Supporting Information to:

Ring-opening Polymerization of a 2,3-Disubstituted Oxirane Leading to a Polyether Having a Carbonyl-Aromatic π-Stacked Structure

Maia Merlani[a,b], Yasuhito Koyama[a], Hiroyasu Sato[c], Li Geng[a], Vakhtang Barbakadze[b], Bezhan Chankvetadze[d], Tamaki Nakano[*a]

[a]*Catalysis Research Center (CRC) and Graduate School of Chemical Sciences and Engineering, Hokkaido University, N 21, W 10, Kita-ku, Sapporo 001-0021, Japan. Email:tamaki.nakano@cat.hokudai.ac.jp; Fax: +81-11-7069156; Tel: +81-11-7069155*

[b]*Kutateladze Institute of Pharmacochemistry, Tbilisi State Medical University, 36 str. P. Sarajishvili, 0159 Tbilisi, Georgia*

[c]*Rigaku Corporation 4-14-4, Sendagaya, Shibuya-Ku, Tokyo 151-0051, Japan*

[d]*Department of Physical and Analytical Chemistry, School of Exact and Natural Sciences, Tbilisi State University, Chavchavadze Ave 1, 0179 Tbilisi, Georgia*

*To whom correspondence should be addressed
(E-mail: tamaki.nakano@cat.hokudai.ac.jp)

Contents

Experimental Section

Measurements

^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded on a JEOL JNM-ESC400 spectrometer using $CDCl_3$ as a solvent, calibrated using tetramethylsilane as the internal standard. SEC analyses were carried out using a chromatographic system consisting of a Hitachi L-7100 chromatographic pump, a Hitachi L-7420 UV detector (254 nm), and a Hitachi L-7490 RI detector equipped with TOSOH TSK gel G3000H HR and G6000H HR columns (30 x 0.72(i.d.) cm) connected in series (eluent THF, flow rate 1.0 mL/min). SEC-VISC-RALLS analyses were performed using Wyatt Technology DAWN EOS-N MALLS detector and Viscotek Model TDA300 on-line RI and viscometric detectors. Right-angle scattering information was obtained from the MALLS detector and integrated into the TDA detector system to calculate molar masses and Mark–Houwink–Sakurada coefficients. FT–IR spectra were measured using a ThermoFischer Scientific Nexus 870 spectrometer. Differential scanning calorimetry (DSC) analyses were performed on Rigaku Thermo plus TG8120 and DSC8230 apparatuses using Thermo plus 2 software for data analyses. Melting points were determines on Yanaco (model MP-S3) apparatus.

MD simulation

Molecular mechanics structure optimization was effected using the COMPASS[1] force field implemented in the Discover module of the Material Studio 2.0 (Accelrys) software package with the Fletcher-Reeves[2] conjugate gradient algorithm until the RMS residue went below 0.01 kcal/mol/Å. Molecular dynamic simulation was performed under a constant NVT condition in which the numbers of atoms, volume, and thermodynamic temperature were held constant. Berendsen's thermocouple[3] was used for coupling to a thermal bath. The step time was 1 fs and the decay constant was 0.1 ps. Conformations obtained through MD simulation were saved in trajectory files at every 5 or 10 ps and were optimized by MM simulation.

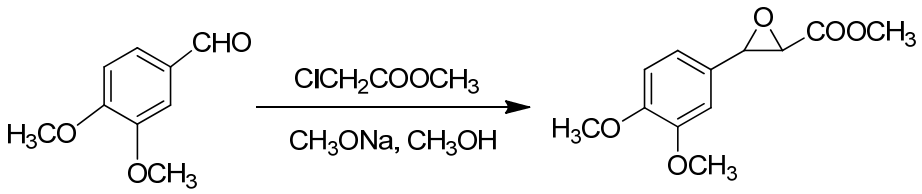
Materials

CH_2Cl_2 (Kanto Chemical) and toluene (Kanto Chemical) were distilled over CaH_2 and stored with MS 4A (Wako). 2-Ethoxycarbonyl-3-phenyloxirane (ethyl 3-phenylglycidate) (Sigma-Aldrich) and $BF_3{\cdot}OEt_2$ (Kanto Chemical) were distilled over CaH_2 under reduced pressure. Methanol (Wako) was dehydrated using Mg. Veratraldehyde (TCI), methyl chloroacetate (TCI), trifluoromethanesulfonic anhydride (TCI), acetic acid (Wako), diethyl ether (Wako), $CHCl_3$ (Wako), hexane (Wako), ethyl acetate (Wako), $NaHCO_3$ (Wako), $MgSO_4$

(Wako), Tf_2O (Wako), and CaH_2 (Wako) were used as obtained. For preparative silica-gel chromatography, Silica Gel 60N (spherical, neutral; 63~210 mm) (Kako Chemical) was used.

Synthesis of 2-methoxycarbonyl-3-(3,4-dimethoxyphenyl)oxirane (MCDMPO) (methyl 3-(3,4-dimethoxyphenyl)glycidate) (Scheme S1)

A solution of veratraldehyde (2.99 g, 18 mmol) and methyl chloroacetate (2.92 g, 27 mmol) was added to a suspension of Na (0.69 g, 27 mmol) in dehydrated methanol (9.0 mL) at −10 °C during a period of 30 min. The mixture was stirred at −5 °C for 2 h and then at room temperature for 3 h. The mixture was poured into 50 mL ice water containing acetic acid (0.2 mL). Crude products precipitated as a white powder, which was collected by filtration, washed with water, and dried in vacuo. The crude material was recrystallized from diethyl ether to yield MCDMPO (4.28 g, 18 mmol, 50%) as a white solid (m.p. 65–66 °C [lit.[4] 66–67 °C]); IR (KBr) υ 3007, 2960, 2846, 1726, 1616, 1599, 1524, 1443, 1309, 1273,1231, 1147, 1025, 871, 824, 776, 757 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$, TMS, 25 °C) δ 3.49 (d, J = 1.4 Hz, 1H), 3.82 (s, 3H), 3.87 (s, 3H), 3.88 (s, 3H), 4.06 (d, J = 1.4 Hz, 1H), 6.71 (d, J = 8.3 Hz, 1H), 6.82 (d, J = 2.0 Hz, 1H), 6.88 (dd, J = 8.3, 2.0 Hz) ppm; ^{13}C NMR (100 MHz, $CDCl_3$, TMS, 25 °C) δ 52.7, 55.9, 56.0, 56.7, 58.7, 108.2, 111.1, 119.0, 127.2, 149.3, 149. 8, 168.8 ppm.



Scheme S1. Synthesis of MCDMPO.

Polymerization of MCDMPO (run 7 in Table 1)

To a solution of MCDMPO (0.200 g, 0.84 mmol) in dry CH_2Cl_2 (0.8 mL) at 0 °C was added $BF_3{\cdot}OEt_2$ (5.0 μL, 20% solution in CH_2Cl_2) with stirring under nitrogen atmosphere. The reaction mixture was stirred at 0 °C for 5 h, quenched with methanol (0.2 mL), and concentrated in vacuo. The obtained product was dissolved in 2.0 mL of $CHCl_3$ and precipitated into a large excess of hexane (100 mL). The hexane-insoluble and -soluble parts were separated with a centrifuge. Hexane-insoluble part: 0.190 g, (95.1%); IR (KBr) υ 3010, 2950, 2845, 1748, 1607, 1597, 1518, 1466, 1424, 1266, 1202, 1140, 1027, 872, 815, 765 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$, TMS, 25 °C) δ 3.82 (brd, 11H), 6.73 (brd, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$, TMS,

25 °C) δ 51.9, 56.0, 80.8–82.0 (brd), 110.4, 120.8, 128.8, 148.9, 170.4 ppm; M_n 1350, M_w 3350, M_w/M_n 2.48 (estimated by SEC based on PS standards); M_n 8700, M_w 8950, M_w/M_n 1.02 (estimated by SEC-VISC-RALS); T_m 88.1 °C ;T_{d5} 263.4 °C; T_{d10} 285.3 °C.

Synthesis of diethyl 3,6-diphenyl-1,4-dioxane-2,5-dicarboxylate **I**

To a solution of 2-ethoxycarbonyl-3-phenyloxirane (ethyl 3-phenylglycidate) (1.92 g, 10 mmol) in dry toluene (5.0 mL), $BF_3 \cdot OEt_2$ (0.12 mL, 1.0 mmol) was added and reaction mixture was heated at 110 °C under nitrogen for 12 h, neutralized with sat. aq. $NaHCO_3$ solution and extracted with ethyl acetate. The organic layer was dried over $MgSO_4$, filtered, and concentrated in vacuo. The crude material was first purified by silica-gel column chromatography with a mixture of ethyl acetate and *n*-hexane (1/10, v/v) as eluent and then was recrystallized from Et_2O : hexane (1:2) to afford white crystals of compound **I** (m.p.154–155 °C). Yield: 0.49 g, 13%. IR (KBr) υ: 3071, 3038, 2974, 2924, 1744, 1479, 1415, 1351, 1271, 1029, 758, 701. ^{1}H NMR (400 MHz, $CDCl_3$, TMS, 25 °C,): δ 0.97 (t, 3H, J = 6.8 Hz), 3.96 (q, 2H, J = 6.8 Hz), 4.39 (d, 1H, J = 9.6 Hz), 4.84 (d, 1H, J = 9.6 Hz), 7.3–7.5 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS, 25 °C): δ 13.81, 61.19, 79.56, 81.15, 127.61, 128.48, 129.12, 135.56, 167.66. MS (ESI) m/z: 407 (M^++Na).

Synthesis of ethyl 2-benzyl-4-hydroxy-5-oxo-3-phenyl-2,5-dihydrofuran-2-carboxylate **II**

To a solution of 2-ethoxycarbonyl-3-phenyloxirane (ethyl 3-phenylglycidate) (1.92 g, 10 mmol) in dry toluene (5.0 mL), Tf_2O (0.282 g, 1.0 mmol) was added and resultant mixture was heated at 110 °C under nitrogen for 12 h, neutralized with sat. aq. $NaHCO_3$ solution and extracted with ethyl acetate. The organic layer was dried over $MgSO_4$, filtered, and concentrated in vacuo. The crude material was first purified by silica-gel column chromatography with a mixture of ethyl acetate and *n*-hexane (1/10, v/v) as eluent and was then recrystallized from Et_2O : hexane (1:2) to afford white crystals of compound **II** (m.p. 141–142 °C [lit.[5] 141.0–141.3 °C]). Yield: 3.03 g, 89.8 %. IR (KBr) υ: 3313, 3063, 2963, 1739, 1671, 1498, 1397, 1184, 1100, 1008, 760, 704. ^{1}H NMR (400 MHz, $CDCl_3$, TMS, 25 °C): δ 1.23 (t, 3H, J = 7.2 Hz), 3.56 (d, 2H, J = 10.8 Hz), 3.67 (d, 2H, J = 10.8 Hz), 4.27 (q, 2H, J = 7.2 Hz), 6.12 (s, 1H), 7.09–7.69 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS, 25 °C): δ 13.99, 39.21, 63.09, 86.14, 96.14, 127.38, 127.76, 128.08, 128.89, 129.12, 129.42, 129.57, 130.48, 132.79, 138.52, 169.01. MS (ESI) m/z: 339 (M^{+1}).

IR spectra

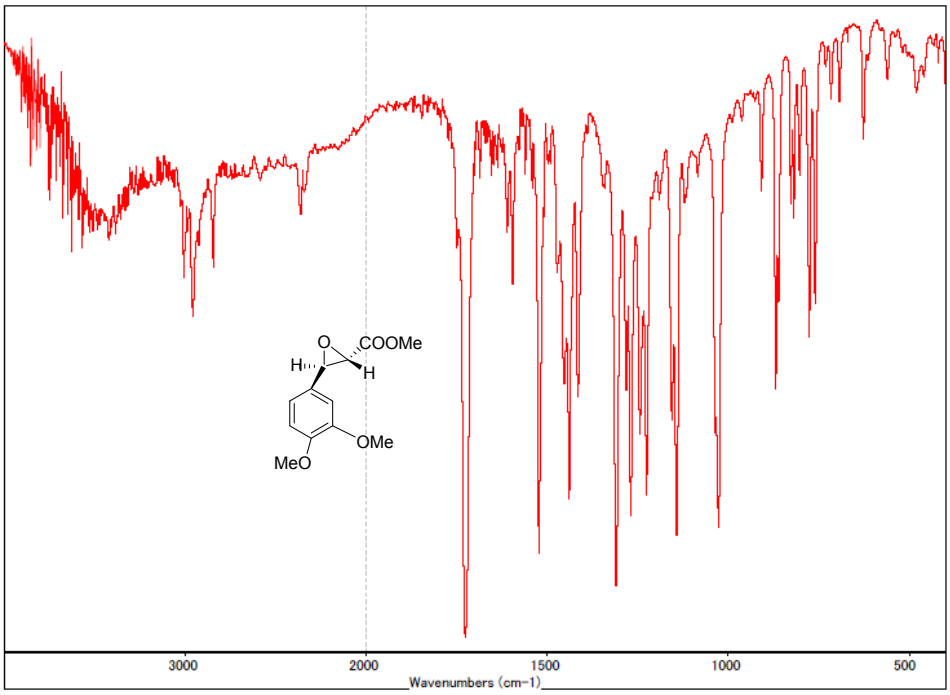


Figure S1. IR spectrum of MCDMPO (KBr).

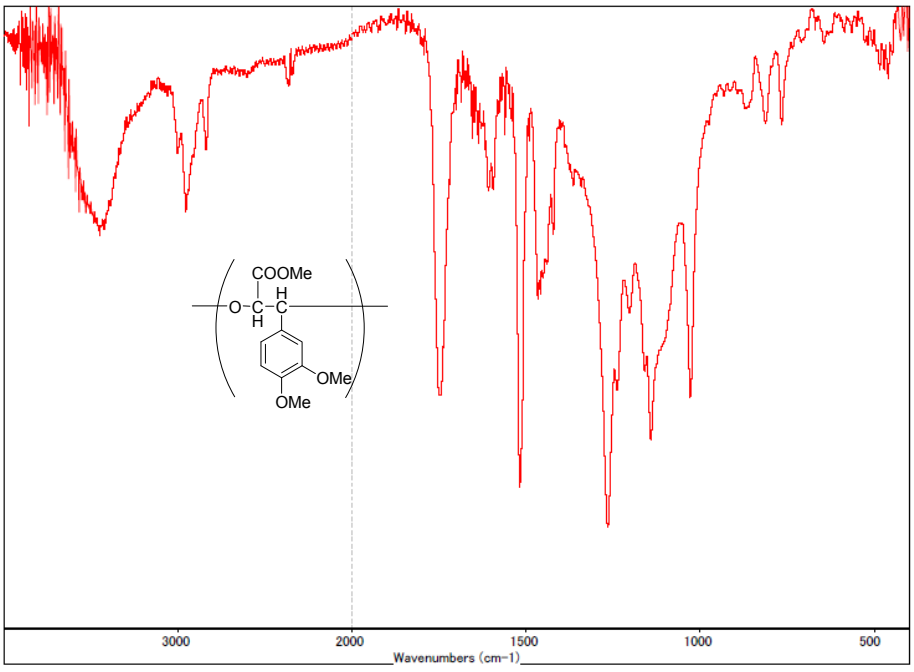


Figure S2. IR spectrum of poly(MCDMPO) (run 7 in Table 1) (KBr).

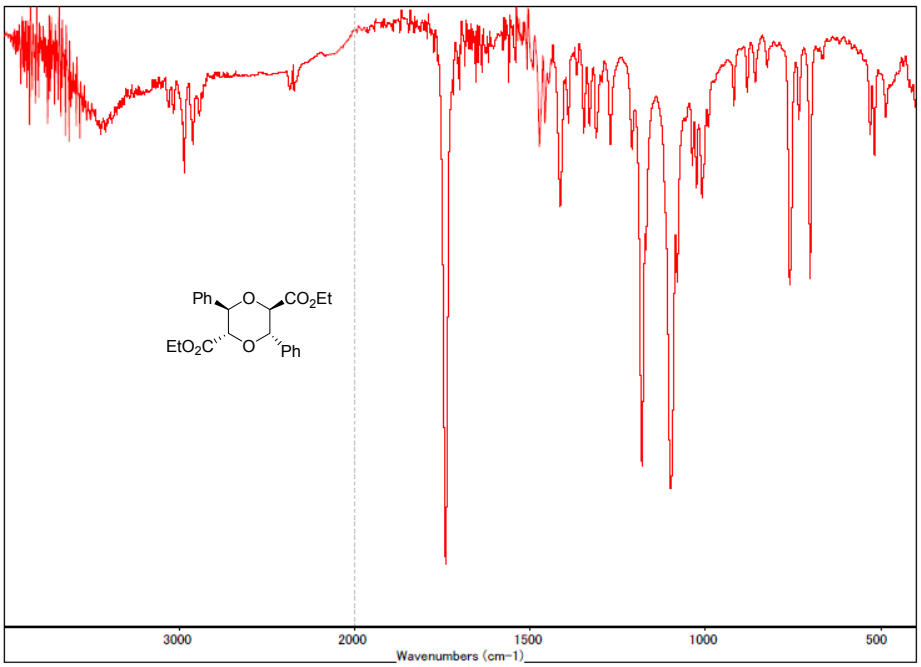


Figure S3. IR spectrum of **I** (KBr).

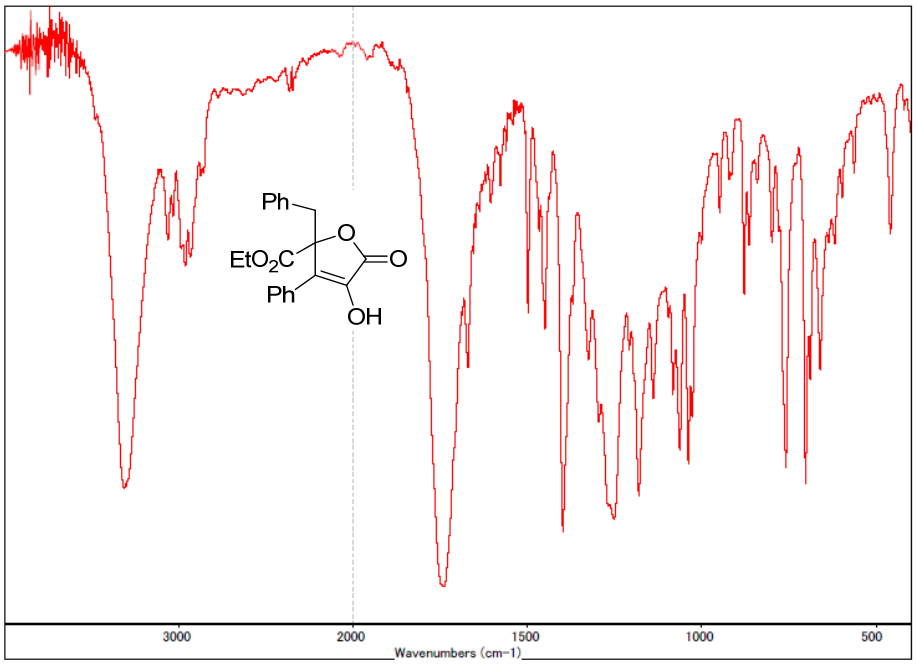


Figure S4. IR spectrum of **II** (KBr).

NMR spectra

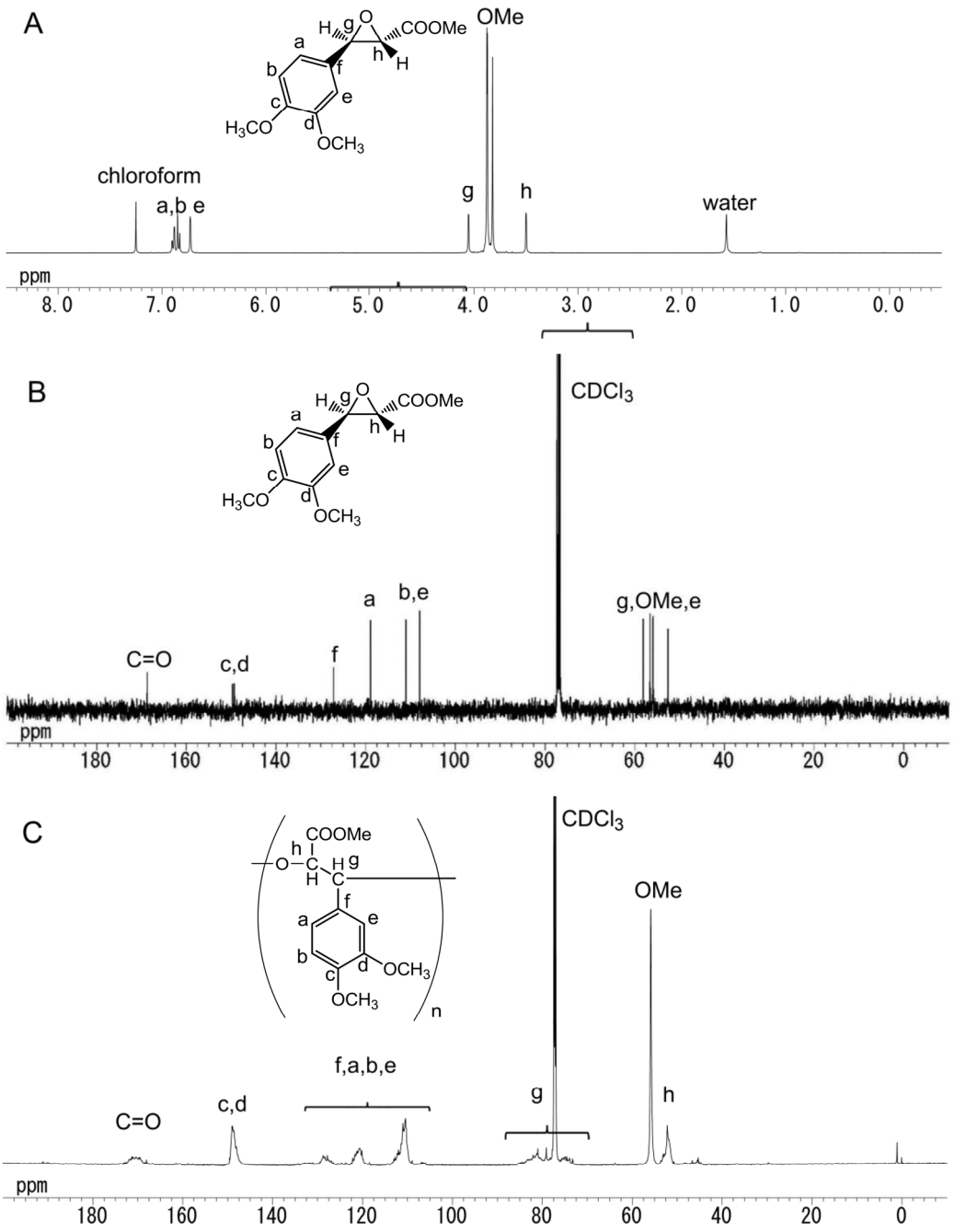


Figure S5. ^{1}H NMR (A) and ^{13}C NMR spectra of MCDMPO (B) and ^{13}C NMR spectrum of poly(MCDMPO) (run 7 in Table 1) (C) (KBr) [100 MHz, 23^{o}C, $CDCl_3$].

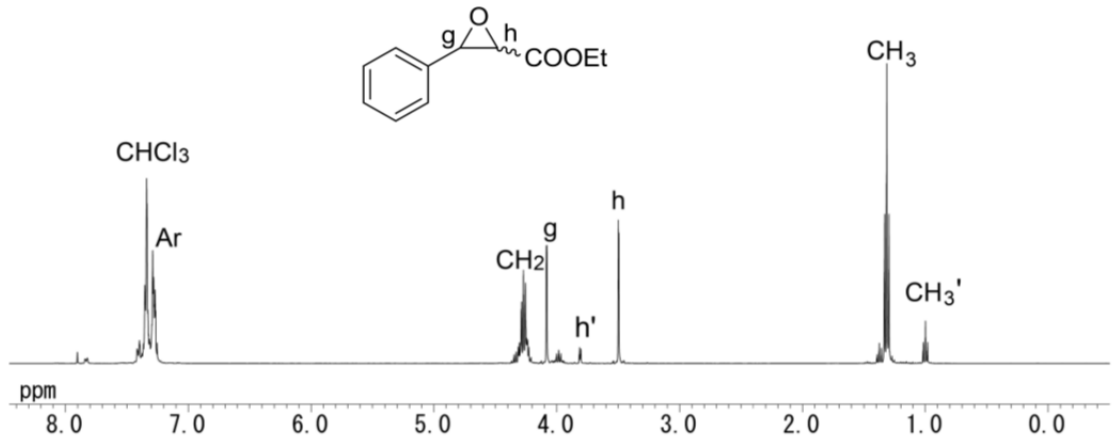


Figure S6. ^{1}H NMR spectrum of 2-ethoxycarbonyl-3-phenyloxirane [400 MHz, $CDCl_3$, 25 ^{o}C].

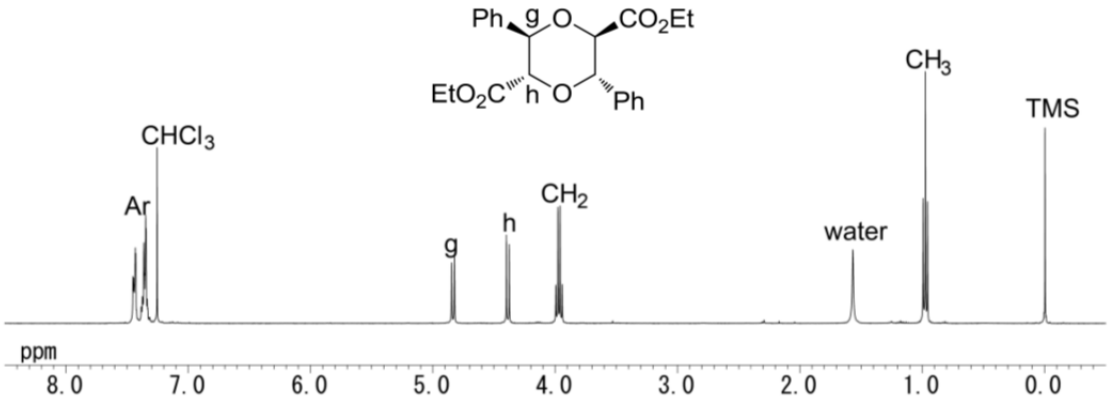


Figure S7. ^{1}H NMR spectrum of **I** [400 MHz, $CDCl_3$, 25 ^{o}C].

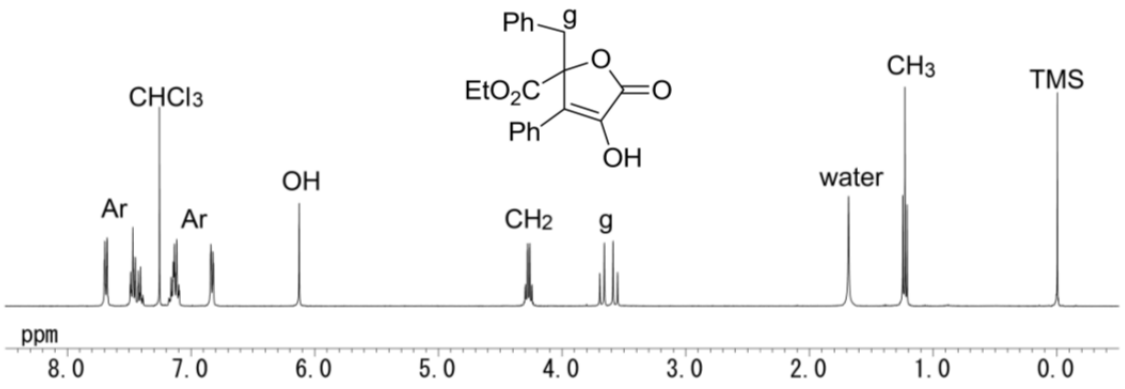


Figure S8. ^{1}H NMR spectrum of **II** [400 MHz, $CDCl_3$, 25 ^{o}C]. The signal at 1.7 ppm has been assigned to water but it might be based on some impurity whose identity is not known.

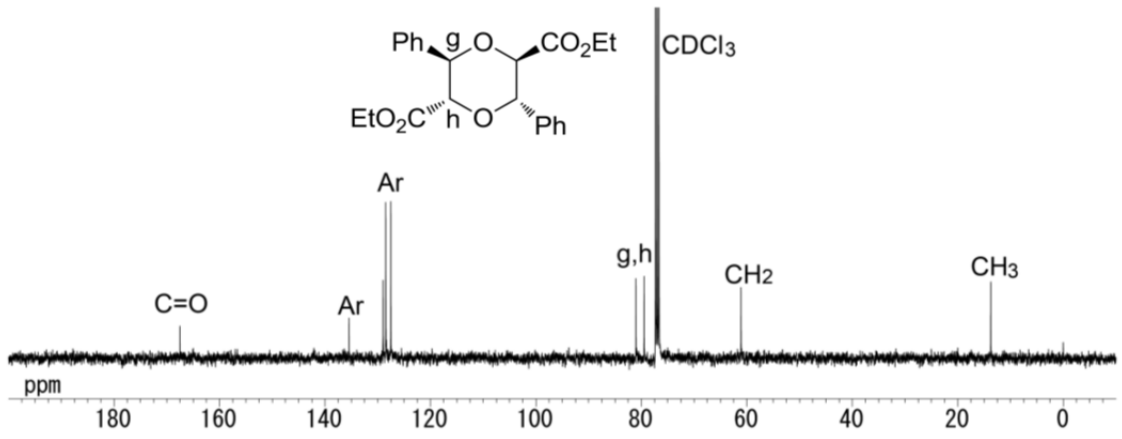


Figure S9. ^{13}C NMR spectrum of **I** [100 MHz, $CDCl_3$, 25 ^{o}C].

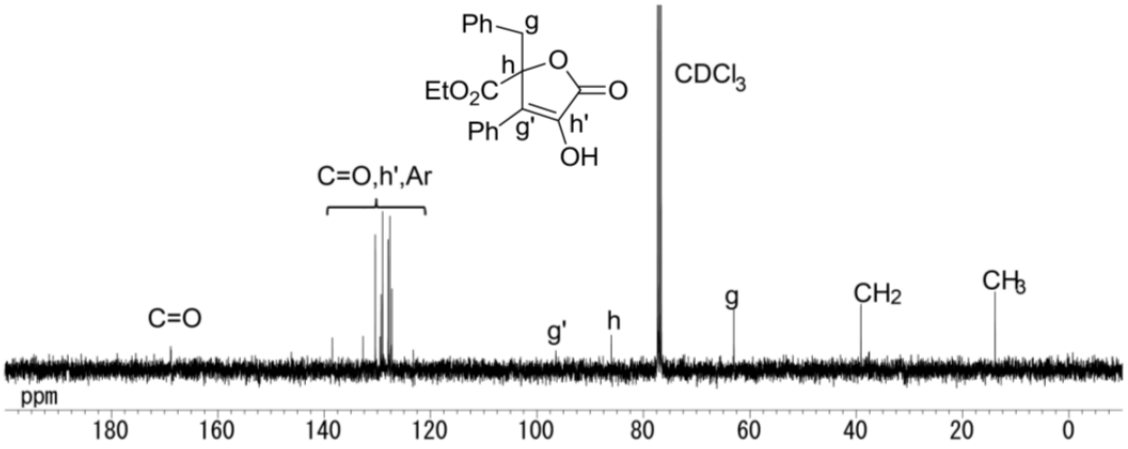


Figure S10. ^{13}C NMR spectrum of **II** [100 MHz, $CDCl_3$, 25 ^{o}C].

TGA profiles

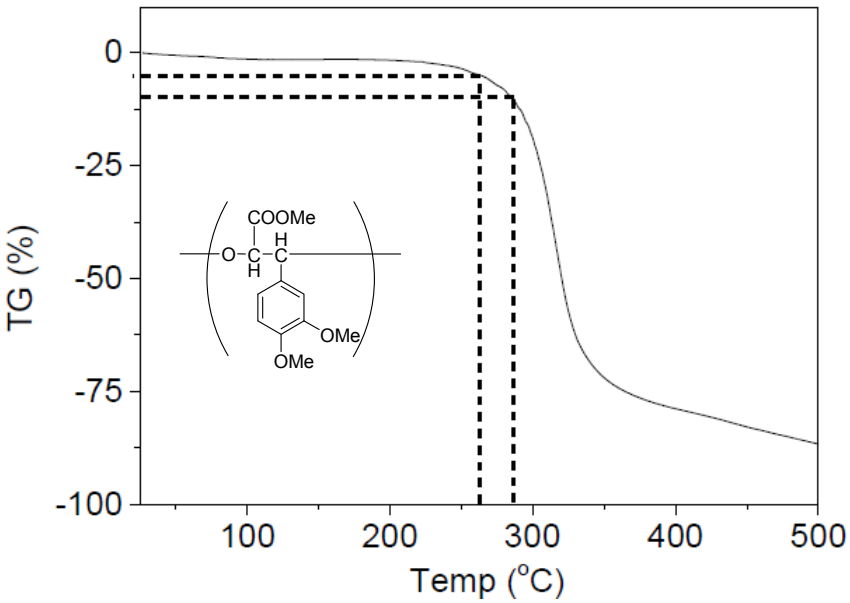


Figure S11. TGA profile of poly(MCDMPO) (run 7 in Table 1) (N_2 atmosphere, heating rate 10 °C/min). T_{d5} = 263.4°C, T_{d10} = 285.3°C.

DSC profiles

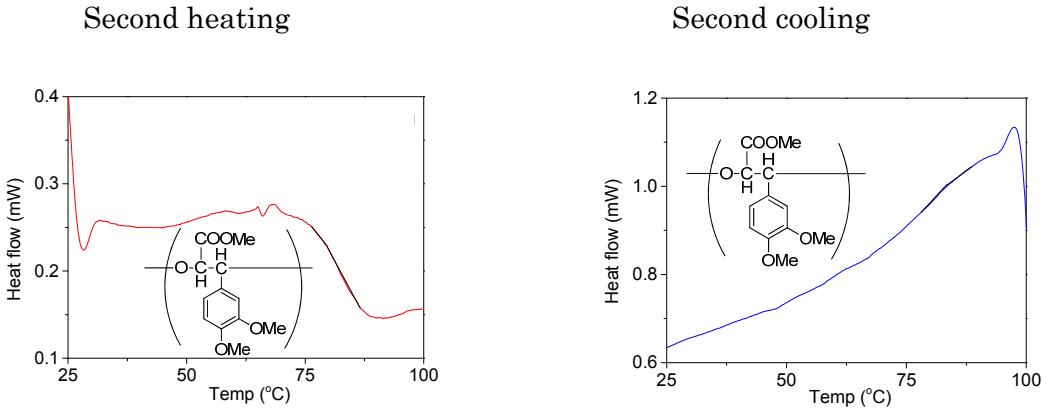


Figure S12. T_g = 79.3°C, T_m = 88.1°C, T_g = 81.4°C, DSC profiles of poly(MCDMPO) (run 7 in Table 1) obtained on second heating (left) and on second cooling (right), (N_2: atmosphere, 20 mL/min, –25 °C–100 °C, heating/ cooling rate = 5 °C /min).

MD Simulations

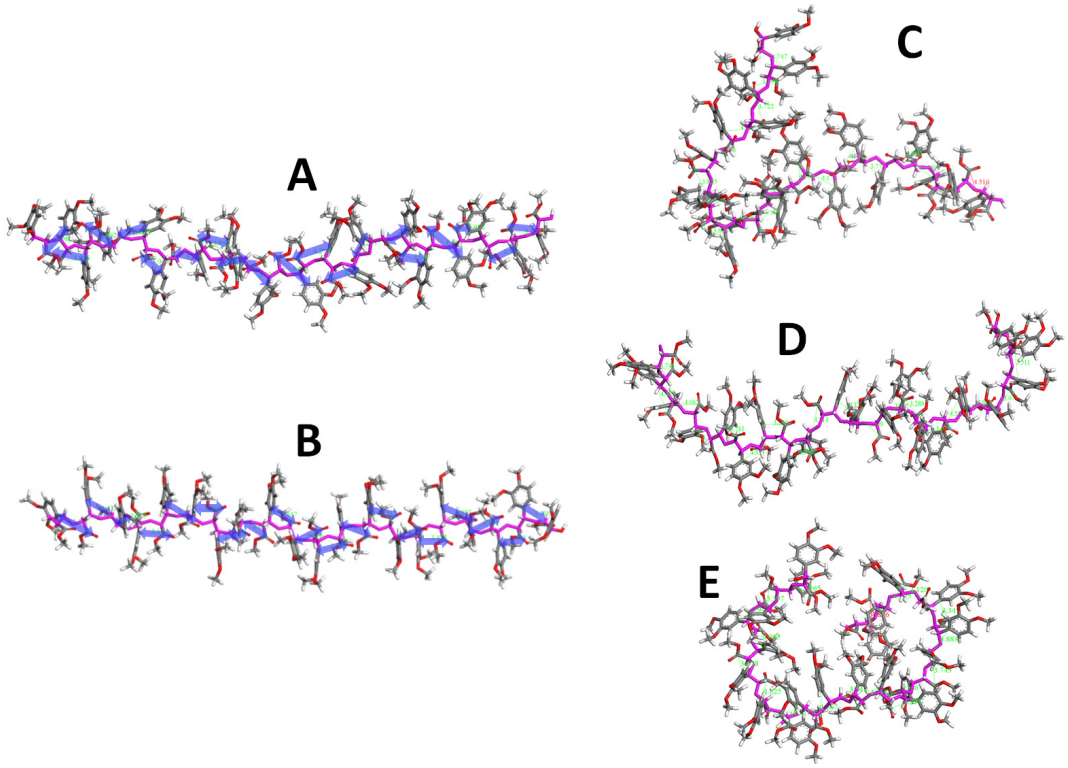


Figure S13. Structures obtained through MD simulations for 5 nsec for at 297 K (S,S)-20-mer (A), (S,S)/(R,R)-20-mer (B), (R,S)-20-mer (C), (R,S)/(S,R)-20-mer (D), and (S,S)/(R,R)/(R,S)/(S,R)-20-mer (E). In A-E, white, gray, and red correspond to H, C, and O, respectively, and pink the main chain. Blue arrows indicate carbonyl-aromatic combinations that may contribute to ICT interactions.

Note: As representative head-to-tail type chain structures, five 20-mer models were constructed: chains consisting of twenty (S,S)-units ((S,S)-20-mer), randomly distributed ten (S,S)-units and ten (R,R)-units ((S,S)/(R,R)-20-mer), twenty (R,S)-units ((R,S)-20-mer), randomly distributed ten (R,S)-units and ten (S,R)-units ((R,S)/(S,R)-20-mer), and randomly distributed five (S,S)-units, five (R,R)-units, five (R,S)-units, and five (S,R)-units ((S,S)/(R,R,)/(R,S)/(S,R)-20-mer) (Fig. S13 A-E, respectively).

During simulations for 5 nsec at 297 K, (S,S)-20-mer and (S,S)/(R,R)-20-mer remained as stretched chains and showed no bending or folding of the chain (Fig. S13A,B). In contrast, (R,S)-20-mer and (S,S)/(R,R)/(R,S)/(S,R)-20-mer curled up

into compact shapes (Fig. S13C,E), and (R,S)/(S,R)-20-mer also significantly deviated from a stretched conformation (Fig. S13D). Because a rather stiff conformation is proposed based on the α values, chain models consisting only of (S,S) and/or (R,R) unit may be more plausible.

For all models, the distance between neighboring dimethoxyphenyl groups seems too long to form a stack. On the other hand, as for (S,S)-20-mer and (S,S)/(R,R)-20-mer, carbonyl and aromatic groups belonging to adjacent units are located close enough to interact with each other through local bond rotations. The averaged distance between such carbonyl and aromatic groups as defined as a distance between the carbonyl carbon and the 1-position carbon of phenyl group was 3.5 Å for (S,S)-20-mer and 3.7 Å for (S,S)/(R,R)-20-mer which are rather close to the stacking distance in DNAs (3.4 Å). The number of carbonyl-aromatic combinations with a distance of 3.4 Å or shorter out of total 19 combinations per chain was 8 for both (S,S)-20-mer (S,S)/(R,R)-20-mer. On the other hand, the averaged distance and the number of carbonyl-aromatic combinations were 4.1 Å and 1 set for (R,S)-20-mer, 4.0 Å and 3 sets for (R,S)/(S,R)-20-mer, and 4.0 Å and 3 sets for (S,S)/(R,R)/(R,S)/(S,R)-20-mer, respectively.

Thus, (S,S)-20-mer and (S,S)/(R,R)-20-mer models resulted from retention of configuration on monomer addition may explain the stiff conformation and the occurrence of the ICT band through interaction between carbonyl and aromatic groups in the polymer.

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Crystal Data for 3,6-diphenyl-1,4-dioxane-2,5-dicarboxylate (I)

Experimental

Data Collection

A colorless platelet crystal of $C_{22}H_{24}O_6$ having approximate dimensions of 0.428 x 0.072 x 0.010 mm was mounted on a glass fiber. All measurements were made on a Rigaku R-AXIS RAPID diffractometer using multi-layer mirror monochromated Cu-Kα radiation.

The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive triclinic cell with dimensions:

a	= 6.0103(4) Å	α	= 94.344(7)^o
b	= 8.0802(6) Å	β	= 93.489(6)^o
c	= 22.1231(14) Å	γ	= 111.753(5)^o
V	= 990.44(12) Å^3		

For Z = 2 and F.W. = 384.43, the calculated density is 1.289 g/cm^3. 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-1 (#2)

The data were collected at a temperature of -180 $\pm$ 1^{o}C to a maximum 2θ value of 136.3^o. A total of 90 oscillation images were collected. A sweep of data was done using ω scans from 80.0 to 260.0^o in 10.00^o step, at χ=54.0^o and ϕ = 0.0^o. The exposure rate was 10.0 [sec./^{o}]. A second sweep was performed using ω scans from 80.0 to 260.0^o in 10.00^o step, at χ=54.0^o and ϕ = 90.0^o. The exposure rate was 10.0 [sec./^{o}]. Another sweep was performed using ω scans from 80.0 to 260.0^o in 10.00^o step, at χ=54.0^o and ϕ = 180.0^o. The exposure rate was 10.0 [sec./^{o}]. Another sweep was performed using ω scans from 80.0 to 260.0^o in 10.00^o step, at χ=54.0^o and ϕ = 270.0^o. The exposure rate was 10.0 [sec./^{o}]. Another sweep was performed using ω scans from 80.0 to 260.0^o in 10.00^o step, at χ=0.0^o and ϕ = 0.0^o. The exposure rate was 10.0 [sec./^{o}]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 11495 reflections were collected, where 3498 were unique (R_{int} = 0.1280); equivalent reflections were merged.

The linear absorption coefficient, μ, for Cu-Kα radiation is 7.729 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.486 to 0.992. 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. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[2] on F^2 was based on 3498 observed reflections and 256 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.1069$$

$$wR2 = [\Sigma (w (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.3079$$

The standard deviation of an observation of unit weight[3] was 1.06. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.57 and -0.35 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[4]. Anomalous dispersion effects were included in Fcalc[5]; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley[6]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[7]. All calculations were performed using the CrystalStructure[8] crystallographic software package except for refinement, which was performed using SHELXL-97[9].

References

(1) SHELX97: Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

(2) Least Squares function minimized: (SHELXL97)

$\Sigma w(F_o^2-F_c^2)^2$ where w = Least Squares weights.

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

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

where: N_o = number of observations
N_v = number of variables

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EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{22}H_{24}O_6$
Formula Weight	384.43
Crystal Color, Habit	colorless, platelet
Crystal Dimensions	0.428 X 0.072 X 0.010 mm
Crystal System	triclinic
Lattice Type	Primitive
Lattice Parameters	a = 6.0103(4) Å b = 8.0802(6) Å c = 22.1231(14) Å α = 94.344(7) ^{o} β = 93.489(6) ^{o} γ = 111.753(5) ^{o} V = 990.44(12) Å^3
Space Group	P-1 (#2)
Z value	2
D_{calc}	1.289 g/cm^3
F_{000}	408.00
μ(CuKα)	7.729 cm^{-1}

B. Intensity Measurements

Diffractometer	R-AXIS RAPID
Radiation	CuKα (λ = 1.54187 Å) multi-layer mirror monochromated
Voltage, Current	40kV, 30mA
Temperature	-180.0^{o}C
Detector Aperture	460 x 256 mm
Data Images	90 exposures
ω oscillation Range (χ=54.0, ϕ=0.0)	80.0 - 260.0^{o}
Exposure Rate	10.0 sec./^{o}
ω oscillation Range (χ=54.0, ϕ=90.0)	80.0 - 260.0^{o}
Exposure Rate	10.0 sec./^{o}
ω oscillation Range (χ=54.0, ϕ=180.0)	80.0 - 260.0^{o}
Exposure Rate	10.0 sec./^{o}
ω oscillation Range (χ=54.0, ϕ=270.0)	80.0 - 260.0^{o}
Exposure Rate	10.0 sec./^{o}
ω oscillation Range (χ=0.0, ϕ=0.0)	80.0 - 260.0^{o}
Exposure Rate	10.0 sec./^{o}
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	135.0^{o}
No. of Reflections Measured	Total: 11495 Unique: 3498 (R_{int} = 0.1280)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.486 - 0.992)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELX97)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1/ [\sigma^2(Fo^2) + (0.1573 \cdot P)^2 + 1.0829 \cdot P]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$
$2\theta_{max}$ cutoff	135.0^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	3498
No. Variables	256
Reflection/Parameter Ratio	13.66
Residuals: R1 (I>2.00σ(I))	0.1069
Residuals: R (All reflections)	0.1232
Residuals: wR2 (All reflections)	0.3079
Goodness of Fit Indicator	1.060
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.57 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.35 $e^-/Å^3$

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

atom	x	y	z	B_{eq}
O1	0.1179(5)	0.1694(4)	1.03332(12)	3.00(6)
O2	0.3352(5)	0.3831(4)	0.94008(14)	3.64(6)
O3	0.6077(5)	0.2542(4)	0.95434(13)	3.06(6)
O4	-0.0446(5)	0.3483(4)	0.53151(12)	2.99(6)
O5	-0.0775(5)	0.0792(4)	0.43898(15)	3.84(7)
O6	0.3276(5)	0.2115(4)	0.44910(13)	3.25(6)
C1	-0.1452(7)	0.0891(6)	1.11325(19)	2.95(8)
C2	-0.1480(8)	0.0254(6)	1.16950(19)	3.33(8)
C3	-0.2691(8)	0.0761(6)	1.2148(2)	3.71(9)
C4	-0.3839(8)	0.1920(6)	1.2043(2)	3.65(9)
C5	-0.3781(8)	0.2590(6)	1.1487(2)	3.48(8)
C6	-0.2580(7)	0.2085(6)	1.1031(2)	3.25(8)
C7	-0.0336(7)	0.0218(6)	1.06246(18)	2.90(8)
C8	0.2267(7)	0.1047(6)	0.98581(19)	3.07(8)
C9	0.3926(7)	0.2665(6)	0.95785(18)	2.91(8)
C10	0.7854(7)	0.3945(6)	0.92465(19)	3.22(8)
C11	0.7552(9)	0.3452(7)	0.8574(2)	4.15(10)
C12	-0.2069(8)	0.4704(6)	0.61224(19)	3.12(8)
C13	-0.1278(8)	0.5607(6)	0.6700(2)	3.38(8)
C14	-0.2859(9)	0.5345(6)	0.7154(2)	3.85(9)
C15	-0.5222(8)	0.4134(6)	0.7029(2)	3.81(9)
C16	-0.6003(8)	0.3204(6)	0.6458(2)	3.64(9)
C17	-0.4429(8)	0.3488(6)	0.6003(2)	3.32(8)
C18	-0.0374(7)	0.5134(6)	0.56268(19)	3.03(8)
C19	0.1165(7)	0.3851(6)	0.48454(18)	3.05(8)
C20	0.1045(7)	0.2058(6)	0.45520(19)	3.16(8)
C21	0.3451(8)	0.0501(6)	0.4190(2)	3.25(8)
C22	0.3280(10)	0.0502(7)	0.3509(2)	4.46(10)

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

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}
H2	-0.06726	-0.05311	1.17738	3.998
H3	-0.27234	0.03029	1.25321	4.456
H4	-0.46665	0.22562	1.23525	4.377
H5	-0.45589	0.33961	1.14133	4.175
H6	-0.25316	0.25580	1.06495	3.895
H7	0.06472	-0.04300	1.07965	3.484
H8	0.32323	0.03941	1.00359	3.679
H10A	0.94977	0.41019	0.94102	3.867
H10B	0.76463	0.50954	0.93356	3.867
H11A	0.75016	0.22298	0.84884	4.985
H11B	0.89098	0.42870	0.83901	4.985
H11C	0.60480	0.35152	0.84019	4.985
H13	0.03492	0.64098	0.67887	4.059
H14	-0.23212	0.59916	0.75464	4.615
H15	-0.62999	0.39458	0.73364	4.569
H16	-0.76146	0.23686	0.63737	4.364
H17	-0.49747	0.28477	0.56098	3.983
H18	0.13013	0.58655	0.58098	3.641
H19	0.28429	0.45808	0.50272	3.665
H21A	0.21396	-0.05684	0.43012	3.900
H21B	0.50031	0.04299	0.43324	3.900
H22A	0.45156	0.15960	0.33993	5.355
H22B	0.16842	0.04561	0.33628	5.355
H22C	0.35284	-0.05471	0.33217	5.355

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O1	0.0297(14)	0.0321(16)	0.0485(17)	0.0070(12)	0.0115(12)	0.0003(12)
O2	0.0369(16)	0.0423(18)	0.0619(19)	0.0158(14)	0.0128(13)	0.0110(15)
O3	0.0291(14)	0.0354(16)	0.0514(17)	0.0109(12)	0.0079(12)	0.0049(13)
O4	0.0369(15)	0.0331(16)	0.0459(16)	0.0143(13)	0.0128(12)	0.0061(12)
O5	0.0366(17)	0.0394(18)	0.067(2)	0.0118(14)	0.0102(14)	-0.0021(15)
O6	0.0350(15)	0.0384(17)	0.0531(17)	0.0159(13)	0.0109(12)	0.0067(13)
C1	0.032(2)	0.033(2)	0.045(2)	0.0097(17)	0.0078(16)	-0.0001(17)
C2	0.041(2)	0.041(2)	0.045(2)	0.0148(19)	0.0075(17)	0.0027(19)
C3	0.052(3)	0.044(3)	0.043(2)	0.014(2)	0.0109(19)	0.0045(19)
C4	0.043(2)	0.048(3)	0.046(2)	0.015(2)	0.0136(19)	-0.003(2)
C5	0.040(2)	0.040(2)	0.054(3)	0.016(2)	0.0096(18)	0.002(2)
C6	0.038(2)	0.039(2)	0.044(2)	0.0110(19)	0.0084(17)	0.0017(18)
C7	0.030(2)	0.035(2)	0.044(2)	0.0108(18)	0.0080(16)	0.0044(18)
C8	0.034(2)	0.036(2)	0.047(2)	0.0120(18)	0.0120(17)	0.0015(18)
C9	0.030(2)	0.037(2)	0.044(2)	0.0122(18)	0.0082(16)	0.0017(18)
C10	0.031(2)	0.033(2)	0.055(3)	0.0073(18)	0.0105(17)	0.0038(19)
C11	0.049(3)	0.048(3)	0.054(3)	0.008(2)	0.013(2)	0.010(2)
C12	0.039(2)	0.038(2)	0.045(2)	0.0173(19)	0.0109(17)	0.0046(18)
C13	0.046(2)	0.034(2)	0.049(2)	0.0154(19)	0.0071(19)	0.0024(19)
C14	0.059(3)	0.044(3)	0.042(2)	0.018(2)	0.012(2)	0.002(2)
C15	0.050(3)	0.045(3)	0.052(3)	0.018(2)	0.019(2)	0.008(2)
C16	0.041(2)	0.044(3)	0.053(3)	0.014(2)	0.0118(19)	0.006(2)
C17	0.042(2)	0.040(2)	0.044(2)	0.0158(19)	0.0077(18)	0.0025(19)
C18	0.037(2)	0.032(2)	0.046(2)	0.0127(18)	0.0049(17)	0.0021(18)
C19	0.035(2)	0.039(2)	0.043(2)	0.0142(18)	0.0096(17)	0.0047(18)
C20	0.038(2)	0.037(2)	0.048(2)	0.016(2)	0.0097(18)	0.0070(18)
C21	0.039(2)	0.028(2)	0.059(3)	0.0144(18)	0.0144(19)	0.0069(19)
C22	0.069(3)	0.050(3)	0.055(3)	0.026(3)	0.020(2)	0.006(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 4. Fragment Analysis

fragment: 1

O(1)	O(2)	O(3)	C(1)	C(2)
C(3)	C(4)	C(5)	C(6)	C(7)
C(8)	C(9)	C(10)	C(11)	

fragment: 2

O(4)	O(5)	O(6)	C(12)	C(13)
C(14)	C(15)	C(16)	C(17)	C(18)
C(19)	C(20)	C(21)	C(22)	

Table 5. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
O1	C7	1.432(5)	O1	C8	1.435(6)
O2	C9	1.201(6)	O3	C9	1.339(6)
O3	C10	1.469(5)	O4	C18	1.439(5)
O4	C19	1.436(5)	O5	C20	1.199(4)
O6	C20	1.341(6)	O6	C21	1.461(6)
C1	C2	1.381(6)	C1	C6	1.393(7)
C1	C7	1.509(6)	C2	C3	1.397(7)
C3	C4	1.379(8)	C4	C5	1.379(7)
C5	C6	1.396(7)	C7	C8[1]	1.529(5)
C8	C9	1.523(6)	C10	C11	1.493(6)
C12	C13	1.386(6)	C12	C17	1.389(5)
C12	C18	1.514(6)	C13	C14	1.398(7)
C14	C15	1.390(6)	C15	C16	1.380(6)
C16	C17	1.397(7)	C18	C19[2]	1.532(7)
C19	C20	1.516(7)	C21	C22	1.503(7)

Symmetry Operators:

(1) -X,-Y,-Z+2 (2) -X,-Y+1,-Z+1

Table 6. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C2	H2	0.950	C3	H3	0.950
C4	H4	0.950	C5	H5	0.950
C6	H6	0.950	C7	H7	1.000
C8	H8	1.000	C10	H10A	0.990
C10	H10B	0.990	C11	H11A	0.980
C11	H11B	0.980	C11	H11C	0.980
C13	H13	0.950	C14	H14	0.950
C15	H15	0.950	C16	H16	0.950
C17	H17	0.950	C18	H18	1.000
C19	H19	1.000	C21	H21A	0.990
C21	H21B	0.990	C22	H22A	0.980
C22	H22B	0.980	C22	H22C	0.980

Table 7. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
C7	O1	C8	109.6(3)	C9	O3	C10	116.4(4)
C18	O4	C19	110.1(3)	C20	O6	C21	116.1(3)
C2	C1	C6	119.2(4)	C2	C1	C7	120.2(5)
C6	C1	C7	120.5(4)	C1	C2	C3	120.2(5)
C2	C3	C4	120.5(4)	C3	C4	C5	119.6(5)
C4	C5	C6	120.1(5)	C1	C6	C5	120.3(4)
O1	C7	C1	110.0(3)	O1	C7	C8[1]	107.8(3)
C1	C7	C8[1]	111.1(3)	O1	C8	C7[1]	110.4(3)
O1	C8	C9	107.7(4)	C7[1]	C8	C9	110.5(3)
O2	C9	O3	125.8(4)	O2	C9	C8	124.8(4)
O3	C9	C8	109.3(4)	O3	C10	C11	110.4(3)
C13	C12	C17	119.4(4)	C13	C12	C18	119.1(3)
C17	C12	C18	121.4(4)	C12	C13	C14	120.4(4)
C13	C14	C15	119.7(4)	C14	C15	C16	120.1(4)
C15	C16	C17	120.1(4)	C12	C17	C16	120.3(4)
O4	C18	C12	108.9(3)	O4	C18	C19[2]	107.4(3)
C12	C18	C19[2]	110.8(4)	O4	C19	C18[2]	109.8(4)
O4	C19	C20	107.0(3)	C18[2]	C19	C20	110.3(3)
O5	C20	O6	125.2(5)	O5	C20	C19	125.1(4)
O6	C20	C19	109.7(3)	O6	C21	C22	111.4(4)

Symmetry Operators:

(1) -X,-Y,-Z+2 (2) -X,-Y+1,-Z+1

Table 8. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C1	C2	H2	119.9	C3	C2	H2	119.9
C2	C3	H3	119.7	C4	C3	H3	119.7
C3	C4	H4	120.2	C5	C4	H4	120.2
C4	C5	H5	119.9	C6	C5	H5	119.9
C1	C6	H6	119.8	C5	C6	H6	119.8
O1	C7	H7	109.3	C1	C7	H7	109.3
C8[1]	C7	H7	109.3	O1	C8	H8	109.4
C7[1]	C8	H8	109.4	C9	C8	H8	109.4
O3	C10	H10A	109.6	O3	C10	H10B	109.6
C11	C10	H10A	109.6	C11	C10	H10B	109.6
H10A	C10	H10B	108.1	C10	C11	H11A	109.5
C10	C11	H11B	109.5	C10	C11	H11C	109.5
H11A	C11	H11B	109.5	H11A	C11	H11C	109.5
H11B	C11	H11C	109.5	C12	C13	H13	119.8
C14	C13	H13	119.8	C13	C14	H14	120.2
C15	C14	H14	120.2	C14	C15	H15	120.0
C16	C15	H15	120.0	C15	C16	H16	119.9
C17	C16	H16	119.9	C12	C17	H17	119.9
C16	C17	H17	119.9	O4	C18	H18	109.9
C12	C18	H18	109.9	C19[2]	C18	H18	109.9
O4	C19	H19	109.9	C18[2]	C19	H19	109.9
C20	C19	H19	109.9	O6	C21	H21A	109.4
O6	C21	H21B	109.4	C22	C21	H21A	109.4
C22	C21	H21B	109.4	H21A	C21	H21B	108.0
C21	C22	H22A	109.5	C21	C22	H22B	109.5
C21	C22	H22C	109.5	H22A	C22	H22B	109.5
H22A	C22	H22C	109.5	H22B	C22	H22C	109.5

Symmetry Operators:

(1) -X,-Y,-Z+2

(2) -X,-Y+1,-Z+1

Table 9. Torsion Angles(O)
(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C7	O1	C8	C7[1]	61.4(4)	C7	O1	C8	C9	-178.0(3)
C8	O1	C7	C1	179.0(3)	C8	O1	C7	C8[1]	-59.8(4)
C9	O3	C10	C11	-86.5(4)	C10	O3	C9	O2	-1.6(5)
C10	O3	C9	C8	176.2(3)	C18	O4	C19	C18[2]	-61.9(3)
C18	O4	C19	C20	178.4(3)	C19	O4	C18	C12	-179.5(3)
C19	O4	C18	C19[2]	60.4(4)	C20	O6	C21	C22	85.6(4)
C21	O6	C20	O5	1.4(6)	C21	O6	C20	C19	-177.4(3)
C2	C1	C6	C5	-1.8(5)	C6	C1	C2	C3	2.0(5)
C2	C1	C7	O1	-134.1(3)	C2	C1	C7	C8[1]	106.6(4)
C7	C1	C2	C3	-174.6(3)	C6	C1	C7	O1	49.4(4)
C6	C1	C7	C8[1]	-69.9(4)	C7	C1	C6	C5	174.7(3)
C1	C2	C3	C4	-1.0(5)	C2	C3	C4	C5	-0.3(5)
C3	C4	C5	C6	0.5(5)	C4	C5	C6	C1	0.6(5)
O1	C7	C8[1]	O1[1]	60.3(5)	O1	C7	C8[1]	C9[1]	179.2(3)
C1	C7	C8[1]	O1[1]	-179.2(3)	C1	C7	C8[1]	C9[1]	-60.2(5)
O1	C8	C9	O2	-51.1(5)	O1	C8	C9	O3	131.1(3)
C7[1]	C8	C9	O2	69.5(5)	C7[1]	C8	C9	O3	-108.3(4)
C13	C12	C17	C16	1.2(8)	C17	C12	C13	C14	-2.1(8)
C13	C12	C18	O4	133.0(4)	C13	C12	C18	C19[2]	-109.0(5)
C18	C12	C13	C14	174.9(4)	C17	C12	C18	O4	-50.1(6)
C17	C12	C18	C19[2]	67.9(6)	C18	C12	C17	C16	-175.7(4)
C12	C13	C14	C15	1.7(8)	C13	C14	C15	C16	-0.4(8)
C14	C15	C16	C17	-0.6(8)	C15	C16	C17	C12	0.1(8)
O4	C18	C19[2]	O4[2]	-60.3(3)	O4	C18	C19[2]	C20[2]	-177.9(3)
C12	C18	C19[2]	O4[2]	-179.1(3)	C12	C18	C19[2]	C20[2]	63.2(4)
O4	C19	C20	O5	47.4(5)	O4	C19	C20	O6	-133.7(3)
C18[2]	C19	C20	O5	-72.0(5)	C18[2]	C19	C20	O6	106.8(4)

Symmetry Operators:

(1) -X,-Y,-Z+2

(2) -X,-Y+1,-Z+1

Table 10. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
O1	O1[1]	2.817(3)	O1	O2	2.853(4)
O1	O3	3.391(4)	O1	C2	3.567(5)
O1	C6	2.910(6)	O2	C7[1]	3.092(5)
O2	C10	2.716(6)	O2	C11	3.296(6)
O3	C1[1]	3.287(4)	O3	C7[1]	3.322(4)
O4	O4[2]	2.807(4)	O4	O5	2.813(4)
O4	O6	3.402(5)	O4	C13	3.552(5)
O4	C17	2.915(6)	O5	C18[2]	3.107(6)
O5	C21	2.696(6)	O5	C22	3.274(7)
O6	C12[2]	3.281(6)	O6	C18[2]	3.311(6)
C1	C4	2.791(7)	C1	C9[1]	2.967(5)
C2	C5	2.771(8)	C2	C8[1]	3.467(6)
C2	C9[1]	3.403(5)	C3	C6	2.762(7)
C6	C8[1]	3.154(7)	C7	C7[1]	2.832(6)
C8	C8[1]	2.763(5)	C9	C11	3.142(6)
C12	C15	2.789(7)	C12	C20[2]	2.993(7)
C13	C16	2.774(6)	C13	C19[2]	3.482(6)
C13	C20[2]	3.449(7)	C14	C17	2.777(6)
C17	C19[2]	3.150(6)	C18	C18[2]	2.841(6)
C19	C19[2]	2.780(7)	C20	C22	3.146(8)

Symmetry Operators:

(1) -X,-Y,-Z+2 (2) -X,-Y+1,-Z+1

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O1	H6	2.687	O1	H7[1]	2.635
O1	H8[1]	2.599	O2	H2[1]	3.423
O2	H7[1]	2.884	O2	H8	3.186
O2	H10B	2.416	O2	H11C	2.869
O3	H8	2.332	O3	H11A	2.561
O3	H11B	3.270	O3	H11C	2.702
O4	H17	2.707	O4	H18[2]	2.642
O4	H19[2]	2.610	O5	H13[2]	3.538
O5	H18[2]	2.896	O5	H19	3.182
O5	H21A	2.398	O5	H22B	2.834
O6	H19	2.347	O6	H22A	2.619
O6	H22B	2.682	O6	H22C	3.285
C1	H3	3.262	C1	H5	3.274
C1	H8[1]	2.709	C2	H4	3.268
C2	H6	3.250	C2	H7	2.561
C2	H11A[1]	3.407	C2	H11C[1]	3.239
C3	H5	3.243	C3	H11A[1]	3.161
C3	H11C[1]	3.374	C4	H2	3.264
C4	H6	3.260	C4	H11A[1]	3.330
C5	H3	3.241	C6	H2	3.252
C6	H4	3.264	C6	H7	3.323
C6	H8[1]	2.885	C7	H2	2.660
C7	H6	2.676	C7	H7[1]	3.159
C7	H8	2.541	C8	H6[1]	3.105
C8	H7	2.538	C8	H8[1]	3.101
C9	H2[1]	3.429	C9	H7[1]	2.706
C9	H10A	3.168	C9	H10B	2.489
C9	H11A	3.409	C9	H11C	2.983
C12	H14	3.272	C12	H16	3.272
C12	H19[2]	2.701	C13	H15	3.270
C13	H17	3.254	C13	H18	2.554
C13	H22A[2]	3.496	C13	H22B[2]	3.292
C14	H16	3.257	C14	H22A[2]	3.276
C14	H22B[2]	3.497	C15	H13	3.268
C15	H17	3.261	C15	H22A[2]	3.527
C16	H14	3.257	C17	H13	3.254
C17	H15	3.264	C17	H18	3.337

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C17	H19[2]	2.870	C18	H13	2.644
C18	H17	2.693	C18	H18[2]	3.182
C18	H19	2.561	C19	H17[2]	3.087
C19	H18	2.562	C19	H19[2]	3.127
C20	H13[2]	3.472	C20	H18[2]	2.695
C20	H21A	2.479	C20	H21B	3.161
C20	H22A	3.475	C20	H22B	2.953
H2	H3	2.342	H2	H7	2.345
H2	H11C[1]	3.215	H3	H4	2.328
H3	H11A[1]	3.419	H3	H11C[1]	3.427
H4	H5	2.331	H5	H6	2.343
H6	H8[1]	2.607	H7	H8	2.326
H7	H8[1]	2.892	H10A	H11A	2.403
H10A	H11B	2.288	H10A	H11C	2.837
H10B	H11A	2.838	H10B	H11B	2.394
H10B	H11C	2.296	H13	H14	2.348
H13	H18	2.325	H13	H22B[2]	3.224
H14	H15	2.342	H14	H22A[2]	3.479
H14	H22B[2]	3.548	H15	H16	2.329
H16	H17	2.344	H17	H19[2]	2.576
H18	H19	2.360	H18	H19[2]	2.904
H21A	H22A	2.847	H21A	H22B	2.330
H21A	H22C	2.368	H21B	H22A	2.379
H21B	H22B	2.846	H21B	H22C	2.320

Symmetry Operators:

(1) -X,-Y,-Z+2 (2) -X,-Y+1,-Z+1

Table 12. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
O1	C10[1]	3.397(5)	O2	O2[1]	3.217(4)
O2	O3[1]	3.505(4)	O2	C5[2]	3.557(6)
O2	C9[1]	3.307(5)	O2	C10[3]	3.338(6)
O3	O2[1]	3.505(4)	O3	C6[4]	3.420(5)
O4	C21[5]	3.360(5)	O5	O5[5]	3.301(5)
O5	O6[5]	3.553(4)	O5	C16[6]	3.420(5)
O5	C17[6]	3.588(5)	O5	C20[5]	3.376(6)
O5	C21[3]	3.389(6)	O6	O5[5]	3.553(4)
O6	C17[4]	3.473(5)	C5	O2[2]	3.557(6)
C6	O3[3]	3.420(5)	C6	C10[1]	3.535(5)
C9	O2[1]	3.307(5)	C10	O1[1]	3.397(5)
C10	O2[4]	3.338(6)	C10	C6[1]	3.535(5)
C16	O5[6]	3.420(5)	C17	O5[6]	3.588(5)
C17	O6[3]	3.473(5)	C17	C21[5]	3.485(7)
C20	O5[5]	3.376(6)	C21	O4[5]	3.360(5)
C21	O5[4]	3.389(6)	C21	C17[5]	3.485(7)

Symmetry Operators:

(1)	-X+1,-Y+1,-Z+2	(2)	-X,-Y+1,-Z+2
(3)	X-1,Y,Z	(4)	X+1,Y,Z
(5)	-X,-Y,-Z+1	(6)	-X-1,-Y,-Z+1

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O1	H5[1]	3.211	O1	H6[1]	3.593
O1	H10A[2]	3.284	O1	H10A[3]	3.576
O1	H10B[3]	2.461	O2	H5[4]	2.891
O2	H6[4]	3.143	O2	H10A[2]	2.408
O2	H10B[3]	3.025	O2	H11B[2]	3.520
O3	H6[1]	2.535	O3	H7[5]	3.135
O3	H8[5]	2.772	O3	H10A[6]	3.569
O4	H16[1]	3.188	O4	H17[1]	3.542
O4	H21A[7]	2.436	O4	H21B[2]	3.429
O4	H21B[7]	3.510	O5	H16[8]	2.762
O5	H17[8]	3.100	O5	H21A[7]	3.058
O5	H21B[2]	2.438	O6	H17[1]	2.565
O6	H18[9]	3.196	O6	H19[9]	2.890
C1	H10B[3]	3.482	C2	H14[4]	3.296
C3	H13[4]	3.023	C3	H14[4]	3.169
C3	H22A[10]	3.472	C3	H22C[10]	3.518
C4	H13[4]	3.100	C4	H14[11]	3.450
C4	H14[4]	3.480	C4	H15[11]	3.483
C4	H22A[10]	3.215	C5	H7[2]	3.501
C5	H8[2]	3.570	C5	H11B[3]	3.055
C6	H8[2]	3.061	C6	H10A[3]	3.213
C6	H10B[3]	3.195	C6	H11B[3]	3.065
C7	H10B[3]	3.515	C8	H6[1]	3.242
C8	H8[5]	3.321	C8	H10B[3]	3.454
C9	H6[1]	3.116	C9	H10A[2]	3.287
C9	H10B[3]	3.290	C10	H6[1]	3.371
C10	H6[3]	3.143	C10	H7[5]	3.279
C10	H10A[6]	3.272	C11	H2[5]	3.586
C11	H7[5]	3.371	C11	H14[1]	3.162
C12	H21A[7]	3.386	C12	H22C[7]	3.484
C13	H3[4]	3.514	C14	H4[11]	3.029
C14	H11B[2]	3.164	C14	H11C[2]	3.218
C15	H3[12]	3.565	C15	H4[11]	3.153
C15	H11C[2]	3.208	C15	H22C[7]	3.461
C16	H18[2]	3.474	C16	H19[2]	3.546
C16	H21B[7]	3.563	C16	H22C[7]	3.082
C17	H19[2]	3.009	C17	H21A[7]	3.192
C17	H21B[7]	3.082	C17	H22C[7]	3.093
C18	H21A[7]	3.459	C19	H17[1]	3.166
C19	H19[9]	3.331	C19	H21A[7]	3.443
C20	H17[1]	3.093	C20	H21A[7]	3.301
C20	H21B[2]	3.361	C21	H16[8]	3.498
C21	H17[1]	3.430	C21	H17[7]	3.213
C21	H18[9]	3.421	C21	H21B[13]	3.555
C22	H3[14]	3.367	C22	H4[14]	3.118
C22	H16[8]	3.374	H2	C11[5]	3.586
H2	H4[1]	3.566	H2	H11A[5]	2.794
H2	H11B[5]	3.568	H2	H14[4]	3.591
H2	H15[12]	3.512	H3	C13[4]	3.514
H3	C15[12]	3.565	H3	C22[10]	3.367
H3	H13[4]	2.770	H3	H14[4]	3.406

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
H3	H15[12]	3.301	H3	H16[12]	3.408
H3	H22A[10]	2.982	H3	H22B[15]	3.089
H3	H22C[10]	2.848	H4	C14[11]	3.029
H4	C15[11]	3.153	H4	C22[10]	3.118
H4	H2[2]	3.566	H4	H13[4]	2.919
H4	H14[11]	2.690	H4	H15[11]	2.922
H4	H22A[10]	2.454	H4	H22B[10]	3.263
H4	H22C[10]	3.178	H5	O1[2]	3.211
H5	O2[4]	2.891	H5	H7[2]	3.464
H5	H8[2]	3.596	H5	H10B[4]	3.047
H5	H11B[3]	3.195	H5	H11C[4]	2.957
H5	H14[11]	3.178	H5	H15[11]	3.261
H6	O1[2]	3.593	H6	O2[4]	3.143
H6	O3[2]	2.535	H6	C8[2]	3.242
H6	C9[2]	3.116	H6	C10[2]	3.371
H6	C10[3]	3.143	H6	H8[2]	2.702
H6	H10A[2]	3.218	H6	H10A[3]	2.657
H6	H10B[3]	2.841	H6	H11B[3]	3.202
H7	O3[5]	3.135	H7	C5[1]	3.501
H7	C10[5]	3.279	H7	C11[5]	3.371
H7	H5[1]	3.464	H7	H10A[5]	2.936
H7	H11A[5]	2.681	H8	O3[5]	2.772
H8	C5[1]	3.570	H8	C6[1]	3.061
H8	C8[5]	3.321	H8	H5[1]	3.596
H8	H6[1]	2.702	H8	H8[5]	2.444
H10A	O1[1]	3.284	H10A	O1[3]	3.576
H10A	O2[1]	2.408	H10A	O3[6]	3.569
H10A	C6[3]	3.213	H10A	C9[1]	3.287
H10A	C10[6]	3.272	H10A	H6[1]	3.218
H10A	H6[3]	2.657	H10A	H7[5]	2.936
H10A	H10A[6]	2.805	H10A	H10B[6]	3.064
H10B	O1[3]	2.461	H10B	O2[3]	3.025
H10B	C1[3]	3.482	H10B	C6[3]	3.195
H10B	C7[3]	3.515	H10B	C8[3]	3.454
H10B	C9[3]	3.290	H10B	H5[4]	3.047
H10B	H6[3]	2.841	H10B	H10A[6]	3.064
H11A	H2[5]	2.794	H11A	H7[5]	2.681
H11B	O2[1]	3.520	H11B	C5[3]	3.055
H11B	C6[3]	3.065	H11B	C14[1]	3.164
H11B	H2[5]	3.568	H11B	H5[3]	3.195
H11B	H6[3]	3.202	H11B	H14[1]	2.628
H11C	C14[1]	3.218	H11C	C15[1]	3.208
H11C	H5[4]	2.957	H11C	H14[1]	2.809
H11C	H15[1]	2.784	H13	C3[4]	3.023
H13	C4[4]	3.100	H13	H3[4]	2.770
H13	H4[4]	2.919	H13	H15[1]	3.540
H13	H22A[9]	2.978	H14	C2[4]	3.296
H14	C3[4]	3.169	H14	C4[11]	3.450
H14	C4[4]	3.480	H14	C11[2]	3.162
H14	H2[4]	3.591	H14	H3[4]	3.406
H14	H4[11]	2.690	H14	H5[11]	3.178

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
H14	H11B[2]	2.628	H14	H11C[2]	2.809
H15	C4[11]	3.483	H15	H2[12]	3.512
H15	H3[12]	3.301	H15	H4[11]	2.922
H15	H5[11]	3.261	H15	H11C[2]	2.784
H15	H13[2]	3.540	H16	O4[2]	3.188
H16	O5[8]	2.762	H16	C21[8]	3.498
H16	C22[8]	3.374	H16	H3[12]	3.408
H16	H18[2]	3.430	H16	H19[2]	3.562
H16	H21A[8]	2.826	H16	H21B[7]	3.523
H16	H22B[8]	2.787	H16	H22C[8]	3.438
H16	H22C[7]	3.359	H17	O4[2]	3.542
H17	O5[8]	3.100	H17	O6[2]	2.565
H17	C19[2]	3.166	H17	C20[2]	3.093
H17	C21[2]	3.430	H17	C21[7]	3.213
H17	H19[2]	2.602	H17	H21A[7]	2.945
H17	H21B[2]	3.308	H17	H21B[7]	2.655
H17	H22C[7]	3.378	H18	O6[9]	3.196
H18	C16[1]	3.474	H18	C21[9]	3.421
H18	H16[1]	3.430	H18	H21B[9]	3.048
H18	H22A[9]	2.948	H19	O6[9]	2.890
H19	C16[1]	3.546	H19	C17[1]	3.009
H19	C19[9]	3.331	H19	H16[1]	3.562
H19	H17[1]	2.602	H19	H19[9]	2.438
H21A	O4[7]	2.436	H21A	O5[7]	3.058
H21A	C12[7]	3.386	H21A	C17[7]	3.192
H21A	C18[7]	3.459	H21A	C19[7]	3.443
H21A	C20[7]	3.301	H21A	H16[8]	2.826
H21A	H17[7]	2.945	H21A	H21B[13]	3.358
H21B	O4[1]	3.429	H21B	O4[7]	3.510
H21B	O5[1]	2.438	H21B	C16[7]	3.563
H21B	C17[7]	3.082	H21B	C20[1]	3.361
H21B	C21[13]	3.555	H21B	H16[7]	3.523
H21B	H17[1]	3.308	H21B	H17[7]	2.655
H21B	H18[9]	3.048	H21B	H21A[13]	3.358
H21B	H21B[13]	3.085	H22A	C3[14]	3.472
H22A	C4[14]	3.215	H22A	H3[14]	2.982
H22A	H4[14]	2.454	H22A	H13[9]	2.978
H22A	H18[9]	2.948	H22B	H3[16]	3.089
H22B	H4[14]	3.263	H22B	H16[8]	2.787
H22C	C3[14]	3.518	H22C	C12[7]	3.484
H22C	C15[7]	3.461	H22C	C16[7]	3.082
H22C	C17[7]	3.093	H22C	H3[14]	2.848
H22C	H4[14]	3.178	H22C	H16[8]	3.438
H22C	H16[7]	3.359	H22C	H17[7]	3.378

Symmetry Operators:

(1)	X+1,Y,Z	(2)	X-1,Y,Z
(3)	-X+1,-Y+1,-Z+2	(4)	-X,-Y+1,-Z+2
(5)	-X+1,-Y,-Z+2	(6)	-X+2,-Y+1,-Z+2

(7) -X,-Y,-Z+1
(8) -X-1,-Y,-Z+1
(9) -X+1,-Y+1,-Z+1
(10) X-1,Y,Z+1
(11) -X-1,-Y+1,-Z+2
(12) -X-1,-Y,-Z+2
(13) -X+1,-Y,-Z+1
(14) X+1,Y,Z-1
(15) X,Y,Z+1
(16) X,Y,Z-1

Crystal Data for ethyl 2-benzyl-4-hydroxy-5-oxo-3-phenyl-2,5-dihydrofuran-2-carboxylate (II)

Experimental

Data Collection

A colorless block crystal of $C_{20}H_{18}O_5$ having approximate dimensions of 0.168 x 0.137 x 0.125 mm was mounted on a glass fiber. All measurements were made on a Rigaku R-AXIS RAPID diffractometer using multi-layer mirror monochromated Cu-Kα radiation.

The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive triclinic cell with dimensions:

a	=	9.6559(3) Å	α	=	84.288(6)^o
b	=	9.9869(3) Å	β	=	81.394(6)^o
c	=	18.8822(5) Å	γ	=	73.131(5)^o
V	=	1719.86(10) Å^3			

For Z = 4 and F.W. = 338.36, the calculated density is 1.307 g/cm^3. 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-1 (#2)

The data were collected at a temperature of -180 $\pm$ 1^{o}C to a maximum 2θ value of 136.4^o. A total of 90 oscillation images were collected. A sweep of data was done using ω scans from 80.0 to 260.0^o in 10.00^o step, at χ=54.0^o and ϕ = 0.0^o. The exposure rate was 5.0 [sec./^{o}]. A second sweep was performed using ω scans from 80.0 to 260.0^o in 10.00^o step, at χ=54.0^o and ϕ = 90.0^o. The exposure rate was 5.0 [sec./^{o}]. Another sweep was performed using ω scans from 80.0 to 260.0^o in 10.00^o step, at χ=54.0^o and ϕ = 180.0^o. The exposure rate was 5.0 [sec./^{o}]. Another sweep was performed using ω scans from 80.0 to 260.0^o in 10.00^o step, at χ=54.0^o and ϕ = 270.0^o. The exposure rate was 5.0 [sec./^{o}]. Another sweep was performed using ω scans from 80.0 to 260.0^o in 10.00^o step, at χ=0.0^o and ϕ = 0.0^o. The exposure rate was 5.0 [sec./^{o}]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 20584 reflections were collected, where 6171 were unique (R_{int} = 0.0339); equivalent reflections were merged.

The linear absorption coefficient, μ, for Cu-Kα radiation is 7.764 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.612 to 0.908. 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. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[2] on F^2 was based on 6171 observed reflections and 455 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \; ||Fo| - |Fc|| \; / \; \Sigma \; |Fo| = 0.0522$$

$$wR2 = [\; \Sigma \; (\; w \; (Fo^2 - Fc^2)^2 \;) / \; \Sigma \; w(Fo^2)^2]^{1/2} = 0.1585$$

The standard deviation of an observation of unit weight[3] was 1.05. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.25 and -0.24 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[4]. Anomalous dispersion effects were included in Fcalc[5]; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley[6]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[7]. All calculations were performed using the CrystalStructure[8] crystallographic software package except for refinement, which was performed using SHELXL-97[9].

References

(1) SHELX97: Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

(2) Least Squares function minimized: (SHELXL97)

$\Sigma w(F_o^2-F_c^2)^2$ where w = Least Squares weights.

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

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

where: N_o = number of observations
N_v = number of variables

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

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

(6) 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).

(7) 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).

(8) CrystalStructure 4.1: Crystal Structure Analysis Package, Rigaku Corporation (2000-2013). Tokyo 196-8666, Japan.

(9) SHELX97: Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{20}H_{18}O_5$
Formula Weight	338.36
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.168 X 0.137 X 0.125 mm
Crystal System	triclinic
Lattice Type	Primitive
Lattice Parameters	a = 9.6559(3) Å b = 9.9869(3) Å c = 18.8822(5) Å α = 84.288(6) ^{o} β = 81.394(6) ^{o} γ = 73.131(5) ^{o} V = 1719.86(10) Å^3
Space Group	P-1 (#2)
Z value	4
D_{calc}	1.307 g/cm^3
F_{000}	712.00
μ(CuKα)	7.764 cm^{-1}

B. Intensity Measurements

Diffractometer	R-AXIS RAPID
Radiation	CuKα (λ = 1.54187 Å) multi-layer mirror monochromated
Voltage, Current	40kV, 30mA
Temperature	-180.0^{o}C
Detector Aperture	460 x 256 mm
Data Images	90 exposures
ω oscillation Range (χ=54.0, ϕ=0.0)	80.0 - 260.0^{o}
Exposure Rate	5.0 sec./^{o}
ω oscillation Range (χ=54.0, ϕ=90.0)	80.0 - 260.0^{o}
Exposure Rate	5.0 sec./^{o}
ω oscillation Range (χ=54.0, ϕ=180.0)	80.0 - 260.0^{o}
Exposure Rate	5.0 sec./^{o}
ω oscillation Range (χ=54.0, ϕ=270.0)	80.0 - 260.0^{o}
Exposure Rate	5.0 sec./^{o}
ω oscillation Range (χ=0.0, ϕ=0.0)	80.0 - 260.0^{o}
Exposure Rate	5.0 sec./^{o}
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	136.4^{o}
No. of Reflections Measured	Total: 20584 Unique: 6171 (R_{int} = 0.0339)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.612 - 0.908)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELX97)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\Sigma\ w\ (Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1/\ [\ \sigma^2(Fo^2) + (0.0886 \cdot P)^2 + 0.0000 \cdot P\ ]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$
$2\theta_{max}$ cutoff	136.4^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	6171
No. Variables	455
Reflection/Parameter Ratio	13.56
Residuals: R1 (I>2.00σ(I))	0.0522
Residuals: R (All reflections)	0.0741
Residuals: wR2 (All reflections)	0.1585
Goodness of Fit Indicator	1.049
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	$0.25\ e^-/Å^3$
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}
O1	0.38040(15)	0.21775(14)	0.71107(7)	3.27(3)
O2	0.46453(16)	0.37458(15)	0.75443(7)	3.83(3)
O3	0.37547(15)	0.55149(14)	0.62569(7)	3.30(3)
O4	0.13712(16)	0.07694(17)	0.66457(9)	4.64(4)
O5	0.08594(16)	0.28266(17)	0.71468(8)	4.40(3)
O6	0.58357(13)	-0.15971(13)	0.75411(6)	2.78(3)
O7	0.48920(14)	-0.30779(14)	0.70904(7)	3.18(3)
O8	0.57202(16)	-0.49154(15)	0.83661(7)	3.60(3)
O9	0.84682(14)	-0.04580(15)	0.80756(7)	3.54(3)
O10	0.87836(14)	-0.23689(15)	0.74605(7)	3.35(3)
C1	0.5688(2)	0.0987(2)	0.58139(11)	3.33(4)
C2	0.6774(2)	0.0666(2)	0.62629(12)	3.88(5)
C3	0.8156(3)	0.0809(2)	0.60191(13)	4.30(5)
C4	0.8461(3)	0.1264(2)	0.53148(14)	4.45(5)
C5	0.7406(3)	0.1593(2)	0.48588(13)	4.23(5)
C6	0.6021(2)	0.1468(2)	0.51097(11)	3.68(4)
C7	0.4194(2)	0.0810(2)	0.60850(11)	3.56(4)
C8	0.3175(2)	0.2065(2)	0.64717(10)	3.10(4)
C9	0.3065(2)	0.3464(2)	0.60497(10)	2.95(4)
C10	0.3612(2)	0.4224(2)	0.64230(10)	2.86(4)
C11	0.4082(2)	0.3432(2)	0.70798(11)	3.20(4)
C12	0.2537(2)	0.3782(2)	0.53452(10)	2.97(4)
C13	0.2847(2)	0.4872(2)	0.48929(10)	3.25(4)
C14	0.2417(2)	0.5144(2)	0.42135(11)	3.70(4)
C15	0.1660(3)	0.4334(2)	0.39748(11)	4.00(5)
C16	0.1351(3)	0.3254(3)	0.44157(12)	5.16(6)
C17	0.1771(3)	0.2975(3)	0.50948(12)	4.39(5)
C18	0.1696(2)	0.1797(2)	0.67584(11)	3.34(4)
C19	-0.0539(2)	0.2662(3)	0.75096(12)	4.36(5)
C20	-0.0809(3)	0.3266(3)	0.82046(13)	5.30(6)
C21	0.4081(2)	-0.0498(2)	0.89002(10)	3.12(4)
C22	0.3854(2)	-0.1009(2)	0.96077(11)	3.51(4)
C23	0.2536(3)	-0.1262(2)	0.98821(13)	4.40(5)
C24	0.1441(3)	-0.1015(3)	0.94545(14)	4.58(5)
C25	0.1633(2)	-0.0491(2)	0.87537(13)	4.29(5)
C26	0.2953(2)	-0.0233(2)	0.84802(12)	3.69(4)
C27	0.5547(2)	-0.0288(2)	0.85839(10)	3.01(4)
C28	0.6508(2)	-0.1553(2)	0.81741(9)	2.68(4)
C29	0.6575(2)	-0.2968(2)	0.85792(10)	2.75(4)
C30	0.5952(2)	-0.3658(2)	0.82041(10)	2.87(4)
C31	0.5489(2)	-0.2817(2)	0.75569(10)	2.84(4)
C32	0.7133(2)	-0.3375(2)	0.92751(10)	2.98(4)
C33	0.7909(2)	-0.2593(2)	0.95484(11)	3.73(5)
C34	0.8335(3)	-0.2939(3)	1.02237(12)	4.76(6)
C35	0.8035(3)	-0.4062(3)	1.06349(12)	4.99(6)
C36	0.7310(3)	-0.4869(3)	1.03659(11)	4.28(5)
C37	0.6854(2)	-0.4524(2)	0.96943(10)	3.37(4)
C38	0.8027(2)	-0.1376(2)	0.79012(10)	2.81(4)
C39	1.0317(2)	-0.2419(2)	0.72260(10)	3.45(4)
C40	1.1248(2)	-0.3338(2)	0.77521(11)	3.98(5)

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

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}
H2	0.65649	0.03413	0.67462	4.654
H3	0.88822	0.05956	0.63334	5.164
H3A	0.41966	0.57237	0.65616	3.960
H4	0.94082	0.13524	0.51424	5.346
H5	0.76267	0.19032	0.43742	5.072
H6	0.52900	0.17138	0.47968	4.421
H7A	0.43035	-0.00353	0.64174	4.276
H7B	0.37435	0.06548	0.56738	4.276
H8	0.53712	-0.51370	0.80281	4.317
H13	0.33617	0.54396	0.50526	3.905
H14	0.26437	0.58902	0.39107	4.434
H15	0.13573	0.45227	0.35102	4.797
H16	0.08383	0.26887	0.42514	6.189
H17	0.15378	0.22277	0.53938	5.264
H19A	-0.13320	0.31496	0.72193	5.229
H19B	-0.05013	0.16572	0.75735	5.229
H20A	-0.17270	0.31461	0.84638	6.358
H20B	-0.08741	0.42673	0.81350	6.358
H20C	-0.00065	0.27918	0.84832	6.358
H22	0.46083	-0.11846	0.99043	4.211
H23	0.23868	-0.16078	1.03664	5.279
H24	0.05455	-0.12066	0.96436	5.495
H25	0.08703	-0.03094	0.84621	5.148
H26	0.30882	0.01308	0.79985	4.429
H27A	0.60556	-0.01388	0.89745	3.618
H27B	0.53917	0.05621	0.82538	3.618
H33	0.81414	-0.18256	0.92675	4.473
H34	0.88420	-0.23925	1.04079	5.715
H35	0.83249	-0.42829	1.11015	5.991
H36	0.71251	-0.56610	1.06427	5.140
H37	0.63443	-0.50758	0.95160	4.047
H39A	1.04347	-0.14621	0.71962	4.137
H39B	1.06223	-0.27952	0.67440	4.137
H40A	1.22691	-0.33521	0.76047	4.771
H40B	1.11562	-0.42928	0.77657	4.771
H40C	1.09282	-0.29719	0.82300	4.771

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O1	0.0527(9)	0.0434(9)	0.0396(8)	-0.0284(7)	-0.0171(7)	0.0047(6)
O2	0.0655(10)	0.0497(10)	0.0442(8)	-0.0313(8)	-0.0255(7)	0.0053(7)
O3	0.0523(9)	0.0405(9)	0.0426(8)	-0.0245(7)	-0.0178(7)	0.0028(6)
O4	0.0532(10)	0.0570(11)	0.0783(12)	-0.0327(8)	-0.0137(8)	-0.0002(9)
O5	0.0540(10)	0.0678(11)	0.0556(10)	-0.0367(9)	0.0041(8)	-0.0092(8)
O6	0.0387(8)	0.0422(8)	0.0328(7)	-0.0221(6)	-0.0111(6)	0.0028(6)
O7	0.0447(8)	0.0486(9)	0.0369(8)	-0.0240(7)	-0.0148(6)	0.0008(6)
O8	0.0607(10)	0.0459(9)	0.0441(8)	-0.0317(8)	-0.0225(7)	0.0063(7)
O9	0.0411(8)	0.0495(9)	0.0524(9)	-0.0251(7)	-0.0098(7)	-0.0002(7)
O10	0.0359(8)	0.0557(10)	0.0418(8)	-0.0223(7)	-0.0041(6)	-0.0045(7)
C1	0.0507(13)	0.0346(12)	0.0472(12)	-0.0191(10)	-0.0127(10)	0.0003(9)
C2	0.0544(14)	0.0467(14)	0.0551(14)	-0.0272(11)	-0.0172(11)	0.0099(11)
C3	0.0521(14)	0.0466(14)	0.0711(16)	-0.0212(11)	-0.0191(12)	0.0066(12)
C4	0.0482(14)	0.0449(14)	0.0774(17)	-0.0192(11)	0.0004(13)	-0.0039(12)
C5	0.0657(16)	0.0425(14)	0.0522(14)	-0.0187(12)	0.0016(12)	-0.0046(11)
C6	0.0536(14)	0.0405(13)	0.0481(13)	-0.0145(11)	-0.0091(11)	-0.0052(10)
C7	0.0523(13)	0.0449(13)	0.0485(13)	-0.0263(11)	-0.0166(10)	0.0021(10)
C8	0.0441(12)	0.0468(13)	0.0352(11)	-0.0223(10)	-0.0149(9)	0.0020(9)
C9	0.0365(11)	0.0430(13)	0.0386(11)	-0.0194(9)	-0.0092(9)	0.0015(9)
C10	0.0400(11)	0.0370(12)	0.0381(11)	-0.0200(9)	-0.0093(9)	0.0013(9)
C11	0.0440(12)	0.0447(13)	0.0411(12)	-0.0231(10)	-0.0120(9)	0.0003(10)
C12	0.0380(11)	0.0428(12)	0.0373(11)	-0.0176(10)	-0.0091(9)	-0.0011(9)
C13	0.0471(12)	0.0435(13)	0.0374(11)	-0.0156(10)	-0.0126(9)	-0.0026(9)
C14	0.0568(14)	0.0501(14)	0.0406(12)	-0.0237(11)	-0.0144(10)	0.0028(10)
C15	0.0646(15)	0.0582(15)	0.0393(12)	-0.0292(12)	-0.0185(11)	0.0033(10)
C16	0.0885(19)	0.0786(19)	0.0527(15)	-0.0523(16)	-0.0341(14)	0.0091(13)
C17	0.0703(16)	0.0674(17)	0.0498(13)	-0.0483(14)	-0.0267(12)	0.0147(12)
C18	0.0499(13)	0.0474(14)	0.0396(12)	-0.0257(11)	-0.0181(10)	0.0061(10)
C19	0.0480(14)	0.0745(18)	0.0514(14)	-0.0338(13)	-0.0047(11)	0.0050(12)
C20	0.0533(15)	0.085(2)	0.0650(17)	-0.0234(14)	0.0015(13)	-0.0126(14)
C21	0.0426(12)	0.0401(12)	0.0425(12)	-0.0209(10)	-0.0062(9)	-0.0051(9)
C22	0.0450(12)	0.0493(14)	0.0431(12)	-0.0199(10)	-0.0026(10)	-0.0061(10)
C23	0.0637(16)	0.0557(16)	0.0518(14)	-0.0289(13)	0.0089(12)	-0.0094(11)
C24	0.0468(14)	0.0606(17)	0.0746(18)	-0.0304(12)	0.0063(13)	-0.0166(13)
C25	0.0400(13)	0.0584(16)	0.0710(17)	-0.0209(11)	-0.0096(12)	-0.0086(13)
C26	0.0417(12)	0.0515(14)	0.0539(13)	-0.0202(11)	-0.0134(10)	-0.0028(11)
C27	0.0429(12)	0.0420(12)	0.0370(11)	-0.0210(10)	-0.0103(9)	-0.0017(9)
C28	0.0374(11)	0.0428(12)	0.0302(10)	-0.0217(9)	-0.0124(8)	0.0014(9)
C29	0.0338(11)	0.0420(12)	0.0357(11)	-0.0204(9)	-0.0086(8)	0.0002(9)
C30	0.0384(11)	0.0384(12)	0.0386(11)	-0.0202(9)	-0.0081(9)	0.0003(9)
C31	0.0322(11)	0.0452(13)	0.0363(11)	-0.0196(9)	-0.0059(8)	-0.0011(9)
C32	0.0394(11)	0.0443(13)	0.0345(11)	-0.0188(10)	-0.0093(9)	0.0016(9)
C33	0.0529(14)	0.0580(15)	0.0438(12)	-0.0342(12)	-0.0180(10)	0.0096(10)
C34	0.0785(17)	0.0741(18)	0.0486(14)	-0.0470(15)	-0.0314(13)	0.0136(12)
C35	0.093(2)	0.0726(18)	0.0443(14)	-0.0486(16)	-0.0343(13)	0.0196(12)
C36	0.0725(16)	0.0600(16)	0.0424(13)	-0.0374(13)	-0.0192(11)	0.0145(11)
C37	0.0498(13)	0.0504(14)	0.0367(11)	-0.0257(11)	-0.0123(10)	0.0019(10)
C38	0.0385(11)	0.0416(12)	0.0326(11)	-0.0186(10)	-0.0135(9)	0.0062(9)
C39	0.0345(11)	0.0610(15)	0.0406(12)	-0.0231(10)	-0.0026(9)	-0.0012(10)
C40	0.0460(13)	0.0602(15)	0.0475(13)	-0.0186(11)	-0.0084(10)	-0.0007(11)

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 4. Fragment Analysis

fragment: 1

O(1)	O(2)	O(3)	O(4)	O(5)
C(1)	C(2)	C(3)	C(4)	C(5)
C(6)	C(7)	C(8)	C(9)	C(10)
C(11)	C(12)	C(13)	C(14)	C(15)
C(16)	C(17)	C(18)	C(19)	C(20)

fragment: 2

O(6)	O(7)	O(8)	O(9)	O(10)
C(21)	C(22)	C(23)	C(24)	C(25)
C(26)	C(27)	C(28)	C(29)	C(30)
C(31)	C(32)	C(33)	C(34)	C(35)
C(36)	C(37)	C(38)	C(39)	C(40)

Table 5. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
O1	C8	1.457(3)	O1	C11	1.349(3)
O2	C11	1.208(3)	O3	C10	1.338(3)
O4	C18	1.203(3)	O5	C18	1.319(2)
O5	C19	1.465(3)	O6	C28	1.453(2)
O6	C31	1.350(3)	O7	C31	1.209(3)
O8	C30	1.337(3)	O9	C38	1.208(3)
O10	C38	1.331(2)	O10	C39	1.468(2)
C1	C2	1.392(3)	C1	C6	1.392(3)
C1	C7	1.511(3)	C2	C3	1.388(3)
C3	C4	1.379(3)	C4	C5	1.378(4)
C5	C6	1.387(3)	C7	C8	1.525(3)
C8	C9	1.521(3)	C8	C18	1.538(3)
C9	C10	1.342(3)	C9	C12	1.468(3)
C10	C11	1.461(3)	C12	C13	1.389(3)
C12	C17	1.395(4)	C13	C14	1.384(3)
C14	C15	1.381(4)	C15	C16	1.371(3)
C16	C17	1.381(3)	C19	C20	1.455(4)
C21	C22	1.391(3)	C21	C26	1.391(3)
C21	C27	1.516(3)	C22	C23	1.384(3)
C23	C24	1.376(4)	C24	C25	1.380(3)
C25	C26	1.387(3)	C27	C28	1.535(3)
C28	C29	1.527(3)	C28	C38	1.535(3)
C29	C30	1.343(3)	C29	C32	1.470(3)
C30	C31	1.463(3)	C32	C33	1.405(4)
C32	C37	1.397(3)	C33	C34	1.379(3)
C34	C35	1.376(4)	C35	C36	1.383(4)
C36	C37	1.383(3)	C39	C40	1.498(3)

Table 6. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
O3	H3A	0.840	O8	H8	0.840
C2	H2	0.950	C3	H3	0.950
C4	H4	0.950	C5	H5	0.950
C6	H6	0.950	C7	H7A	0.990
C7	H7B	0.990	C13	H13	0.950
C14	H14	0.950	C15	H15	0.950
C16	H16	0.950	C17	H17	0.950
C19	H19A	0.990	C19	H19B	0.990
C20	H20A	0.980	C20	H20B	0.980
C20	H20C	0.980	C22	H22	0.950
C23	H23	0.950	C24	H24	0.950
C25	H25	0.950	C26	H26	0.950
C27	H27A	0.990	C27	H27B	0.990
C33	H33	0.950	C34	H34	0.950
C35	H35	0.950	C36	H36	0.950
C37	H37	0.950	C39	H39A	0.990
C39	H39B	0.990	C40	H40A	0.980
C40	H40B	0.980	C40	H40C	0.980

Table 7. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
C8	O1	C11	109.48(15)	C18	O5	C19	117.3(2)
C28	O6	C31	110.00(14)	C38	O10	C39	116.69(18)
C2	C1	C6	118.1(2)	C2	C1	C7	120.74(18)
C6	C1	C7	121.1(2)	C1	C2	C3	121.4(2)
C2	C3	C4	119.2(2)	C3	C4	C5	120.7(2)
C4	C5	C6	119.8(2)	C1	C6	C5	120.8(2)
C1	C7	C8	113.4(2)	O1	C8	C7	107.28(15)
O1	C8	C9	104.60(18)	O1	C8	C18	104.81(15)
C7	C8	C9	114.00(15)	C7	C8	C18	110.82(19)
C9	C8	C18	114.44(16)	C8	C9	C10	107.00(17)
C8	C9	C12	122.9(2)	C10	C9	C12	129.96(19)
O3	C10	C9	128.38(18)	O3	C10	C11	121.4(2)
C9	C10	C11	110.18(19)	O1	C11	O2	121.33(18)
O1	C11	C10	108.72(19)	O2	C11	C10	130.0(2)
C9	C12	C13	120.4(2)	C9	C12	C17	121.67(19)
C13	C12	C17	117.90(19)	C12	C13	C14	121.1(2)
C13	C14	C15	120.2(2)	C14	C15	C16	119.3(2)
C15	C16	C17	121.0(3)	C12	C17	C16	120.5(2)
O4	C18	O5	125.3(2)	O4	C18	C8	123.77(18)
O5	C18	C8	111.0(2)	O5	C19	C20	107.9(2)
C22	C21	C26	118.7(2)	C22	C21	C27	120.70(19)
C26	C21	C27	120.59(17)	C21	C22	C23	120.4(2)
C22	C23	C24	120.1(2)	C23	C24	C25	120.6(2)
C24	C25	C26	119.2(2)	C21	C26	C25	121.0(2)
C21	C27	C28	111.68(18)	O6	C28	C27	107.42(14)
O6	C28	C29	104.35(17)	O6	C28	C38	106.34(13)
C27	C28	C29	114.20(14)	C27	C28	C38	111.32(18)
C29	C28	C38	112.52(15)	C28	C29	C30	106.96(17)
C28	C29	C32	123.68(19)	C30	C29	C32	129.20(19)
O8	C30	C29	128.24(18)	O8	C30	C31	121.5(2)
C29	C30	C31	110.24(19)	O6	C31	O7	121.84(17)
O6	C31	C30	108.42(18)	O7	C31	C30	129.7(2)
C29	C32	C33	121.14(19)	C29	C32	C37	120.5(2)
C33	C32	C37	118.28(19)	C32	C33	C34	120.1(2)
C33	C34	C35	121.0(3)	C34	C35	C36	119.7(2)
C35	C36	C37	120.1(2)	C32	C37	C36	120.8(2)
O9	C38	O10	125.18(18)	O9	C38	C28	123.64(16)
O10	C38	C28	111.18(19)	O10	C39	C40	109.27(16)

Table 8. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C10	O3	H3A	109.5	C30	O8	H8	109.5
C1	C2	H2	119.3	C3	C2	H2	119.3
C2	C3	H3	120.4	C4	C3	H3	120.4
C3	C4	H4	119.6	C5	C4	H4	119.7
C4	C5	H5	120.1	C6	C5	H5	120.1
C1	C6	H6	119.6	C5	C6	H6	119.6
C1	C7	H7A	108.9	C1	C7	H7B	108.9
C8	C7	H7A	108.9	C8	C7	H7B	108.9
H7A	C7	H7B	107.7	C12	C13	H13	119.4
C14	C13	H13	119.4	C13	C14	H14	119.9
C15	C14	H14	119.9	C14	C15	H15	120.4
C16	C15	H15	120.4	C15	C16	H16	119.5
C17	C16	H16	119.5	C12	C17	H17	119.8
C16	C17	H17	119.7	O5	C19	H19A	110.1
O5	C19	H19B	110.1	C20	C19	H19A	110.1
C20	C19	H19B	110.1	H19A	C19	H19B	108.4
C19	C20	H20A	109.5	C19	C20	H20B	109.5
C19	C20	H20C	109.5	H20A	C20	H20B	109.5
H20A	C20	H20C	109.5	H20B	C20	H20C	109.5
C21	C22	H22	119.8	C23	C22	H22	119.8
C22	C23	H23	119.9	C24	C23	H23	119.9
C23	C24	H24	119.7	C25	C24	H24	119.7
C24	C25	H25	120.4	C26	C25	H25	120.4
C21	C26	H26	119.5	C25	C26	H26	119.5
C21	C27	H27A	109.3	C21	C27	H27B	109.3
C28	C27	H27A	109.3	C28	C27	H27B	109.3
H27A	C27	H27B	107.9	C32	C33	H33	120.0
C34	C33	H33	120.0	C33	C34	H34	119.5
C35	C34	H34	119.5	C34	C35	H35	120.1
C36	C35	H35	120.1	C35	C36	H36	120.0
C37	C36	H36	120.0	C32	C37	H37	119.6
C36	C37	H37	119.6	O10	C39	H39A	109.8
O10	C39	H39B	109.8	C40	C39	H39A	109.8
C40	C39	H39B	109.8	H39A	C39	H39B	108.3
C39	C40	H40A	109.5	C39	C40	H40B	109.5
C39	C40	H40C	109.5	H40A	C40	H40B	109.5
H40A	C40	H40C	109.5	H40B	C40	H40C	109.5

Table 9. Torsion Angles(O)
(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C8	O1	C11	O2	-177.97(15)	C8	O1	C11	C10	1.39(18)
C11	O1	C8	C7	119.85(15)	C11	O1	C8	C9	-1.56(17)
C11	O1	C8	C18	-122.29(14)	C18	O5	C19	C20	142.91(17)
C19	O5	C18	O4	4.3(3)	C19	O5	C18	C8	-174.79(14)
C28	O6	C31	O7	178.65(14)	C28	O6	C31	C30	-1.18(16)
C31	O6	C28	C27	-119.72(14)	C31	O6	C28	C29	1.86(15)
C31	O6	C28	C38	121.00(13)	C38	O10	C39	C40	88.84(18)
C39	O10	C38	O9	6.1(3)	C39	O10	C38	C28	-173.17(12)
C2	C1	C6	C5	1.4(3)	C6	C1	C2	C3	-0.4(3)
C2	C1	C7	C8	84.5(2)	C7	C1	C2	C3	179.28(17)
C6	C1	C7	C8	-95.9(2)	C7	C1	C6	C5	-178.25(16)
C1	C2	C3	C4	-0.7(3)	C2	C3	C4	C5	0.9(3)
C3	C4	C5	C6	0.2(3)	C4	C5	C6	C1	-1.3(3)
C1	C7	C8	O1	-64.0(2)	C1	C7	C8	C9	51.3(2)
C1	C7	C8	C18	-177.86(14)	O1	C8	C9	C10	1.13(17)
O1	C8	C9	C12	177.42(13)	O1	C8	C18	O4	-120.1(2)
O1	C8	C18	O5	59.06(19)	C7	C8	C9	C10	-115.73(18)
C7	C8	C9	C12	60.6(2)	C7	C8	C18	O4	-4.7(3)
C7	C8	C18	O5	174.47(15)	C9	C8	C18	O4	125.9(2)
C9	C8	C18	O5	-54.9(2)	C18	C8	C9	C10	115.24(18)
C18	C8	C9	C12	-68.5(2)	C8	C9	C10	O3	179.22(15)
C8	C9	C10	C11	-0.35(19)	C8	C9	C12	C13	-161.15(15)
C8	C9	C12	C17	16.0(2)	C10	C9	C12	C13	14.2(3)
C10	C9	C12	C17	-168.62(17)	C12	C9	C10	O3	3.3(3)
C12	C9	C10	C11	-176.28(16)	O3	C10	C11	O1	179.75(14)
O3	C10	C11	O2	-1.0(3)	C9	C10	C11	O1	-0.6(2)
C9	C10	C11	O2	178.64(18)	C9	C12	C13	C14	176.89(14)
C9	C12	C17	C16	-176.73(15)	C13	C12	C17	C16	0.5(3)
C17	C12	C13	C14	-0.4(3)	C12	C13	C14	C15	0.4(3)
C13	C14	C15	C16	-0.5(3)	C14	C15	C16	C17	0.6(3)
C15	C16	C17	C12	-0.6(3)	C22	C21	C26	C25	-1.0(3)
C26	C21	C22	C23	0.8(3)	C22	C21	C27	C28	95.5(2)
C27	C21	C22	C23	-176.85(16)	C26	C21	C27	C28	-82.1(2)
C27	C21	C26	C25	176.62(16)	C21	C22	C23	C24	0.2(3)
C22	C23	C24	C25	-1.1(3)	C23	C24	C25	C26	0.9(3)
C24	C25	C26	C21	0.2(3)	C21	C27	C28	O6	67.96(18)
C21	C27	C28	C29	-47.2(2)	C21	C27	C28	C38	-176.00(13)
O6	C28	C29	C30	-1.88(16)	O6	C28	C29	C32	-177.73(12)
O6	C28	C38	O9	126.47(17)	O6	C28	C38	O10	-54.27(18)
C27	C28	C29	C30	115.11(17)	C27	C28	C29	C32	-60.7(2)
C27	C28	C38	O9	9.8(2)	C27	C28	C38	O10	-170.97(13)
C29	C28	C38	O9	-119.88(19)	C29	C28	C38	O10	59.4(2)
C38	C28	C29	C30	-116.73(16)	C38	C28	C29	C32	67.4(2)
C28	C29	C30	O8	-177.49(15)	C28	C29	C30	C31	1.25(18)
C28	C29	C32	C33	-12.7(2)	C28	C29	C32	C37	164.82(14)
C30	C29	C32	C33	172.39(16)	C30	C29	C32	C37	-10.1(3)
C32	C29	C30	O8	-1.9(3)	C32	C29	C30	C31	176.79(15)
O8	C30	C31	O6	178.74(14)	O8	C30	C31	O7	-1.1(3)
C29	C30	C31	O6	-0.09(19)	C29	C30	C31	O7	-179.91(16)
C29	C32	C33	C34	175.54(14)	C29	C32	C37	C36	-176.61(14)

Table 9. Torsion angles (°) (continued)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C33	C32	C37	C36	1.0(2)	C37	C32	C33	C34	-2.1(3)
C32	C33	C34	C35	1.3(3)	C33	C34	C35	C36	0.7(3)
C34	C35	C36	C37	-1.7(3)	C35	C36	C37	C32	0.9(3)

Table 10. Possible hydrogen bonds

Donor	H	Acceptor	D...A	D-H	H...A	D-H...A	
O3	H3A	O2	2.9426(18)	0.84	2.56	109.10	intramol.
O3	H3A	O7[1]	2.741(2)	0.84	1.95	157.53	
O8	H8	O2[2]	2.641(2)	0.84	1.85	156.49	
O8	H8	O7	2.9418(18)	0.84	2.56	109.04	intramol.

Symmetry Operators:

(1) X,Y+1,Z (2) X,Y-1,Z

Table 11. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
O1	O3	3.5457(19)	O1	O4	3.324(3)
O1	O5	2.718(2)	O1	C1	2.951(2)
O1	C2	3.108(2)	O2	O3	2.9426(18)
O2	C8	3.443(3)	O2	C9	3.474(3)
O3	C12	3.117(3)	O3	C13	3.024(3)
O4	C7	2.782(3)	O4	C9	3.547(3)
O4	C17	3.528(3)	O4	C19	2.707(3)
O5	C9	2.905(2)	O5	C10	3.400(3)
O5	C11	3.320(3)	O6	O8	3.5435(19)
O6	O9	3.384(2)	O6	O10	2.7092(18)
O6	C21	2.973(2)	O6	C26	3.091(2)
O7	O8	2.9418(18)	O7	C28	3.449(3)
O7	C29	3.477(3)	O8	C32	3.101(3)
O8	C37	2.984(3)	O9	C27	2.802(2)
O9	C29	3.492(3)	O9	C33	3.407(2)
O9	C39	2.708(2)	O9	C40	3.351(2)
O10	C29	2.917(2)	O10	C30	3.412(3)
O10	C31	3.315(3)	C1	C4	2.785(3)
C1	C9	2.995(3)	C1	C10	3.475(3)
C1	C11	3.454(3)	C2	C5	2.759(3)
C2	C8	3.327(3)	C2	C11	3.500(3)
C3	C6	2.771(3)	C6	C8	3.429(3)
C6	C9	3.340(3)	C6	C12	3.478(3)
C7	C10	3.400(3)	C7	C11	3.339(3)
C7	C12	3.226(3)	C7	C17	3.348(3)
C8	C17	3.048(3)	C10	C13	3.050(3)
C10	C18	3.412(3)	C11	C18	3.338(4)
C12	C15	2.796(3)	C12	C18	3.308(3)
C13	C16	2.746(4)	C14	C17	2.758(3)
C17	C18	3.241(3)	C18	C20	3.496(3)
C21	C24	2.778(3)	C21	C29	2.943(2)
C21	C30	3.429(3)	C21	C31	3.457(3)
C21	C32	3.570(3)	C22	C25	2.774(3)
C22	C28	3.405(3)	C22	C29	3.278(3)
C22	C32	3.370(3)	C23	C26	2.758(3)
C26	C28	3.284(3)	C26	C31	3.413(3)
C27	C30	3.410(3)	C27	C31	3.351(3)
C27	C32	3.258(3)	C27	C33	3.344(3)
C28	C33	3.057(3)	C30	C37	3.039(3)
C30	C38	3.404(3)	C31	C38	3.348(3)
C32	C35	2.792(3)	C32	C38	3.280(3)
C33	C36	2.771(3)	C33	C38	3.223(3)
C34	C37	2.758(4)	C38	C40	3.147(3)

Table 12. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O1	H2	2.794	O1	H7A	2.569
O1	H7B	3.255	O2	H3A	2.559
O3	H13	2.373	O4	H7A	2.689
O4	H7B	2.694	O4	H17	2.659
O4	H19A	3.115	O4	H19B	2.357
O5	H17	3.356	O5	H20A	3.219
O5	H20B	2.560	O5	H20C	2.537
O6	H26	2.778	O6	H27A	3.260
O6	H27B	2.552	O7	H8	2.559
O8	H37	2.317	O9	H27A	2.627
O9	H27B	2.826	O9	H33	2.541
O9	H39A	2.377	O9	H39B	3.589
O9	H40C	2.938	O10	H33	3.444
O10	H40A	3.266	O10	H40B	2.625
O10	H40C	2.601	C1	H3	3.280
C1	H5	3.272	C2	H4	3.245
C2	H6	3.248	C2	H7A	2.643
C2	H7B	3.285	C3	H5	3.252
C4	H2	3.242	C4	H6	3.247
C5	H3	3.254	C6	H2	3.247
C6	H4	3.249	C6	H7A	3.253
C6	H7B	2.606	C7	H2	2.672
C7	H6	2.683	C7	H17	2.968
C8	H2	3.318	C8	H6	3.488
C8	H17	2.723	C9	H3A	3.066
C9	H6	3.223	C9	H7A	3.386
C9	H7B	2.827	C9	H13	2.636
C9	H17	2.672	C10	H13	2.760
C11	H2	3.363	C11	H3A	2.424
C12	H6	2.970	C12	H7B	3.036
C12	H14	3.269	C12	H16	3.264
C13	H6	3.349	C13	H15	3.256
C13	H17	3.247	C14	H16	3.232
C15	H13	3.250	C15	H17	3.247
C16	H14	3.233	C17	H6	3.245
C17	H7B	2.784	C17	H13	3.247
C17	H15	3.253	C18	H7A	2.679
C18	H7B	2.711	C18	H17	2.590
C18	H19A	2.883	C18	H19B	2.458
C18	H20C	3.520	C21	H23	3.264
C21	H25	3.275	C22	H24	3.248
C22	H26	3.251	C22	H27A	2.609
C22	H27B	3.258	C23	H25	3.252
C24	H22	3.248	C24	H26	3.243
C25	H23	3.250	C26	H22	3.254
C26	H24	3.245	C26	H27A	3.298
C26	H27B	2.660	C27	H22	2.681
C27	H26	2.674	C27	H33	2.940
C28	H22	3.497	C28	H26	3.292
C28	H33	2.727	C29	H8	3.066
C29	H22	3.247	C29	H27A	2.877

Table 12. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
C29	H27B	3.399	C29	H33	2.676
C29	H37	2.650	C30	H37	2.749
C31	H8	2.426	C31	H26	3.269
C32	H22	2.949	C32	H27A	3.116
C32	H34	3.268	C32	H36	3.273
C33	H22	3.088	C33	H27A	2.809
C33	H35	3.253	C33	H37	3.266
C33	H40C	3.495	C34	H36	3.244
C35	H33	3.252	C35	H37	3.251
C36	H34	3.242	C37	H22	3.434
C37	H33	3.267	C37	H35	3.255
C38	H27A	2.690	C38	H27B	2.749
C38	H33	2.587	C38	H39A	2.483
C38	H39B	3.159	C38	H40B	3.534
C38	H40C	2.916	H2	H3	2.334
H2	H7A	2.495	H2	H7B	3.557
H3	H4	2.330	H3A	H13	3.135
H4	H5	2.325	H5	H6	2.333
H6	H7A	3.515	H6	H7B	2.433
H6	H17	3.532	H7B	H17	2.348
H8	H37	3.104	H13	H14	2.325
H14	H15	2.335	H15	H16	2.319
H16	H17	2.319	H19A	H20A	2.323
H19A	H20B	2.309	H19A	H20C	2.818
H19B	H20A	2.306	H19B	H20B	2.818
H19B	H20C	2.326	H22	H23	2.331
H22	H27A	2.424	H22	H27B	3.508
H22	H33	3.339	H23	H24	2.321
H24	H25	2.331	H25	H26	2.336
H26	H27A	3.565	H26	H27B	2.507
H27A	H33	2.319	H33	H34	2.321
H33	H40C	3.077	H34	H35	2.321
H35	H36	2.335	H36	H37	2.327
H39A	H40A	2.340	H39A	H40B	2.848
H39A	H40C	2.364	H39B	H40A	2.358
H39B	H40B	2.345	H39B	H40C	2.848

Table 13. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
O1	C26	3.526(3)	O2	O7[1]	3.270(2)
O2	O8[1]	2.641(2)	O2	C30[1]	3.589(3)
O3	O7[1]	2.741(2)	O3	C5[2]	3.414(2)
O3	C13[2]	3.596(2)	O3	C14[2]	3.551(3)
O3	C40[3]	3.483(2)	O4	C3[4]	3.473(3)
O6	C2	3.347(3)	O7	O2[5]	3.270(2)
O7	O3[5]	2.741(2)	O7	C15[6]	3.574(2)
O8	O2[5]	2.641(2)	O8	C20[7]	3.301(3)
O8	C36[8]	3.536(3)	O9	C19[9]	3.541(3)
O9	C25[9]	3.476(3)	C2	O6	3.347(3)
C3	O4[9]	3.473(3)	C4	C4[10]	3.436(3)
C5	O3[2]	3.414(2)	C5	C13[2]	3.542(3)
C6	C7[6]	3.427(3)	C7	C6[6]	3.427(3)
C13	O3[2]	3.596(2)	C13	C5[2]	3.542(3)
C14	O3[2]	3.551(3)	C15	O7[6]	3.574(2)
C19	O9[4]	3.541(3)	C20	O8[3]	3.301(3)
C24	C24[11]	3.456(3)	C25	O9[4]	3.476(3)
C26	O1	3.526(3)	C30	O2[5]	3.589(3)
C36	O8[8]	3.536(3)	C40	O3[7]	3.483(2)

Symmetry Operators:

(1) X,Y+1,Z
(2) -X+1,-Y+1,-Z+1
(3) X-1,Y+1,Z
(4) X-1,Y,Z
(5) X,Y-1,Z
(6) -X+1,-Y,-Z+1
(7) X+1,Y-1,Z
(8) -X+1,-Y-1,-Z+2
(9) X+1,Y,Z
(10) -X+2,-Y,-Z+1
(11) -X,-Y,-Z+2

Table 14. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O1	H26	2.694	O1	H27B	2.911
O2	H8[1]	1.849	O2	H14[2]	3.568
O2	H27B	3.246	O2	H35[3]	3.498
O2	H40A[4]	3.131	O2	H40B[4]	3.358
O3	H5[2]	2.778	O3	H6[2]	3.508
O3	H13[2]	3.412	O3	H14[2]	3.330
O3	H39B[4]	3.059	O3	H40A[4]	2.890
O3	H40B[4]	3.484	O4	H3[5]	2.615
O4	H4[5]	3.554	O4	H5[6]	3.277
O4	H25	3.497	O4	H26	3.164
O4	H39A[5]	2.700	O5	H15[7]	3.159
O5	H26	3.358	O5	H40B[4]	3.312
O6	H2	2.528	O6	H7A	2.840
O7	H3A[8]	1.946	O7	H7A	3.101
O7	H14[6]	3.599	O7	H15[6]	3.542
O7	H40A[5]	2.654	O8	H19A[9]	3.510
O8	H20A[9]	2.675	O8	H20B[9]	3.123
O8	H36[10]	3.023	O9	H2	3.232
O9	H3	3.354	O9	H19B[11]	2.625
O9	H25[11]	2.581	O10	H2	3.242
O10	H3	3.486	O10	H15[6]	3.006
O10	H16[6]	3.236	O10	H20B[9]	3.413
C1	H6[6]	3.453	C1	H7B[6]	3.289
C3	H4[12]	3.384	C3	H14[2]	3.172
C3	H16[6]	3.412	C3	H19A[11]	3.593
C3	H39A	3.534	C4	H4[12]	2.933
C4	H13[2]	3.311	C4	H14[2]	3.155
C4	H16[11]	3.358	C4	H17[11]	3.405
C5	H4[12]	3.586	C5	H7B[6]	3.084
C5	H13[2]	2.855	C5	H14[2]	3.575
C6	H7A[6]	3.437	C6	H7B[6]	2.642
C6	H13[2]	3.288	C7	H6[6]	3.038
C8	H26	3.311	C10	H14[2]	3.547
C10	H39B[4]	3.523	C10	H40A[4]	3.303
C10	H40B[4]	3.340	C11	H8[1]	3.001
C11	H27B	3.493	C11	H40A[4]	3.349
C11	H40B[4]	3.254	C13	H5[2]	3.513
C13	H13[2]	3.600	C14	H3A[2]	3.280
C14	H19A[7]	3.196	C15	H19A[7]	3.182
C15	H39B[6]	3.512	C16	H4[5]	3.128
C16	H39B[6]	3.254	C17	H4[5]	3.146
C18	H3[5]	3.493	C18	H26	2.988
C19	H3[5]	3.385	C19	H14[7]	3.545
C19	H15[7]	3.213	C19	H25	3.350
C20	H23[13]	3.419	C20	H25	3.485
C20	H34[3]	3.362	C20	H35[3]	3.334
C20	H37[4]	3.545	C20	H40B[4]	3.470
C21	H22[3]	3.517	C21	H34[3]	3.595
C22	H22[3]	3.248	C22	H27A[3]	3.037
C23	H20A[13]	3.586	C23	H24[13]	3.335
C23	H27A[3]	3.325	C23	H33[3]	3.467

Table 14. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
C23	H34[3]	3.521	C23	H36[10]	3.233
C24	H24[13]	2.968	C24	H33[5]	3.581
C24	H34[5]	3.405	C24	H34[3]	3.367
C24	H36[10]	3.217	C24	H40C[5]	3.349
C25	H19B	3.417	C25	H20C	3.227
C25	H34[3]	3.307	C25	H40C[5]	3.041
C26	H20C	3.504	C26	H34[3]	3.414
C27	H2	3.508	C27	H22[3]	3.303
C28	H2	3.139	C29	H20B[9]	3.208
C30	H20A[9]	3.364	C30	H20B[9]	3.155
C31	H3A[8]	3.085	C31	H7A	3.370
C31	H15[6]	3.474	C31	H40A[5]	3.291
C32	H20B[9]	3.320	C33	H24[11]	3.265
C34	H20C[3]	3.159	C34	H24[11]	3.133
C35	H20C[3]	3.283	C35	H40B[14]	3.377
C35	H40C[14]	3.468	C36	H37[10]	3.518
C36	H40C[14]	3.557	C37	H20A[9]	3.311
C37	H20B[9]	3.489	C37	H37[10]	3.337
C38	H2	2.909	C38	H3	3.518
C38	H25[11]	3.547	C39	H3	3.330
C39	H5[12]	3.439	C39	H15[6]	3.491
C39	H16[6]	3.218	C39	H25[11]	3.490
C40	H3A[9]	3.315	C40	H20B[9]	3.535
C40	H25[11]	3.332	C40	H35[14]	3.029
C40	H36[14]	3.545	H2	O6	2.528
H2	O9	3.232	H2	O10	3.242
H2	C27	3.508	H2	C28	3.139
H2	C38	2.909	H2	H27B	2.907
H3	O4[11]	2.615	H3	O9	3.354
H3	O10	3.486	H3	C18[11]	3.493
H3	C19[11]	3.385	H3	C38	3.518
H3	C39	3.330	H3	H4[12]	3.480
H3	H14[2]	3.403	H3	H16[6]	3.488
H3	H17[11]	3.587	H3	H19A[11]	3.128
H3	H19B[11]	2.870	H3	H39A	2.720
H3	H39B	3.385	H3A	O7[1]	1.946
H3A	C14[2]	3.280	H3A	C31[1]	3.085
H3A	C40[4]	3.315	H3A	H5[2]	3.085
H3A	H6[2]	3.525	H3A	H8[1]	3.097
H3A	H14[2]	3.053	H3A	H39B[4]	3.317
H3A	H40A[4]	2.542	H3A	H40B[4]	3.437
H4	O4[11]	3.554	H4	C3[12]	3.384
H4	C4[12]	2.933	H4	C5[12]	3.586
H4	C16[11]	3.128	H4	C17[11]	3.146
H4	H3[12]	3.480	H4	H4[12]	2.684
H4	H13[2]	3.555	H4	H14[2]	3.385
H4	H16[11]	2.539	H4	H17[11]	2.576
H5	O3[2]	2.778	H5	O4[6]	3.277
H5	C13[2]	3.513	H5	C39[12]	3.439
H5	H3A[2]	3.085	H5	H7A[6]	3.549
H5	H7B[6]	3.214	H5	H13[2]	2.814

Table 14. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
H5	H16[11]	3.383	H5	H39A[12]	3.257
H5	H39B[12]	2.746	H6	O3[2]	3.508
H6	C1[6]	3.453	H6	C7[6]	3.038
H6	H3A[2]	3.525	H6	H7A[6]	2.887
H6	H7B[6]	2.479	H6	H13[2]	3.504
H7A	O6	2.840	H7A	O7	3.101
H7A	C6[6]	3.437	H7A	C31	3.370
H7A	H5[6]	3.549	H7A	H6[6]	2.887
H7A	H26	3.042	H7B	C1[6]	3.289
H7B	C5[6]	3.084	H7B	C6[6]	2.642
H7B	H5[6]	3.214	H7B	H6[6]	2.479
H7B	H7B[6]	3.324	H8	O2[8]	1.849
H8	C11[8]	3.001	H8	H3A[8]	3.097
H8	H19A[9]	3.361	H8	H20A[9]	3.015
H8	H20B[9]	3.533	H8	H36[10]	3.196
H8	H40A[5]	3.182	H13	O3[2]	3.412
H13	C4[2]	3.311	H13	C5[2]	2.855
H13	C6[2]	3.288	H13	C13[2]	3.600
H13	H4[2]	3.555	H13	H5[2]	2.814
H13	H6[2]	3.504	H13	H13[2]	3.009
H14	O2[2]	3.568	H14	O3[2]	3.330
H14	O7[6]	3.599	H14	C3[2]	3.172
H14	C4[2]	3.155	H14	C5[2]	3.575
H14	C10[2]	3.547	H14	C19[7]	3.545
H14	H3[2]	3.403	H14	H3A[2]	3.053
H14	H4[2]	3.385	H14	H19A[7]	2.602
H15	O5[7]	3.159	H15	O7[6]	3.542
H15	O10[6]	3.006	H15	C19[7]	3.213
H15	C31[6]	3.474	H15	C39[6]	3.491
H15	H19A[7]	2.577	H15	H20B[7]	3.269
H15	H39B[6]	3.035	H16	O10[6]	3.236
H16	C3[6]	3.412	H16	C4[5]	3.358
H16	C39[6]	3.218	H16	H3[6]	3.488
H16	H4[5]	2.539	H16	H5[5]	3.383
H16	H39B[6]	2.490	H17	C4[5]	3.405
H17	H3[5]	3.587	H17	H4[5]	2.576
H19A	O8[4]	3.510	H19A	C3[5]	3.593
H19A	C14[7]	3.196	H19A	C15[7]	3.182
H19A	H3[5]	3.128	H19A	H8[4]	3.361
H19A	H14[7]	2.602	H19A	H15[7]	2.577
H19B	O9[5]	2.625	H19B	C25	3.417
H19B	H3[5]	2.870	H19B	H25	2.632
H19B	H26	3.536	H19B	H39A[5]	3.105
H20A	O8[4]	2.675	H20A	C23[13]	3.586
H20A	C30[4]	3.364	H20A	C37[4]	3.311
H20A	H8[4]	3.015	H20A	H23[13]	2.668
H20A	H37[4]	2.871	H20B	O8[4]	3.123
H20B	O10[4]	3.413	H20B	C29[4]	3.208
H20B	C30[4]	3.155	H20B	C32[4]	3.320
H20B	C37[4]	3.489	H20B	C40[4]	3.535
H20B	H8[4]	3.533	H20B	H15[7]	3.269

Table 14. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
H20B	H35[3]	3.037	H20B	H37[4]	3.416
H20B	H40B[4]	2.725	H20C	C25	3.227
H20C	C26	3.504	H20C	C34[3]	3.159
H20C	C35[3]	3.283	H20C	H23[13]	3.312
H20C	H25	2.967	H20C	H26	3.448
H20C	H34[3]	2.467	H20C	H35[3]	2.736
H20C	H40B[4]	3.497	H22	C21[3]	3.517
H22	C22[3]	3.248	H22	C27[3]	3.303
H22	H22[3]	2.755	H22	H27A[3]	2.510
H22	H27B[3]	3.593	H23	C20[13]	3.419
H23	H20A[13]	2.668	H23	H20C[13]	3.312
H23	H24[13]	3.362	H23	H27A[3]	3.061
H23	H33[3]	3.442	H23	H36[10]	3.363
H23	H37[10]	3.178	H24	C23[13]	3.335
H24	C24[13]	2.968	H24	C33[5]	3.265
H24	C34[5]	3.133	H24	H23[13]	3.362
H24	H24[13]	2.735	H24	H33[5]	2.773
H24	H34[5]	2.504	H24	H36[10]	3.321
H24	H40C[5]	3.261	H25	O4	3.497
H25	O9[5]	2.581	H25	C19	3.350
H25	C20	3.485	H25	C38[5]	3.547
H25	C39[5]	3.490	H25	C40[5]	3.332
H25	H19B	2.632	H25	H20C	2.967
H25	H33[5]	3.494	H25	H39A[5]	2.889
H25	H40A[5]	3.409	H25	H40C[5]	2.721
H26	O1	2.694	H26	O4	3.164
H26	O5	3.358	H26	C8	3.311
H26	C18	2.988	H26	H7A	3.042
H26	H19B	3.536	H26	H20C	3.448
H27A	C22[3]	3.037	H27A	C23[3]	3.325
H27A	H22[3]	2.510	H27A	H23[3]	3.061
H27B	O1	2.911	H27B	O2	3.246
H27B	C11	3.493	H27B	H2	2.907
H27B	H22[3]	3.593	H33	C23[3]	3.467
H33	C24[11]	3.581	H33	H23[3]	3.442
H33	H24[11]	2.773	H33	H25[11]	3.494
H34	C20[3]	3.362	H34	C21[3]	3.595
H34	C23[3]	3.521	H34	C24[11]	3.405
H34	C24[3]	3.367	H34	C25[3]	3.307
H34	C26[3]	3.414	H34	H20C[3]	2.467
H34	H24[11]	2.504	H35	O2[3]	3.498
H35	C20[3]	3.334	H35	C40[14]	3.029
H35	H20B[3]	3.037	H35	H20C[3]	2.736
H35	H40A[14]	3.331	H35	H40B[14]	2.480
H35	H40C[14]	2.840	H36	O8[10]	3.023
H36	C23[10]	3.233	H36	C24[10]	3.217
H36	C40[14]	3.545	H36	H8[10]	3.196
H36	H23[10]	3.363	H36	H24[10]	3.321
H36	H37[10]	3.268	H36	H40A[14]	3.444
H36	H40C[14]	3.010	H37	C20[9]	3.545
H37	C36[10]	3.518	H37	C37[10]	3.337

Table 14. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
H37	H20A[9]	2.871	H37	H20B[9]	3.416
H37	H23[10]	3.178	H37	H36[10]	3.268
H37	H37[10]	2.917	H39A	O4[11]	2.700
H39A	C3	3.534	H39A	H3	2.720
H39A	H5[12]	3.257	H39A	H19B[11]	3.105
H39A	H25[11]	2.889	H39B	O3[9]	3.059
H39B	C10[9]	3.523	H39B	C15[6]	3.512
H39B	C16[6]	3.254	H39B	H3	3.385
H39B	H3A[9]	3.317	H39B	H5[12]	2.746
H39B	H15[6]	3.035	H39B	H16[6]	2.490
H40A	O2[9]	3.131	H40A	O3[9]	2.890
H40A	O7[11]	2.654	H40A	C10[9]	3.303
H40A	C11[9]	3.349	H40A	C31[11]	3.291
H40A	H3A[9]	2.542	H40A	H8[11]	3.182
H40A	H25[11]	3.409	H40A	H35[14]	3.331
H40A	H36[14]	3.444	H40B	O2[9]	3.358
H40B	O3[9]	3.484	H40B	O5[9]	3.312
H40B	C10[9]	3.340	H40B	C11[9]	3.254
H40B	C20[9]	3.470	H40B	C35[14]	3.377
H40B	H3A[9]	3.437	H40B	H20B[9]	2.725
H40B	H20C[9]	3.497	H40B	H35[14]	2.480
H40C	C24[11]	3.349	H40C	C25[11]	3.041
H40C	C35[14]	3.468	H40C	C36[14]	3.557
H40C	H24[11]	3.261	H40C	H25[11]	2.721
H40C	H35[14]	2.840	H40C	H36[14]	3.010

Symmetry Operators:

(1) X,Y+1,Z
(2) -X+1,-Y+1,-Z+1
(3) -X+1,-Y,-Z+2
(4) X-1,Y+1,Z
(5) X-1,Y,Z
(6) -X+1,-Y,-Z+1
(7) -X,-Y+1,-Z+1
(8) X,Y-1,Z
(9) X+1,Y-1,Z
(10) -X+1,-Y-1,-Z+2
(11) X+1,Y,Z
(12) -X+2,-Y,-Z+1
(13) -X,-Y,-Z+2
(14) -X+2,-Y-1,-Z+2