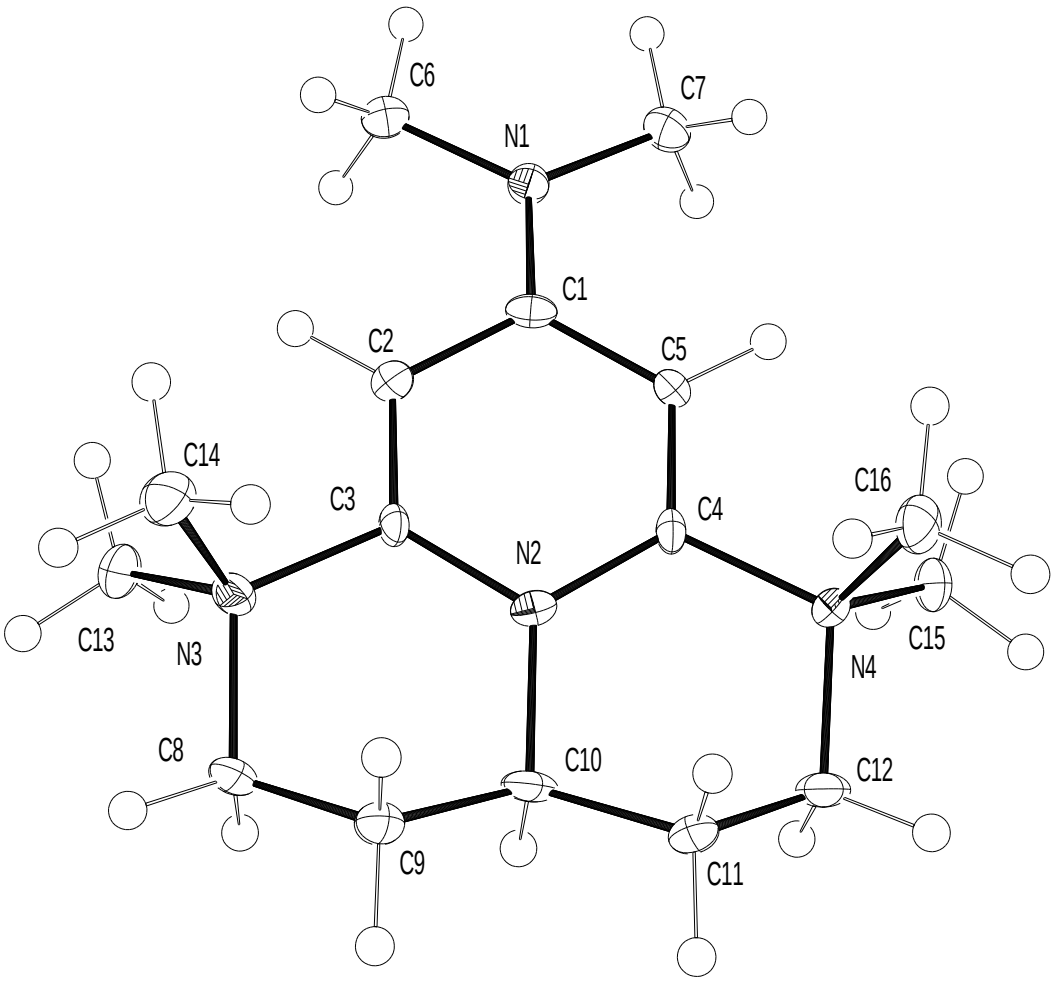
Symmetry transformations used to generate equivalent atoms:



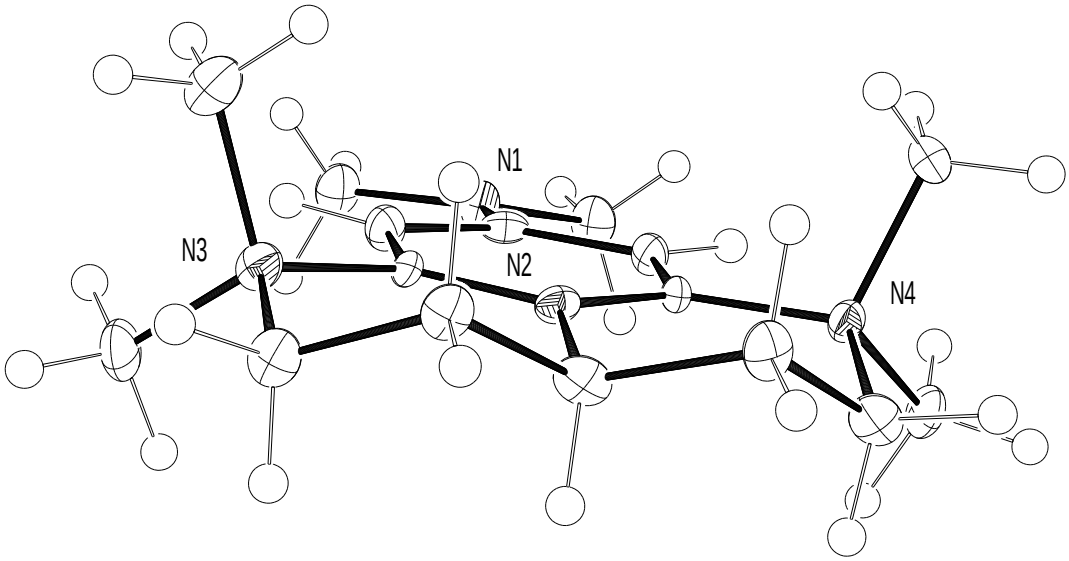

Above – contents of the asymmetric unit.



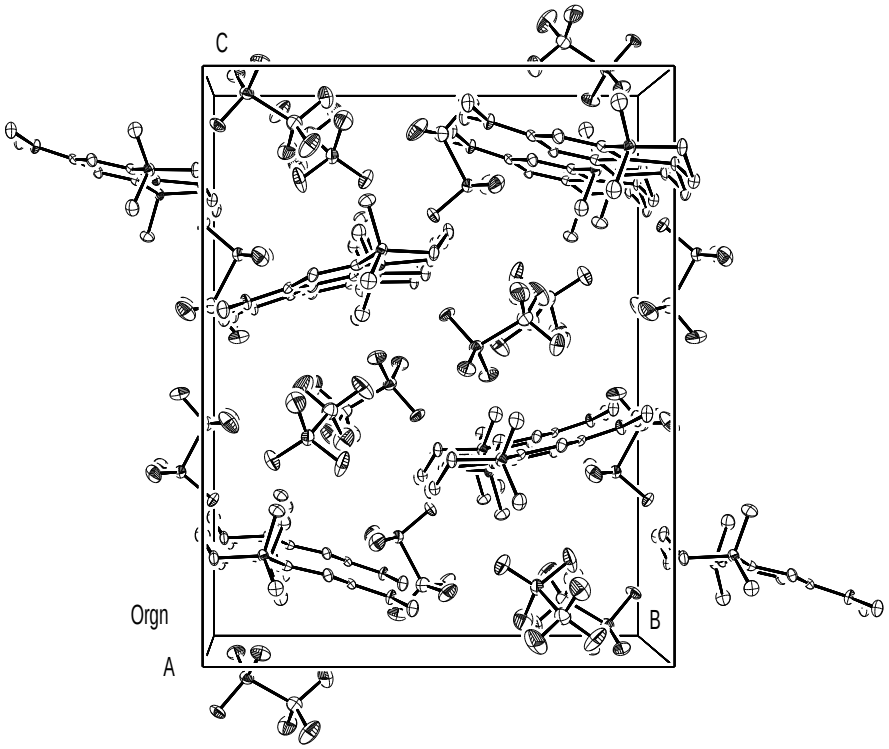

Above – view of the cation.




Above – cation with atomiv labels.




Above – end on view of cation.




Above – packed structure.

# Appendix 5

Crystallographic Data for Disalt **8.57**

Table 1. Crystal data and structure refinement for cs_jm08feb09.

| | | |
|---|---|---|
| Identification code | cs_jm08feb09 | |
| Empirical formula | C23 H20 Cl2 F6 N2 O6 S2 | |
| Formula weight | 669.43 | |
| Temperature | 123(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Monoclinic | |
| Space group | P21/c | |
| Unit cell dimensions | a = 7.9991(5) Å | α= 90°. |
| | b = 34.1785(19) Å | β= 97.268(6)°. |
| | c = 10.2197(7) Å | γ = 90°. |
| Volume | 2771.6(3) $Å^3$ | |
| Z | 4 | |
| Density (calculated) | 1.604 $Mg/m^3$ | |
| Absorption coefficient | 0.468 $mm^{-1}$ | |
| F(000) | 1360 | |
| Crystal size | 0.30 x 0.12 x 0.09 $mm^3$ | |
| Theta range for data collection | 2.69 to 27.00°. | |
| Index ranges | -10<=h<=9, -43<=k<=36, -9<=l<=13 | |
| Reflections collected | 12676 | |
| Independent reflections | 6030 [R(int) = 0.0528] | |
| Completeness to theta = 27.00° | 99.6 % | |
| Absorption correction | Semi-empirical from equivalents | |
| Max. and min. transmission | 1.00000 and 0.97343 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 6030 / 27 / 381 | |
| Goodness-of-fit on $F^2$ | 0.836 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0449, wR2 = 0.0880 | |
| R indices (all data) | R1 = 0.1203, wR2 = 0.0977 | |
| Largest diff. peak and hole | 0.360 and -0.384 $e.Å^{-3}$ | |

Table 2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for cs_jm08feb09. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Cl(1) | 7585(1) | 44(1) | 5512(1) | 60(1) |
| Cl(2) | 6099(1) | -105(1) | 7904(1) | 47(1) |
| S(1) | 11545(1) | 861(1) | -1404(1) | 31(1) |
| S(2) | 5859(1) | 1559(1) | 3433(1) | 29(1) |
| F(1) | 11825(8) | 752(2) | -3954(4) | 61(2) |
| F(2) | 13388(7) | 1218(2) | -3032(6) | 52(2) |
| F(3) | 10756(9) | 1295(2) | -3516(6) | 50(2) |
| F(4) | 7307(3) | 871(1) | 3659(2) | 48(1) |
| F(5) | 4911(2) | 896(1) | 4408(2) | 44(1) |
| F(6) | 7111(3) | 1147(1) | 5510(2) | 49(1) |
| O(1) | 12880(3) | 602(1) | -952(3) | 64(1) |
| O(2) | 9922(3) | 681(1) | -1681(3) | 47(1) |
| O(3) | 11569(3) | 1224(1) | -707(2) | 46(1) |
| O(4) | 4982(3) | 1436(1) | 2185(2) | 37(1) |
| O(5) | 7508(3) | 1722(1) | 3370(3) | 42(1) |
| O(6) | 4833(3) | 1765(1) | 4269(2) | 32(1) |
| N(1) | 8672(3) | 1107(1) | 1065(3) | 22(1) |
| N(2) | 10310(3) | 1648(1) | 1803(3) | 24(1) |
| C(1) | 7115(4) | 988(1) | 475(3) | 24(1) |
| C(2) | 6708(4) | 606(1) | 432(3) | 30(1) |
| C(3) | 7859(4) | 329(1) | 977(4) | 31(1) |
| C(4) | 9386(4) | 454(1) | 1623(3) | 30(1) |
| C(5) | 9806(4) | 841(1) | 1681(3) | 25(1) |
| C(6) | 11403(4) | 1004(1) | 2394(3) | 28(1) |
| C(7) | 11077(4) | 1404(1) | 2928(3) | 28(1) |
| C(8) | 9122(4) | 1516(1) | 955(3) | 23(1) |
| C(9) | 8180(4) | 1750(1) | -80(3) | 23(1) |
| C(10) | 7939(4) | 1630(1) | -1400(3) | 28(1) |
| C(11) | 7058(4) | 1866(1) | -2331(4) | 36(1) |
| C(12) | 6429(4) | 2219(1) | -1995(4) | 36(1) |
| C(13) | 6657(4) | 2342(1) | -683(4) | 34(1) |
| C(14) | 7514(4) | 2109(1) | 271(3) | 29(1) |
| C(15) | 11009(4) | 2041(1) | 1666(3) | 25(1) |

| | | | | |
|---|---|---|---|---|
| C(16) | 12046(4) | 2100(1) | 715(3) | 27(1) |
| C(17) | 12643(4) | 2475(1) | 557(4) | 34(1) |
| C(18) | 12231(4) | 2775(1) | 1333(4) | 36(1) |
| C(19) | 11223(4) | 2708(1) | 2332(4) | 35(1) |
| C(20) | 10607(4) | 2335(1) | 2510(3) | 31(1) |
| C(21) | 11921(10) | 1032(2) | -3069(7) | 44(3) |
| C(22) | 6313(4) | 1099(1) | 4295(4) | 31(1) |
| C(23) | 6531(5) | 254(1) | 6759(4) | 43(1) |
| F(1A) | 12382(9) | 642(2) | -3582(8) | 67(3) |
| F(2A) | 13784(11) | 1120(3) | -2749(10) | 72(3) |
| F(3A) | 11224(14) | 1219(3) | -3653(12) | 83(4) |
| C(21A) | 12241(13) | 979(3) | -2962(8) | 50(4) |

Table 3. Bond lengths [Å] and angles [°] for cs_jm08feb09.

| | |
|---|---|
| Cl(1)-C(23) | 1.768(4) |
| Cl(2)-C(23) | 1.761(4) |
| S(1)-O(1) | 1.419(2) |
| S(1)-O(3) | 1.429(2) |
| S(1)-O(2) | 1.432(2) |
| S(1)-C(21A) | 1.797(8) |
| S(1)-C(21) | 1.859(7) |
| S(2)-O(4) | 1.437(2) |
| S(2)-O(5) | 1.441(2) |
| S(2)-O(6) | 1.442(2) |
| S(2)-C(22) | 1.817(3) |
| F(1)-C(21) | 1.314(7) |
| F(2)-C(21) | 1.331(7) |
| F(3)-C(21) | 1.333(8) |
| F(4)-C(22) | 1.338(4) |
| F(5)-C(22) | 1.336(4) |
| F(6)-C(22) | 1.333(4) |
| N(1)-C(1) | 1.374(4) |
| N(1)-C(5) | 1.378(4) |
| N(1)-C(8) | 1.451(4) |
| N(2)-C(8) | 1.285(4) |
| N(2)-C(15) | 1.468(4) |
| N(2)-C(7) | 1.489(4) |
| C(1)-C(2) | 1.346(4) |
| C(1)-H(1) | 0.9500 |
| C(2)-C(3) | 1.388(4) |
| C(2)-H(2) | 0.9500 |
| C(3)-C(4) | 1.381(4) |
| C(3)-H(3) | 0.9500 |
| C(4)-C(5) | 1.365(4) |
| C(4)-H(4) | 0.9500 |
| C(5)-C(6) | 1.494(4) |
| C(6)-C(7) | 1.507(4) |
| C(6)-H(6A) | 0.9900 |
| C(6)-H(6B) | 0.9900 |
| C(7)-H(7A) | 0.9900 |

| | |
|---|---|
| C(7)-H(7B) | 0.9900 |
| C(8)-C(9) | 1.458(4) |
| C(9)-C(10) | 1.400(4) |
| C(9)-C(14) | 1.401(4) |
| C(10)-C(11) | 1.372(4) |
| C(10)-H(10) | 0.9500 |
| C(11)-C(12) | 1.365(5) |
| C(11)-H(11) | 0.9500 |
| C(12)-C(13) | 1.396(5) |
| C(12)-H(12) | 0.9500 |
| C(13)-C(14) | 1.375(4) |
| C(13)-H(13) | 0.9500 |
| C(14)-H(14) | 0.9500 |
| C(15)-C(16) | 1.370(4) |
| C(15)-C(20) | 1.389(4) |
| C(16)-C(17) | 1.384(4) |
| C(16)-H(16) | 0.9500 |
| C(17)-C(18) | 1.362(4) |
| C(17)-H(17) | 0.9500 |
| C(18)-C(19) | 1.398(5) |
| C(18)-H(18) | 0.9500 |
| C(19)-C(20) | 1.388(4) |
| C(19)-H(19) | 0.9500 |
| C(20)-H(20) | 0.9500 |
| C(23)-H(23A) | 0.9900 |
| C(23)-H(23B) | 0.9900 |
| F(1A)-C(21A) | 1.327(8) |
| F(2A)-C(21A) | 1.316(8) |
| F(3A)-C(21A) | 1.300(9) |

| | |
|---|---|
| O(1)-S(1)-O(3) | 114.65(16) |
| O(1)-S(1)-O(2) | 114.99(15) |
| O(3)-S(1)-O(2) | 115.18(15) |
| O(1)-S(1)-C(21A) | 97.1(4) |
| O(3)-S(1)-C(21A) | 105.2(3) |
| O(2)-S(1)-C(21A) | 107.2(4) |
| O(1)-S(1)-C(21) | 107.0(3) |
| O(3)-S(1)-C(21) | 101.0(3) |

| | |
|---|---|
| O(2)-S(1)-C(21) | 101.6(3) |
| C(21A)-S(1)-C(21) | 9.9(5) |
| O(4)-S(2)-O(5) | 114.82(15) |
| O(4)-S(2)-O(6) | 114.66(14) |
| O(5)-S(2)-O(6) | 115.53(14) |
| O(4)-S(2)-C(22) | 102.95(15) |
| O(5)-S(2)-C(22) | 103.17(15) |
| O(6)-S(2)-C(22) | 103.25(15) |
| C(1)-N(1)-C(5) | 120.9(2) |
| C(1)-N(1)-C(8) | 118.0(2) |
| C(5)-N(1)-C(8) | 120.9(2) |
| C(8)-N(2)-C(15) | 121.0(3) |
| C(8)-N(2)-C(7) | 121.4(3) |
| C(15)-N(2)-C(7) | 117.5(2) |
| C(2)-C(1)-N(1) | 120.2(3) |
| C(2)-C(1)-H(1) | 119.9 |
| N(1)-C(1)-H(1) | 119.9 |
| C(1)-C(2)-C(3) | 120.2(3) |
| C(1)-C(2)-H(2) | 119.9 |
| C(3)-C(2)-H(2) | 119.9 |
| C(4)-C(3)-C(2) | 118.9(3) |
| C(4)-C(3)-H(3) | 120.6 |
| C(2)-C(3)-H(3) | 120.6 |
| C(5)-C(4)-C(3) | 121.2(3) |
| C(5)-C(4)-H(4) | 119.4 |
| C(3)-C(4)-H(4) | 119.4 |
| C(4)-C(5)-N(1) | 118.3(3) |
| C(4)-C(5)-C(6) | 124.9(3) |
| N(1)-C(5)-C(6) | 116.8(3) |
| C(5)-C(6)-C(7) | 109.7(3) |
| C(5)-C(6)-H(6A) | 109.7 |
| C(7)-C(6)-H(6A) | 109.7 |
| C(5)-C(6)-H(6B) | 109.7 |
| C(7)-C(6)-H(6B) | 109.7 |
| H(6A)-C(6)-H(6B) | 108.2 |
| N(2)-C(7)-C(6) | 107.6(3) |
| N(2)-C(7)-H(7A) | 110.2 |
| C(6)-C(7)-H(7A) | 110.2 |

| | |
|---|---|
| N(2)-C(7)-H(7B) | 110.2 |
| C(6)-C(7)-H(7B) | 110.2 |
| H(7A)-C(7)-H(7B) | 108.5 |
| N(2)-C(8)-N(1) | 117.2(3) |
| N(2)-C(8)-C(9) | 124.5(3) |
| N(1)-C(8)-C(9) | 118.3(3) |
| C(10)-C(9)-C(14) | 119.5(3) |
| C(10)-C(9)-C(8) | 122.3(3) |
| C(14)-C(9)-C(8) | 118.2(3) |
| C(11)-C(10)-C(9) | 119.5(3) |
| C(11)-C(10)-H(10) | 120.3 |
| C(9)-C(10)-H(10) | 120.3 |
| C(12)-C(11)-C(10) | 121.2(3) |
| C(12)-C(11)-H(11) | 119.4 |
| C(10)-C(11)-H(11) | 119.4 |
| C(11)-C(12)-C(13) | 120.1(3) |
| C(11)-C(12)-H(12) | 120.0 |
| C(13)-C(12)-H(12) | 120.0 |
| C(14)-C(13)-C(12) | 119.8(3) |
| C(14)-C(13)-H(13) | 120.1 |
| C(12)-C(13)-H(13) | 120.1 |
| C(13)-C(14)-C(9) | 119.9(3) |
| C(13)-C(14)-H(14) | 120.1 |
| C(9)-C(14)-H(14) | 120.1 |
| C(16)-C(15)-C(20) | 122.8(3) |
| C(16)-C(15)-N(2) | 118.4(3) |
| C(20)-C(15)-N(2) | 118.8(3) |
| C(15)-C(16)-C(17) | 118.0(3) |
| C(15)-C(16)-H(16) | 121.0 |
| C(17)-C(16)-H(16) | 121.0 |
| C(18)-C(17)-C(16) | 121.1(3) |
| C(18)-C(17)-H(17) | 119.5 |
| C(16)-C(17)-H(17) | 119.5 |
| C(17)-C(18)-C(19) | 120.4(3) |
| C(17)-C(18)-H(18) | 119.8 |
| C(19)-C(18)-H(18) | 119.8 |
| C(20)-C(19)-C(18) | 119.6(3) |
| C(20)-C(19)-H(19) | 120.2 |

| | |
|---|---|
| C(18)-C(19)-H(19) | 120.2 |
| C(19)-C(20)-C(15) | 118.0(3) |
| C(19)-C(20)-H(20) | 121.0 |
| C(15)-C(20)-H(20) | 121.0 |
| F(1)-C(21)-F(2) | 110.1(7) |
| F(1)-C(21)-F(3) | 106.1(7) |
| F(2)-C(21)-F(3) | 105.3(6) |
| F(1)-C(21)-S(1) | 113.4(5) |
| F(2)-C(21)-S(1) | 111.6(5) |
| F(3)-C(21)-S(1) | 109.8(5) |
| F(6)-C(22)-F(5) | 106.9(3) |
| F(6)-C(22)-F(4) | 106.3(3) |
| F(5)-C(22)-F(4) | 107.3(3) |
| F(6)-C(22)-S(2) | 112.8(2) |
| F(5)-C(22)-S(2) | 111.8(2) |
| F(4)-C(22)-S(2) | 111.3(2) |
| Cl(2)-C(23)-Cl(1) | 110.35(17) |
| Cl(2)-C(23)-H(23A) | 109.6 |
| Cl(1)-C(23)-H(23A) | 109.6 |
| Cl(2)-C(23)-H(23B) | 109.6 |
| Cl(1)-C(23)-H(23B) | 109.6 |
| H(23A)-C(23)-H(23B) | 108.1 |
| F(3A)-C(21A)-F(2A) | 111.6(9) |
| F(3A)-C(21A)-F(1A) | 111.9(9) |
| F(2A)-C(21A)-F(1A) | 105.1(9) |
| F(3A)-C(21A)-S(1) | 112.4(8) |
| F(2A)-C(21A)-S(1) | 108.9(7) |
| F(1A)-C(21A)-S(1) | 106.5(6) |

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Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for cs_jm08feb09. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| Cl(1) | 82(1) | 57(1) | 41(1) | 7(1) | 13(1) | 3(1) |
| Cl(2) | 52(1) | 37(1) | 56(1) | -2(1) | 20(1) | 0(1) |
| S(1) | 30(1) | 29(1) | 34(1) | 1(1) | 3(1) | 1(1) |
| S(2) | 28(1) | 26(1) | 35(1) | 0(1) | 11(1) | 1(1) |
| F(1) | 110(5) | 57(3) | 18(3) | -18(2) | 17(3) | 8(3) |
| F(2) | 34(3) | 53(3) | 76(4) | 24(3) | 33(3) | -4(3) |
| F(3) | 55(4) | 71(4) | 22(3) | 28(3) | 1(3) | 8(3) |
| F(4) | 64(2) | 35(1) | 50(1) | 8(1) | 26(1) | 19(1) |
| F(5) | 47(1) | 40(1) | 46(1) | 10(1) | 8(1) | -14(1) |
| F(6) | 54(1) | 51(1) | 37(1) | 1(1) | -11(1) | 4(1) |
| O(1) | 42(2) | 39(2) | 103(3) | 17(2) | -18(2) | 6(1) |
| O(2) | 31(2) | 60(2) | 50(2) | -2(1) | 4(1) | -18(1) |
| O(3) | 55(2) | 43(2) | 41(2) | -17(1) | 10(1) | -4(1) |
| O(4) | 44(2) | 42(1) | 26(1) | 0(1) | 4(1) | 6(1) |
| O(5) | 27(1) | 35(1) | 69(2) | 7(1) | 19(1) | -5(1) |
| O(6) | 25(1) | 34(1) | 38(2) | -8(1) | 8(1) | 1(1) |
| N(1) | 20(2) | 21(1) | 25(2) | -2(1) | 4(1) | 0(1) |
| N(2) | 22(2) | 25(1) | 25(2) | -4(1) | 0(1) | -1(1) |
| C(1) | 20(2) | 29(2) | 23(2) | 0(2) | 1(2) | -1(1) |
| C(2) | 29(2) | 33(2) | 27(2) | -3(2) | 2(2) | -6(2) |
| C(3) | 30(2) | 24(2) | 41(2) | -1(2) | 11(2) | -4(2) |
| C(4) | 28(2) | 25(2) | 37(2) | 5(2) | 8(2) | 3(2) |
| C(5) | 24(2) | 28(2) | 26(2) | 1(2) | 8(2) | 4(2) |
| C(6) | 26(2) | 28(2) | 30(2) | 3(2) | -2(2) | 3(2) |
| C(7) | 30(2) | 33(2) | 21(2) | 7(2) | -1(2) | 0(2) |
| C(8) | 21(2) | 24(2) | 24(2) | -4(2) | 7(2) | 0(2) |
| C(9) | 19(2) | 22(2) | 26(2) | 7(2) | 0(2) | 0(1) |
| C(10) | 31(2) | 29(2) | 24(2) | -4(2) | 4(2) | -6(2) |
| C(11) | 38(2) | 46(2) | 23(2) | 4(2) | 0(2) | -7(2) |
| C(12) | 33(2) | 41(2) | 31(2) | 17(2) | -3(2) | -1(2) |
| C(13) | 28(2) | 26(2) | 49(3) | 10(2) | 6(2) | 4(2) |
| C(14) | 30(2) | 30(2) | 27(2) | -1(2) | 0(2) | -3(2) |
| C(15) | 19(2) | 26(2) | 28(2) | -2(2) | -4(2) | -2(1) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C(16) | 26(2) | 35(2) | 21(2) | -4(2) | 8(2) | -1(2) |
| C(17) | 31(2) | 34(2) | 36(2) | 1(2) | 4(2) | -2(2) |
| C(18) | 34(2) | 30(2) | 41(2) | 6(2) | -3(2) | -9(2) |
| C(19) | 39(2) | 28(2) | 38(2) | -12(2) | -1(2) | -4(2) |
| C(20) | 31(2) | 37(2) | 25(2) | -4(2) | 5(2) | 0(2) |
| C(22) | 33(2) | 36(2) | 25(2) | -5(2) | 6(2) | -3(2) |
| C(23) | 45(2) | 27(2) | 53(3) | 2(2) | -7(2) | 5(2) |

Table 5. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters ($Å^2$x $10^3$) for cs_jm08feb09.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(1) | 6325 | 1177 | 96 | 29 |
| H(2) | 5628 | 526 | 26 | 36 |
| H(3) | 7601 | 58 | 908 | 37 |
| H(4) | 10160 | 267 | 2034 | 36 |
| H(6A) | 12266 | 1022 | 1782 | 34 |
| H(6B) | 11834 | 827 | 3127 | 34 |
| H(7A) | 10301 | 1384 | 3607 | 34 |
| H(7B) | 12146 | 1523 | 3337 | 34 |
| H(10) | 8381 | 1387 | -1650 | 33 |
| H(11) | 6883 | 1783 | -3225 | 43 |
| H(12) | 5836 | 2379 | -2655 | 43 |
| H(13) | 6221 | 2588 | -450 | 41 |
| H(14) | 7655 | 2190 | 1167 | 35 |
| H(16) | 12345 | 1890 | 180 | 32 |
| H(17) | 13352 | 2524 | -104 | 40 |
| H(18) | 12631 | 3032 | 1194 | 43 |
| H(19) | 10962 | 2917 | 2886 | 42 |
| H(20) | 9929 | 2282 | 3189 | 37 |
| H(23A) | 7242 | 462 | 7218 | 52 |
| H(23B) | 5463 | 375 | 6357 | 52 |

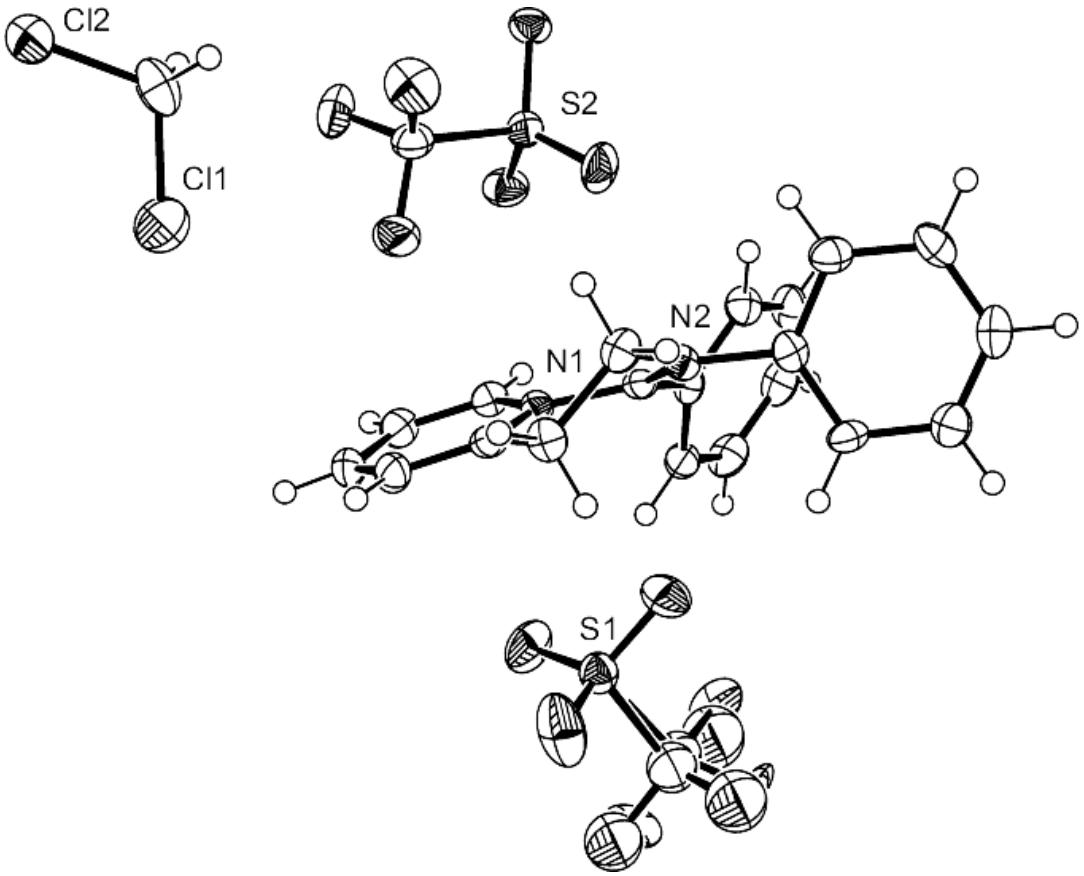
Table 6. Torsion angles [°] for cs_jm08feb09.

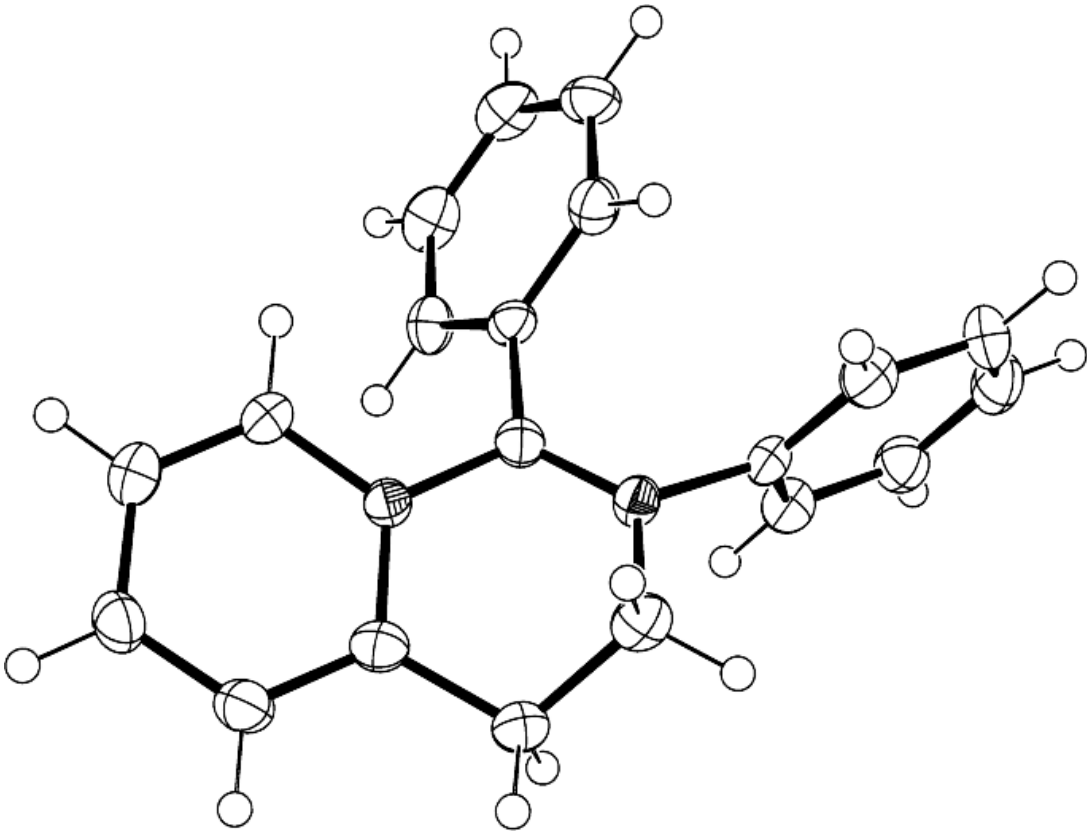
| | |
|---|---|
| C(5)-N(1)-C(1)-C(2) | -3.4(5) |
| C(8)-N(1)-C(1)-C(2) | 173.1(3) |
| N(1)-C(1)-C(2)-C(3) | -0.3(5) |
| C(1)-C(2)-C(3)-C(4) | 3.4(5) |
| C(2)-C(3)-C(4)-C(5) | -2.9(5) |
| C(3)-C(4)-C(5)-N(1) | -0.7(5) |
| C(3)-C(4)-C(5)-C(6) | 177.9(3) |
| C(1)-N(1)-C(5)-C(4) | 3.9(4) |
| C(8)-N(1)-C(5)-C(4) | -172.5(3) |
| C(1)-N(1)-C(5)-C(6) | -174.8(3) |
| C(8)-N(1)-C(5)-C(6) | 8.7(4) |
| C(4)-C(5)-C(6)-C(7) | -147.3(3) |
| N(1)-C(5)-C(6)-C(7) | 31.4(4) |
| C(8)-N(2)-C(7)-C(6) | 45.1(4) |
| C(15)-N(2)-C(7)-C(6) | -132.4(3) |
| C(5)-C(6)-C(7)-N(2) | -55.1(3) |
| C(15)-N(2)-C(8)-N(1) | 171.6(3) |
| C(7)-N(2)-C(8)-N(1) | -5.8(4) |
| C(15)-N(2)-C(8)-C(9) | -10.0(4) |
| C(7)-N(2)-C(8)-C(9) | 172.6(3) |
| C(1)-N(1)-C(8)-N(2) | 159.9(3) |
| C(5)-N(1)-C(8)-N(2) | -23.6(4) |
| C(1)-N(1)-C(8)-C(9) | -18.7(4) |
| C(5)-N(1)-C(8)-C(9) | 157.8(3) |
| N(2)-C(8)-C(9)-C(10) | 130.3(3) |
| N(1)-C(8)-C(9)-C(10) | -51.3(4) |
| N(2)-C(8)-C(9)-C(14) | -49.0(4) |
| N(1)-C(8)-C(9)-C(14) | 129.4(3) |
| C(14)-C(9)-C(10)-C(11) | 0.2(5) |
| C(8)-C(9)-C(10)-C(11) | -179.2(3) |
| C(9)-C(10)-C(11)-C(12) | 0.8(5) |
| C(10)-C(11)-C(12)-C(13) | -0.8(5) |
| C(11)-C(12)-C(13)-C(14) | -0.1(5) |
| C(12)-C(13)-C(14)-C(9) | 1.1(5) |
| C(10)-C(9)-C(14)-C(13) | -1.1(5) |
| C(8)-C(9)-C(14)-C(13) | 178.3(3) |

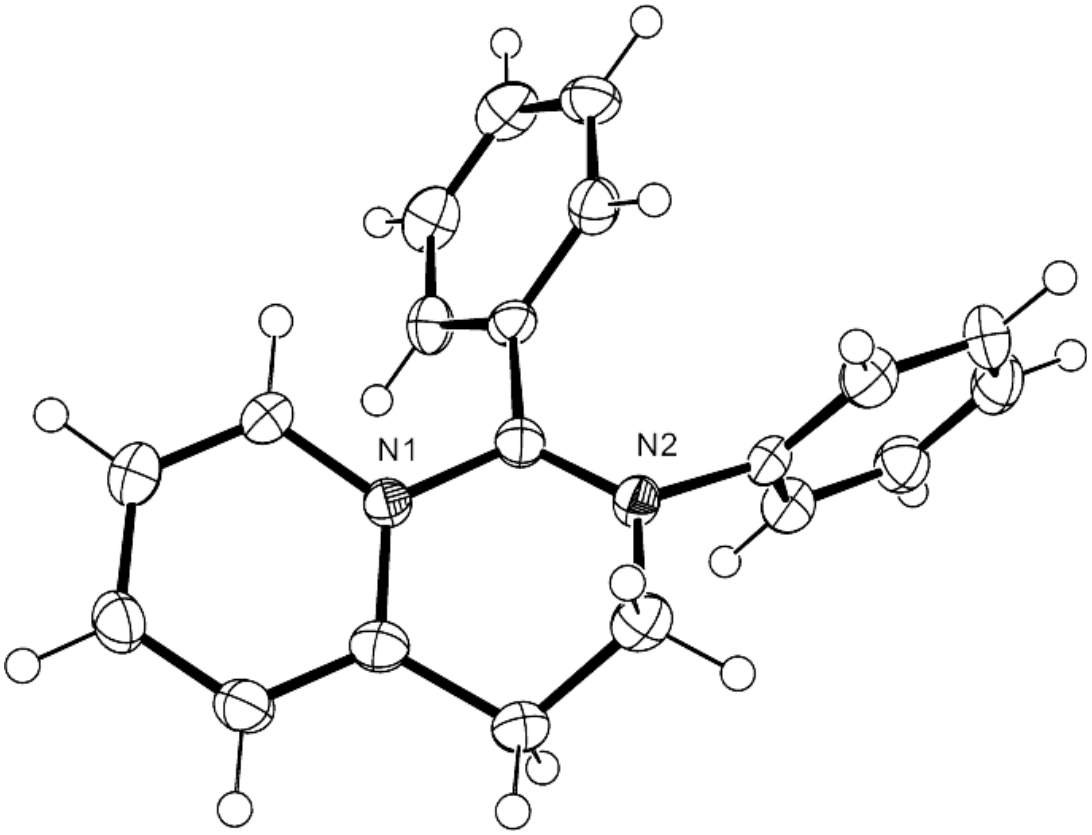
| | |
|---|---|
| C(8)-N(2)-C(15)-C(16) | -73.9(4) |
| C(7)-N(2)-C(15)-C(16) | 103.6(3) |
| C(8)-N(2)-C(15)-C(20) | 106.7(4) |
| C(7)-N(2)-C(15)-C(20) | -75.8(4) |
| C(20)-C(15)-C(16)-C(17) | -3.2(5) |
| N(2)-C(15)-C(16)-C(17) | 177.4(3) |
| C(15)-C(16)-C(17)-C(18) | 0.8(5) |
| C(16)-C(17)-C(18)-C(19) | 1.6(5) |
| C(17)-C(18)-C(19)-C(20) | -1.6(5) |
| C(18)-C(19)-C(20)-C(15) | -0.7(5) |
| C(16)-C(15)-C(20)-C(19) | 3.1(5) |
| N(2)-C(15)-C(20)-C(19) | -177.5(3) |
| O(1)-S(1)-C(21)-F(1) | 64.9(6) |
| O(3)-S(1)-C(21)-F(1) | -174.8(6) |
| O(2)-S(1)-C(21)-F(1) | -56.0(6) |
| C(21A)-S(1)-C(21)-F(1) | 69(3) |
| O(1)-S(1)-C(21)-F(2) | -60.1(6) |
| O(3)-S(1)-C(21)-F(2) | 60.1(6) |
| O(2)-S(1)-C(21)-F(2) | 179.0(5) |
| C(21A)-S(1)-C(21)-F(2) | -56(3) |
| O(1)-S(1)-C(21)-F(3) | -176.5(4) |
| O(3)-S(1)-C(21)-F(3) | -56.3(5) |
| O(2)-S(1)-C(21)-F(3) | 62.6(5) |
| C(21A)-S(1)-C(21)-F(3) | -172(3) |
| O(4)-S(2)-C(22)-F(6) | -178.7(2) |
| O(5)-S(2)-C(22)-F(6) | 61.6(3) |
| O(6)-S(2)-C(22)-F(6) | -59.1(3) |
| O(4)-S(2)-C(22)-F(5) | -58.1(3) |
| O(5)-S(2)-C(22)-F(5) | -177.8(2) |
| O(6)-S(2)-C(22)-F(5) | 61.5(3) |
| O(4)-S(2)-C(22)-F(4) | 61.9(3) |
| O(5)-S(2)-C(22)-F(4) | -57.8(3) |
| O(6)-S(2)-C(22)-F(4) | -178.5(2) |
| O(1)-S(1)-C(21A)-F(3A) | 179.2(8) |
| O(3)-S(1)-C(21A)-F(3A) | -62.8(8) |
| O(2)-S(1)-C(21A)-F(3A) | 60.2(8) |
| C(21)-S(1)-C(21A)-F(3A) | 3(2) |
| O(1)-S(1)-C(21A)-F(2A) | -56.6(7) |

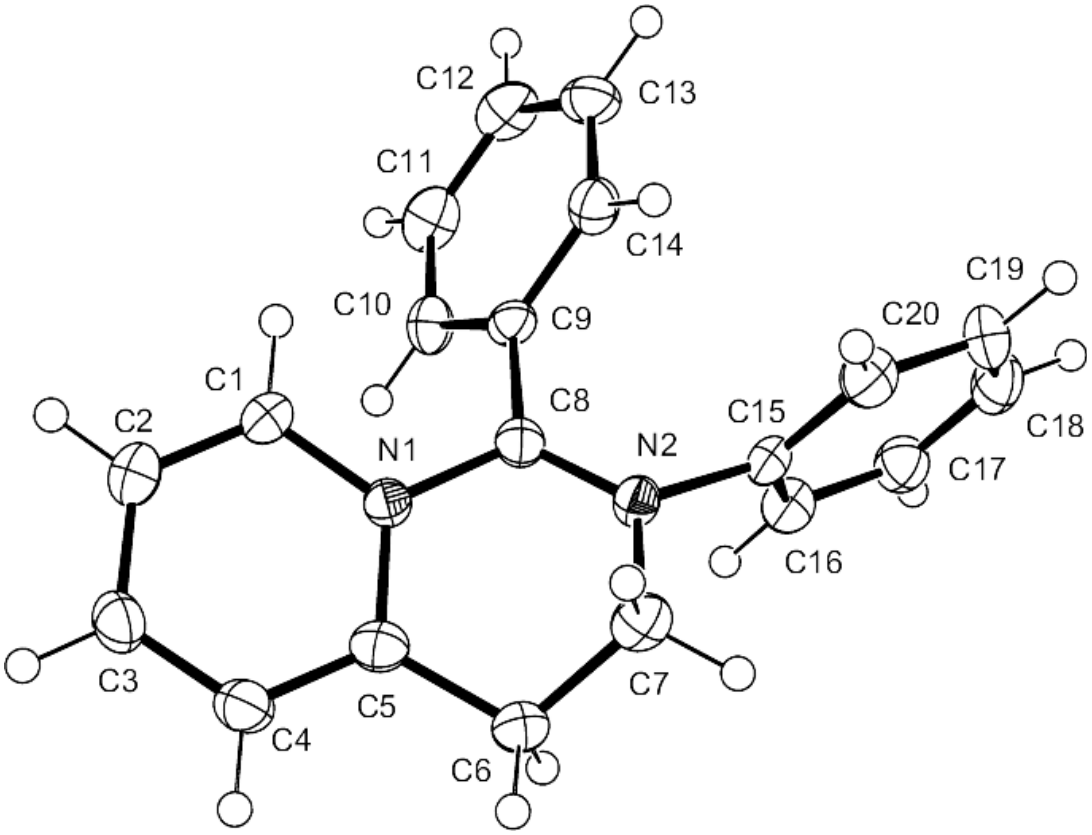
| | |
|---|---|
| O(3)-S(1)-C(21A)-F(2A) | 61.4(7) |
| O(2)-S(1)-C(21A)-F(2A) | -175.5(6) |
| C(21)-S(1)-C(21A)-F(2A) | 128(3) |
| O(1)-S(1)-C(21A)-F(1A) | 56.3(7) |
| O(3)-S(1)-C(21A)-F(1A) | 174.3(6) |
| O(2)-S(1)-C(21A)-F(1A) | -62.6(7) |
| C(21)-S(1)-C(21A)-F(1A) | -120(3) |

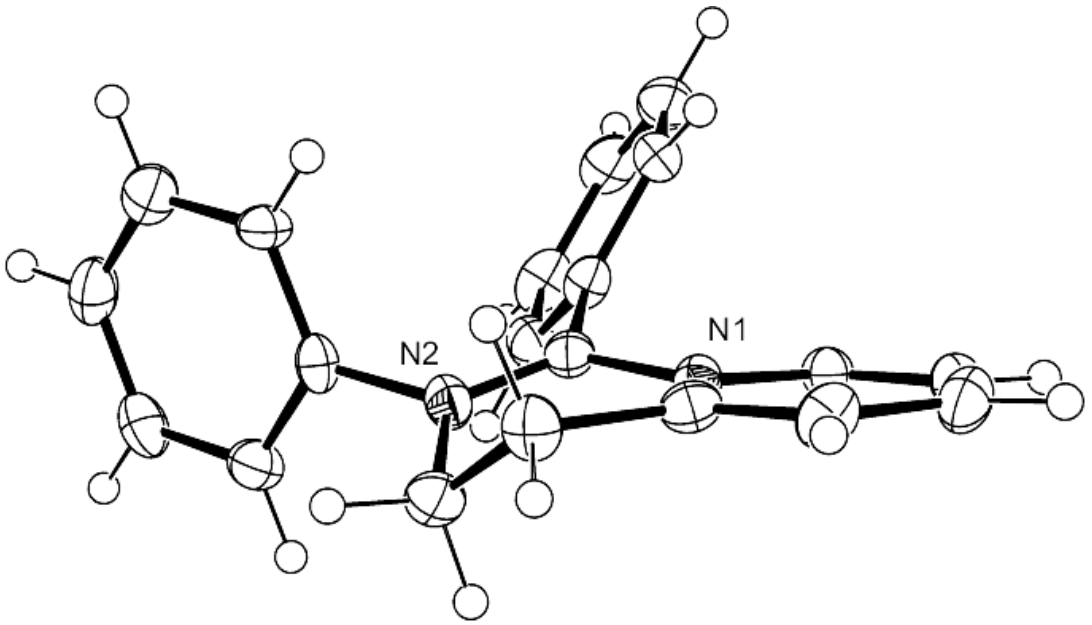
Symmetry transformations used to generate equivalent atoms:






N1
N2


C12
C13
C11
C14
C19
C10
C9
C20
C1
C8
C15
C18
C2
N1
N2
C17
C16
C3
C5
C7
C4
C6


N2
N1

# Appendix 6

Data for Kinetic Study of Reaction of 2-DMAP Disalt **4.16** and *N*,*N*-di-*iso*-propyl-ethylamine **5.21**

Table A1: Data for Figure 5.3.

| **Time/ min.** | **Relative Integral[a]** | | |
|---|---|---|---|
| | **4.16[b]** | **4.22[c]** | **5.31[d]** |
| 8 | 0.2444 | 0.0065 | 0.0040 |
| 17 | 0.2126 | 0.0115 | 0.0051 |
| 33 | 0.2010 | 0.0186 | 0.0082 |
| 50 | 0.1916 | 0.0244 | 0.0086 |
| 67 | 0.1846 | 0.0294 | 0.0081 |
| 81 | 0.1786 | 0.0340 | 0.0080 |
| 98 | 0.1721 | 0.0383 | 0.0085 |
| 115 | 0.1666 | 0.0420 | 0.0084 |
| 131 | 0.1614 | 0.0461 | 0.0084 |
| 148 | 0.1564 | 0.0493 | 0.0077 |
| 165 | 0.1513 | 0.0522 | 0.0083 |
| 181 | 0.1465 | 0.0552 | 0.0081 |
| 198 | 0.1440 | 0.0587 | 0.0085 |
| 215 | 0.1397 | 0.0607 | 0.0080 |
| 231 | 0.1367 | 0.0628 | 0.0075 |
| 248 | 0.1351 | 0.0658 | 0.0078 |
| 265 | 0.1321 | 0.0683 | 0.0077 |
| 281 | 0.1283 | 0.0704 | 0.0080 |
| 298 | 0.1259 | 0.0727 | 0.0079 |
| 315 | 0.1205 | 0.0751 | 0.0080 |
| 331 | 0.1172 | 0.0764 | 0.0080 |
| 348 | 0.1151 | 0.0786 | 0.0081 |
| 364 | 0.1124 | 0.0797 | 0.0083 |
| 381 | 0.1086 | 0.0815 | 0.0083 |
| 398 | 0.1053 | 0.0834 | 0.0083 |

[a] Integral taken relative to 1,3,5,7-cyclooctatetraene **5.29** $\delta_H(CD_3CN)$ 5.77 ppm set at 1.000. [b] Aromatic peak at $\delta_H(CD_3CN)$ 8.63 ppm used. [c] Aromatic peak at $\delta_H(CD_3CN)$ 8.22 ppm used. [d] Peak at $\delta_H(CD_3CN)$ 6.43 ppm used.

Table A2: Data for figure 5.4.

| **Time/ s** | **mmol 4.16** | **Concentration 4.16/ mol.L$^{-1}$** | **1/[4.16] / L.mol$^{-1}$** |
|---|---|---|---|
| 0 | 0.1069 | 0.2138 | 4.6774 |
| 480 | 0.0987 | 0.1975 | 5.0641 |
| 1020 | 0.0856 | 0.1712 | 5.8394 |
| 1980 | 0.0809 | 0.1617 | 6.1827 |
| 3000 | 0.0773 | 0.1545 | 6.4709 |
| 4020 | 0.0744 | 0.1488 | 6.7212 |
| 4860 | 0.0720 | 0.1440 | 6.9461 |
| 5880 | 0.0693 | 0.1387 | 7.2109 |
| 6900 | 0.0671 | 0.1343 | 7.4467 |
| 7860 | 0.0650 | 0.1300 | 7.6933 |
| 8880 | 0.0631 | 0.1261 | 7.9273 |
| 9900 | 0.0610 | 0.1220 | 8.1989 |
| 10860 | 0.0590 | 0.1180 | 8.4744 |
| 11880 | 0.0581 | 0.1162 | 8.6081 |
| 12900 | 0.0561 | 0.1122 | 8.9089 |
| 13860 | 0.0547 | 0.1095 | 9.1343 |
| 14880 | 0.0538 | 0.1076 | 9.2936 |
| 15900 | 0.0524 | 0.1048 | 9.5431 |
| 16860 | 0.0511 | 0.1022 | 9.7819 |
| 17880 | 0.0500 | 0.1000 | 9.9987 |
| 18900 | 0.0483 | 0.0967 | 10.3426 |
| 19860 | 0.0470 | 0.0939 | 10.6478 |
| 20880 | 0.0462 | 0.0924 | 10.8246 |
| 21840 | 0.0452 | 0.0904 | 11.0594 |
| 22860 | 0.0438 | 0.0877 | 11.4035 |
| 23880 | 0.0427 | 0.0854 | 11.7109 |