X-Ray Crystalographic data for Compound 11f

Experimental

Data Collection

A clear crystal of $C_{39}H_{56}N_3O_{12}FS \cdot ½(C_3H_6O)$ having approximate dimensions of 0.40 x 0.40 x 0.40 mm was mounted on a glass fiber. All measurements were made on a Bruker CCD area detectorwith graphite monochromated Mo-KALPHA radiation.

Cell constants and an orientation matrix for data collection corresponded to atetragonal cell with dimensions:

a = 13.7274(2) Å
c = 46.843(1) Å
V = 8828.1(3) $Å^3$

For Z = 8 and F.W. = 837.97, the calculated density is 1.26 g/cm^3. Based on the systematic absences of:

00l: l ± 4n

and the successful solution and refinement of thestructure, the space group was determined to be:

$P4_1$ (#76)

The data were collected at ambient temperature to a maximum 2THETA value of 46.6°.Data were collected in 0.30° oscillations with 30.0 second exposures.The crystal-to-detector distance was 60.00 mm with the detector at the zero swing position.

Data Reduction

Of the 33920 reflections which were collected, 7038 were unique (R_{int} = 0.097); equivalent reflections were merged.

The linear absorption coefficient, MU, for Mo-KALPHA radiation is 1.4 cm^{-1}. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-squares refinement[3] was based on 4753 observed reflections (I > 3.00sigma(I)) and 1045 variable parameters and converged (largest parameter shift was 0.99 times its esd) with unweighted and weighted agreement factors of:

$$R = \text{SIGMA } ||Fo| - |Fc|| / \text{SIGMA } |Fo| = 0.065$$

$$\backslash R_w = [(\text{SIGMA } w (|Fo| - |Fc|)^2 / \text{SIGMA } w Fo^2)]^{1/2} = 0.063$$

The standard deviation of an observation of unit weight[4] was 2.12. The weighting scheme was based on counting statistics. Plots of SIGMA w $(|Fo| - |Fc|)^2$ versus |Fo|,reflection order in data collection, sin THETA/LAMBDA and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.30 and -0.29 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for DELTAf' and DELTAf" were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbel[8]. All calculations were performed using the teXsan[9] crystallographic software package of Molecular Structure Corporation.

References

(1) SHELXS86: Sheldrick, G.M. (1985). In: "Crystallographic Computing 3" (Eds G.M. Sheldrick, C. Kruger and R. Goddard) Oxford University Press, pp. 175-189.

(2) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least-Squares:

Function minimized: $\sum w(|F_o|-|F_c|)^2$

(4) Standard deviation of an observation of unit weight:

$[\sum w(|F_o|-|F_c|)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations

N_v = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1992).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{39}H_{56}N_3O_{12}FS \cdot ½(C_3H_6O)$
Formula Weight	837.97
Crystal Color, Habit	clear, fragment
Crystal Dimensions	0.40 X 0.40 X 0.40 mm
Crystal System	tetragonal
Lattice Type	Primitive
Lattice Parameters	a = 13.7274(2)Å
	c = 46.843(1) Å
	V = 8828.1(3) $Å^3$
Space Group	$P4_1$ (#76)
Z value	8
D_{calc}	1.261 g/cm^3
F_{000}	3576.00
MU(MoKALPHA)	1.40 cm^{-1}

B. Intensity Measurements

Diffractometer	Bruker CCD
Radiation	MoKALPHA (LAMBDA = 0.71069 Å) graphite monochromated
Data Images	1271 exposures @ 30.0 seconds
Detector Position	60.00 mm
$2THETA_{max}$	46.6^{o}
No. of Reflections Measured	Total: 33920 Unique: 7038
(R_{int} = 0.097)	
Corrections	Lorentz-polarization

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELXS86)
Refinement	Full-matrix least-squares
Function Minimized	SIGMA w $(\|Fo\| - \|Fc\|)^2$
Least Squares Weights	$1/sigma^2(Fo) = 4Fo^2/sigma^2(Fo^2)$
p-factor	0.0000
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>3.00sigma(I))	4753
No. Variables	1045
Reflection/Parameter Ratio	4.55
Residuals: R; Rw	0.065 ; 0.063
Goodness of Fit Indicator	2.12
Max Shift/Error in Final Cycle	0.99
Maximum peak in Final Diff. Map	0.30 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.29 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
S(1)	0.2481(2)	-0.6331(1)	0.11722(5)	5.29(6)
S(2)	-0.4766(2)	-0.1329(2)	-0.04455(5)	5.92(6)
F(1)	0.0908(3)	-1.2877(3)	0.09068(9)	5.2(1)
F(2)	-0.2960(3)	-0.7844(2)	-0.01433(9)	5.2(1)
O(1)	0.1573(3)	-1.4271(3)	-0.0054(1)	3.8(1)
O(2)	0.2680(3)	-1.4726(3)	-0.0387(1)	4.1(1)
O(3)	0.1192(3)	-1.6216(3)	0.0083(1)	5.2(1)
O(4)	0.1606(4)	-0.5824(4)	0.1122(1)	7.0(2)
O(5)	0.3333(4)	-0.5748(4)	0.1181(1)	9.0(2)
O(6)	0.1785(3)	-1.1667(3)	0.0091(1)	3.9(1)
O(7)	0.3107(3)	-1.2558(3)	0.0537(1)	4.8(1)
O(8)	0.0814(3)	-1.1067(3)	0.0848(1)	4.5(1)
O(9)	0.2404(3)	-1.0815(3)	0.0921(1)	6.0(2)
O(10)	0.0451(4)	-1.0158(3)	-0.0294(1)	6.0(2)
O(11)	-0.0493(3)	-0.8881(3)	0.0636(1)	4.8(1)
O(12)	-0.0122(3)	-0.7571(3)	0.0375(1)	6.1(1)
O(13)	-0.4741(3)	-0.9647(3)	0.1167(1)	4.5(1)
O(14)	-0.3401(3)	-1.1172(3)	0.0660(1)	4.9(1)
O(15)	-0.3671(3)	-0.9220(3)	0.0813(1)	3.7(1)
O(16)	-0.3897(3)	-0.6602(3)	0.0675(1)	3.5(1)
O(17)	-0.2626(3)	-0.5147(3)	0.1093(1)	6.5(2)
O(18)	-0.2062(3)	-0.2485(3)	0.0428(1)	6.0(1)
O(19)	-0.1567(3)	-0.3830(3)	0.0172(1)	5.0(1)
O(20)	-0.5197(3)	-0.7513(3)	0.0209(1)	5.6(1)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
O(21)	-0.2840(3)	-0.6035(3)	-0.0041(1)	4.6(1)
O(22)	-0.4373(3)	-0.5727(3)	-0.0195(1)	7.1(2)
O(24)	-0.4046(4)	-0.0616(3)	-0.0356(1)	8.2(2)
O(25)	-0.5732(4)	-0.1026(4)	-0.0511(1)	8.4(2)
O(26)	0.0610(5)	-1.2139(6)	0.1610(2)	12.4(3)
N(1)	0.1680(5)	-1.7678(4)	-0.0338(1)	5.7(2)
N(2)	0.0390(4)	-0.9102(4)	0.0245(1)	3.9(1)
N(3)	-0.3892(4)	-1.2637(4)	0.1073(1)	5.1(2)
N(4)	-0.2530(4)	-0.4029(4)	0.0559(1)	4.8(2)
N(5)	0.3146(4)	-0.8024(4)	0.0993(1)	4.3(2)
N(6)	-0.5280(4)	-0.3051(4)	-0.0251(1)	4.1(2)
C(1)	0.2205(4)	-1.5004(5)	-0.0123(2)	3.9(2)
C(2)	0.1612(5)	-1.5940(5)	-0.0181(2)	4.2(2)
C(3)	0.2251(5)	-1.6756(5)	-0.0305(2)	4.8(2)
C(4)	0.2800(5)	-1.6380(5)	-0.0581(2)	4.7(2)
C(5)	0.3340(4)	-1.5469(5)	-0.0491(2)	4.0(2)
C(6)	0.3908(5)	-1.4980(6)	-0.0734(2)	6.1(2)
C(7)	0.2323(6)	-1.8511(5)	-0.0379(2)	7.6(3)
C(8)	0.0878(6)	-1.7621(6)	-0.0558(2)	9.4(3)
C(9)	0.2355(7)	-0.6991(6)	0.1493(2)	8.4(3)
C(10)	0.2607(5)	-0.7252(5)	0.0907(2)	4.0(2)
C(12)	0.2199(5)	-0.7138(4)	0.0640(2)	5.2(2)
C(13)	0.3244(5)	-0.8737(5)	0.0799(2)	4.3(2)
C(14)	0.2328(5)	-0.7870(5)	0.0450(2)	4.6(2)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
C(15)	0.2796(5)	-0.8755(5)	0.0532(2)	3.8(2)
C(16)	0.2834(4)	-0.9572(5)	0.0342(2)	3.8(2)
C(17)	0.2800(4)	-1.0302(4)	0.0215(2)	3.8(2)
C(18)	0.2705(5)	-1.1205(5)	0.0034(2)	4.7(2)
C(19)	0.1966(5)	-1.3451(4)	0.0089(2)	3.5(2)
C(20)	0.1555(5)	-1.2505(4)	-0.0074(2)	3.5(1)
C(21)	0.1998(5)	-1.2487(4)	-0.0384(2)	3.7(2)
C(22)	0.2302(5)	-1.2762(4)	0.0584(2)	3.4(2)
C(23)	0.1836(5)	-1.2441(5)	0.0876(2)	4.3(2)
C(24)	0.1740(5)	-1.1330(5)	0.0876(2)	3.9(2)
C(25)	0.1868(5)	-1.4500(5)	0.0539(2)	5.4(2)
C(26)	0.1695(4)	-1.3475(5)	0.0404(2)	3.2(2)
C(27)	0.2437(6)	-1.2800(5)	0.1126(2)	6.3(2)
C(28)	0.0597(5)	-0.9999(5)	0.0852(2)	4.5(2)
C(29)	0.0403(5)	-0.9675(5)	0.1155(2)	6.0(2)
C(30)	0.1200(7)	-0.9569(8)	0.1356(2)	10.5(3)
C(31)	0.0448(5)	-1.2557(4)	-0.0094(2)	3.8(2)
C(32)	-0.0063(5)	-1.1781(4)	-0.0284(1)	3.5(2)
C(33)	-0.1147(5)	-1.2085(5)	-0.0330(2)	5.0(2)
C(34)	0.0003(5)	-1.0785(5)	-0.0168(2)	4.2(2)
C(35)	-0.0579(5)	-1.0527(5)	0.0100(2)	4.8(2)
C(36)	-0.1436(6)	-0.9860(6)	0.0022(2)	8.9(3)
C(37)	0.0125(4)	-1.0068(4)	0.0324(2)	3.9(2)
C(38)	-0.0249(5)	-0.9924(5)	0.0633(2)	4.5(2)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
C(39)	-0.1146(5)	-1.0470(5)	0.0714(2)	6.1(2)
C(40)	-0.0054(5)	-0.8434(5)	0.0402(2)	4.6(2)
C(41)	-0.3113(6)	-1.2688(6)	0.1276(2)	8.1(3)
C(42)	-0.4406(4)	-1.1694(5)	0.1067(2)	3.7(2)
C(43)	-0.3749(4)	-1.0932(4)	0.0931(2)	3.5(2)
C(44)	-0.4854(5)	-1.1329(5)	0.1338(2)	5.5(2)
C(45)	-0.5380(5)	-1.0349(5)	0.1293(2)	4.7(2)
C(46)	-0.5790(6)	-0.9874(6)	0.1546(2)	7.0(3)
C(47)	-0.4331(5)	-0.9957(5)	0.0907(2)	3.7(2)
C(48)	-0.4079(5)	-0.8342(4)	0.0670(2)	3.9(2)
C(49)	-0.3774(5)	-0.8412(5)	0.0363(2)	3.7(2)
C(50)	-0.3970(6)	-0.9432(5)	0.0228(2)	6.4(2)
C(51)	-0.4174(5)	-0.7432(5)	0.1138(2)	5.1(2)
C(52)	-0.3667(5)	-0.7471(5)	0.0838(2)	3.9(2)
C(53)	-0.2605(5)	-0.7523(4)	0.0872(1)	3.6(2)
C(54)	-0.4591(5)	-1.3423(5)	0.1121(2)	7.5(3)
C(55)	-0.2132(5)	-0.6789(5)	0.1086(2)	4.1(2)
C(56)	-0.1030(5)	-0.7056(5)	0.1146(2)	5.9(2)
C(57)	-0.2167(5)	-0.5737(5)	0.0974(2)	4.0(2)
C(58)	-0.1534(5)	-0.5483(5)	0.0704(2)	4.9(2)
C(59)	-0.0733(5)	-0.4792(5)	0.0807(2)	7.6(3)
C(60)	-0.2214(5)	-0.5059(4)	0.0479(2)	3.8(2)
C(61)	-0.2051(5)	-0.3355(5)	0.0394(2)	4.3(2)
C(62)	-0.1811(5)	-0.4891(5)	0.0189(2)	4.3(2)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
C(63)	-0.0910(5)	-0.5436(5)	0.0104(2)	5.9(2)
C(64)	-0.4319(5)	-0.7712(4)	0.0169(2)	3.7(2)
C(65)	-0.3855(5)	-0.7411(5)	-0.0120(2)	5.0(2)
C(66)	-0.4512(6)	-0.7735(5)	-0.0362(2)	6.5(2)
C(67)	-0.3751(5)	-0.6295(5)	-0.0119(2)	4.3(2)
C(68)	-0.2627(5)	-0.5017(5)	-0.0052(2)	4.4(2)
C(69)	-0.4825(5)	-0.6115(4)	0.0712(2)	4.5(2)
C(70)	-0.4860(4)	-0.5267(5)	0.0535(2)	3.6(2)
C(71)	-0.4939(5)	-0.4540(5)	0.0390(2)	4.2(2)
C(72)	-0.4937(4)	-0.3703(5)	0.0205(2)	3.7(2)
C(73)	-0.5339(5)	-0.3754(4)	-0.0067(2)	3.8(2)
C(74)	-0.4466(5)	-0.2847(5)	0.0284(2)	5.1(2)
C(75)	-0.4402(5)	-0.2083(5)	0.0091(2)	5.1(2)
C(77)	-0.4840(5)	-0.2261(5)	-0.0177(2)	4.7(2)
C(79)	-0.2321(6)	-0.4680(6)	-0.0362(2)	6.4(2)
C(80)	-0.3022(7)	-0.4126(8)	-0.0513(2)	13.3(4)
C(82)	-0.0305(8)	-1.2278(9)	0.1634(3)	12.1(4)
C(83)	-0.0566(9)	-1.3238(9)	0.1489(3)	14.5(5)
C(84)	-0.0919(9)	-1.1594(9)	0.1745(3)	16.6(5)
C(85)	-0.4307(6)	-0.1917(5)	-0.0759(2)	6.2(2)
H(1)	0.0840	-0.8957	0.0092	4.2187
H(2)	-0.3008	-0.3876	0.0701	4.9635
H(3)	0.2675	-1.5109	0.0021	3.6699
H(4)	0.1112	-1.5818	-0.0312	4.1531

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(5)	0.2766	-1.6868	-0.0169	4.5396
H(6)	0.3209	-1.6860	-0.0651	5.0246
H(7)	0.2309	-1.6221	-0.0721	5.0246
H(8)	0.3787	-1.5641	-0.0345	4.1696
H(9)	0.4364	-1.5423	-0.0810	6.5043
H(10)	0.3452	-1.4803	-0.0882	6.5043
H(11)	0.4217	-1.4417	-0.0666	6.5043
H(12)	0.1940	-1.9070	-0.0398	7.4682
H(13)	0.2680	-1.8396	-0.0552	7.4682
H(14)	0.2748	-1.8547	-0.0224	7.4682
H(15)	0.0497	-1.7085	-0.0504	8.7475
H(16)	0.1179	-1.7531	-0.0734	8.7475
H(17)	0.0535	-1.8205	-0.0547	8.7475
H(19)	0.1818	-0.6569	0.0591	4.4117
H(20)	0.3636	-0.9270	0.0859	4.7436
H(21)	0.2118	-0.7793	0.0255	5.0762
H(22)	0.3232	-1.1632	0.0075	5.6833
H(23)	0.2745	-1.1022	-0.0165	5.6833
H(24)	0.2658	-1.3452	0.0074	4.8848
H(25)	0.1812	-1.3081	-0.0482	4.2753
H(26)	0.1778	-1.1951	-0.0486	4.2753
H(27)	0.2694	-1.2483	-0.0371	4.2753
H(28)	0.1657	-1.4485	0.0731	5.1997
H(29)	0.1503	-1.4965	0.0434	5.1997

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(30)	0.2537	-1.4640	0.0528	5.1997
H(31)	0.1011	-1.3302	0.0419	3.1744
H(32)	0.2461	-1.3480	0.1131	7.3436
H(33)	0.3086	-1.2542	0.1106	7.3436
H(34)	0.2163	-1.2548	0.1297	7.3436
H(35)	0.0271	-1.3182	-0.0162	3.8442
H(36)	0.0200	-1.2480	0.0097	3.8442
H(37)	0.0242	-1.1781	-0.0464	3.5522
H(38)	-0.1443	-1.2119	-0.0142	4.9662
H(39)	-0.1453	-1.1643	-0.0442	4.9662
H(40)	-0.1150	-1.2725	-0.0408	4.9662
H(41)	-0.0803	-1.1135	0.0185	5.0900
H(42)	-0.1225	-0.9297	-0.0064	10.4018
H(43)	-0.1873	-1.0213	-0.0103	10.4018
H(44)	-0.1806	-0.9710	0.0193	10.4018
H(45)	0.0705	-1.0434	0.0330	2.7696
H(46)	-0.1326	-1.0286	0.0907	6.7005
H(47)	-0.1662	-1.0322	0.0591	6.7005
H(48)	-0.1036	-1.1161	0.0716	6.7005
H(49)	-0.2832	-1.3328	0.1249	9.7304
H(50)	-0.2669	-1.2212	0.1232	9.7304
H(51)	-0.3384	-1.2639	0.1456	9.7304
H(52)	-0.4925	-1.1754	0.0933	3.6531
H(53)	-0.3204	-1.0835	0.1055	3.4923

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(54)	-0.5301	-1.1809	0.1390	5.0244
H(55)	-0.4349	-1.1259	0.1467	5.0244
H(56)	-0.5900	-1.0456	0.1160	6.9311
H(57)	-0.5251	-0.9743	0.1682	8.6199
H(58)	-0.6083	-0.9283	0.1503	8.6199
H(59)	-0.6227	-1.0297	0.1643	8.6199
H(60)	-0.4847	-1.0035	0.0766	4.6699
H(61)	-0.4781	-0.8346	0.0686	4.7569
H(62)	-0.3080	-0.8285	0.0349	3.8372
H(63)	-0.4671	-0.9551	0.0241	6.7433
H(64)	-0.3766	-0.9472	0.0044	6.7433
H(65)	-0.3666	-0.9923	0.0346	6.7433
H(66)	-0.3921	-0.6890	0.1239	6.0431
H(67)	-0.4856	-0.7343	0.1109	6.0431
H(68)	-0.4055	-0.8012	0.1237	6.0431
H(69)	-0.2451	-0.8159	0.0936	4.4519
H(70)	-0.2314	-0.7412	0.0690	4.4519
H(71)	-0.4825	-1.3369	0.1322	7.8931
H(72)	-0.5117	-1.3407	0.1002	7.8931
H(73)	-0.4262	-1.4041	0.1113	7.8931
H(74)	-0.2498	-0.6800	0.1260	4.7179
H(75)	-0.1039	-0.7704	0.1233	7.2608
H(76)	-0.0704	-0.7090	0.0973	7.2608
H(77)	-0.0773	-0.6614	0.1273	7.2608

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(78)	-0.1278	-0.6080	0.0630	5.8478
H(79)	-0.0352	-0.5122	0.0954	8.5565
H(80)	-0.0314	-0.4634	0.0656	8.5565
H(81)	-0.1006	-0.4227	0.0889	8.5565
H(82)	-0.2775	-0.5446	0.0472	3.8658
H(83)	-0.1017	-0.6117	0.0109	5.4943
H(84)	-0.0698	-0.5246	-0.0080	5.4943
H(85)	-0.0406	-0.5278	0.0239	5.4943
H(86)	-0.5108	-0.7430	-0.0341	7.9533
H(87)	-0.4199	-0.7535	-0.0535	7.9533
H(88)	-0.4557	-0.8415	-0.0356	7.9533
H(89)	-0.3168	-0.4642	0.0004	4.1930
H(90)	-0.5333	-0.6549	0.0652	4.9027
H(91)	-0.4915	-0.5940	0.0902	4.9027
H(92)	-0.5655	-0.4325	-0.0115	4.1254
H(93)	-0.4190	-0.2784	0.0475	5.5807
H(94)	-0.4077	-0.1509	0.0141	5.1600
H(96)	0.1156	-0.9686	0.0782	5.7915
H(97)	0.0123	-0.9009	0.1131	6.1976
H(98)	-0.0113	-1.0062	0.1229	6.1976
H(99)	-0.1716	-0.4332	-0.0338	7.5327

$B_{eq} = 8/3\ PI^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos GAMMA + 2U_{13}(aa^*cc^*)\cos BETA + 2U_{23}(bb^*cc^*)\cos ALPHA)$

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	0.094(2)	0.051(1)	0.056(2)	0.019(1)	0.010(2)	-0.001(1)
S(2)	0.097(2)	0.064(1)	0.064(2)	-0.012(1)	0.015(2)	-0.003(1)
F(1)	0.062(2)	0.055(3)	0.081(4)	-0.010(2)	0.013(3)	-0.003(3)
F(2)	0.072(3)	0.048(3)	0.079(4)	0.001(2)	0.024(3)	0.002(2)
O(1)	0.041(3)	0.031(2)	0.073(4)	-0.003(2)	0.012(3)	-0.003(3)
O(2)	0.058(3)	0.038(3)	0.061(4)	-0.011(2)	-0.001(3)	-0.016(3)
O(3)	0.076(3)	0.050(3)	0.072(4)	0.019(3)	0.021(3)	0.009(3)
O(4)	0.099(3)	0.101(4)	0.068(4)	0.040(3)	0.014(4)	-0.004(4)
O(5)	0.125(4)	0.092(4)	0.126(6)	-0.038(3)	-0.009(5)	-0.010(4)
O(6)	0.051(3)	0.034(3)	0.062(4)	-0.003(2)	0.010(3)	-0.011(2)
O(7)	0.050(3)	0.048(3)	0.086(4)	-0.008(3)	0.011(3)	-0.009(3)
O(8)	0.052(3)	0.041(2)	0.079(4)	0.002(2)	-0.002(3)	0.010(3)
O(9)	0.068(3)	0.055(3)	0.103(5)	-0.009(3)	-0.028(3)	0.003(3)
O(10)	0.098(4)	0.045(3)	0.085(5)	-0.015(3)	-0.003(4)	0.010(3)
O(11)	0.050(3)	0.045(3)	0.087(4)	0.012(2)	0.008(3)	-0.004(3)
O(12)	0.089(4)	0.037(3)	0.108(5)	0.009(3)	0.007(4)	0.023(3)
O(13)	0.064(3)	0.043(3)	0.064(4)	0.004(2)	0.016(3)	0.000(3)
O(14)	0.067(3)	0.044(3)	0.076(4)	-0.012(3)	0.015(3)	-0.017(3)
O(15)	0.050(3)	0.039(3)	0.052(4)	-0.002(2)	-0.006(3)	0.004(2)
O(16)	0.047(3)	0.032(3)	0.053(3)	0.000(2)	0.003(3)	-0.012(2)
O(17)	0.089(4)	0.046(3)	0.111(5)	0.020(3)	0.010(4)	-0.032(3)
O(18)	0.072(3)	0.044(3)	0.112(5)	-0.008(3)	0.015(3)	0.000(3)
O(19)	0.053(3)	0.041(2)	0.096(4)	-0.011(2)	0.027(3)	-0.008(3)
O(20)	0.065(3)	0.080(4)	0.067(4)	0.019(3)	0.002(3)	0.007(3)

Table 2. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(21)	0.053(3)	0.039(2)	0.083(4)	-0.008(2)	0.022(3)	-0.002(3)
O(22)	0.056(3)	0.061(3)	0.153(6)	0.006(3)	-0.008(4)	-0.006(4)
O(24)	0.165(5)	0.076(4)	0.072(5)	-0.075(3)	0.011(4)	-0.006(3)
O(25)	0.103(3)	0.106(4)	0.110(5)	0.036(3)	0.033(4)	0.050(4)
O(26)	0.122(5)	0.211(7)	0.139(6)	-0.013(6)	-0.001(5)	0.013(6)
N(1)	0.117(5)	0.027(3)	0.072(5)	-0.019(3)	0.018(4)	-0.002(4)
N(2)	0.047(4)	0.045(3)	0.056(5)	-0.007(3)	0.004(3)	-0.005(3)
N(3)	0.059(4)	0.048(3)	0.087(6)	0.003(3)	0.003(3)	0.003(4)
N(4)	0.059(4)	0.041(3)	0.084(6)	0.013(3)	0.029(4)	0.004(3)
N(5)	0.060(4)	0.042(3)	0.063(5)	-0.003(3)	0.007(4)	0.010(3)
N(6)	0.053(4)	0.050(3)	0.051(5)	0.002(3)	0.004(4)	-0.006(3)
C(1)	0.015(4)	0.051(4)	0.084(6)	0.002(2)	0.011(4)	0.002(4)
C(2)	0.046(5)	0.050(4)	0.062(5)	-0.004(3)	-0.009(4)	-0.015(4)
C(3)	0.061(5)	0.031(4)	0.090(6)	0.004(3)	-0.002(4)	0.009(4)
C(4)	0.068(5)	0.044(4)	0.067(6)	-0.007(4)	-0.023(4)	-0.011(4)
C(5)	0.031(4)	0.053(4)	0.067(6)	-0.006(3)	-0.008(4)	-0.008(4)
C(6)	0.092(6)	0.089(6)	0.052(6)	-0.014(5)	0.014(5)	-0.006(5)
C(7)	0.156(8)	0.027(4)	0.108(8)	0.002(4)	0.032(7)	-0.020(5)
C(8)	0.099(7)	0.077(6)	0.18(1)	-0.032(5)	-0.061(6)	-0.039(7)
C(9)	0.20(1)	0.082(6)	0.036(5)	0.051(6)	-0.024(6)	0.003(4)
C(10)	0.050(5)	0.037(4)	0.065(5)	-0.010(3)	0.004(4)	0.000(3)
C(12)	0.115(6)	0.014(4)	0.068(5)	0.029(4)	-0.024(5)	0.003(4)
C(13)	0.062(5)	0.038(4)	0.062(5)	0.017(4)	-0.010(4)	0.015(4)
C(14)	0.108(6)	0.030(4)	0.036(5)	-0.022(4)	-0.006(5)	0.009(3)

Table 2. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(15)	0.044(5)	0.045(4)	0.057(5)	-0.015(3)	0.009(4)	0.003(3)
C(16)	0.047(4)	0.030(4)	0.067(6)	0.004(4)	0.009(5)	0.016(3)
C(17)	0.041(4)	0.024(4)	0.080(7)	0.001(4)	0.004(5)	0.024(3)
C(18)	0.049(4)	0.047(4)	0.082(7)	-0.009(3)	0.001(5)	-0.008(4)
C(19)	0.049(4)	0.033(3)	0.050(4)	0.022(3)	0.000(4)	-0.010(3)
C(20)	0.058(3)	0.030(3)	0.046(4)	0.006(3)	0.007(4)	0.000(3)
C(21)	0.057(5)	0.034(4)	0.050(5)	-0.004(4)	0.021(4)	0.009(4)
C(22)	0.043(3)	0.037(4)	0.051(5)	0.010(3)	-0.017(3)	0.012(4)
C(23)	0.055(4)	0.047(3)	0.060(5)	-0.001(3)	-0.008(4)	-0.010(4)
C(24)	0.054(3)	0.050(3)	0.044(5)	0.012(3)	-0.007(5)	-0.023(4)
C(25)	0.072(5)	0.068(5)	0.065(6)	0.021(4)	0.013(5)	0.024(4)
C(26)	0.015(4)	0.058(4)	0.050(4)	0.008(3)	-0.002(4)	0.002(4)
C(27)	0.103(6)	0.075(6)	0.061(6)	0.023(5)	-0.022(5)	0.012(5)
C(28)	0.055(5)	0.046(3)	0.071(5)	0.020(4)	0.003(4)	0.016(4)
C(29)	0.070(5)	0.076(6)	0.081(5)	0.009(5)	0.030(4)	-0.013(5)
C(30)	0.104(7)	0.22(1)	0.073(8)	-0.052(7)	0.008(5)	-0.054(8)
C(31)	0.057(3)	0.034(4)	0.054(6)	0.024(3)	0.035(4)	0.003(4)
C(32)	0.056(4)	0.045(3)	0.031(5)	0.009(3)	0.003(4)	-0.006(3)
C(33)	0.060(4)	0.049(5)	0.080(7)	-0.003(4)	-0.016(5)	0.010(5)
C(34)	0.058(5)	0.038(3)	0.064(6)	-0.001(3)	-0.036(4)	0.000(3)
C(35)	0.052(4)	0.040(4)	0.089(6)	-0.002(3)	-0.011(3)	-0.024(4)
C(36)	0.077(6)	0.101(6)	0.16(1)	0.051(5)	-0.061(6)	-0.011(7)
C(37)	0.022(4)	0.034(3)	0.092(5)	0.005(3)	0.007(4)	-0.024(4)
C(38)	0.041(4)	0.046(3)	0.085(5)	0.006(3)	0.001(3)	-0.001(5)

Table 2. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(39)	0.060(5)	0.070(6)	0.102(8)	-0.013(4)	0.035(5)	0.004(5)
C(40)	0.071(5)	0.034(3)	0.071(6)	0.008(4)	-0.009(4)	-0.009(4)
C(41)	0.080(6)	0.077(6)	0.152(9)	0.002(5)	-0.064(5)	0.014(6)
C(42)	0.037(4)	0.049(4)	0.057(5)	-0.007(3)	-0.026(4)	0.010(4)
C(43)	0.033(4)	0.035(4)	0.065(5)	-0.001(3)	-0.024(4)	-0.006(4)
C(44)	0.081(6)	0.060(5)	0.067(6)	0.005(4)	0.010(5)	0.007(5)
C(45)	0.059(5)	0.073(5)	0.048(5)	0.001(3)	0.007(4)	0.031(4)
C(46)	0.118(7)	0.067(6)	0.082(7)	0.016(5)	0.057(6)	0.003(5)
C(47)	0.044(4)	0.036(4)	0.061(5)	0.006(2)	-0.001(4)	0.004(4)
C(48)	0.070(5)	0.027(3)	0.051(4)	-0.006(3)	0.019(4)	0.004(3)
C(49)	0.050(4)	0.043(4)	0.049(4)	0.003(4)	0.010(4)	-0.011(4)
C(50)	0.148(7)	0.035(4)	0.059(6)	-0.002(5)	-0.014(6)	0.003(4)
C(51)	0.070(5)	0.061(5)	0.062(5)	-0.007(4)	0.024(5)	0.028(5)
C(52)	0.059(4)	0.035(3)	0.056(5)	-0.016(4)	0.009(4)	0.003(3)
C(53)	0.063(4)	0.044(4)	0.031(5)	0.006(4)	-0.004(4)	0.001(4)
C(54)	0.069(5)	0.056(5)	0.16(1)	-0.012(4)	-0.007(6)	0.007(6)
C(55)	0.063(4)	0.037(4)	0.055(6)	-0.015(4)	0.003(4)	-0.007(3)
C(56)	0.073(4)	0.088(6)	0.062(6)	0.013(5)	-0.021(5)	-0.001(5)
C(57)	0.054(5)	0.036(3)	0.060(5)	0.004(3)	-0.025(4)	-0.013(3)
C(58)	0.057(5)	0.053(5)	0.078(6)	0.004(3)	-0.002(3)	0.003(4)
C(59)	0.080(6)	0.069(6)	0.139(9)	-0.029(4)	-0.029(6)	-0.001(6)
C(60)	0.048(5)	0.029(3)	0.068(5)	-0.016(3)	0.017(4)	0.008(4)
C(61)	0.039(4)	0.044(3)	0.078(6)	0.002(4)	-0.004(4)	0.005(4)
C(62)	0.054(4)	0.040(3)	0.069(5)	-0.002(3)	0.020(3)	0.012(4)

Table 2. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(63)	0.056(5)	0.041(5)	0.128(8)	0.002(3)	0.037(5)	0.007(5)
C(64)	0.066(4)	0.016(4)	0.060(5)	0.001(3)	0.001(4)	-0.026(3)
C(65)	0.066(4)	0.047(3)	0.076(5)	-0.009(4)	0.013(4)	0.010(5)
C(66)	0.103(7)	0.050(5)	0.093(7)	0.002(5)	-0.022(5)	-0.017(5)
C(67)	0.058(4)	0.048(3)	0.060(6)	-0.007(3)	0.014(4)	-0.012(4)
C(68)	0.051(5)	0.033(3)	0.086(5)	0.013(4)	0.006(4)	0.000(4)
C(69)	0.051(4)	0.034(4)	0.086(7)	0.007(3)	0.003(5)	-0.011(3)
C(70)	0.022(4)	0.039(4)	0.075(6)	-0.002(4)	0.013(4)	-0.009(3)
C(71)	0.049(5)	0.051(4)	0.058(6)	0.010(4)	-0.005(5)	-0.011(3)
C(72)	0.037(4)	0.047(4)	0.058(5)	-0.007(3)	0.025(4)	-0.013(3)
C(73)	0.053(5)	0.029(4)	0.061(5)	-0.003(4)	0.010(4)	-0.018(3)
C(74)	0.051(5)	0.056(4)	0.085(7)	-0.026(4)	-0.011(5)	0.000(4)
C(75)	0.061(5)	0.071(5)	0.062(5)	-0.020(4)	0.009(5)	0.000(4)
C(77)	0.056(5)	0.063(4)	0.060(5)	-0.019(4)	0.019(4)	-0.010(3)
C(79)	0.084(6)	0.082(6)	0.079(6)	-0.016(4)	0.028(5)	-0.022(5)
C(80)	0.137(9)	0.28(1)	0.094(9)	0.104(8)	0.035(7)	0.092(8)
C(82)	0.122(6)	0.18(1)	0.15(1)	-0.001(7)	0.019(9)	-0.028(8)
C(83)	0.20(1)	0.20(1)	0.15(1)	-0.05(1)	-0.05(1)	-0.021(9)
C(84)	0.18(1)	0.20(1)	0.25(1)	-0.032(8)	0.12(1)	-0.13(1)
C(85)	0.107(7)	0.071(6)	0.059(5)	-0.014(5)	0.024(5)	-0.007(4)

The general temperature factor expression:

$\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$