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Absolute Stereochemistry of Amphidinolides G and H

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Experimental Procedure

Cultivation and Isolation. The dinoflagellate *Amphidinium* sp. (strain number Y-72) was isolated from the inside cells of the marine acoel flatworm *Amphiscolops* sp. collected off Zanpa, Okinawa. The dinoflagellate was unialgally cultured at 25 °C for two weeks in a seawater medium enriched with 1% ES supplement. The harvested cells of the cultured dinoflagellate (50 g, wet weight, from 120 L of culture) were extracted with MeOH/toluene (4:1, 150 mL x 2). After addition of 1 M NaCl aq. (150 mL), the mixture was extracted with toluene (150 mL x 3). The toluene-soluble materials (1.2 g) were subjected to a silica gel column ($CHCl_3$/MeOH, 95:5) and a Sep-Pak C_{18} cartridge (CH_3CN/H_2O, 8:2) followed by C_{18} HPLC [Mightysil RP-18, 5 μm, Kanto Chemical Co. Inc., 10 x 250 mm; eluent, CH_3CN/H_2O, 70:30; flow rate, 3.0 mL/min; UV detection at 240 nm] to afford amphidinolides G (**1**, 23 mg, 0.046 %, wet weight, t_R 19.2 min) and H (**2**, 41 mg, 0.082 %, t_R 16.4 min).

Tris-(*S*)-MTPA Ester 3. To a solution of amphidinolide H (**2**, 2.9 mg) in EtOH (300 μL) was added $NaBH_4$ (4.2 mg) at 0 °C, and stirring was continued for 20 min. After evaporation of the solvent, the residue was partitioned between EtOAc (300 μL x 3) and 1 M phosphate buffer (pH 6.9, 300 μL), and then the organic phase was evaporated in vacuo to give a crude residue. To a solution of the crude residue in THF/1 M phosphate buffer (1:1, 300 μL) was added $NaIO_4$ (3.8 mg) at 0 °C, and the mixture was stirred for 1 h. After evaporation of the solvent, the reaction mixture

was extracted with EtOAc (300 μL), and then the extract was evaporated in vacuo. To a solution of the extract in EtOH (300 μL) was added $NaBH_4$ (4.3 mg) at 0 °C, and stirring was continued for 45 min. After evaporation of the solvent, the residue was dissolved in 0.1% DMAP solution in CH_2Cl_2 (200 μL). To the mixture were added Et_3N (15 μL) and (*R*)-(-)-MTPACl (16 μL), and the mixture was stirred at room temperature for 2 h. After addition of *N*,*N*-dimethyl-1,3-propanediamine (8 μL), the solvent was evaporated in vacuo. The residue was subjected to silica gel chromatography (hexane/acetone, 2:1) and then C_{18} HPLC (Mightysil RP-18, Kanto Chemical Co. Inc., 10 x 250 mm; eluent CH_3CN/H_2O, 85:15; flow rate, 3.0 mL/min; UV detection at 220 nm) to afford compound **3** (0.2 mg, t_R 26.4 min). **3:** ^{1}H NMR ($CDCl_3$) δ 0.92 (3H, d, *J* = 6.5 Hz), 1.36 (1H, m), 1.74 ~ 1.85 (2H, m), 3.37 (3H, s), 3.42 (3H, s), 3.50 (3H, s), 4.03 (1H, dd, *J* = 6.1 and 10.9 Hz), 4.14 ~ 4.18 (2H, m), 4.47 (1H, dd, *J* = 2.7 and 12.3 Hz), 5.37 (1H, m), and 7.25 ~ 7.48 (15H, m); FABMS *m/z* 805 $(M+Na)^+$; HRFABMS *m/z* 805.2012 [$(M+Na)^+$, calcd for $C_{36}H_{35}O_9F_9Na$, 805.2035].

Tris-(*S*)-MTPA Ester (3) of (2*R*,4*R*)-4-Methylpentane-1,2,5-triol. To a solution of (2*R*, 4*R*)-4,5-(isopropylidenedioxy)-2-methylpentan-1-ol (**5**, 20 mg) in THF (1 mL) was added 1N HCl aq. (1 mL), and stirring was continued at room temperature for 2 h. The solvent was evaporated in vacuo to afford (2*R*,4*R*)-4-methylpentane-1,2,5-triol (15 mg). To a solution of the triol (1.4 mg) in pyridine (40

μL) was added (*R*)-(-)-MTPACl (16.3 μL), and the mixture was stirred at room temperature for 15 min. After addition of *N,N*-dimethyl-1,3-propanediamine (11 μL), the solvent was evaporated in vacuo. The mixture was partitioned between $CHCl_3$ (300 μL x 3) and H_2O (300 μL), and the organic phase was subjected to silica gel chromatography (hexane/EtOAc, 4:1) and then C_{18} HPLC (Mightysil RP-18, 10 x 250 mm; eluent CH_3CN/H_2O, 85:15; flow rate, 3.0 mL/min; UV detection at 220 nm) to afford tris-(*S*)-MTPA ester (**3**, 2.8 mg, t_R 26.4 min). **3:** colorless oil; $[\alpha]_D^{24}$ -37° (*c* 0.5, $CHCl_3$); IR (KBr) ν_{max} 3425, 1750, 1270, and 1175 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.92 (3H, d, *J* = 6.5 Hz), 1.36 (1H, m), 1.74 ~ 1.85 (2H, m), 3.37 (3H, s), 3.42 (3H, s), 3.50 (3H, s), 4.03 (1H, dd, *J* = 6.1 and 10.9 Hz), 4.14 ~ 4.18 (2H, m), 4.47 (1H, dd, *J* = 2.7 and 12.3 Hz), 5.37 (1H, m), and 7.25 ~ 7.48 (15H, m); FABMS *m/z* 805 $(M+Na)^+$; HRFABMS *m/z* 805.2029 [$(M+Na)^+$, calcd for $C_{36}H_{35}O_9F_9Na$, 805.2035].

Tris-(*R*)-MTPA Ester (4) of (2*R*,4*R*)-4-Methylpentane-1,2,5-triol. Tris-(*R*)-MTPA ester (**4**, 1.7 mg) was prepared from (2*R*,4*R*)-4-methylpentane-1,2,5-triol (0.7 mg) and (*S*)-(+)-MTPACl (16.0 μL) by the same procedure as described above. **4**: colorless oil; $[\alpha]_D^{24}$ +43° (*c* 0.6, $CHCl_3$); IR (KBr) ν_{max} 3425, 1750, 1270, and 1175 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.86 (3H, d, *J* = 6.7 Hz), 1.27 (1H, m), 1.60 (1H, m), 1.70 (1H, ddd, *J* = 4.3, 10.0, and 14.3 Hz), 3.39 (3H, s), 3.46 (3H, s), 3.48 (3H, s), 4.00 (2H, m), 4.26 (1H, dd, *J* = 5.6 and 12.3 Hz), 4.56 (1H, dd, *J* = 3.0 and 12.3 Hz), 5.35 (1H, m), and 7.30 ~ 7.48 (15H, m); FABMS *m/z* 805 $(M+Na)^+$; HRFABMS *m/z*

805.2012 [$(M+Na)^+$, calcd for $C_{36}H_{35}O_9F_9Na$, 805.2035].

Interconversion between Amphidinolides G (1) and H (2). To a solution of amphidinolide H (**2**, 0.6 mg) in EtOH (60 μL) was added K_2CO_3 (0.15 mg), and stirring was continued at 4 °C for 18 h. After evaporation of the solvent, the residue was partitioned between EtOAc and H_2O. The EtOAc-soluble materials were subjected to C_{18} HPLC [Mightysil RP-18, 5 μm, 10 x 250 mm; eluent, CH_3CN/H_2O, 70:30; flow rate, 3.0 mL/min; UV detection at 240 nm] to afford amphidinolides G (**1**, 0.1 mg, t_R 19.2 min) and H (**2**, 0.1 mg, t_R 16.4 min). **1**: $[\alpha]_D^{24}$ -67° (*c* 0.1, $CHCl_3$); FABMS *m/z* 668 $(M+diethanolamine+H)^+$; HRFABMS *m/z* 668.4352 [$(M+diethanolamine+H)^+$, calcd for $C_{36}H_{62}O_{10}N$, 668.4374].

Amphidinolide G (**1**, 0.5 mg) was subjected to treatment with K_2CO_3 followed by separation using HPLC as the same condition as described above to afford **1** (0.1 mg, t_R 19.2 min) and **2** (0.1 mg, t_R 16.4 min). **2**: $[\alpha]_D^{24}$ -35° (*c* 0.1, $CHCl_3$); FABMS *m/z* 668 $(M+diethanolamine+H)^+$; HRFABMS *m/z* 668.4386 [$(M+diethanolamine+H)^+$, calcd for $C_{36}H_{62}O_{10}N$, 668.4374].

X-ray Diffraction Analysis. Amphidinolide H (**2**) was crystallized as colorless needles (mp. 131 ~ 132 °C) from *n*-hexane/benzene. Crystal data: $C_{44}H_{62}O_8$, M_r = 718.97, crystal dimensions 0.30 x 0.30 x 0.08 mm, orthorhombic, space group $P2_12_12_1$ (no. 19), a = 11.766(1) Å, b = 31.797(2) Å, c = 11.233(1) Å, V = 4202.3(6) Å^3, Z = 4, D_{calc} = 1.14g/cm^{-1}. A crystal was coated with liquid paraffin. All measurements was made on Rigaku RAXIS-RAPID Imaging Plate diffractometer with graphite monochromated Mo-Kα radiation. The data were collected at a temperature of -150 ± 1 °C to maximum 2θ value of 55.0°. A total of 145 images, corresponding to 290.0° oscillation angles, were collected with 3 different goniometer settings. Exposure time was 2.00 min per degree. The camera radius was 127.40 mm. Of the 43252 reflections which were collected, 5363 were unique (R_{int} = 7.0 %); equivalent reflections were merged. The linear absorption coefficient, m, for Mo-Kα radiation was 0.8 cm^{-1}. A symmetry-related absorption correction using the program ABSCOR was applied which resulted in transmission factors ranging from 0.88 to 0.99. The data were corrected for Lorentz and polarization effects. A correction for secondary extinction was applied (coefficient = 6.99710 x 10^{-7}).

The structure was solved by direct methods (SIR97) and expanded using Fourier technique. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were including but not refined. The final cycle of full matrix least-squares refinement was based on 3853 observed reflections ($I > 2.00\sigma(I)$) and 470 variable parameters and

converged with unweighted and weighted agreement factors of $R = 0.0045$, $R_w = 0.037$. All calculations were performed using the teXsan crystallographic software package of Molecular Structure Corporation.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{44}H_{62}O_8$
Formula Weight	718.97
Crystal Color, Habit	colorless, ~~unknown~~
Crystal Dimensions	0.30 X 0.30 X 0.08 mm
Crystal System	orthorhombic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	79220 (3.6 - 55.0°)
Indexing Images	3 oscillations at 1.0 minutes
Camera Radius	127.40 mm
Lattice Parameters	a = 11.766(1) Å b = 31.797(2) Å c = 11.233(1) Å V = 4202.3(6) Å^3
Space Group	$P2_12_12_1$ (#19)
Z value	4
D_{calc}	1.136 g/cm^3
F_{000}	1560.00
μ(MoKα)	0.76 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID Imaging Plate
Radiation	MoKα (λ = 0.71069 Å) graphite monochromated

Temperature	-150.0 °C
Voltage, Current	60 kV, 40 mA
Collimator Size	0.5 mm
Detector Aperture	270.0 mm x 256.0 mm
Data Images	145 exposures at 2.0 minutes per degree
Oscillation Range (ϕ=0.0°,χ=45.0°)	ω 140.0 - 200.0° with 2.0° step
Oscillation Range (ϕ=180.0°,χ=45.0°)	ω 0.0 - 50.0° with 2.0° step
Oscillation Range (ϕ=90.0°,χ=0.0°)	ω 0.0 - 180.0° with 2.0° step
Camera Radius	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	55.0°
No. of Reflections Measured	Total: 43252 Unique: 5363 (R_{int} = 0.070)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.8774 - 0.9939) Secondary Extinction (coefficient: 6.99710e-07)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR97)
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(\|Fo\| - \|Fc\|)^2$
Least Squares Weights	$w = \frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$
p-factor	0.0130
No. Observations (I>2.00σ(I))	3853
No. Variables	470
Reflection/Parameter Ratio	8.20
Residuals: R; Rw	0.045 ; 0.037

Goodness of Fit Indicator	1.22
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.30 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.24 $e^-/Å^3$

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

atom	x	y	z	B_{eq}
O(1)	-0.2156(2)	-0.18835(6)	0.0192(2)	1.59(4)
O(2)	-0.3912(2)	-0.21440(7)	-0.0101(2)	2.40(5)
O(3)	0.0026(2)	-0.03958(7)	0.0289(2)	1.75(4)
O(4)	-0.2347(2)	-0.01583(7)	0.4782(2)	1.98(5)
O(5)	-0.1714(2)	0.00049(7)	0.2004(2)	1.55(4)
O(6)	-0.2081(2)	-0.07480(7)	0.0906(2)	1.71(4)
O(7)	-0.3415(2)	-0.07181(6)	0.3144(2)	1.87(4)
O(8)	-0.1272(2)	-0.26938(7)	0.0583(2)	2.68(5)
C(1)	-0.3102(3)	-0.19470(9)	-0.0454(3)	1.55(6)
C(2)	-0.3042(2)	-0.17448(10)	-0.1653(3)	1.65(6)
C(3)	-0.2309(3)	-0.1437(1)	-0.1856(3)	1.78(6)
C(4)	-0.2156(3)	-0.1185(1)	-0.2972(3)	2.12(7)
C(5)	-0.0903(3)	-0.11657(10)	-0.3368(3)	2.00(7)
C(6)	-0.0111(3)	-0.09587(10)	-0.2501(3)	1.69(6)
C(7)	-0.0369(2)	-0.06536(10)	-0.1742(3)	1.54(6)
C(8)	0.0445(2)	-0.0459(1)	-0.0930(2)	1.51(6)
C(9)	0.0260(2)	-0.0033(1)	-0.0452(2)	1.52(6)
C(10)	0.1196(3)	0.0246(1)	-0.0026(2)	1.73(6)
C(11)	0.0889(2)	0.05007(10)	0.1083(2)	1.42(6)
C(12)	0.1940(2)	0.07204(10)	0.1592(3)	1.59(6)
C(13)	0.1745(2)	0.09048(10)	0.2823(2)	1.45(6)
C(14)	0.1457(2)	0.05896(10)	0.3732(2)	1.37(6)
C(15)	0.0773(2)	0.06166(10)	0.4685(2)	1.45(6)
C(16)	0.0647(2)	0.02419(10)	0.5497(2)	1.42(6)

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

atom	x	y	z	B_{eq}
C(17)	-0.0584(2)	0.00901(10)	0.5671(2)	1.35(6)
C(18)	-0.1248(2)	0.00270(10)	0.4528(2)	1.40(6)
C(19)	-0.0694(2)	-0.02695(9)	0.3647(2)	1.31(6)
C(20)	-0.1381(2)	-0.03083(9)	0.2517(2)	1.16(6)
C(21)	-0.1621(2)	-0.07493(10)	0.2069(2)	1.35(6)
C(22)	-0.2460(2)	-0.09771(9)	0.2913(2)	1.45(6)
C(23)	-0.2872(2)	-0.13950(9)	0.2393(3)	1.56(6)
C(24)	-0.1878(3)	-0.1699(1)	0.2243(3)	1.64(6)
C(25)	-0.2086(2)	-0.20630(10)	0.1386(3)	1.57(6)
C(26)	-0.1091(3)	-0.2360(1)	0.1399(3)	2.18(7)
C(27)	-0.3908(3)	-0.1909(1)	-0.2525(3)	2.50(7)
C(28)	-0.0056(3)	0.0814(1)	0.0860(3)	2.62(8)
C(29)	0.1858(3)	0.1312(1)	0.3031(3)	2.25(7)
C(30)	0.0118(3)	0.1007(1)	0.5025(3)	2.33(7)
C(31)	0.1169(3)	0.0335(1)	0.6726(3)	2.18(7)
C(32)	-0.3801(3)	-0.1587(1)	0.3190(3)	3.22(8)
C(33)	0.1265(3)	-0.1351(1)	0.1050(4)	4.3(1)
C(34)	0.1478(3)	-0.1119(1)	0.2094(5)	5.2(1)
C(35)	0.2213(4)	-0.1263(1)	0.2938(4)	5.1(1)
C(36)	0.2767(4)	-0.1643(1)	0.2758(4)	4.6(1)
C(37)	0.2558(4)	-0.1867(1)	0.1772(4)	4.8(1)
C(38)	0.1798(4)	-0.1724(1)	0.0918(3)	4.2(1)
C(39)	0.0601(5)	-0.2179(1)	0.5033(4)	5.8(1)
C(40)	0.1331(4)	-0.2094(1)	0.5938(5)	4.9(1)

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

atom	x	y	z	B_{eq}
C(41)	0.0989(4)	-0.2139(1)	0.7104(4)	4.0(1)
C(42)	-0.0090(4)	-0.2267(1)	0.7350(3)	3.71(10)
C(43)	-0.0845(4)	-0.2348(1)	0.6429(4)	3.76(9)
C(44)	-0.0487(4)	-0.2306(1)	0.5264(4)	4.7(1)
H(3)	-0.1819	-0.1368	-0.1213	2.1
H(4O)	-0.2537	-0.0066	0.5250	2.1
H(4a)	-0.2592	-0.1309	-0.3589	2.5
H(4b)	-0.2418	-0.0906	-0.2832	2.5
H(5a)	-0.0646	-0.1446	-0.3493	2.4
H(5b)	-0.0867	-0.1015	-0.4096	2.4
H(6)	0.0653	-0.1056	-0.2494	2.0
H(6O)	-0.1755	-0.0651	0.0584	1.8
H(7)	-0.1131	-0.0555	-0.1727	1.8
H(7O)	-0.3256	-0.0561	0.3539	2.0
H(8)	0.1213	-0.0547	-0.1006	1.8
H(8O)	-0.0699	-0.2795	0.0401	2.9
H(9)	-0.0392	0.0109	-0.0750	1.8
H(10a)	0.1837	0.0076	0.0152	2.0
H(10b)	0.1384	0.0437	-0.0648	2.0
H(11)	0.0626	0.0308	0.1668	1.6
H(12a)	0.2540	0.0522	0.1640	1.9
H(12b)	0.2151	0.0942	0.1068	1.9
H(14)	0.1813	0.0324	0.3628	1.6
H(16)	0.1065	0.0017	0.5156	1.7

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

atom	x	y	z	B_{eq}
H(17a)	-0.0563	-0.0171	0.6084	1.6
H(17b)	-0.0973	0.0292	0.6141	1.6
H(18)	-0.1358	0.0292	0.4157	1.6
H(19a)	0.0041	-0.0166	0.3455	1.5
H(19b)	-0.0626	-0.0540	0.4003	1.5
H(21)	-0.0927	-0.0902	0.2057	1.6
H(22)	-0.2083	-0.1032	0.3645	1.7
H(23)	-0.3191	-0.1342	0.1630	1.8
H(24a)	-0.1246	-0.1543	0.1957	1.9
H(24b)	-0.1704	-0.1813	0.3001	1.9
H(25)	-0.2769	-0.2206	0.1582	1.8
H(26a)	-0.1002	-0.2473	0.2177	2.6
H(26b)	-0.0421	-0.2212	0.1181	2.6
H(27a)	-0.3820	-0.1768	-0.3266	2.9
H(27b)	-0.4651	-0.1859	-0.2224	2.9
H(27c)	-0.3800	-0.2202	-0.2634	2.9
H(28a)	0.0191	0.1017	0.0298	3.1
H(28b)	-0.0249	0.0950	0.1586	3.1
H(28c)	-0.0703	0.0671	0.0558	3.1
H(29a)	0.1753	0.1418	0.3814	2.6
H(29b)	0.2045	0.1497	0.2399	2.6
H(30a)	-0.0638	0.0932	0.5230	2.7
H(30b)	0.0474	0.1138	0.5687	2.7
H(30c)	0.0106	0.1196	0.4371	2.7

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

atom	x	y	z	B_{eq}
H(31a)	0.0773	0.0562	0.7087	2.6
H(31b)	0.1111	0.0092	0.7214	2.6
H(31c)	0.1947	0.0408	0.6632	2.6
H(32a)	-0.3965	-0.1865	0.2929	3.8
H(32b)	-0.4469	-0.1420	0.3143	3.8
H(32c)	-0.3541	-0.1595	0.3991	3.8
H(33)	0.0761	-0.1248	0.0456	5.1
H(34)	0.1104	-0.0857	0.2212	6.2
H(35)	0.2344	-0.1105	0.3642	6.0
H(36)	0.3293	-0.1743	0.3332	5.4
H(37)	0.2935	-0.2128	0.1657	5.7
H(38)	0.1651	-0.1891	0.0233	5.0
H(39)	0.0849	-0.2149	0.4233	6.9
H(40)	0.2083	-0.2004	0.5766	5.9
H(41)	0.1504	-0.2079	0.7734	4.7
H(42)	-0.0326	-0.2301	0.8153	4.4
H(43)	-0.1602	-0.2433	0.6598	4.5
H(44)	-0.0992	-0.2365	0.4627	5.6

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

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.018(1)	0.022(1)	0.020(1)	-0.001(1)	-0.0012(9)	0.002(1)
O(2)	0.024(1)	0.039(1)	0.028(1)	-0.011(1)	-0.002(1)	0.006(1)
O(3)	0.024(1)	0.029(1)	0.0144(10)	-0.005(1)	0.0051(9)	0.0008(10)
O(4)	0.019(1)	0.038(1)	0.018(1)	-0.006(1)	0.0050(9)	-0.009(1)
O(5)	0.023(1)	0.021(1)	0.0149(10)	0.000(1)	-0.0036(9)	0.0004(10)
O(6)	0.024(1)	0.028(1)	0.0127(10)	-0.008(1)	0.0024(9)	-0.0004(10)
O(7)	0.020(1)	0.023(1)	0.028(1)	0.002(1)	0.008(1)	-0.009(1)
O(8)	0.029(1)	0.031(1)	0.041(1)	0.008(1)	0.001(1)	-0.010(1)
C(1)	0.019(1)	0.017(2)	0.023(2)	0.000(1)	0.000(1)	-0.003(1)
C(2)	0.019(1)	0.021(2)	0.022(1)	-0.001(2)	0.001(1)	-0.002(1)
C(3)	0.022(2)	0.026(2)	0.020(2)	-0.001(2)	-0.005(1)	0.000(1)
C(4)	0.030(2)	0.025(2)	0.025(2)	-0.004(2)	-0.007(2)	-0.001(2)
C(5)	0.039(2)	0.017(2)	0.020(2)	-0.004(2)	0.001(2)	0.001(1)
C(6)	0.020(1)	0.024(2)	0.021(1)	-0.002(2)	0.003(1)	0.002(2)
C(7)	0.017(1)	0.021(2)	0.021(1)	0.000(1)	0.002(1)	0.006(1)
C(8)	0.014(1)	0.028(2)	0.015(1)	-0.004(2)	0.006(1)	0.003(1)
C(9)	0.017(1)	0.028(2)	0.012(1)	0.000(2)	-0.001(1)	0.003(1)
C(10)	0.020(2)	0.030(2)	0.015(1)	-0.004(2)	0.001(1)	0.003(1)
C(11)	0.016(1)	0.023(2)	0.015(1)	-0.001(2)	-0.002(1)	0.000(1)
C(12)	0.016(1)	0.025(2)	0.020(1)	-0.006(2)	-0.001(1)	0.003(1)
C(13)	0.011(1)	0.025(2)	0.019(1)	-0.003(1)	-0.001(1)	-0.003(1)
C(14)	0.012(1)	0.023(2)	0.017(1)	0.000(1)	-0.004(1)	-0.001(1)
C(15)	0.015(1)	0.023(2)	0.017(1)	-0.001(1)	-0.006(1)	-0.004(1)
C(16)	0.017(1)	0.025(2)	0.012(1)	0.002(2)	-0.001(1)	-0.002(1)

Table 2. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(17)	0.016(1)	0.025(2)	0.010(1)	-0.003(1)	0.001(1)	-0.002(1)
C(18)	0.015(1)	0.022(2)	0.016(1)	-0.002(1)	0.002(1)	0.002(1)
C(19)	0.014(1)	0.022(2)	0.014(1)	-0.001(1)	0.002(1)	0.000(1)
C(20)	0.008(1)	0.021(2)	0.016(1)	-0.001(1)	0.006(1)	-0.002(1)
C(21)	0.014(1)	0.021(2)	0.016(1)	0.001(1)	0.001(1)	-0.003(1)
C(22)	0.019(1)	0.021(2)	0.015(1)	0.002(1)	0.003(1)	0.000(1)
C(23)	0.020(1)	0.015(2)	0.024(2)	-0.002(1)	0.004(1)	-0.001(1)
C(24)	0.021(1)	0.021(2)	0.021(1)	-0.001(1)	-0.002(1)	0.005(1)
C(25)	0.022(2)	0.020(2)	0.018(1)	0.000(2)	0.002(1)	0.007(1)
C(26)	0.036(2)	0.018(2)	0.028(2)	0.005(2)	-0.003(2)	-0.003(2)
C(27)	0.035(2)	0.037(2)	0.023(2)	-0.015(2)	-0.002(2)	0.000(2)
C(28)	0.031(2)	0.036(2)	0.032(2)	0.004(2)	-0.009(2)	-0.010(2)
C(29)	0.031(2)	0.031(2)	0.023(2)	-0.007(2)	0.004(2)	0.002(2)
C(30)	0.030(2)	0.030(2)	0.028(2)	-0.002(2)	0.008(2)	-0.001(2)
C(31)	0.028(2)	0.040(2)	0.015(1)	-0.004(2)	-0.006(1)	0.000(2)
C(32)	0.043(2)	0.028(2)	0.051(2)	-0.014(2)	0.023(2)	-0.007(2)
C(33)	0.034(2)	0.042(3)	0.089(3)	0.005(2)	0.016(3)	0.020(3)
C(34)	0.026(2)	0.035(3)	0.138(5)	0.003(2)	0.012(3)	0.001(3)
C(35)	0.053(3)	0.053(3)	0.087(4)	-0.008(3)	0.010(3)	-0.012(3)
C(36)	0.063(3)	0.047(3)	0.062(3)	0.000(3)	0.003(3)	0.015(3)
C(37)	0.069(3)	0.048(3)	0.065(3)	0.020(3)	0.025(3)	0.015(3)
C(38)	0.057(3)	0.056(3)	0.048(2)	0.011(3)	0.014(2)	0.014(2)
C(39)	0.103(4)	0.061(3)	0.057(3)	-0.020(3)	0.030(3)	-0.021(3)
C(40)	0.050(3)	0.051(3)	0.087(4)	-0.009(3)	0.014(3)	-0.017(3)

Table 2. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(41)	0.056(3)	0.024(2)	0.070(3)	-0.001(2)	-0.015(3)	0.005(2)
C(42)	0.068(3)	0.027(2)	0.046(2)	-0.003(2)	-0.003(2)	0.012(2)
C(43)	0.054(2)	0.029(2)	0.060(3)	-0.003(2)	-0.001(2)	0.009(2)
C(44)	0.083(3)	0.042(3)	0.053(3)	-0.010(3)	-0.009(3)	-0.011(2)

The general temperature factor expression:

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

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
O(1)	C(1)	1.345(3)	O(1)	C(25)	1.459(3)
O(2)	C(1)	1.207(3)	O(3)	C(8)	1.469(3)
O(3)	C(9)	1.449(3)	O(4)	C(18)	1.449(3)
O(5)	C(20)	1.215(3)	O(6)	C(21)	1.414(3)
O(7)	C(22)	1.417(3)	O(8)	C(26)	1.418(4)
C(1)	C(2)	1.494(4)	C(2)	C(3)	1.324(4)
C(2)	C(27)	1.507(4)	C(3)	C(4)	1.501(4)
C(4)	C(5)	1.541(4)	C(5)	C(6)	1.500(4)
C(6)	C(7)	1.327(4)	C(7)	C(8)	1.461(4)
C(8)	C(9)	1.474(4)	C(9)	C(10)	1.492(4)
C(10)	C(11)	1.530(4)	C(11)	C(12)	1.531(4)
C(11)	C(28)	1.514(4)	C(12)	C(13)	1.519(4)
C(13)	C(14)	1.470(4)	C(13)	C(29)	1.322(4)
C(14)	C(15)	1.342(4)	C(15)	C(16)	1.508(4)
C(15)	C(30)	1.510(4)	C(16)	C(17)	1.539(4)
C(16)	C(31)	1.539(4)	C(17)	C(18)	1.517(4)
C(18)	C(19)	1.514(4)	C(19)	C(20)	1.511(4)
C(20)	C(21)	1.516(4)	C(21)	C(22)	1.549(4)
C(22)	C(23)	1.531(4)	C(23)	C(24)	1.526(4)
C(23)	C(32)	1.539(4)	C(24)	C(25)	1.526(4)
C(25)	C(26)	1.505(4)	C(33)	C(34)	1.409(6)
C(33)	C(38)	1.350(5)	C(34)	C(35)	1.362(6)
C(35)	C(36)	1.387(6)	C(36)	C(37)	1.341(6)
C(37)	C(38)	1.387(6)	C(39)	C(40)	1.357(6)

Table 3. Bond Lengths($\AA$) (continued)

atom	atom	distance	atom	atom	distance
C(39)	C(44)	1.368(6)	C(40)	C(41)	1.377(6)
C(41)	C(42)	1.362(6)	C(42)	C(43)	1.388(6)
C(43)	C(44)	1.381(6)			

Table 4. Bond Lengths($\AA$)

atom	atom	distance	atom	atom	distance
O(4)	H(4O)	0.64	O(6)	H(6O)	0.61
O(7)	H(7O)	0.69	O(8)	H(8O)	0.77
C(3)	H(3)	0.95	C(4)	H(4a)	0.95
C(4)	H(4b)	0.95	C(5)	H(5a)	0.95
C(5)	H(5b)	0.95	C(6)	H(6)	0.95
C(7)	H(7)	0.95	C(8)	H(8)	0.95
C(9)	H(9)	0.95	C(10)	H(10a)	0.95
C(10)	H(10b)	0.95	C(11)	H(11)	0.95
C(12)	H(12a)	0.95	C(12)	H(12b)	0.95
C(14)	H(14)	0.95	C(16)	H(16)	0.95
C(17)	H(17a)	0.95	C(17)	H(17b)	0.95
C(18)	H(18)	0.95	C(19)	H(19a)	0.95
C(19)	H(19b)	0.95	C(21)	H(21)	0.95
C(22)	H(22)	0.95	C(23)	H(23)	0.95
C(24)	H(24a)	0.95	C(24)	H(24b)	0.95
C(25)	H(25)	0.95	C(26)	H(26a)	0.95
C(26)	H(26b)	0.95	C(27)	H(27a)	0.95
C(27)	H(27b)	0.95	C(27)	H(27c)	0.95
C(28)	H(28a)	0.95	C(28)	H(28b)	0.95
C(28)	H(28c)	0.95	C(29)	H(29a)	0.95
C(29)	H(29b)	0.95	C(30)	H(30a)	0.95
C(30)	H(30b)	0.95	C(30)	H(30c)	0.95
C(31)	H(31a)	0.95	C(31)	H(31b)	0.95
C(31)	H(31c)	0.95	C(32)	H(32a)	0.95

Table 4. Bond Lengths($\AA$) (continued)

atom	atom	distance	atom	atom	distance
C(32)	H(32b)	0.95	C(32)	H(32c)	0.95
C(33)	H(33)	0.95	C(34)	H(34)	0.95
C(35)	H(35)	0.95	C(36)	H(36)	0.95
C(37)	H(37)	0.95	C(38)	H(38)	0.95
C(39)	H(39)	0.95	C(40)	H(40)	0.95
C(41)	H(41)	0.95	C(42)	H(42)	0.95
C(43)	H(43)	0.95	C(44)	H(44)	0.95

Table 5. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(1)	O(1)	C(25)	118.9(2)	C(8)	O(3)	C(9)	60.7(2)
O(1)	C(1)	O(2)	123.7(3)	O(1)	C(1)	C(2)	112.5(2)
O(2)	C(1)	C(2)	123.8(3)	C(1)	C(2)	C(3)	120.3(3)
C(1)	C(2)	C(27)	113.9(3)	C(3)	C(2)	C(27)	125.8(3)
C(2)	C(3)	C(4)	128.2(3)	C(3)	C(4)	C(5)	112.2(3)
C(4)	C(5)	C(6)	115.1(3)	C(5)	C(6)	C(7)	126.6(3)
C(6)	C(7)	C(8)	124.1(3)	O(3)	C(8)	C(7)	114.8(2)
O(3)	C(8)	C(9)	59.0(2)	C(7)	C(8)	C(9)	121.3(3)
O(3)	C(9)	C(8)	60.3(2)	O(3)	C(9)	C(10)	115.4(2)
C(8)	C(9)	C(10)	123.6(3)	C(9)	C(10)	C(11)	113.7(2)
C(10)	C(11)	C(12)	110.8(2)	C(10)	C(11)	C(28)	112.8(2)
C(12)	C(11)	C(28)	110.8(3)	C(11)	C(12)	C(13)	113.2(2)
C(12)	C(13)	C(14)	113.8(3)	C(12)	C(13)	C(29)	121.6(3)
C(14)	C(13)	C(29)	124.6(3)	C(13)	C(14)	C(15)	130.4(3)
C(14)	C(15)	C(16)	119.4(3)	C(14)	C(15)	C(30)	124.1(3)
C(16)	C(15)	C(30)	116.5(2)	C(15)	C(16)	C(17)	114.7(2)
C(15)	C(16)	C(31)	110.6(3)	C(17)	C(16)	C(31)	108.8(2)
C(16)	C(17)	C(18)	114.8(2)	O(4)	C(18)	C(17)	110.3(2)
O(4)	C(18)	C(19)	105.0(2)	C(17)	C(18)	C(19)	114.4(2)
C(18)	C(19)	C(20)	111.7(2)	O(5)	C(20)	C(19)	120.3(3)
O(5)	C(20)	C(21)	122.7(2)	C(19)	C(20)	C(21)	117.0(2)
O(6)	C(21)	C(20)	112.0(2)	O(6)	C(21)	C(22)	108.8(2)
C(20)	C(21)	C(22)	110.4(2)	O(7)	C(22)	C(21)	110.2(2)
O(7)	C(22)	C(23)	108.8(2)	C(21)	C(22)	C(23)	112.0(2)

Table 5. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(22)	C(23)	C(24)	110.4(2)	C(22)	C(23)	C(32)	110.3(2)
C(24)	C(23)	C(32)	111.0(3)	C(23)	C(24)	C(25)	115.3(2)
O(1)	C(25)	C(24)	106.9(2)	O(1)	C(25)	C(26)	107.3(2)
C(24)	C(25)	C(26)	110.2(2)	O(8)	C(26)	C(25)	110.3(3)
C(34)	C(33)	C(38)	118.0(4)	C(33)	C(34)	C(35)	121.0(4)
C(34)	C(35)	C(36)	119.3(4)	C(35)	C(36)	C(37)	119.9(4)
C(36)	C(37)	C(38)	121.0(4)	C(33)	C(38)	C(37)	120.8(4)
C(40)	C(39)	C(44)	120.5(4)	C(39)	C(40)	C(41)	120.5(4)
C(40)	C(41)	C(42)	119.7(4)	C(41)	C(42)	C(43)	120.1(4)
C(42)	C(43)	C(44)	119.5(4)	C(39)	C(44)	C(43)	119.6(4)

Table 6. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(18)	O(4)	H(4O)	106.6	C(21)	O(6)	H(6O)	108.0
C(22)	O(7)	H(7O)	108.7	C(26)	O(8)	H(8O)	110.5
C(2)	C(3)	H(3)	115.9	C(4)	C(3)	H(3)	115.9
C(3)	C(4)	H(4a)	108.8	C(3)	C(4)	H(4b)	108.8
C(5)	C(4)	H(4a)	108.8	C(5)	C(4)	H(4b)	108.8
H(4a)	C(4)	H(4b)	109.5	C(4)	C(5)	H(5a)	108.1
C(4)	C(5)	H(5b)	108.1	C(6)	C(5)	H(5a)	108.0
C(6)	C(5)	H(5b)	108.0	H(5a)	C(5)	H(5b)	109.5
C(5)	C(6)	H(6)	116.7	C(7)	C(6)	H(6)	116.7
C(6)	C(7)	H(7)	117.9	C(8)	C(7)	H(7)	118.0
O(3)	C(8)	H(8)	116.4	C(7)	C(8)	H(8)	116.3
C(9)	C(8)	H(8)	116.4	O(3)	C(9)	H(9)	115.2
C(8)	C(9)	H(9)	115.3	C(10)	C(9)	H(9)	115.3
C(9)	C(10)	H(10a)	108.4	C(9)	C(10)	H(10b)	108.4
C(11)	C(10)	H(10a)	108.5	C(11)	C(10)	H(10b)	108.4
H(10a)	C(10)	H(10b)	109.4	C(10)	C(11)	H(11)	107.4
C(12)	C(11)	H(11)	107.4	C(28)	C(11)	H(11)	107.4
C(11)	C(12)	H(12a)	108.6	C(11)	C(12)	H(12b)	108.5
C(13)	C(12)	H(12a)	108.5	C(13)	C(12)	H(12b)	108.5
H(12a)	C(12)	H(12b)	109.5	C(13)	C(14)	H(14)	114.8
C(15)	C(14)	H(14)	114.8	C(15)	C(16)	H(16)	107.5
C(17)	C(16)	H(16)	107.6	C(31)	C(16)	H(16)	107.5
C(16)	C(17)	H(17a)	108.2	C(16)	C(17)	H(17b)	108.2
C(18)	C(17)	H(17a)	108.1	C(18)	C(17)	H(17b)	108.1

Table 6. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
H(17a)	C(17)	H(17b)	109.4	O(4)	C(18)	H(18)	109.0
C(17)	C(18)	H(18)	108.9	C(19)	C(18)	H(18)	109.0
C(18)	C(19)	H(19a)	109.0	C(18)	C(19)	H(19b)	108.9
C(20)	C(19)	H(19a)	108.9	C(20)	C(19)	H(19b)	108.9
H(19a)	C(19)	H(19b)	109.4	O(6)	C(21)	H(21)	108.5
C(20)	C(21)	H(21)	108.5	C(22)	C(21)	H(21)	108.5
O(7)	C(22)	H(22)	108.6	C(21)	C(22)	H(22)	108.6
C(23)	C(22)	H(22)	108.6	C(22)	C(23)	H(23)	108.4
C(24)	C(23)	H(23)	108.4	C(32)	C(23)	H(23)	108.3
C(23)	C(24)	H(24a)	107.9	C(23)	C(24)	H(24b)	108.0
C(25)	C(24)	H(24a)	108.0	C(25)	C(24)	H(24b)	108.0
H(24a)	C(24)	H(24b)	109.5	O(1)	C(25)	H(25)	110.7
C(24)	C(25)	H(25)	110.7	C(26)	C(25)	H(25)	110.8
O(8)	C(26)	H(26a)	109.2	O(8)	C(26)	H(26b)	109.2
C(25)	C(26)	H(26a)	109.3	C(25)	C(26)	H(26b)	109.3
H(26a)	C(26)	H(26b)	109.5	C(2)	C(27)	H(27a)	109.5
C(2)	C(27)	H(27b)	109.5	C(2)	C(27)	H(27c)	109.5
H(27a)	C(27)	H(27b)	109.5	H(27a)	C(27)	H(27c)	109.5
H(27b)	C(27)	H(27c)	109.5	C(11)	C(28)	H(28a)	109.4
C(11)	C(28)	H(28b)	109.4	C(11)	C(28)	H(28c)	109.4
H(28a)	C(28)	H(28b)	109.5	H(28a)	C(28)	H(28c)	109.5
H(28b)	C(28)	H(28c)	109.5	C(13)	C(29)	H(29a)	120.0
C(13)	C(29)	H(29b)	120.0	H(29a)	C(29)	H(29b)	120.0
C(15)	C(30)	H(30a)	109.4	C(15)	C(30)	H(30b)	109.5

Table 6. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(15)	C(30)	H(30c)	109.4	H(30a)	C(30)	H(30b)	109.5
H(30a)	C(30)	H(30c)	109.5	H(30b)	C(30)	H(30c)	109.5
C(16)	C(31)	H(31a)	109.4	C(16)	C(31)	H(31b)	109.4
C(16)	C(31)	H(31c)	109.4	H(31a)	C(31)	H(31b)	109.5
H(31a)	C(31)	H(31c)	109.5	H(31b)	C(31)	H(31c)	109.5
C(23)	C(32)	H(32a)	109.5	C(23)	C(32)	H(32b)	109.5
C(23)	C(32)	H(32c)	109.4	H(32a)	C(32)	H(32b)	109.5
H(32a)	C(32)	H(32c)	109.5	H(32b)	C(32)	H(32c)	109.5
C(34)	C(33)	H(33)	121.0	C(38)	C(33)	H(33)	121.0
C(33)	C(34)	H(34)	119.5	C(35)	C(34)	H(34)	119.5
C(34)	C(35)	H(35)	120.3	C(36)	C(35)	H(35)	120.4
C(35)	C(36)	H(36)	120.0	C(37)	C(36)	H(36)	120.1
C(36)	C(37)	H(37)	119.5	C(38)	C(37)	H(37)	119.5
C(33)	C(38)	H(38)	119.6	C(37)	C(38)	H(38)	119.7
C(40)	C(39)	H(39)	119.7	C(44)	C(39)	H(39)	119.8
C(39)	C(40)	H(40)	119.7	C(41)	C(40)	H(40)	119.7
C(40)	C(41)	H(41)	120.1	C(42)	C(41)	H(41)	120.2
C(41)	C(42)	H(42)	119.9	C(43)	C(42)	H(42)	120.0
C(42)	C(43)	H(43)	120.3	C(44)	C(43)	H(43)	120.2
C(39)	C(44)	H(44)	120.1	C(43)	C(44)	H(44)	120.3

Table 7. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
O(1)	C(1)	C(2)	C(3)	-19.8(4)	O(1)	C(1)	C(2)	C(27)	163.1(3)
O(1)	C(25)	C(24)	C(23)	68.8(3)	O(1)	C(25)	C(26)	O(8)	-62.6(3)
O(2)	C(1)	O(1)	C(25)	0.4(4)	O(2)	C(1)	C(2)	C(3)	159.9(3)
O(2)	C(1)	C(2)	C(27)	-17.2(4)	O(3)	C(8)	C(7)	C(6)	-136.0(3)
O(3)	C(8)	C(9)	C(10)	102.4(3)	O(3)	C(9)	C(8)	C(7)	102.0(3)
O(3)	C(9)	C(10)	C(11)	-71.4(3)	O(4)	C(18)	C(17)	C(16)	173.8(2)
O(4)	C(18)	C(19)	C(20)	60.5(3)	O(5)	C(20)	C(19)	C(18)	49.1(3)
O(5)	C(20)	C(21)	O(6)	10.7(4)	O(5)	C(20)	C(21)	C(22)	-110.8(3)
O(6)	C(21)	C(20)	C(19)	-169.1(2)	O(6)	C(21)	C(22)	O(7)	-73.8(3)
O(6)	C(21)	C(22)	C(23)	47.5(3)	O(7)	C(22)	C(21)	C(20)	49.5(3)
O(7)	C(22)	C(23)	C(24)	-174.6(2)	O(7)	C(22)	C(23)	C(32)	-51.7(3)
O(8)	C(26)	C(25)	C(24)	-178.7(2)	C(1)	O(1)	C(25)	C(24)	-122.2(3)
C(1)	O(1)	C(25)	C(26)	119.5(3)	C(1)	C(2)	C(3)	C(4)	-177.1(3)
C(2)	C(1)	O(1)	C(25)	-179.9(2)	C(2)	C(3)	C(4)	C(5)	-130.1(3)
C(3)	C(4)	C(5)	C(6)	-62.1(4)	C(4)	C(3)	C(2)	C(27)	-0.4(5)
C(4)	C(5)	C(6)	C(7)	-30.2(5)	C(5)	C(6)	C(7)	C(8)	-179.0(3)
C(6)	C(7)	C(8)	C(9)	156.5(3)	C(7)	C(8)	O(3)	C(9)	-113.0(3)
C(7)	C(8)	C(9)	C(10)	-155.6(3)	C(8)	O(3)	C(9)	C(10)	-115.8(3)
C(8)	C(9)	C(10)	C(11)	-141.4(3)	C(9)	C(10)	C(11)	C(12)	170.4(3)
C(9)	C(10)	C(11)	C(28)	-64.8(3)	C(10)	C(11)	C(12)	C(13)	-168.4(2)
C(11)	C(12)	C(13)	C(14)	61.8(3)	C(11)	C(12)	C(13)	C(29)	-119.7(3)
C(12)	C(13)	C(14)	C(15)	-146.3(3)	C(13)	C(12)	C(11)	C(28)	65.7(3)
C(13)	C(14)	C(15)	C(16)	-178.8(3)	C(13)	C(14)	C(15)	C(30)	0.1(5)
C(14)	C(15)	C(16)	C(17)	-123.8(3)	C(14)	C(15)	C(16)	C(31)	112.7(3)

Table 7. Torsion Angles(°) (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(15)	C(14)	C(13)	C(29)	35.4(5)	C(15)	C(16)	C(17)	C(18)	49.9(4)
C(16)	C(17)	C(18)	C(19)	55.7(4)	C(17)	C(16)	C(15)	C(30)	57.2(3)
C(17)	C(18)	C(19)	C(20)	-178.4(2)	C(18)	C(17)	C(16)	C(31)	174.2(3)
C(18)	C(19)	C(20)	C(21)	-131.2(3)	C(19)	C(20)	C(21)	C(22)	69.5(3)
C(20)	C(21)	C(22)	C(23)	170.8(2)	C(21)	C(22)	C(23)	C(24)	63.3(3)
C(21)	C(22)	C(23)	C(32)	-173.7(3)	C(22)	C(23)	C(24)	C(25)	-161.2(2)
C(23)	C(24)	C(25)	C(26)	-174.8(2)	C(25)	C(24)	C(23)	C(32)	76.2(3)
C(30)	C(15)	C(16)	C(31)	-66.2(3)	C(33)	C(34)	C(35)	C(36)	0.6(7)
C(33)	C(38)	C(37)	C(36)	1.2(7)	C(34)	C(33)	C(38)	C(37)	-1.8(6)
C(34)	C(35)	C(36)	C(37)	-1.3(7)	C(35)	C(34)	C(33)	C(38)	0.9(6)
C(35)	C(36)	C(37)	C(38)	0.4(7)	C(39)	C(40)	C(41)	C(42)	-0.1(7)
C(39)	C(44)	C(43)	C(42)	-1.0(7)	C(40)	C(39)	C(44)	C(43)	0.2(8)
C(40)	C(41)	C(42)	C(43)	-0.7(7)	C(41)	C(40)	C(39)	C(44)	0.4(8)
C(41)	C(42)	C(43)	C(44)	1.2(6)					

Table 8. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	ADC	atom	atom	distance	ADC
O(2)	O(8)	2.876(3)	44503	O(2)	C(26)	3.344(4)	44503
O(3)	C(33)	3.476(5)	1	O(3)	C(34)	3.509(5)	1
O(4)	O(5)	2.773(3)	45502	O(4)	O(6)	3.218(3)	45502
O(4)	C(9)	3.491(3)	45502	O(5)	C(17)	3.527(3)	45402
O(6)	C(18)	3.394(4)	45402	O(6)	C(17)	3.464(4)	45402
O(7)	C(28)	3.555(4)	45502	O(7)	C(9)	3.592(3)	45502
O(8)	C(37)	3.292(5)	44503	O(8)	C(38)	3.379(5)	44503
O(8)	C(29)	3.591(4)	54504	C(9)	C(31)	3.545(4)	55401

The ADC (atom designator code) specifies the position of an atom in a crystal. The 5-digit number shown in the table is a composite of three one-digit numbers and one two-digit number: TA (first digit) + TB (second digit) + TC (third digit) + SN (last two digits). TA, TB and TC are the crystal lattice translation digits along cell edges a, b and c. A translation digit of 5 indicates the origin unit cell. If TA = 4, this indicates a translation of one unit cell length along the a-axis in the negative direction. Each translation digit can range in value from 1 to 9 and thus $\pm$4 lattice translations from the origin (TA=5, TB=5, TC=5) can be represented.

The SN, or symmetry operator number, refers to the number of the symmetry operator used to generate the coordinates of the target atom. A list of symmetry operators relevant to this structure are given below.

For a given intermolecular contact, the first atom (origin atom) is located in the origin unit cell and its position can be generated using the identity operator (SN=1). Thus, the ADC for an origin atom is always 55501. The position of the second atom (target atom) can be generated using the ADC and the coordinates of the atom in the parameter table. For example, an ADC of 47502 refers to the target atom moved through symmetry operator two, then translated -1 cell translations along the a axis, +2 cell translations along the b axis, and 0 cell translations along the c axis.

An ADC of 1 indicates an intermolecular contact between two fragments (eg. cation and anion) that reside in the same asymmetric unit.

Symmetry Operators:

(1)	X,	Y,	Z	(2)	1/2-X,	-Y,	1/2+Z
(3)	1/2+X,	1/2-Y,	-Z	(4)	-X,	1/2+Y,	1/2-Z