Revised

Supporting Information

Crystal Structure of hydrated 4,4'-Bis(1-cyclobutene-4-hydroxy-2,3-dione)biphenyl (BSQB)

Title: Proton-Relay in a One-dimensional Hydrogen-bonded Chain Composed of Water Molecules and a Squaric Acid Derivative.

Authors: Hiroshi Terao, Tadashi Sugawara*, Yasuo Kita[†], Naoki Sato[†], Eriko Kaho[‡], Sadamu Takeda[‡]

Contribution from the Department of Basic Science, Graduate School of Arts and Sciences, The University of Tokyo, Komaba, Meguro, Tokyo 153-8902, Japan, [†]the Institute for Chemical Research, Kyoto University, Gokasho, Uji, Kyoto 611-0011, Japan, and [‡]the Division of Chemistry, Graduate School of Science, Hokkaido University, Sapporo 060-0810, Japan

Contents.

Experimental

Data Collection

A single crystal of **BSQB** with approximate dimensions of 0.40 x 0.40 x 0.20 mm was sealed into a glass fiber with an aliqont of water, then mounted. All measurements were made on a Rigaku R-AXIS-IV imaging plate area detector with graphite monochromated Mo-Kα radiation.

Indexing was performed from 3 oscillations which were exposed for 5.0 minutes. The crystal-to-detector distance was 105.0 mm with the detector at the zero swing position. Readout was performed in the 0.1 mm pixel mode.

Cell constants and an orientation matrix for data collection corresponded to a primitive triclinic cell with dimensions listed in Table 1.

A number of containing water molecules was determined by flotation method. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be $P\bar{1}$ (#2)

The data were collected to a maximum 2θ value of 56.3°. A total of thirtyfive 3.00° oscillation images were collected, each being exposed for 55.0 minutes. The crystal-to-detector distance was 105.0 mm with the detector at the zero swing position. Readout was performed in the 0.1 mm pixel mode.

Data Reduction

The linear absorption coefficient, μ, for Mo-Kα radiation is 19.2 cm^{-1} at 103 K. Azimuthal scans of several reflections indicated no need for an absorption correction. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by a direct method (SIR92) 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 the reflections of $I > 3.00\sigma$ (I) and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \sigma \, ||Fo| - |Fc|| \, / \, \sigma \, |Fo|$$

$$R_w = [(\sigma \, w \, (|Fo| - |Fc|)^2 \, / \, \sigma \, w \, Fo^2)]^{1/2}$$

listed in Table 1

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for $\Delta f'$ and $\Delta f''$ 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) ORIENT: Beurskens, P. T.; Admiraal, G.; Beurskens, G.; Bosman, W. P.; Garcia-Granda, S.;Gould, R. O.; Smits, J. M. M. and Smykalla, C. (1992). The DIRDIF program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(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: $\Sigma\, w(|F_o|-|F_c|)^2$ where
$w = 1/[\sigma^2(Fo)] = [\sigma^2_c(Fo) + p^2Fo^2/4]^{-1}$
$\sigma_c(Fo)$ = e.s.d. based on counting statistics
p = p-factor

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

$[\Sigma\, w(|F_o|-|F_c|)^2/(N_o-N_v)]^{1/2}$
where: N_o = number of observations
N_v = number of variables

(5) Cromer, D. T. and 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. and Hamilton, W. C.; *Acta Cryst.*, 17, 781 (1964).

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

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

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

Experimental Details

Table 1

Formula	$C_{50}H_{45}O_{25}$		
Formula Weight	1045.89		
Crystal Color, Habit	Yellow, Block		
Crystal Dimensions	0.4 x 0.4 x 0.2		
Temperature / K	103(1)	200(1)	273(1)
Crystal System	triclinic	triclinic	triclinic
Lattice Type	P	P	P
Cell Determination (2θ range)		$4 < 2\theta < 20$	
a / Å	14.095(3)	14.123(1)	14.202(2)
b / Å	15.649(4)	15.694(1)	15.805(3)
c / Å	11.585(2)	11.649(1)	11.653(2)
α / °	109.30(1)	109.865(4)	110.15(1)
β / °	97.92(1)	98.362(5)	98.138(10)
γ / °	94.69(1)	94.384(5)	94.54(1)
V / Å^3	2366.3(1)	2380.2(1)	2407.6(1)
Space Group		$P\bar{1}$	
Z value		2	
D_{calc} / gcm^{-3}	1.468	1.459	1.443
D_{obs} / gcm^{-3}			1.44
μ(MoKα) /mm^{-1}	1.917	1.906	1.884
Diffractometer	Rigku R-Axis-IV		
Radiation	Mo-Kα (λ = 0.7107 Å) graphite monochromated		
Crystal-to-Detector Distance / mm	105		
$2\theta_{max}$	55		
No. of Reflections Measured	8845	8592	8474
Corrections	Lorentz-polarization		
Structure Solution	Direct Methods (SIR92)		
Refinement	Fullmatrix leastsquare		
Function Minimized	$\Sigma w (\|Fo\| - \|Fc\|)^2$		
Least Squares Weights	$1/\sigma^2(Fo) = 4Fo^2/\sigma^2(Fo^2)$		
p-factor	0.0100	0.0100	0.0100
No. Observations (I>3.00$\sigma(I)$)	7237	6731	6385
No. Variables	694	694	694
Reflection/Parameter Ratio	10.4	9.7	9.2
Residuals: R; Rw	0.0988	0.0758	0.0764
Residuals: $R1$	0.0873	0.0864	0.0784
No. of Reflections to calc $R1$	7237	6731	6385
Goodness of Fit Indicator	7.111	7.374	5.748
Max Shift/Error in Final Cycle	0.00	0.00	0.00
Maximum peak in Final Diff. Map	0.61	0.32	0.42
Minimum peak in Final Diff. Map	-0.57	-0.26	-0.30

Numbering Scheme

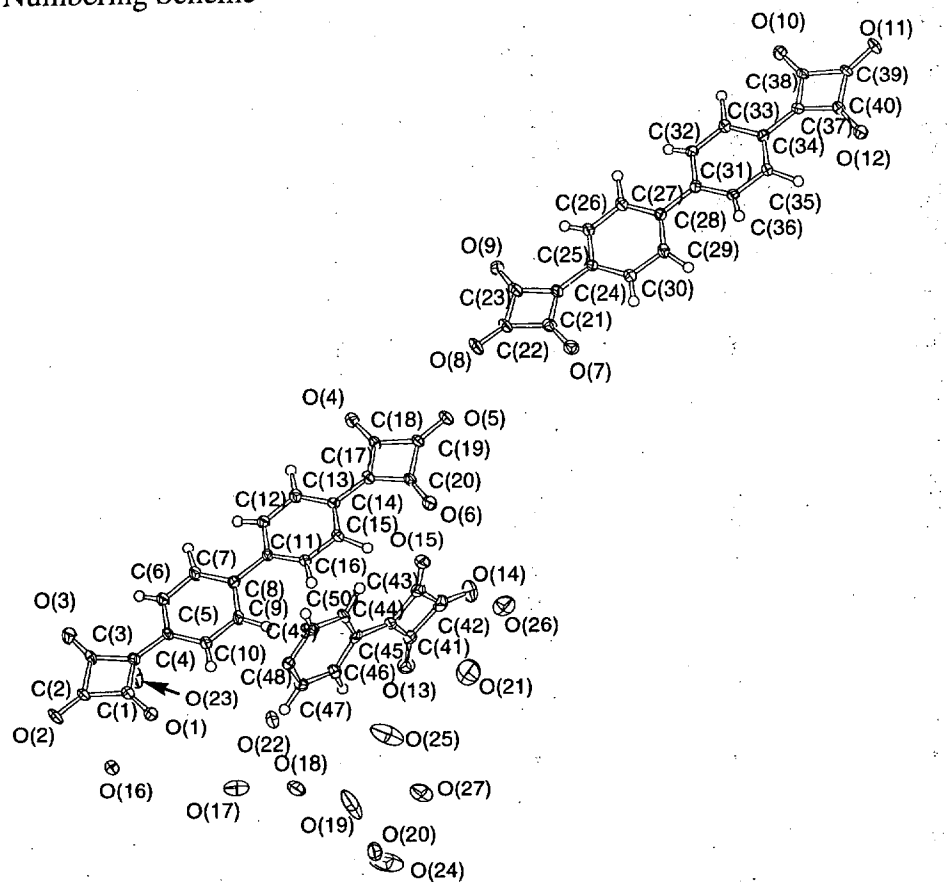

Table 2. Atomic coordinates, B_{eq} and occupancy at 103 K

atom	*x*	*y*	*z*	B_{eq}	*occ*
O(1)	0.2951(2)	0.3771(2)	0.7137(3)	1.76(7)	1.0000
O(2)	0.0965(2)	0.3186(2)	0.7947(3)	2.21(7)	1.0000
O(3)	0.0390(2)	0.1509(2)	0.5240(3)	2.56(8)	1.0000
O(4)	0.4079(2)	-0.1036(2)	-0.3819(3)	2.20(7)	1.0000
O(5)	0.6040(2)	-0.0340(2)	-0.4603(3)	1.94(7)	1.0000
O(6)	0.6603(2)	0.1223(2)	-0.1780(3)	2.57(8)	1.0000
O(7)	0.7936(2)	-0.1273(2)	-0.7828(3)	1.99(7)	1.0000
O(8)	0.5924(2)	-0.1944(2)	-0.7129(3)	2.32(7)	1.0000
O(9)	0.5548(2)	-0.3689(2)	-0.9792(3)	2.11(7)	1.0000
O(10)	0.9229(2)	-0.5961(2)	-1.8673(3)	1.85(7)	1.0000
O(11)	1.1129(2)	-0.5266(2)	-1.9575(2)	1.87(7)	1.0000
O(12)	1.1752(2)	-0.3688(2)	-1.6783(3)	2.26(7)	1.0000
O(13)	0.7904(2)	0.1986(2)	0.3198(3)	2.36(7)	1.0000

Table 2. Atomic coordinates, B_{eq} and occupancy at 103 K (continued)

atom	*x*	*y*	*z*	B_{eq}	*occ*
O(14)	0.8430(3)	0.1759(3)	0.0489(3)	3.16(9)	1.0000
O(15)	0.7064(2)	0.3339(2)	0.0325(3)	2.31(7)	1.0000
O(16)	0.2688(2)	0.4831(2)	0.9191(3)	2.01(7)	1.0000
O(17)	0.5601(3)	0.4371(3)	0.8624(3)	4.03(10)	1.0000
O(18)	0.6920(3)	0.3272(3)	0.7920(3)	3.58(9)	1.0000
O(19)	0.8314(4)	0.1571(4)	0.7628(4)	7.9(2)	1.0000
O(20)	0.9308(3)	0.3120(3)	0.9021(3)	3.07(9)	1.0000
O(21)	0.9322(3)	0.0804(3)	0.2643(4)	5.9(1)	1.0000
O(22)	0.5659(3)	0.2025(3)	0.5942(3)	3.25(9)	1.0000
O(23)	0.2261(3)	0.0303(3)	0.5622(3)	3.37(9)	1.0000
O(24)	0.9732(4)	0.0794(4)	0.8856(4)	3.7(2)	0.5000
O(25)	0.8286(4)	0.1480(3)	0.5275(4)	3.3(1)	0.5000
O(26)	0.9358(6)	0.0045(5)	0.0303(7)	3.7(2)	0.5000
O(27)	0.9704(6)	0.0319(5)	0.6467(6)	3.5(2)	0.5000
C(1)	0.2219(3)	0.3143(3)	0.6618(4)	1.47(9)	1.0000
C(2)	0.1314(3)	0.2889(3)	0.7014(4)	1.53(9)	1.0000
C(3)	0.1072(3)	0.2113(3)	0.5768(4)	1.52(9)	1.0000
C(4)	0.1991(3)	0.2419(3)	0.5487(4)	1.44(9)	1.0000
C(5)	0.2454(3)	0.2077(3)	0.4411(4)	1.47(9)	1.0000
C(6)	0.1985(3)	0.1368(4)	0.3361(4)	2.5(1)	1.0000
C(7)	0.2404(3)	0.1057(4)	0.2302(4)	2.6(1)	1.0000
C(8)	0.3307(3)	0.1466(3)	0.2263(4)	1.39(9)	1.0000
C(9)	0.3777(3)	0.2169(3)	0.3334(4)	2.02(10)	1.0000
C(10)	0.3364(3)	0.2478(3)	0.4403(4)	1.86(9)	1.0000
C(11)	0.3730(3)	0.1164(3)	0.1127(4)	1.31(8)	1.0000
C(12)	0.3293(3)	0.0410(3)	0.0085(4)	1.56(9)	1.0000
C(13)	0.3699(3)	0.0120(3)	-0.0989(4)	1.72(9)	1.0000
C(14)	0.4569(3)	0.0592(3)	-0.1035(4)	1.41(9)	1.0000
C(15)	0.5009(3)	0.1339(3)	-0.0026(4)	1.73(9)	1.0000
C(16)	0.4591(3)	0.1636(3)	0.1044(4)	1.70(9)	1.0000
C(17)	0.5025(3)	0.0297(3)	-0.2123(4)	1.37(9)	1.0000
C(18)	0.4797(3)	-0.0387(3)	-0.3286(4)	1.38(9)	1.0000
C(19)	0.5689(3)	-0.0099(3)	-0.3670(3)	1.41(9)	1.0000
C(20)	0.5936(3)	0.0632(3)	-0.2378(4)	1.55(9)	1.0000
C(21)	0.7247(3)	-0.1910(3)	-0.8364(4)	1.65(9)	1.0000
C(22)	0.6327(3)	-0.2224(3)	-0.8017(4)	1.61(9)	1.0000
C(23)	0.6165(3)	-0.2998(3)	-0.9239(4)	1.71(9)	1.0000
C(24)	0.7042(3)	-0.2647(3)	-0.9518(4)	1.54(9)	1.0000
C(25)	0.7522(3)	-0.2939(3)	-1.0580(4)	1.44(9)	1.0000
C(26)	0.7066(3)	-0.3642(3)	-1.1662(4)	2.06(10)	1.0000
C(27)	0.7503(3)	-0.3898(3)	-1.2698(4)	2.11(10)	1.0000

Table 2. Atomic coordinates, B_{eq} and occupancy at 103 K (continued)

atom	*x*	*y*	*z*	B_{eq}	*occ*
C(28)	0.8413(3)	-0.3473(3)	-1.2691(4)	1.27(8)	1.0000
C(29)	0.8873(3)	-0.2782(3)	-1.1589(4)	2.11(10)	1.0000
C(30)	0.8433(3)	-0.2508(3)	-1.0544(4)	1.98(10)	1.0000
C(31)	0.8867(3)	-0.3757(3)	-1.3817(4)	1.39(9)	1.0000
C(32)	0.8417(3)	-0.4481(3)	-1.4891(4)	1.59(9)	1.0000
C(33)	0.8850(3)	-0.4761(3)	-1.5935(4)	1.54(9)	1.0000
C(34)	0.9747(3)	-0.4309(3)	-1.5936(4)	1.32(8)	1.0000
C(35)	1.0194(3)	-0.3581(3)	-1.4882(4)	1.57(9)	1.0000
C(36)	0.9750(3)	-0.3308(3)	-1.3842(3)	1.53(9)	1.0000
C(37)	1.0193(3)	-0.4610(3)	-1.7025(3)	1.31(8)	1.0000
C(38)	0.9919(3)	-0.5325(3)	-1.8196(4)	1.34(8)	1.0000
C(39)	1.0803(3)	-0.5033(3)	-1.8627(3)	1.36(9)	1.0000
C(40)	1.1055(3)	-0.4304(3)	-1.7345(4)	1.46(9)	1.0000
C(41)	0.7656(3)	0.2355(3)	0.2415(4)	1.72(9)	1.0000
C(42)	0.7919(3)	0.2224(3)	0.1148(4)	2.3(1)	1.0000
C(43)	0.7297(3)	0.2955(3)	0.1099(4)	1.84(9)	1.0000
C(44)	0.7073(3)	0.3020(3)	0.2294(4)	1.62(9)	1.0000
C(45)	0.6472(3)	0.3585(3)	0.3076(4)	1.64(9)	1.0000
C(46)	0.5962(3)	0.4208(3)	0.2693(4)	1.78(9)	1.0000
C(47)	0.5405(3)	0.4751(3)	0.3432(4)	1.96(10)	1.0000
C(48)	0.5308(3)	0.4707(3)	0.4606(4)	1.50(8)	1.0000
C(49)	0.5821(3)	0.4077(3)	0.4986(4)	1.62(9)	1.0000
C(50)	0.6394(3)	0.3533(3)	0.4246(4)	1.80(9)	1.0000
H(1)	0.3710	0.2916	0.5092	1.3773	1.0000
H(2)	0.4472	0.2342	0.3368	3.7566	1.0000
H(3)	0.1437	0.1106	0.3426	9.2728	1.0000
H(4)	0.1966	0.0491	0.1522	3.5198	1.0000
H(5)	0.4959	0.2148	0.1706	0.4609	1.0000
H(6)	0.5767	0.1781	0.0141	6.1958	1.0000
H(7)	0.2725	0.0104	0.0243	2.8431	1.0000
H(8)	0.3314	-0.0471	-0.1778	3.4689	1.0000
H(9)	0.8765	-0.2075	-0.9821	0.7818	1.0000
H(10)	0.9569	-0.2386	-1.1269	7.2793	1.0000
H(11)	0.6514	-0.3812	-1.1589	3.7850	1.0000
H(12)	0.7110	-0.4380	-1.3529	3.6991	1.0000
H(13)	1.0091	-0.2849	-1.3242	0.3554	1.0000
H(14)	1.0972	-0.3259	-1.4785	3.6943	1.0000
H(15)	0.7901	-0.4785	-1.4795	1.9257	1.0000
H(16)	0.8460	-0.5372	-1.6839	1.3670	1.0000
H(17)	0.6771	0.3170	0.4599	2.9671	1.0000
H(18)	0.5758	0.3983	0.5856	3.6913	1.0000

Table 2. Atomic coordinates, B_{eq} and occupancy at 103 K (continued)

atom	x	y	z	B_{eq}	occ
H(19)	0.5998	0.4270	0.1782	1.9489	1.0000
H(20)	0.5101	0.5269	0.3127	4.8992	1.0000
H(21)	0.4265	-0.1360	-0.4637	6.7562	1.0000
H(22)	0.4938	0.1820	0.5864	8.3516	1.0000
H(23)	0.8817	0.1097	0.2864	5.3804	1.0000
H(24)	0.9872	0.3107	0.9147	7.8769	1.0000

Table 3. Anisotropic Displacement Parameters at 103 K

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.021(2)	0.023(2)	0.020(1)	-0.002(1)	0.004(1)	0.006(1)
O(2)	0.036(2)	0.037(2)	0.019(1)	0.015(2)	0.018(1)	0.012(1)
O(3)	0.028(2)	0.036(2)	0.029(2)	-0.009(2)	0.008(2)	0.009(2)
O(4)	0.026(2)	0.034(2)	0.023(2)	-0.002(2)	0.007(1)	0.009(1)
O(5)	0.030(2)	0.030(2)	0.016(1)	0.008(2)	0.011(1)	0.007(1)
O(6)	0.025(2)	0.043(2)	0.022(2)	-0.012(2)	0.004(1)	0.007(2)
O(7)	0.027(2)	0.025(2)	0.021(2)	-0.002(2)	0.004(1)	0.006(1)
O(8)	0.037(2)	0.037(2)	0.022(2)	0.017(2)	0.020(2)	0.012(1)
O(9)	0.022(2)	0.031(2)	0.022(1)	-0.001(2)	0.004(1)	0.002(1)
O(10)	0.023(2)	0.027(2)	0.019(1)	-0.002(2)	0.005(1)	0.006(1)
O(11)	0.029(2)	0.028(2)	0.016(1)	0.007(2)	0.013(1)	0.005(1)
O(12)	0.023(2)	0.037(2)	0.019(1)	-0.010(2)	0.003(1)	0.006(1)
O(13)	0.030(2)	0.031(2)	0.030(2)	0.002(2)	0.002(1)	0.014(1)
O(14)	0.037(2)	0.044(2)	0.043(2)	0.015(2)	0.021(2)	0.011(2)
O(15)	0.028(2)	0.037(2)	0.023(2)	0.000(2)	0.005(1)	0.013(2)
O(16)	0.024(2)	0.031(2)	0.021(1)	0.001(2)	0.006(1)	0.009(1)
O(17)	0.064(3)	0.044(3)	0.036(2)	0.000(2)	-0.009(2)	0.010(2)
O(18)	0.045(2)	0.062(3)	0.024(2)	-0.010(2)	0.009(2)	0.012(2)
O(19)	0.107(4)	0.097(4)	0.056(3)	-0.057(3)	0.059(3)	-0.028(3)
O(20)	0.031(2)	0.054(3)	0.042(2)	0.009(2)	0.015(2)	0.026(2)
O(21)	0.063(3)	0.070(3)	0.066(3)	0.016(3)	0.015(3)	-0.015(3)
O(22)	0.029(2)	0.060(3)	0.031(2)	-0.003(2)	0.014(2)	0.009(2)
O(23)	0.047(3)	0.043(2)	0.052(2)	0.012(2)	0.031(2)	0.026(2)
O(24)	0.136(6)	0.076(4)	0.043(3)	-0.045(4)	-0.007(3)	0.015(3)
O(25)	0.147(5)	0.049(3)	0.037(2)	-0.046(3)	0.013(3)	0.010(2)
O(26)	0.047(5)	0.033(4)	0.042(4)	0.006(4)	0.000(4)	-0.009(3)
O(27)	0.065(6)	0.027(4)	0.032(4)	-0.009(4)	0.010(4)	0.000(3)
C(1)	0.021(2)	0.023(2)	0.019(2)	0.012(2)	0.007(2)	0.013(2)
C(2)	0.024(2)	0.024(2)	0.014(2)	0.009(2)	0.009(2)	0.008(2)
C(3)	0.020(2)	0.022(2)	0.017(2)	0.007(2)	0.011(2)	0.005(2)
C(4)	0.018(2)	0.025(2)	0.016(2)	0.006(2)	0.005(2)	0.012(2)
C(5)	0.018(2)	0.028(3)	0.016(2)	0.006(2)	0.007(2)	0.013(2)

Table 3. Anisotropic Displacement Parameters at 103 K (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(6)	0.024(3)	0.042(3)	0.018(2)	-0.014(2)	0.006(2)	0.001(2)
C(7)	0.024(3)	0.046(3)	0.020(2)	-0.015(2)	0.007(2)	0.004(2)
C(8)	0.015(2)	0.021(2)	0.019(2)	0.000(2)	0.002(2)	0.011(2)
C(9)	0.018(2)	0.033(3)	0.023(2)	-0.007(2)	0.008(2)	0.006(2)
C(10)	0.019(2)	0.030(3)	0.018(2)	-0.004(2)	0.007(2)	0.004(2)
C(11)	0.014(2)	0.023(2)	0.018(2)	0.005(2)	0.005(2)	0.012(2)
C(12)	0.020(2)	0.020(2)	0.018(2)	-0.002(2)	0.004(2)	0.005(2)
C(13)	0.021(2)	0.024(2)	0.021(2)	0.000(2)	0.006(2)	0.008(2)
C(14)	0.017(2)	0.024(2)	0.015(2)	0.005(2)	0.004(2)	0.008(2)
C(15)	0.022(2)	0.027(3)	0.016(2)	0.000(2)	0.007(2)	0.006(2)
C(16)	0.017(2)	0.028(3)	0.016(2)	-0.002(2)	0.005(2)	0.005(2)
C(17)	0.021(2)	0.017(2)	0.017(2)	0.009(2)	0.007(2)	0.007(2)
C(18)	0.022(2)	0.015(2)	0.015(2)	0.002(2)	0.007(2)	0.003(2)
C(19)	0.021(2)	0.023(2)	0.013(2)	0.006(2)	0.007(2)	0.009(2)
C(20)	0.022(2)	0.021(2)	0.015(2)	0.006(2)	0.008(2)	0.003(2)
C(21)	0.025(3)	0.024(3)	0.019(2)	0.009(2)	0.006(2)	0.013(2)
C(22)	0.028(3)	0.023(2)	0.016(2)	0.012(2)	0.011(2)	0.008(2)
C(23)	0.027(3)	0.027(3)	0.015(2)	0.013(2)	0.008(2)	0.009(2)
C(24)	0.018(2)	0.031(3)	0.016(2)	0.006(2)	0.005(2)	0.014(2)
C(25)	0.016(2)	0.027(3)	0.015(2)	0.004(2)	0.003(2)	0.012(2)
C(26)	0.020(3)	0.037(3)	0.017(2)	-0.006(2)	0.006(2)	0.005(2)
C(27)	0.022(3)	0.039(3)	0.016(2)	-0.005(2)	0.005(2)	0.007(2)
C(28)	0.015(2)	0.019(2)	0.017(2)	0.002(2)	0.004(2)	0.010(2)
C(29)	0.022(3)	0.037(3)	0.020(2)	-0.006(2)	0.006(2)	0.009(2)
C(30)	0.020(2)	0.035(3)	0.017(2)	-0.003(2)	0.005(2)	0.005(2)
C(31)	0.015(2)	0.024(2)	0.020(2)	0.005(2)	0.003(2)	0.015(2)
C(32)	0.018(2)	0.024(2)	0.016(2)	-0.004(2)	0.002(2)	0.006(2)
C(33)	0.018(2)	0.023(2)	0.018(2)	0.002(2)	0.004(2)	0.009(2)
C(34)	0.014(2)	0.020(2)	0.017(2)	0.000(2)	0.001(2)	0.009(2)
C(35)	0.016(2)	0.029(3)	0.016(2)	0.002(2)	0.005(2)	0.009(2)
C(36)	0.018(2)	0.023(2)	0.014(2)	-0.005(2)	0.002(2)	0.004(2)
C(37)	0.020(2)	0.017(2)	0.013(2)	0.004(2)	0.002(2)	0.005(2)
C(38)	0.019(2)	0.018(2)	0.014(2)	0.005(2)	0.005(2)	0.004(2)
C(39)	0.022(2)	0.022(2)	0.012(2)	0.007(2)	0.007(2)	0.007(2)
C(40)	0.022(2)	0.018(2)	0.016(2)	0.004(2)	0.007(2)	0.004(2)
C(41)	0.019(2)	0.021(2)	0.018(2)	-0.009(2)	0.001(2)	0.001(2)
C(42)	0.023(3)	0.031(3)	0.032(2)	-0.003(2)	0.004(2)	0.012(2)
C(43)	0.020(2)	0.025(3)	0.023(2)	-0.005(2)	0.000(2)	0.008(2)
C(44)	0.019(2)	0.019(2)	0.014(2)	-0.008(2)	0.004(2)	-0.004(2)
C(45)	0.013(2)	0.023(2)	0.023(2)	-0.007(2)	-0.001(2)	0.009(2)
C(46)	0.024(2)	0.026(2)	0.017(2)	0.008(2)	0.010(2)	0.003(2)

Table 3. Anisotropic Displacement Parameters at 103 K (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(47)	0.026(3)	0.029(3)	0.021(2)	0.001(2)	0.003(2)	0.013(2)
C(48)	0.017(2)	0.019(2)	0.017(2)	-0.006(2)	0.001(2)	0.004(2)
C(49)	0.023(2)	0.019(2)	0.016(2)	0.002(2)	0.004(2)	0.002(2)
C(50)	0.023(2)	0.023(2)	0.021(2)	0.005(2)	0.005(2)	0.006(2)

Table 4. Bond Lengths (Å) at 103 K

atom	atom	distance	atom	atom	distance
O(1)	C(1)	1.290(6)	O(2)	C(2)	1.216(4)
O(3)	C(3)	1.224(6)	O(4)	C(18)	1.298(6)
O(5)	C(19)	1.209(4)	O(6)	C(20)	1.214(6)
O(7)	C(21)	1.260(6)	O(8)	C(22)	1.215(4)
O(9)	C(23)	1.253(6)	O(10)	C(38)	1.253(5)
O(11)	C(39)	1.204(4)	O(12)	C(40)	1.253(5)
O(13)	C(41)	1.252(5)	O(14)	C(42)	1.225(5)
O(15)	C(43)	1.257(5)	C(1)	C(2)	1.481(6)
C(1)	C(4)	1.395(6)	C(2)	C(3)	1.521(6)
C(3)	C(4)	1.459(6)	C(4)	C(5)	1.452(5)
C(5)	C(6)	1.384(6)	C(5)	C(10)	1.383(6)
C(6)	C(7)	1.393(6)	C(7)	C(8)	1.391(6)
C(8)	C(9)	1.392(6)	C(8)	C(11)	1.471(5)
C(9)	C(10)	1.398(5)	C(11)	C(12)	1.401(6)
C(11)	C(16)	1.400(6)	C(12)	C(13)	1.395(6)
C(13)	C(14)	1.395(6)	C(14)	C(15)	1.377(6)
C(14)	C(17)	1.450(5)	C(15)	C(16)	1.402(5)
C(17)	C(18)	1.391(5)	C(17)	C(20)	1.453(6)
C(18)	C(19)	1.475(6)	C(19)	C(20)	1.527(6)
C(21)	C(22)	1.502(6)	C(21)	C(24)	1.421(6)
C(22)	C(23)	1.502(6)	C(23)	C(24)	1.433(6)
C(24)	C(25)	1.444(5)	C(25)	C(26)	1.395(6)
C(25)	C(30)	1.390(6)	C(26)	C(27)	1.380(5)
C(27)	C(28)	1.394(6)	C(28)	C(29)	1.401(6)
C(28)	C(31)	1.483(5)	C(29)	C(30)	1.393(6)
C(31)	C(32)	1.403(6)	C(31)	C(36)	1.388(6)
C(32)	C(33)	1.388(5)	C(33)	C(34)	1.398(6)
C(34)	C(35)	1.391(6)	C(34)	C(37)	1.444(5)
C(35)	C(36)	1.391(5)	C(37)	C(38)	1.423(5)
C(37)	C(40)	1.411(6)	C(38)	C(39)	1.498(6)
C(39)	C(40)	1.518(5)	C(41)	C(42)	1.515(6)
C(41)	C(51)	1.409(6)	C(42)	C(43)	1.508(6)
C(43)	C(44)	1.436(6)	C(45)	C(46)	1.405(6)
C(45)	C(50)	1.403(6)	C(45)	C(44)	1.457(6)

Table 4. Bond Lengths (Å) at 103 K (continued)

atom	atom	distance	atom	atom	distance
C(46)	C(47)	1.374(6)	C(47)	C(48)	1.409(6)
C(48)	C(49)	1.415(6)	C(50)	C(49)	1.389(6)

Table 5. Bond Angles(°) at 103 K

atom	atom	atom	angle	atom	atom	atom	angle
O(1)	C(1)	C(2)	133.9(4)	O(1)	C(1)	C(4)	133.9(4)
C(2)	C(1)	C(4)	92.2(4)	O(2)	C(2)	C(1)	134.6(4)
O(2)	C(2)	C(3)	138.5(5)	C(1)	C(2)	C(3)	86.9(3)
O(3)	C(3)	C(2)	135.1(4)	O(3)	C(3)	C(4)	136.7(4)
C(2)	C(3)	C(4)	88.2(4)	C(1)	C(4)	C(3)	92.7(3)
C(1)	C(4)	C(5)	135.3(4)	C(3)	C(4)	C(5)	132.0(4)
C(4)	C(5)	C(6)	120.9(4)	C(4)	C(5)	C(10)	120.4(4)
C(6)	C(5)	C(10)	118.7(4)	C(5)	C(6)	C(7)	121.6(5)
C(6)	C(7)	C(8)	120.6(4)	C(7)	C(8)	C(9)	117.1(4)
C(7)	C(8)	C(11)	120.5(4)	C(9)	C(8)	C(11)	122.4(4)
C(8)	C(9)	C(10)	122.6(4)	C(5)	C(10)	C(9)	119.4(4)
C(8)	C(11)	C(12)	122.2(4)	C(8)	C(11)	C(16)	120.5(4)
C(12)	C(11)	C(16)	117.3(4)	C(11)	C(12)	C(13)	122.1(4)
C(12)	C(13)	C(14)	119.4(4)	C(13)	C(14)	C(15)	119.6(4)
C(13)	C(14)	C(17)	120.9(4)	C(15)	C(14)	C(17)	119.5(4)
C(14)	C(15)	C(16)	120.9(4)	C(11)	C(16)	C(15)	120.7(4)
C(14)	C(17)	C(18)	135.7(4)	C(14)	C(17)	C(20)	131.6(4)
C(18)	C(17)	C(20)	92.7(3)	O(4)	C(18)	C(17)	133.5(4)
O(4)	C(18)	C(19)	133.9(4)	C(17)	C(18)	C(19)	92.6(4)
O(5)	C(19)	C(18)	135.7(4)	O(5)	C(19)	C(20)	137.7(5)
C(18)	C(19)	C(20)	86.5(3)	O(6)	C(20)	C(17)	134.6(4)
O(6)	C(20)	C(19)	137.3(4)	C(17)	C(20)	C(19)	88.1(4)
O(7)	C(21)	C(22)	134.6(4)	O(7)	C(21)	C(24)	134.9(4)
C(22)	C(21)	C(24)	90.5(4)	O(8)	C(22)	C(21)	134.7(5)
O(8)	C(22)	C(23)	138.5(5)	C(21)	C(22)	C(23)	86.8(3)
O(9)	C(23)	C(22)	136.1(4)	O(9)	C(23)	C(24)	133.8(4)
C(22)	C(23)	C(24)	90.0(4)	C(21)	C(24)	C(23)	92.6(3)
C(21)	C(24)	C(25)	134.3(4)	C(23)	C(24)	C(25)	133.0(4)
C(24)	C(25)	C(26)	120.4(4)	C(24)	C(25)	C(30)	120.3(4)
C(26)	C(25)	C(30)	119.2(4)	C(25)	C(26)	C(27)	120.7(4)
C(26)	C(27)	C(28)	121.3(4)	C(27)	C(28)	C(29)	117.5(4)
C(27)	C(28)	C(31)	120.4(4)	C(29)	C(28)	C(31)	122.2(4)
C(28)	C(29)	C(30)	121.8(4)	C(25)	C(30)	C(29)	119.5(4)
C(28)	C(31)	C(32)	121.3(4)	C(28)	C(31)	C(36)	120.9(4)
C(32)	C(31)	C(36)	117.8(4)	C(31)	C(32)	C(33)	121.4(4)
C(32)	C(33)	C(34)	119.9(4)	C(33)	C(34)	C(35)	119.2(4)

Table 5. Bond Angles(°) at 103 K (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(33)	C(34)	C(37)	119.4(4)	C(35)	C(34)	C(37)	121.4(4)
C(34)	C(35)	C(36)	120.2(4)	C(31)	C(36)	C(35)	121.4(4)
C(34)	C(37)	C(38)	133.3(4)	C(34)	C(37)	C(40)	134.0(4)
C(38)	C(37)	C(40)	92.7(3)	O(10)	C(38)	C(37)	134.9(4)
O(10)	C(38)	C(39)	134.1(4)	C(37)	C(38)	C(39)	91.0(4)
O(11)	C(39)	C(38)	136.2(4)	O(11)	C(39)	C(40)	138.2(4)
C(38)	C(39)	C(40)	85.6(3)	O(12)	C(40)	C(37)	134.4(4)
O(12)	C(40)	C(39)	135.0(4)	C(37)	C(40)	C(39)	90.7(4)
O(13)	C(41)	C(42)	131.4(4)	O(13)	C(41)	C(44)	138.1(4)
C(42)	C(41)	C(44)	90.5(4)	O(14)	C(42)	C(41)	136.7(5)
O(14)	C(42)	C(43)	136.9(5)	C(41)	C(42)	C(43)	86.3(3)
O(15)	C(43)	C(42)	135.0(4)	O(15)	C(43)	C(44)	135.1(4)
C(42)	C(43)	C(44)	89.8(4)	C(46)	C(45)	C(50)	117.7(4)
C(46)	C(45)	C(44)	121.3(4)	C(50)	C(45)	C(44)	121.0(4)
C(45)	C(46)	C(47)	121.6(4)	C(46)	C(47)	C(48)	121.8(4)
C(47)	C(48)	C(48)	121.2(5)	C(47)	C(48)	C(49)	116.4(4)
C(48)	C(48)	C(49)	122.4(5)	C(45)	C(50)	C(49)	120.5(4)
C(41)	C(44)	C(43)	93.3(3)	C(41)	C(44)	C(45)	134.4(4)
C(43)	C(44)	C(45)	132.3(4)	C(48)	C(49)	C(50)	122.0(4)

Table 6 Atomic coordinates, B_{eq} and occupancy at 200 K

atom	*x*	*y*	*z*	*B*eq	occ
O(1)	0.2932(3)	-0.6244(3)	0.7121(3)	2.23(9)	1.0000
O(2)	0.0965(3)	-0.6810(3)	0.7928(3)	3.0(1)	1.0000
O(3)	0.0397(3)	-0.8491(3)	0.5219(4)	3.3(1)	1.0000
O(4)	0.4096(3)	-1.1047(3)	-0.3820(3)	2.80(10)	1.0000
O(5)	0.6042(3)	-1.0357(3)	-0.4620(3)	2.72(10)	1.0000
O(6)	0.6610(3)	-0.8779(3)	-0.1794(3)	3.2(1)	1.0000
O(7)	0.7911(3)	-1.1287(3)	-0.7841(3)	2.9(1)	1.0000
O(8)	0.5916(3)	-1.1955(3)	-0.7138(3)	3.2(1)	1.0000
O(9)	0.5552(3)	-1.3699(3)	-0.9796(3)	2.9(1)	1.0000
O(10)	0.9241(3)	-1.5957(3)	-1.8664(3)	2.52(10)	1.0000
O(11)	1.1126(3)	-1.5268(3)	-1.9569(3)	2.69(10)	1.0000
O(12)	1.1747(3)	-1.3679(3)	-1.6781(3)	2.84(10)	1.0000
O(13)	0.7885(3)	-0.7985(3)	0.3215(4)	3.2(1)	1.0000
O(14)	0.8426(3)	-0.8221(3)	0.0509(4)	4.1(1)	1.0000
O(15)	0.7076(3)	-0.6656(3)	0.0339(3)	2.93(10)	1.0000
O(16)	0.2677(3)	-0.5167(3)	0.9165(3)	2.66(9)	1.0000
O(17)	0.5612(4)	-0.5628(3)	0.8632(4)	4.9(1)	1.0000
O(18)	0.6925(3)	-0.6738(3)	0.7909(4)	4.3(1)	1.0000
O(19)	0.8334(4)	-0.8445(5)	0.7622(5)	8.8(2)	1.0000

Table 6 Atomic coordinates, B_{eq} and occupancy at 200 K (continued)

atom	*x*	*y*	*z*	*B*eq	occ
O(20)	0.9330(3)	-0.6910(3)	0.9035(4)	4.1(1)	1.0000
O(21)	0.9263(5)	-0.9185(4)	0.2637(5)	7.7(2)	1.0000
O(22)	0.5658(3)	-0.7972(3)	0.5944(4)	4.2(1)	1.0000
O(23)	0.2281(3)	-0.9702(3)	0.5635(4)	4.3(1)	1.0000
O(24)	0.9690(5)	-0.9223(4)	0.8873(5)	4.3(2)	0.5000
O(25)	0.8281(5)	-0.8524(4)	0.5259(4)	3.5(2)	0.5000
O(26)	0.9385(8)	-0.9955(7)	0.0350(10)	6.0(3)	0.5000
O(27)	0.9689(8)	-0.9658(7)	0.6490(8)	5.2(3)	0.5000
C(1)	0.2213(4)	-0.6865(4)	0.6607(4)	1.6(1)	1.0000
C(2)	0.1306(4)	-0.7120(4)	0.7008(5)	2.0(1)	1.0000
C(3)	0.1071(4)	-0.7891(4)	0.5741(5)	2.0(1)	1.0000
C(4)	0.1981(4)	-0.7601(4)	0.5468(4)	1.7(1)	1.0000
C(5)	0.2454(4)	-0.7932(4)	0.4392(4)	1.5(1)	1.0000
C(6)	0.1996(4)	-0.8647(5)	0.3366(5)	3.0(2)	1.0000
C(7)	0.2418(4)	-0.8949(4)	0.2302(5)	2.8(1)	1.0000
C(8)	0.3299(4)	-0.8537(4)	0.2249(4)	1.5(1)	1.0000
C(9)	0.3765(4)	-0.7819(4)	0.3325(5)	2.4(1)	1.0000
C(10)	0.3345(4)	-0.7507(4)	0.4387(5)	2.4(1)	1.0000
C(11)	0.3730(4)	-0.8827(4)	0.1129(4)	1.4(1)	1.0000
C(12)	0.3299(4)	-0.9584(4)	0.0075(5)	2.1(1)	1.0000
C(13)	0.3700(4)	-0.9859(4)	-0.0980(4)	1.9(1)	1.0000
C(14)	0.4566(4)	-0.9412(4)	-0.1035(4)	1.6(1)	1.0000
C(15)	0.5016(4)	-0.8654(4)	0.0004(5)	2.0(1)	1.0000
C(16)	0.4600(4)	-0.8377(4)	0.1047(5)	2.2(1)	1.0000
C(17)	0.5023(4)	-0.9707(4)	-0.2115(4)	1.7(1)	1.0000
C(18)	0.4795(4)	-1.0398(4)	-0.3299(5)	1.8(1)	1.0000
C(19)	0.5689(4)	-1.0119(4)	-0.3683(5)	2.2(1)	1.0000
C(20)	0.5942(4)	-0.9375(4)	-0.2389(5)	1.9(1)	1.0000
C(21)	0.7224(4)	-1.1941(4)	-0.8386(5)	2.0(1)	1.0000
C(22)	0.6318(4)	-1.2225(4)	-0.8032(5)	2.0(1)	1.0000
C(23)	0.6166(4)	-1.3018(4)	-0.9255(5)	2.0(1)	1.0000
C(24)	0.7038(4)	-1.2667(4)	-0.9537(4)	1.6(1)	1.0000
C(25)	0.7515(4)	-1.2949(4)	-1.0592(4)	1.7(1)	1.0000
C(26)	0.7071(4)	-1.3658(4)	-1.1659(5)	2.4(1)	1.0000
C(27)	0.7512(4)	-1.3907(4)	-1.2695(5)	2.5(1)	1.0000
C(28)	0.8407(4)	-1.3477(4)	-1.2690(4)	1.3(1)	1.0000
C(29)	0.8843(4)	-1.2775(4)	-1.1600(5)	2.5(1)	1.0000
C(30)	0.8413(4)	-1.2510(4)	-1.0567(4)	2.2(1)	1.0000
C(31)	0.8869(4)	-1.3767(4)	-1.3831(4)	1.4(1)	1.0000
C(32)	0.8410(4)	-1.4476(4)	-1.4892(4)	1.8(1)	1.0000
C(33)	0.8858(4)	-1.4757(4)	-1.5938(4)	1.6(1)	1.0000

Table 6 Atomic coordinates, B_{eq} and occupancy at 200 K (continued)

atom	*x*	*y*	*z*	*B*eq	occ
C(34)	0.9740(4)	-1.4308(4)	-1.5933(4)	1.4(1)	1.0000
C(35)	1.0202(4)	-1.3589(4)	-1.4876(4)	1.8(1)	1.0000
C(36)	0.9753(4)	-1.3310(4)	-1.3824(4)	1.7(1)	1.0000
C(37)	1.0194(4)	-1.4610(4)	-1.7032(4)	1.4(1)	1.0000
C(38)	0.9921(4)	-1.5321(4)	-1.8196(5)	1.8(1)	1.0000
C(39)	1.0799(4)	-1.5021(4)	-1.8619(4)	1.7(1)	1.0000
C(40)	1.1053(4)	-1.4295(4)	-1.7341(5)	2.0(1)	1.0000
C(41)	0.7639(4)	-0.7619(4)	0.2434(5)	2.3(1)	1.0000
C(42)	0.7917(4)	-0.7754(4)	0.1163(5)	2.5(1)	1.0000
C(43)	0.7288(4)	-0.7035(4)	0.1114(5)	2.2(1)	1.0000
C(44)	0.7071(4)	-0.6973(4)	0.2300(4)	1.9(1)	1.0000
C(45)	0.6461(4)	-0.6404(4)	0.3089(5)	2.0(1)	1.0000
C(46)	0.5965(4)	-0.5789(4)	0.2705(4)	2.1(1)	1.0000
C(47)	0.5413(4)	-0.5241(4)	0.3460(5)	2.8(2)	1.0000
C(48)	0.5314(4)	-0.5300(4)	0.4598(5)	1.7(1)	1.0000
C(49)	0.5815(4)	-0.5920(4)	0.4985(4)	2.3(1)	1.0000
C(50)	0.6383(4)	-0.6465(4)	0.4230(5)	2.2(1)	1.0000
H(1)	0.1385	-0.8944	0.3370	3.5669	1.0000
H(2)	0.8552	-1.5261	-1.6654	1.8839	1.0000
H(3)	0.5092	-0.4812	0.3192	3.3643	1.0000
H(4)	0.5767	-0.5971	0.5765	2.8116	1.0000
H(5)	0.2086	-0.9451	0.1600	3.3360	1.0000
H(6)	0.4386	-0.7533	0.3337	2.9348	1.0000
H(7)	0.3670	-0.7007	0.5096	2.9167	1.0000
H(8)	0.2713	-0.9917	0.0089	2.5529	1.0000
H(9)	0.3377	-1.0365	-0.1683	2.2456	1.0000
H(10)	0.5609	-0.8331	-0.0012	2.4516	1.0000
H(11)	0.4915	-0.7859	0.1739	2.5954	1.0000
H(12)	0.6006	-0.5745	0.1919	2.5667	1.0000
H(13)	0.6720	-0.6882	0.4502	2.6125	1.0000
H(14)	0.6463	-1.3977	-1.1685	2.9324	1.0000
H(15)	0.7189	-1.4388	-1.3429	3.0028	1.0000
H(16)	0.9458	-1.2465	-1.1565	3.0074	1.0000
H(17)	0.8733	-1.2026	-0.9836	2.5826	1.0000
H(18)	0.7792	-1.4774	-1.4916	2.1772	1.0000
H(19)	1.0816	-1.3286	-1.4859	2.1047	1.0000
H(20)	1.0059	-1.2806	-1.3107	2.0701	1.0000
H(21)	1.0061	-0.9351	0.8504	2.6682	1.0000
H(22)	0.5311	-0.5924	0.8877	2.6682	1.0000

Table 7. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.031(3)	0.027(3)	0.024(2)	0.005(2)	0.009(2)	0.004(2)
O(2)	0.047(3)	0.050(3)	0.025(2)	0.022(2)	0.024(2)	0.016(2)
O(3)	0.036(3)	0.044(3)	0.040(2)	0.000(3)	0.021(2)	0.005(2)
O(4)	0.034(3)	0.037(3)	0.029(2)	-0.003(2)	0.011(2)	0.004(2)
O(5)	0.042(3)	0.047(3)	0.020(2)	0.017(2)	0.025(2)	0.008(2)
O(6)	0.035(3)	0.046(3)	0.029(2)	-0.010(2)	0.012(2)	-0.001(2)
O(7)	0.038(3)	0.034(3)	0.035(2)	0.005(2)	0.008(2)	0.011(2)
O(8)	0.049(3)	0.053(3)	0.029(2)	0.021(3)	0.028(2)	0.015(2)
O(9)	0.029(3)	0.042(3)	0.032(2)	-0.002(2)	0.006(2)	0.004(2)
O(10)	0.030(3)	0.037(3)	0.024(2)	-0.003(2)	0.005(2)	0.007(2)
O(11)	0.036(3)	0.041(3)	0.028(2)	0.008(2)	0.012(2)	0.012(2)
O(12)	0.034(3)	0.039(3)	0.024(2)	-0.013(2)	0.004(2)	0.002(2)
O(13)	0.041(3)	0.044(3)	0.044(3)	0.007(2)	0.010(2)	0.023(2)
O(14)	0.054(3)	0.057(3)	0.052(3)	0.021(3)	0.025(2)	0.018(2)
O(15)	0.042(3)	0.040(3)	0.027(2)	0.003(2)	0.004(2)	0.010(2)
O(16)	0.038(3)	0.036(3)	0.025(2)	0.004(2)	0.010(2)	0.006(2)
O(17)	0.076(4)	0.055(4)	0.047(3)	-0.002(3)	-0.008(2)	0.016(3)
O(18)	0.048(3)	0.077(4)	0.032(2)	-0.007(3)	0.009(2)	0.016(2)
O(19)	0.109(5)	0.110(5)	0.067(4)	-0.058(4)	0.064(4)	-0.033(4)
O(20)	0.048(3)	0.059(3)	0.047(3)	0.000(3)	0.013(2)	0.018(2)
O(21)	0.106(5)	0.086(5)	0.084(4)	0.012(4)	0.010(4)	0.013(4)
O(22)	0.041(3)	0.068(4)	0.034(2)	-0.002(3)	0.020(2)	-0.005(2)
O(23)	0.068(4)	0.046(3)	0.058(3)	0.018(3)	0.040(3)	0.014(2)
O(24)	0.164(7)	0.078(5)	0.058(4)	-0.033(5)	0.013(4)	0.006(3)
O(25)	0.148(6)	0.053(4)	0.048(3)	-0.042(4)	0.016(3)	0.008(3)
O(26)	0.076(8)	0.057(8)	0.084(7)	0.010(6)	-0.006(6)	0.020(6)
O(27)	0.097(9)	0.040(6)	0.051(6)	-0.004(6)	0.029(6)	-0.001(5)
C(1)	0.024(4)	0.023(3)	0.019(3)	0.008(3)	0.008(3)	0.011(3)
C(2)	0.034(4)	0.029(4)	0.020(3)	0.016(3)	0.015(3)	0.010(3)
C(3)	0.026(4)	0.029(4)	0.024(3)	0.008(3)	0.016(3)	0.008(3)
C(4)	0.022(3)	0.026(3)	0.021(3)	0.007(3)	0.004(2)	0.010(3)
C(5)	0.027(3)	0.021(3)	0.009(3)	0.008(3)	0.007(2)	0.002(2)
C(6)	0.028(4)	0.050(5)	0.031(3)	-0.011(3)	0.014(3)	0.010(3)
C(7)	0.032(4)	0.040(4)	0.018(3)	-0.016(3)	0.012(3)	-0.010(3)
C(8)	0.024(3)	0.017(3)	0.016(3)	0.007(3)	0.004(2)	0.006(2)
C(9)	0.024(3)	0.037(4)	0.023(3)	-0.006(3)	0.016(3)	-0.002(3)
C(10)	0.032(4)	0.029(4)	0.022(3)	-0.004(3)	0.007(3)	-0.001(3)
C(11)	0.019(3)	0.019(3)	0.014(3)	0.002(3)	0.002(2)	0.004(2)
C(12)	0.023(3)	0.027(4)	0.030(3)	0.000(3)	0.004(3)	0.010(3)
C(13)	0.028(3)	0.028(3)	0.011(3)	-0.003(3)	0.009(2)	0.000(2)
C(14)	0.023(3)	0.023(3)	0.021(3)	0.009(3)	0.005(2)	0.011(3)

Table 7. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(15)	0.021(3)	0.030(4)	0.023(3)	-0.004(3)	0.009(3)	0.005(3)
C(16)	0.031(4)	0.028(4)	0.018(3)	-0.006(3)	0.004(3)	0.005(3)
C(17)	0.029(3)	0.021(3)	0.017(3)	0.012(3)	0.007(2)	0.007(2)
C(18)	0.020(3)	0.030(4)	0.024(3)	0.006(3)	0.008(3)	0.012(3)
C(19)	0.027(3)	0.029(4)	0.032(3)	0.010(3)	0.002(3)	0.018(3)
C(20)	0.029(4)	0.033(4)	0.016(3)	0.010(3)	0.014(3)	0.010(3)
C(21)	0.027(4)	0.026(4)	0.026(3)	0.006(3)	0.005(3)	0.015(3)
C(22)	0.028(3)	0.029(4)	0.025(3)	0.010(3)	0.009(3)	0.012(3)
C(23)	0.027(4)	0.034(4)	0.019(3)	0.015(3)	0.006(3)	0.011(3)
C(24)	0.026(3)	0.031(4)	0.011(2)	0.015(3)	0.011(2)	0.009(2)
C(25)	0.025(3)	0.023(3)	0.017(3)	0.007(3)	0.003(2)	0.007(3)
C(26)	0.018(3)	0.051(4)	0.020(3)	-0.005(3)	0.015(3)	0.004(3)
C(27)	0.030(4)	0.034(4)	0.019(3)	-0.009(3)	-0.001(3)	-0.002(3)
C(28)	0.015(3)	0.022(3)	0.017(3)	0.005(3)	0.007(2)	0.008(3)
C(29)	0.027(4)	0.034(4)	0.028(3)	-0.008(3)	0.004(3)	0.007(3)
C(30)	0.027(4)	0.029(4)	0.016(3)	-0.005(3)	0.012(2)	-0.006(3)
C(31)	0.023(3)	0.019(3)	0.016(3)	0.009(3)	0.005(2)	0.011(3)
C(32)	0.019(3)	0.027(4)	0.020(3)	-0.002(3)	0.010(2)	0.003(3)
C(33)	0.022(3)	0.017(3)	0.017(3)	-0.002(3)	0.002(2)	0.003(2)
C(34)	0.017(3)	0.019(3)	0.020(3)	0.005(3)	0.007(2)	0.008(3)
C(35)	0.020(3)	0.030(4)	0.017(3)	0.000(3)	0.009(2)	0.007(3)
C(36)	0.022(3)	0.025(3)	0.015(3)	0.001(3)	0.007(2)	0.002(2)
C(37)	0.023(3)	0.018(3)	0.011(3)	0.004(3)	0.004(2)	0.001(2)
C(38)	0.022(3)	0.026(4)	0.022(3)	0.006(3)	0.002(3)	0.012(3)
C(39)	0.027(3)	0.032(4)	0.011(3)	0.014(3)	0.016(2)	0.007(2)
C(40)	0.030(4)	0.026(4)	0.024(3)	0.008(3)	0.008(3)	0.013(3)
C(41)	0.026(4)	0.030(4)	0.025(3)	-0.013(3)	0.002(3)	0.007(3)
C(42)	0.030(4)	0.027(4)	0.030(3)	0.003(3)	0.015(3)	-0.002(3)
C(43)	0.025(3)	0.029(4)	0.024(3)	-0.004(3)	0.003(3)	0.005(3)
C(44)	0.020(3)	0.022(3)	0.020(3)	-0.003(3)	0.007(2)	-0.003(3)
C(45)	0.019(3)	0.021(3)	0.026(3)	-0.008(3)	-0.004(2)	0.002(3)
C(46)	0.033(3)	0.036(4)	0.016(3)	0.013(3)	0.018(3)	0.007(3)
C(47)	0.032(4)	0.042(4)	0.037(3)	0.003(3)	-0.004(3)	0.024(3)
C(48)	0.023(3)	0.021(3)	0.020(3)	0.001(3)	0.008(2)	0.006(2)
C(49)	0.036(4)	0.038(4)	0.014(3)	0.003(3)	0.008(3)	0.007(3)
C(50)	0.025(3)	0.028(4)	0.031(3)	0.001(3)	0.005(3)	0.013(3)

Table 8. Bond Lengths (Å) at 200 K

atom	atom	distance	atom	atom	distance
O(1)	C(1)	1.271(7)	O(2)	C(2)	1.204(5)
O(3)	C(3)	1.212(7)	O(4)	C(18)	1.278(7)
O(5)	C(19)	1.220(6)	O(6)	C(20)	1.216(7)
O(7)	C(21)	1.275(7)	O(8)	C(22)	1.222(6)
O(9)	C(23)	1.238(7)	O(10)	C(38)	1.244(7)
O(11)	C(39)	1.214(5)	O(12)	C(40)	1.249(7)
O(13)	C(41)	1.254(7)	O(14)	C(42)	1.222(6)
O(15)	C(43)	1.253(7)	C(1)	C(2)	1.498(8)
C(1)	C(4)	1.406(7)	C(2)	C(3)	1.527(7)
C(3)	C(4)	1.445(8)	C(4)	C(5)	1.463(7)
C(5)	C(6)	1.364(8)	C(5)	C(10)	1.380(8)
C(6)	C(7)	1.405(7)	C(7)	C(8)	1.377(8)
C(8)	C(9)	1.401(8)	C(8)	C(11)	1.467(7)
C(9)	C(10)	1.403(7)	C(11)	C(12)	1.404(7)
C(11)	C(16)	1.401(8)	C(12)	C(13)	1.377(7)
C(13)	C(14)	1.383(8)	C(14)	C(15)	1.402(8)
C(14)	C(17)	1.449(7)	C(15)	C(16)	1.379(7)
C(17)	C(18)	1.408(7)	C(17)	C(20)	1.479(8)
C(18)	C(19)	1.482(8)	C(19)	C(20)	1.532(8)
C(21)	C(22)	1.481(8)	C(21)	C(24)	1.407(7)
C(22)	C(23)	1.514(7)	C(23)	C(24)	1.438(8)
C(24)	C(25)	1.440(7)	C(25)	C(26)	1.380(8)
C(25)	C(30)	1.388(8)	C(26)	C(27)	1.390(7)
C(27)	C(28)	1.386(8)	C(28)	C(29)	1.387(7)
C(28)	C(31)	1.512(6)	C(29)	C(30)	1.379(7)
C(31)	C(32)	1.382(7)	C(31)	C(36)	1.389(8)
C(32)	C(33)	1.408(7)	C(33)	C(34)	1.381(8)
C(34)	C(35)	1.388(7)	C(34)	C(37)	1.465(6)
C(35)	C(36)	1.414(7)	C(37)	C(38)	1.409(7)
C(37)	C(40)	1.415(8)	C(38)	C(39)	1.499(8)
C(39)	C(40)	1.507(7)	C(41)	C(42)	1.536(8)
C(41)	C(441)	1.375(8)	C(42)	C(43)	1.499(8)
C(43)	C(44)	1.432(7)	C(45)	C(46)	1.389(8)
C(45)	C(50)	1.383(8)	C(45)	C(44)	1.474(7)
C(46)	C(47)	1.383(7)	C(47)	C(48)	1.385(8)
C(48)	C(49)	1.401(8)	C(50)	C(49)	1.395(7)

Table 9. Bond Angles(°) at 200 K

atom	atom	atom	angle	atom	atom	atom	angle
O(1)	C(1)	C(2)	133.7(4)	O(1)	C(1)	C(4)	134.0(5)
C(2)	C(1)	C(4)	92.2(5)	O(2)	C(2)	C(1)	134.2(6)
O(2)	C(2)	C(3)	140.0(6)	C(1)	C(2)	C(3)	85.8(4)
O(3)	C(3)	C(2)	134.0(5)	O(3)	C(3)	C(4)	136.5(5)
C(2)	C(3)	C(4)	89.5(5)	C(1)	C(4)	C(3)	92.5(4)
C(1)	C(4)	C(5)	134.1(5)	C(3)	C(4)	C(5)	133.4(5)
C(4)	C(5)	C(6)	119.7(5)	C(4)	C(5)	C(10)	120.7(5)
C(6)	C(5)	C(10)	119.6(5)	C(5)	C(6)	C(7)	120.7(6)
C(6)	C(7)	C(8)	121.8(5)	C(7)	C(8)	C(9)	116.4(5)
C(7)	C(8)	C(11)	122.2(5)	C(9)	C(8)	C(11)	121.4(5)
C(8)	C(9)	C(10)	122.2(6)	C(5)	C(10)	C(9)	119.4(5)
C(8)	C(11)	C(12)	121.7(5)	C(8)	C(11)	C(16)	122.4(5)
C(12)	C(11)	C(16)	115.9(5)	C(11)	C(12)	C(13)	122.0(6)
C(12)	C(13)	C(14)	121.1(5)	C(13)	C(14)	C(15)	118.3(4)
C(13)	C(14)	C(17)	122.4(5)	C(15)	C(14)	C(17)	119.4(5)
C(14)	C(15)	C(16)	120.0(5)	C(11)	C(16)	C(15)	122.7(5)
C(14)	C(17)	C(18)	135.8(6)	C(14)	C(17)	C(20)	132.8(5)
C(18)	C(17)	C(20)	91.4(4)	O(4)	C(18)	C(17)	132.9(5)
O(4)	C(18)	C(19)	133.6(5)	C(17)	C(18)	C(19)	93.4(5)
O(5)	C(19)	C(18)	136.5(6)	O(5)	C(19)	C(20)	136.9(6)
C(18)	C(19)	C(20)	86.6(4)	O(6)	C(20)	C(17)	134.1(5)
O(6)	C(20)	C(19)	137.2(5)	C(17)	C(20)	C(19)	88.6(5)
O(7)	C(21)	C(22)	133.1(5)	O(7)	C(21)	C(24)	133.9(5)
C(22)	C(21)	C(24)	93.0(5)	O(8)	C(22)	C(21)	136.6(6)
O(8)	C(22)	C(23)	138.0(6)	C(21)	C(22)	C(23)	85.4(4)
O(9)	C(23)	C(22)	135.3(5)	O(9)	C(23)	C(24)	134.3(5)
C(22)	C(23)	C(24)	90.4(5)	C(21)	C(24)	C(23)	91.1(4)
C(21)	C(24)	C(25)	135.0(6)	C(23)	C(24)	C(25)	133.9(5)
C(24)	C(25)	C(26)	119.9(5)	C(24)	C(25)	C(30)	121.2(5)
C(26)	C(25)	C(30)	118.8(5)	C(25)	C(26)	C(27)	120.0(5)
C(26)	C(27)	C(28)	122.0(5)	C(27)	C(28)	C(29)	116.8(5)
C(27)	C(28)	C(31)	120.7(5)	C(29)	C(28)	C(31)	122.5(5)
C(28)	C(29)	C(30)	122.1(6)	C(25)	C(30)	C(29)	120.2(5)
C(28)	C(31)	C(32)	120.4(5)	C(28)	C(31)	C(36)	120.0(5)
C(32)	C(31)	C(36)	119.6(5)	C(31)	C(32)	C(33)	119.9(5)
C(32)	C(33)	C(34)	120.6(5)	C(33)	C(34)	C(35)	119.8(4)
C(33)	C(34)	C(37)	119.8(5)	C(35)	C(34)	C(37)	120.4(5)
C(34)	C(35)	C(36)	119.5(5)	C(31)	C(36)	C(35)	120.5(5)
C(34)	C(37)	C(38)	133.0(5)	C(34)	C(37)	C(40)	134.0(5)
C(38)	C(37)	C(40)	93.0(4)	O(10)	C(38)	C(37)	134.4(5)
O(10)	C(38)	C(39)	134.8(5)	C(37)	C(38)	C(39)	90.8(5)

Table 9. Bond Angles(°) at 200 K (continued)

atom	atom	atom	angle	atom	atom	atom	angle
O(11)	C(39)	C(38)	135.6(5)	O(11)	C(39)	C(40)	138.5(6)
C(38)	C(39)	C(40)	85.9(4)	O(12)	C(40)	C(37)	135.0(5)
O(12)	C(40)	C(39)	134.7(5)	C(37)	C(40)	C(39)	90.3(5)
O(13)	C(41)	C(42)	131.1(5)	O(13)	C(41)	C(44)	138.7(5)
C(42)	C(41)	C(44)	90.2(5)	O(14)	C(42)	C(41)	137.0(6)
O(14)	C(42)	C(43)	137.3(6)	C(41)	C(42)	C(43)	85.6(4)
O(15)	C(43)	C(42)	133.6(5)	O(15)	C(43)	C(44)	136.9(5)
C(42)	C(43)	C(44)	89.5(4)	C(46)	C(45)	C(50)	118.9(5)
C(46)	C(45)	C(44)	120.4(5)	C(50)	C(45)	C(44)	120.7(5)
C(45)	C(46)	C(47)	120.3(5)	C(46)	C(47)	C(48)	121.7(5)
C(47)	C(48)	C(49)	118.0(5)	C(45)	C(50)	C(49)	120.8(5)
C(41)	C(44)	C(43)	94.6(4)	C(41)	C(44)	C(45)	133.4(5)
C(43)	C(44)	C(45)	132.0(5)	C(48)	C(49)	C(50)	120.3(5)

Table 10. Atomic coordinates, *B*eq and occupancy at 273 K

atom	*x*	*y*	*z*	*B*eq	*occ*
O(1)	0.2918(2)	-0.6250(2)	0.7110(2)	3.21(7)	1.0000
O(2)	0.0959(2)	-0.6823(2)	0.7921(2)	3.88(8)	1.0000
O(3)	0.0396(2)	-0.8507(2)	0.5212(3)	4.38(8)	1.0000
O(4)	0.4104(2)	-1.1040(2)	-0.3803(2)	3.86(7)	1.0000
O(5)	0.6034(2)	-1.0361(2)	-0.4611(2)	3.71(7)	1.0000
O(6)	0.6594(2)	-0.8776(2)	-0.1788(3)	4.37(8)	1.0000
O(7)	0.7910(2)	-1.1292(2)	-0.7846(3)	3.75(7)	1.0000
O(8)	0.5912(2)	-1.1959(2)	-0.7154(3)	4.36(8)	1.0000
O(9)	0.5553(2)	-1.3699(2)	-0.9798(3)	4.08(8)	1.0000
O(10)	0.9247(2)	-1.5962(2)	-1.8672(2)	3.36(7)	1.0000
O(11)	1.1129(2)	-1.5263(2)	-1.9567(2)	3.50(7)	1.0000
O(12)	1.1738(2)	-1.3682(2)	-1.6779(2)	3.92(7)	1.0000
O(13)	0.7877(2)	-0.7977(2)	0.3202(3)	4.17(8)	1.0000
O(14)	0.8424(2)	-0.8208(2)	0.0519(3)	5.23(9)	1.0000
O(15)	0.7079(2)	-0.6655(2)	0.0345(2)	3.83(7)	1.0000
O(16)	0.2660(2)	-0.5172(2)	0.9162(2)	3.43(7)	1.0000
O(17)	0.5619(3)	-0.5639(2)	0.8631(3)	6.2(1)	1.0000
O(18)	0.6933(2)	-0.6724(2)	0.7923(3)	5.01(9)	1.0000
O(19)	0.8342(3)	-0.8429(4)	0.7644(4)	11.4(2)	1.0000
O(20)	0.9344(2)	-0.6898(2)	0.9046(3)	5.23(9)	1.0000
O(21)	0.9220(4)	-0.9214(4)	0.2597(5)	12.3(2)	1.0000
O(22)	0.5645(2)	-0.7980(2)	0.5918(3)	5.37(9)	1.0000
O(23)	0.2281(3)	-0.9691(2)	0.5655(3)	5.54(10)	1.0000
O(24)	0.9715(4)	-0.9220(4)	0.8914(4)	5.7(2)	0.5000
O(25)	0.8262(4)	-0.8521(3)	0.5236(4)	4.6(1)	0.5000

Table 10. Atomic coordinates, *B*eq and occupancy at 273 K (continued).

atom	*x*	*y*	*z*	*B*eq	*occ*
O(26)	0.9396(6)	-0.9889(6)	0.0421(8)	8.1(3)	0.5000
O(27)	0.9734(6)	-0.9654(5)	0.6477(7)	7.0(2)	0.5000
C(1)	0.2199(3)	-0.6885(3)	0.6590(3)	2.45(9)	1.0000
C(2)	0.1299(3)	-0.7119(3)	0.6994(3)	2.79(9)	1.0000
C(3)	0.1071(3)	-0.7908(3)	0.5729(4)	2.88(10)	1.0000
C(4)	0.1974(3)	-0.7602(3)	0.5456(3)	2.42(8)	1.0000
C(5)	0.2442(3)	-0.7938(3)	0.4386(3)	2.34(8)	1.0000
C(6)	0.1994(3)	-0.8666(3)	0.3351(4)	3.37(10)	1.0000
C(7)	0.2411(3)	-0.8952(3)	0.2304(3)	3.6(1)	1.0000
C(8)	0.3300(3)	-0.8536(3)	0.2248(3)	2.22(8)	1.0000
C(9)	0.3749(3)	-0.7808(3)	0.3314(4)	3.32(10)	1.0000
C(10)	0.3335(3)	-0.7514(3)	0.4369(3)	3.04(9)	1.0000
C(11)	0.3734(3)	-0.8834(3)	0.1123(3)	2.22(8)	1.0000
C(12)	0.3297(3)	-0.9587(3)	0.0071(3)	2.71(9)	1.0000
C(13)	0.3704(3)	-0.9863(3)	-0.0986(3)	2.60(9)	1.0000
C(14)	0.4563(3)	-0.9408(3)	-0.1042(3)	2.35(8)	1.0000
C(15)	0.5017(3)	-0.8654(3)	-0.0008(3)	2.93(9)	1.0000
C(16)	0.4590(3)	-0.8378(3)	0.1045(3)	3.02(9)	1.0000
C(17)	0.5028(3)	-0.9707(3)	-0.2116(3)	2.41(9)	1.0000
C(18)	0.4801(3)	-1.0392(3)	-0.3286(3)	2.54(9)	1.0000
C(19)	0.5684(3)	-1.0111(3)	-0.3674(3)	2.66(9)	1.0000
C(20)	0.5932(3)	-0.9373(3)	-0.2378(4)	2.80(9)	1.0000
C(21)	0.7221(3)	-1.1930(3)	-0.8377(4)	2.75(9)	1.0000
C(22)	0.6317(3)	-1.2236(3)	-0.8035(3)	2.87(9)	1.0000
C(23)	0.6157(3)	-1.3020(3)	-0.9262(4)	2.99(10)	1.0000
C(24)	0.7025(3)	-1.2671(3)	-0.9536(3)	2.38(9)	1.0000
C(25)	0.7511(3)	-1.2955(3)	-1.0601(3)	2.30(8)	1.0000
C(26)	0.7081(3)	-1.3677(3)	-1.1664(4)	3.6(1)	1.0000
C(27)	0.7525(3)	-1.3924(3)	-1.2690(3)	3.28(9)	1.0000
C(28)	0.8406(3)	-1.3479(3)	-1.2693(3)	2.13(8)	1.0000
C(29)	0.8844(3)	-1.2774(3)	-1.1609(3)	3.13(9)	1.0000
C(30)	0.8400(3)	-1.2512(3)	-1.0581(3)	3.28(10)	1.0000
C(31)	0.8858(3)	-1.3767(3)	-1.3819(3)	2.05(8)	1.0000
C(32)	0.8423(3)	-1.4469(3)	-1.4904(3)	2.58(9)	1.0000
C(33)	0.8850(3)	-1.4748(3)	-1.5933(3)	2.37(8)	1.0000
C(34)	0.9739(3)	-1.4309(3)	-1.5933(3)	2.18(8)	1.0000
C(35)	1.0188(3)	-1.3590(3)	-1.4865(3)	2.55(8)	1.0000
C(36)	0.9756(3)	-1.3325(3)	-1.3829(3)	2.46(8)	1.0000
C(37)	1.0194(3)	-1.4605(3)	-1.7020(3)	2.18(8)	1.0000
C(38)	0.9927(3)	-1.5323(3)	-1.8194(3)	2.48(9)	1.0000
C(39)	1.0804(3)	-1.5022(3)	-1.8620(3)	2.56(9)	1.0000

Table 10. Atomic coordinates, *B*eq and occupancy at 273 K (continued).

atom	*x*	*y*	*z*	*B*eq	*occ*
C(40)	1.1054(3)	-1.4302(3)	-1.7339(3)	2.59(9)	1.0000
C(41)	0.7640(3)	-0.7621(3)	0.2416(4)	2.85(9)	1.0000
C(42)	0.7910(3)	-0.7742(3)	0.1159(4)	3.42(10)	1.0000
C(43)	0.7290(3)	-0.7037(3)	0.1110(4)	2.88(9)	1.0000
C(44)	0.7062(3)	-0.6965(3)	0.2294(3)	2.78(9)	1.0000
C(45)	0.6466(3)	-0.6397(3)	0.3086(3)	2.56(8)	1.0000
C(46)	0.5963(3)	-0.5782(3)	0.2703(4)	3.19(10)	1.0000
C(47)	0.5411(3)	-0.5240(3)	0.3447(4)	3.19(10)	1.0000
C(48)	0.5308(3)	-0.5294(3)	0.4602(3)	2.52(8)	1.0000
C(49)	0.5802(3)	-0.5922(3)	0.4983(3)	3.05(9)	1.0000
C(50)	0.6370(3)	-0.6451(3)	0.4237(3)	2.99(9)	1.0000
H(1)	0.1327	-0.9051	0.3352	4.0331	1.0000
H(2)	0.2161	-0.9600	0.1474	4.2607	1.0000
H(3)	0.4331	-0.7530	0.3289	3.9224	1.0000
H(4)	0.3672	-0.7025	0.5103	3.6329	1.0000
H(5)	0.2659	-0.9887	0.0102	3.2494	1.0000
H(6)	0.3353	-1.0442	-0.1743	3.1091	1.0000
H(7)	0.5630	-0.8344	0.0034	3.5311	1.0000
H(8)	0.4872	-0.7888	0.1725	3.6151	1.0000
H(9)	0.6472	-1.3959	-1.1726	4.2706	1.0000
H(10)	0.7177	-1.4417	-1.3381	3.9124	1.0000
H(11)	0.9579	-1.2484	-1.1515	3.7172	1.0000
H(12)	0.8764	-1.2030	-0.9865	3.9253	1.0000
H(13)	0.7906	-1.4762	-1.4866	3.0798	1.0000
H(14)	0.8546	-1.5324	-1.6723	2.8135	1.0000
H(15)	1.0815	-1.3248	-1.4734	3.0274	1.0000
H(16)	1.0104	-1.2824	-1.3065	2.8936	1.0000
H(17)	0.5053	-0.4738	0.3168	3.8520	1.0000
H(18)	0.6017	-0.5704	0.1677	3.8081	1.0000
H(19)	0.6843	-0.6872	0.4513	3.5502	1.0000
H(20)	0.5764	-0.5929	0.5905	3.6155	1.0000
H(21)	0.7882	-0.8327	0.7498	4.4291	1.0000
H(22)	0.7812	-0.8835	0.7992	4.4291	1.0000
H(23)	0.9351	-0.9315	0.8591	4.4291	1.0000
H(24)	0.9573	-0.7445	0.9265	4.4291	1.0000
H(25)	1.0080	-0.6648	0.9108	4.4291	1.0000
H(26)	0.9045	-0.7186	0.9126	4.4291	1.0000
H(27)	0.6589	-0.6396	0.7275	4.4291	1.0000
H(28)	0.6111	-0.7535	0.6616	4.4291	1.0000
H(29)	0.5006	-0.8057	0.6255	4.4291	1.0000
H(30)	0.2958	-0.9342	0.5572	4.4291	1.0000

Table 10. Atomic coordinates, *B*eq and occupancy at 273 K (continued).

atom	*x*	*y*	*z*	*B*eq	*occ*
H(31)	0.2576	-0.9847	0.6159	4.4291	1.0000
H(32)	0.1995	-1.0306	0.5481	4.4291	1.0000
H(33)	0.9316	-0.9027	0.2626	4.4291	1.0000

Table 11. Anisotropic Displacement Parameters at 273 K

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.042(2)	0.040(2)	0.036(1)	0.001(2)	0.005(1)	0.010(1)
O(2)	0.059(2)	0.063(2)	0.034(1)	0.021(2)	0.032(1)	0.017(1)
O(3)	0.049(2)	0.057(2)	0.051(2)	-0.011(2)	0.019(2)	0.007(2)
O(4)	0.049(2)	0.051(2)	0.037(2)	-0.011(2)	0.010(1)	0.006(1)
O(5)	0.048(2)	0.061(2)	0.035(1)	0.014(2)	0.025(1)	0.013(1)
O(6)	0.045(2)	0.065(2)	0.042(2)	-0.012(2)	0.015(2)	0.003(2)
O(7)	0.053(2)	0.045(2)	0.040(2)	0.006(2)	0.016(2)	0.007(1)
O(8)	0.062(2)	0.068(2)	0.043(2)	0.024(2)	0.035(2)	0.017(2)
O(9)	0.041(2)	0.064(2)	0.042(2)	-0.002(2)	0.012(1)	0.008(2)
O(10)	0.040(2)	0.044(2)	0.035(1)	-0.005(2)	0.010(1)	0.004(1)
O(11)	0.050(2)	0.058(2)	0.029(1)	0.008(2)	0.023(1)	0.014(1)
O(12)	0.043(2)	0.061(2)	0.034(2)	-0.015(2)	0.008(1)	0.007(1)
O(13)	0.053(2)	0.054(2)	0.058(2)	0.013(2)	0.011(2)	0.027(2)
O(14)	0.068(2)	0.072(3)	0.060(2)	0.031(2)	0.032(2)	0.012(2)
O(15)	0.056(2)	0.050(2)	0.037(2)	0.006(2)	0.009(1)	0.013(1)
O(16)	0.047(2)	0.046(2)	0.034(1)	0.005(2)	0.015(1)	0.006(1)
O(17)	0.099(3)	0.064(3)	0.061(2)	0.009(2)	-0.006(2)	0.018(2)
O(18)	0.064(2)	0.077(3)	0.044(2)	-0.005(2)	0.014(2)	0.017(2)
O(19)	0.131(4)	0.167(5)	0.081(3)	-0.078(4)	0.069(3)	-0.022(3)
O(20)	0.187(5)	0.077(3)	0.061(2)	-0.050(3)	0.015(3)	0.018(2)
O(21)	0.169(6)	0.138(5)	0.149(5)	0.035(4)	0.037(4)	0.031(4)
O(22)	0.115(7)	0.092(7)	0.096(6)	0.045(6)	0.023(5)	0.021(5)
O(23)	0.134(8)	0.058(5)	0.064(5)	-0.011(5)	0.033(5)	0.009(4)
O(24)	0.172(6)	0.118(5)	0.102(4)	-0.028(4)	-0.006(4)	0.012(3)
O(25)	0.051(2)	0.078(3)	0.070(2)	-0.004(2)	0.022(2)	0.025(2)
O(26)	0.055(2)	0.083(3)	0.043(2)	-0.017(2)	0.017(2)	-0.003(2)
O(27)	0.085(3)	0.059(2)	0.077(2)	0.010(2)	0.044(2)	0.026(2)
C(1)	0.035(2)	0.034(2)	0.027(2)	0.006(2)	0.006(2)	0.013(2)
C(2)	0.039(2)	0.043(3)	0.031(2)	0.015(2)	0.015(2)	0.017(2)
C(3)	0.034(2)	0.043(3)	0.035(2)	0.004(2)	0.016(2)	0.014(2)
C(4)	0.034(2)	0.033(2)	0.026(2)	0.009(2)	0.010(2)	0.010(2)
C(5)	0.034(2)	0.034(2)	0.022(2)	0.003(2)	0.009(2)	0.011(2)
C(6)	0.037(3)	0.051(3)	0.030(2)	-0.008(2)	0.017(2)	0.002(2)
C(7)	0.042(3)	0.050(3)	0.029(2)	-0.012(2)	0.016(2)	-0.005(2)

Table 11. Anisotropic Displacement Parameters at 273 K (continued).

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(8)	0.029(2)	0.030(2)	0.024(2)	0.002(2)	0.007(2)	0.008(2)
C(9)	0.033(2)	0.049(3)	0.033(2)	-0.007(2)	0.015(2)	0.001(2)
C(10)	0.038(2)	0.040(3)	0.028(2)	-0.006(2)	0.012(2)	0.000(2)
C(11)	0.028(2)	0.033(2)	0.025(2)	0.004(2)	0.007(2)	0.012(2)
C(12)	0.031(2)	0.039(2)	0.029(2)	-0.004(2)	0.011(2)	0.007(2)
C(13)	0.033(2)	0.033(2)	0.026(2)	-0.003(2)	0.008(2)	0.003(2)
C(14)	0.034(2)	0.031(2)	0.023(2)	0.007(2)	0.007(2)	0.007(2)
C(15)	0.030(2)	0.043(3)	0.030(2)	-0.004(2)	0.014(2)	0.003(2)
C(16)	0.038(2)	0.044(3)	0.024(2)	-0.006(2)	0.013(2)	0.001(2)
C(17)	0.033(2)	0.036(2)	0.026(2)	0.007(2)	0.010(2)	0.013(2)
C(18)	0.033(2)	0.036(2)	0.028(2)	0.003(2)	0.009(2)	0.011(2)
C(19)	0.038(2)	0.040(2)	0.028(2)	0.011(2)	0.013(2)	0.016(2)
C(20)	0.036(2)	0.039(3)	0.032(2)	0.005(2)	0.012(2)	0.011(2)
C(21)	0.038(3)	0.040(3)	0.031(2)	0.009(2)	0.008(2)	0.016(2)
C(22)	0.042(3)	0.045(3)	0.031(2)	0.017(2)	0.020(2)	0.018(2)
C(23)	0.040(3)	0.047(3)	0.030(2)	0.015(2)	0.011(2)	0.015(2)
C(24)	0.032(2)	0.039(2)	0.025(2)	0.008(2)	0.009(2)	0.015(2)
C(25)	0.027(2)	0.036(2)	0.025(2)	0.003(2)	0.009(2)	0.012(2)
C(26)	0.033(2)	0.063(3)	0.030(2)	-0.012(2)	0.013(2)	0.005(2)
C(27)	0.034(2)	0.050(3)	0.027(2)	-0.013(2)	0.012(2)	-0.001(2)
C(28)	0.027(2)	0.031(2)	0.024(2)	0.004(2)	0.007(2)	0.010(2)
C(29)	0.034(2)	0.048(3)	0.027(2)	-0.003(2)	0.014(2)	-0.001(2)
C(30)	0.041(3)	0.044(3)	0.028(2)	-0.008(2)	0.011(2)	0.000(2)
C(31)	0.029(2)	0.029(2)	0.022(2)	0.007(2)	0.008(2)	0.009(2)
C(32)	0.029(2)	0.036(2)	0.028(2)	-0.004(2)	0.011(2)	0.004(2)
C(33)	0.029(2)	0.034(2)	0.024(2)	0.002(2)	0.011(2)	0.005(2)
C(34)	0.027(2)	0.032(2)	0.027(2)	0.003(2)	0.008(2)	0.013(2)
C(35)	0.030(2)	0.039(2)	0.023(2)	-0.003(2)	0.010(2)	0.005(2)
C(36)	0.030(2)	0.036(2)	0.023(2)	-0.001(2)	0.011(2)	0.004(2)
C(37)	0.029(2)	0.028(2)	0.024(2)	0.005(2)	0.005(2)	0.007(2)
C(38)	0.034(2)	0.036(2)	0.023(2)	0.003(2)	0.006(2)	0.010(2)
C(39)	0.039(2)	0.038(2)	0.023(2)	0.009(2)	0.011(2)	0.010(2)
C(40)	0.035(2)	0.035(2)	0.027(2)	0.001(2)	0.010(2)	0.010(2)
C(41)	0.033(2)	0.036(3)	0.035(2)	-0.007(2)	0.004(2)	0.012(2)
C(42)	0.041(3)	0.038(3)	0.046(2)	-0.001(2)	0.009(2)	0.010(2)
C(43)	0.034(2)	0.043(3)	0.030(2)	-0.001(2)	0.006(2)	0.011(2)
C(44)	0.030(2)	0.033(2)	0.033(2)	-0.010(2)	0.002(2)	0.005(2)
C(45)	0.028(2)	0.027(2)	0.034(2)	-0.004(2)	0.003(2)	0.003(2)
C(46)	0.045(3)	0.048(3)	0.034(2)	0.009(2)	0.015(2)	0.019(2)
C(47)	0.045(3)	0.045(3)	0.036(2)	0.011(2)	0.009(2)	0.019(2)
C(48)	0.029(2)	0.034(2)	0.030(2)	-0.004(2)	0.004(2)	0.010(2)

Table 11. Anisotropic Displacement Parameters at 273 K (continued).

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(49)	0.043(3)	0.040(2)	0.030(2)	0.008(2)	0.009(2)	0.007(2)
C(50)	0.036(2)	0.044(3)	0.037(2)	0.006(2)	0.011(2)	0.017(2)

Table 12. Bond Lengths (Å) at 273K.

atom	atom	distance	atom	atom	distance
O(1)	C(1)	1.289(5)	O(2)	C(2)	1.206(4)
O(3)	C(3)	1.216(5)	O(4)	C(18)	1.279(5)
O(5)	C(19)	1.218(4)	O(6)	C(20)	1.215(5)
O(7)	C(21)	1.265(5)	O(8)	C(22)	1.214(4)
O(9)	C(23)	1.233(5)	O(10)	C(38)	1.251(5)
O(11)	C(39)	1.207(4)	O(12)	C(40)	1.249(5)
O(13)	C(41)	1.252(5)	O(14)	C(42)	1.221(4)
O(15)	C(43)	1.253(5)	C(1)	C(2)	1.485(6)
C(1)	C(4)	1.387(5)	C(2)	C(3)	1.539(5)
C(3)	C(4)	1.450(6)	C(4)	C(5)	1.454(5)
C(5)	C(6)	1.376(5)	C(5)	C(10)	1.393(6)
C(6)	C(7)	1.382(5)	C(7)	C(8)	1.398(6)
C(8)	C(9)	1.396(5)	C(8)	C(11)	1.472(5)
C(9)	C(10)	1.389(5)	C(11)	C(12)	1.402(5)
C(11)	C(16)	1.394(6)	C(12)	C(13)	1.383(5)
C(13)	C(14)	1.387(6)	C(14)	C(15)	1.399(5)
C(14)	C(17)	1.450(5)	C(15)	C(16)	1.395(5)
C(17)	C(18)	1.392(5)	C(17)	C(20)	1.462(6)
C(18)	C(19)	1.479(6)	C(19)	C(20)	1.529(5)
C(21)	C(22)	1.490(6)	C(21)	C(24)	1.423(5)
C(22)	C(23)	1.508(5)	C(23)	C(24)	1.432(6)
C(24)	C(25)	1.457(5)	C(25)	C(26)	1.382(5)
C(25)	C(30)	1.388(6)	C(26)	C(27)	1.383(5)
C(27)	C(28)	1.387(5)	C(28)	C(29)	1.387(5)
C(28)	C(31)	1.488(4)	C(29)	C(30)	1.385(5)
C(31)	C(32)	1.384(5)	C(31)	C(36)	1.407(5)
C(32)	C(33)	1.373(5)	C(33)	C(34)	1.391(5)
C(34)	C(35)	1.390(5)	C(34)	C(37)	1.454(4)
C(35)	C(36)	1.383(5)	C(37)	C(38)	1.421(5)
C(37)	C(40)	1.421(5)	C(38)	C(39)	1.507(6)
C(39)	C(40)	1.505(5)	C(41)	C(42)	1.518(6)
C(41)	C(44)	1.403(6)	C(42)	C(43)	1.486(6)
C(43)	C(44)	1.430(5)	C(45)	C(46)	1.405(5)
C(45)	C(50)	1.397(5)	C(45)	C(44)	1.465(5)
C(46)	C(47)	1.373(5)	C(47)	C(48)	1.405(5)
C(48)	C(49)	1.414(5)	C(50)	C(49)	1.383(5)

Table 13. Bond Angles(°) at 273 K

atom	atom	atom	angle	atom	atom	atom	angle
O(1)	C(1)	C(2)	132.9(3)	O(1)	C(1)	C(4)	133.7(4)
C(2)	C(1)	C(4)	93.4(3)	O(2)	C(2)	C(1)	135.4(4)
O(2)	C(2)	C(3)	139.2(4)	C(1)	C(2)	C(3)	85.3(3)
O(3)	C(3)	C(2)	133.7(4)	O(3)	C(3)	C(4)	137.5(4)
C(2)	C(3)	C(4)	88.8(3)	C(1)	C(4)	C(3)	92.5(3)
C(1)	C(4)	C(5)	134.9(4)	C(3)	C(4)	C(5)	132.5(4)
C(4)	C(5)	C(6)	120.5(4)	C(4)	C(5)	C(10)	120.8(4)
C(6)	C(5)	C(10)	118.7(3)	C(5)	C(6)	C(7)	120.6(4)
C(6)	C(7)	C(8)	122.4(4)	C(7)	C(8)	C(9)	116.2(3)
C(7)	C(8)	C(11)	122.1(3)	C(9)	C(8)	C(11)	121.7(4)
C(8)	C(9)	C(10)	121.9(4)	C(5)	C(10)	C(9)	120.3(4)
C(8)	C(11)	C(12)	121.4(4)	C(8)	C(11)	C(16)	121.8(4)
C(12)	C(11)	C(16)	116.8(3)	C(11)	C(12)	C(13)	121.3(4)
C(12)	C(13)	C(14)	121.2(4)	C(13)	C(14)	C(15)	118.9(3)
C(13)	C(14)	C(17)	122.4(4)	C(15)	C(14)	C(17)	118.7(4)
C(14)	C(15)	C(16)	119.1(4)	C(11)	C(16)	C(15)	122.7(4)
C(14)	C(17)	C(18)	135.2(4)	C(14)	C(17)	C(20)	132.6(4)
C(18)	C(17)	C(20)	92.2(3)	O(4)	C(18)	C(17)	133.0(4)
O(4)	C(18)	C(19)	133.9(3)	C(17)	C(18)	C(19)	93.1(3)
O(5)	C(19)	C(18)	136.0(4)	O(5)	C(19)	C(20)	137.7(4)
C(18)	C(19)	C(20)	86.3(3)	O(6)	C(20)	C(17)	134.8(4)
O(6)	C(20)	C(19)	136.9(4)	C(17)	C(20)	C(19)	88.4(3)
O(7)	C(21)	C(22)	135.1(4)	O(7)	C(21)	C(24)	133.8(4)
C(22)	C(21)	C(24)	91.0(4)	O(8)	C(22)	C(21)	135.3(4)
O(8)	C(22)	C(23)	137.9(4)	C(21)	C(22)	C(23)	86.7(3)
O(9)	C(23)	C(22)	135.4(4)	O(9)	C(23)	C(24)	134.5(4)
C(22)	C(23)	C(24)	90.0(4)	C(21)	C(24)	C(23)	92.3(3)
C(21)	C(24)	C(25)	133.9(4)	C(23)	C(24)	C(25)	133.9(4)
C(24)	C(25)	C(26)	119.9(4)	C(24)	C(25)	C(30)	121.5(4)
C(26)	C(25)	C(30)	118.6(3)	C(25)	C(26)	C(27)	119.8(4)
C(26)	C(27)	C(28)	122.3(4)	C(27)	C(28)	C(29)	117.4(3)
C(27)	C(28)	C(31)	120.5(3)	C(29)	C(28)	C(31)	122.1(4)
C(28)	C(29)	C(30)	120.9(4)	C(25)	C(30)	C(29)	120.9(4)
C(28)	C(31)	C(32)	122.3(4)	C(28)	C(31)	C(36)	121.0(3)
C(32)	C(31)	C(36)	116.7(3)	C(31)	C(32)	C(33)	122.3(4)
C(32)	C(33)	C(34)	120.6(4)	C(33)	C(34)	C(35)	118.6(3)
C(33)	C(34)	C(37)	120.7(3)	C(35)	C(34)	C(37)	120.8(4)
C(34)	C(35)	C(36)	120.2(4)	C(31)	C(36)	C(35)	121.6(3)
C(34)	C(37)	C(38)	132.9(4)	C(34)	C(37)	C(40)	135.0(4)
C(38)	C(37)	C(40)	92.2(3)	O(10)	C(38)	C(37)	134.5(4)
O(10)	C(38)	C(39)	134.5(3)	C(37)	C(38)	C(39)	91.0(3)

Table 13. Bond Angles(°) at 273 K (continued)

atom	atom	atom	angle	atom	atom	atom	angle
O(11)	C(39)	C(38)	135.8(4)	O(11)	C(39)	C(40)	138.6(4)
C(38)	C(39)	C(40)	85.7(3)	O(12)	C(40)	C(37)	134.3(3)
O(12)	C(40)	C(39)	134.7(4)	C(37)	C(40)	C(39)	91.1(3)
O(13)	C(41)	C(42)	132.5(3)	O(13)	C(41)	C(44)	137.5(4)
C(42)	C(41)	C(44)	90.0(3)	O(14)	C(42)	C(41)	135.4(4)
O(14)	C(42)	C(43)	138.1(4)	C(41)	C(42)	C(43)	86.5(3)
O(15)	C(43)	C(42)	134.2(3)	O(15)	C(43)	C(44)	135.6(4)
C(42)	C(43)	C(44)	90.2(3)	C(46)	C(45)	C(50)	117.7(3)
C(46)	C(45)	C(44)	120.7(4)	C(50)	C(45)	C(44)	121.6(4)
C(45)	C(46)	C(47)	121.0(4)	C(46)	C(47)	C(48)	121.6(4)
C(47)	C(48)	C(49)	117.6(3)	C(45)	C(50)	C(49)	121.8(4)
C(41)	C(44)	C(43)	93.2(3)	C(41)	C(44)	C(45)	133.9(4)
C(43)	C(44)	C(45)	132.9(4)	C(48)	C(49)	C(50)	120.3(4)

$B_{eq} = 8/3\ \pi^2\{U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha\}$

The general temperature factor expression:

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