Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

6-Acetoxymethyl-3-[(2-hydroxy-3-methoxybenzylidene)amino]-3,4,5,6-tetrahydro-2*H*-pyran-2,4,5-triyl triacetate

Yan Fei Wang,[a,b] Shu-Hua Zhang,[c] Zhen Feng Chen[b] and Hong Liang[b]*

[a]College of Chemistry and Chemical Engineering, Central South University, Changsha 410083, People's Republic of China, [b]School of Chemistry and Chemical Engineering, Guangxi Normal University, Guilin 541004, People's Republic of China, and [c]College of Chemistry and Bioengineering, Guilin University of Technology, Guilin 541004, People's Republic of China
Correspondence e-mail: zsh720108@163.com

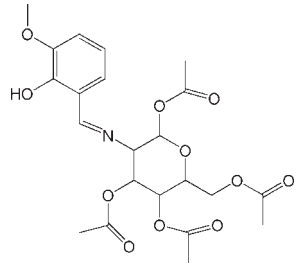
Received 18 March 2010; accepted 26 March 2010

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.007 Å; R factor = 0.046; wR factor = 0.126; data-to-parameter ratio = 7.8.

The title compound, $C_{22}H_{27}NO_{11}$, was synthesized by the reaction of 4,5-diacetoxy-6-acetoxymethyl-3-aminotetrahydropyran-2-yl acetate and 2-hydroxy-3-methoxybenzaldehyde in ethanol. The molecule contains two six-membered rings, one of which is in a chair conformation, and an intramolecular O—H···N hydrogen bond is present.

Related literature

For a Schiff base complex, see: Zhang *et al.* (2003). For macrocyclic Schiff base compounds, see: Frischmann *et al.* (2008); Jiang *et al.* (2010). For 5,5′-dimethoxy-2,2′-[4,5-dimethyl-*o*-phenylenebis(nitrilomethylidyne)]diphenol, which shows similar hydrogen-bonding to the title compound, see: Kargar *et al.* (2010).



Experimental

Crystal data

$C_{22}H_{27}NO_{11}$
M_r = 481.45
Orthorhombic, $P2_12_12_1$
a = 10.806 (3) Å
b = 11.151 (3) Å
c = 20.243 (5) Å
V = 2439.2 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 296 K
0.32 × 0.28 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
12313 measured reflections
2460 independent reflections
1506 reflections with $I > 2\sigma(I)$
R_{int} = 0.061

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.126
S = 1.03
2460 reflections
314 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82	1.90	2.625 (4)	147

Data collection: *SMART* (Bruker 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

We acknowledge financial support by the National Natural Science Foundation of China (No. 20861002) and the 973 Plan of China (2009CB526503).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2250).

References

Bruker (2004). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Frischmann, P. D., Jiang, J., Hui, J. K. H., Grzybowski, J. J. & MacLachlan, M. J. (2008). *Org. Lett.* **10**, 1255–1258.
Jiang, J. & MacLachlan, M. J. (2010). *Org. Lett.* **12**, 1020–1023.
Kargar, H., Kia, R., Khan, I. U., Sahraei, A. & Aberoomand Azar, P. (2010). *Acta Cryst.* E**66**, o728.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Zhang, S. H., Jiang, Y. M., Xiao, Y. & Zhou, Z. Y. (2003). *Chin. J. Inorg. Chem.* **19**, 517–520.

supplementary materials

Acta Cryst. (2010). E**66**, o990 [**doi:10.1107/S1600536810011475**]

6-Acetoxymethyl-3-[(2-hydroxy-3-methoxybenzylidene)amino]-3,4,5,6-tetrahydro-2*H*-pyran-2,4,5-triyl triacetate

Y. F. Wang, S.-H. Zhang, Z. F. Chen and H. Liang

Comment

Schiff base compounds (Zhang, *et al.* 2003; Frischmann, *et al.* 2008; Jiang, *et al.* 2010) have aroused increasing interest because of their antiviral, anticancer and antibacterial activities. Herein, we report the synthesis and crystal structure of a new schiff base compound, (I), prepared by the reaction of Acetic acid 4,5-diacetoxy-6-acetoxymethyl-3-amino]-tetrahydro-pyran-2-yl ester and 2-hydroxy-3-methoxy-benzaldehyde.

The molecular structure of (I) reveals the 2-hydroxy-3-methoxy-benzaldehyde configuration with one acetic acid 4,5-diacetoxy-6-acetoxymethyl-3-amino]-tetrahydro-pyran-2-yl ester molecule on N1-position (Fig. 1). The dihedral angle between the benzene ring of 2-hydroxy-3-methoxy-benzylidene group and the plane of C9, C10, C12, C13 is 56.78 (3) °. The other dihedral angle between the four acetic acid groups and the plane of C9, C10, C12, C13 are in the range of 57.0–111.7°. There is an intramolecular O–H···N hygrogen bond between the phenol and imido-group(Table 1). The distance of N1···H1 is substantially shorter than the van der Waals distance of 2.75 Å for the N and H distance. The hydrogen bond between the phenol and imido-group are similar to those found in the crystal structure of 5,5'-Dimethoxy-2,2'-[4,5-dimethyl-*o*- phenylenebis(nitrilomethylidyne)]diphenol (Kargar, *et al.* 2010). In the molecule, the C9 has S* configuration, while the C10, C11, C12, C13 are in R* configuration which form a R* configuration molecule.

Experimental

The compound Acetic acid 4,5-diacetoxy-6-acetoxymethyl- 3-amino]-tetrahydro-pyran-2-yl ester (0.182 g, 0.5 mmol) was dissolved in ethanol (10 ml). To this solution, 2-hydroxy-3-methoxy-benzaldehyde (0.076 g, 1 mmol) was added and the mixture was stirred and refluxed at 333 K for 3 h. After cooling to room temperature and filtration, the filtrate was left to stand at room temperature. Colourless block crystals suitable for X-ray diffraction were obtained in a yield of 53 %. Analysis found (%): C 54.64, H 5.69, N 2.94; $C_{22}H_{27}NO_{11}$ requires (%) : C 54.88, H 5.65, N 2.91.

Refinement

All H atoms were positioned geometrically and were refined as riding, with (C—H 0.93–0.98 Å, O—H 0.82 Å with U_{iso}(H) = 1.2 U_{eq}(aromatic C) and U_{iso}(H) = 1.5U_{eq}(other C or O).

In the absence of significant anomalous dispersion effects, Friedel pairs were averaged.

Figures

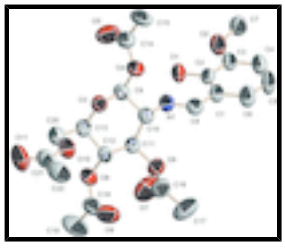


Fig. 1. The molecular structure of (I), showing 30 % probability displacement ellipsoids. H-atoms were omitted.

6-Acetoxymethyl-3-[(2-hydroxy-3-methoxybenzylidene)amino]-3,4,5,6-tetrahydro- 2*H*-pyran-2,4,5-triyl triacetate

Crystal data

$C_{22}H_{27}NO_{11}$	$F(000) = 1016$
$M_r = 481.45$	$D_x = 1.311$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 1384 reflections
$a = 10.806$ (3) Å	$\theta = 2.6$–$18.6°$
$b = 11.151$ (3) Å	$\mu = 0.11$ mm^{-1}
$c = 20.243$ (5) Å	$T = 296$ K
$V = 2439.2$ (11) Å^3	Block, colourless
$Z = 4$	0.32 × 0.28 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer	1506 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.061$
graphite	$\theta_{max} = 25.1°$, $\theta_{min} = 2.0°$
phi and ω scans	$h = -12\rightarrow12$
12313 measured reflections	$k = -12\rightarrow13$
2460 independent reflections	$l = -24\rightarrow24$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.046$	H-atom parameters constrained
$wR(F^2) = 0.126$	$w = 1/[\sigma^2(F_o^2) + (0.0572P)^2 + 0.177P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
2460 reflections	$\Delta\rho_{max} = 0.17$ e Å^{-3}
314 parameters	$\Delta\rho_{min} = -0.14$ e Å^{-3}

0 restraints

Primary atom site location: structure-invariant direct methods

Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0074 (13)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.4441 (3)	0.0108 (3)	−0.01092 (15)	0.0566 (9)
O1	0.2954 (3)	−0.1155 (2)	−0.08619 (14)	0.0650 (8)
H1	0.3485	−0.1041	−0.0580	0.098*
O2	0.1136 (3)	−0.1188 (3)	−0.17050 (16)	0.0796 (10)
O3	0.5742 (3)	−0.0540 (2)	0.15220 (13)	0.0587 (8)
O4	0.3813 (3)	0.0041 (3)	0.12705 (14)	0.0679 (8)
O5	0.2923 (4)	−0.1724 (4)	0.1455 (2)	0.1216 (16)
O6	0.7049 (3)	0.0885 (2)	−0.01748 (13)	0.0653 (8)
O7	0.7871 (5)	−0.0297 (4)	−0.09480 (18)	0.1221 (16)
O8	0.8528 (3)	−0.1002 (3)	0.05512 (15)	0.0654 (8)
O9	0.9690 (4)	0.0649 (4)	0.0508 (2)	0.1069 (14)
O10	0.8042 (3)	−0.0141 (3)	0.21533 (14)	0.0709 (9)
O11	0.9237 (4)	−0.1041 (4)	0.2885 (2)	0.1134 (15)
C1	0.3183 (4)	0.0980 (4)	−0.09509 (19)	0.0564 (11)
C2	0.2611 (4)	−0.0094 (4)	−0.11268 (17)	0.0523 (10)
C3	0.1641 (4)	−0.0085 (4)	−0.1582 (2)	0.0579 (11)
C4	0.1275 (5)	0.0970 (4)	−0.1868 (2)	0.0686 (13)
H4	0.0620	0.0974	−0.2166	0.082*
C5	0.1877 (6)	0.2026 (4)	−0.1716 (2)	0.0830 (17)
H5	0.1651	0.2735	−0.1925	0.100*
C6	0.2796 (5)	0.2030 (4)	−0.1261 (2)	0.0763 (15)
H6	0.3178	0.2752	−0.1153	0.092*
C7	0.0039 (5)	−0.1240 (5)	−0.2101 (2)	0.0794 (15)
H7A	−0.0601	−0.0770	−0.1898	0.119*
H7B	−0.0230	−0.2057	−0.2139	0.119*
H7C	0.0213	−0.0927	−0.2533	0.119*
C8	0.4107 (4)	0.1028 (4)	−0.0435 (2)	0.0593 (11)
H8	0.4473	0.1762	−0.0338	0.071*
C9	0.4872 (4)	−0.0535 (4)	0.1006 (2)	0.0590 (11)

H9	0.4679	−0.1351	0.0859	0.071*
C10	0.5319 (4)	0.0245 (4)	0.0434 (2)	0.0545 (11)
H10	0.5334	0.1086	0.0575	0.065*
C11	0.6604 (4)	−0.0123 (4)	0.02084 (19)	0.0537 (10)
H11	0.6556	−0.0841	−0.0070	0.064*
C12	0.7471 (4)	−0.0339 (3)	0.07825 (18)	0.0524 (10)
H12	0.7737	0.0427	0.0972	0.063*
C13	0.6865 (4)	−0.1114 (4)	0.13127 (19)	0.0562 (11)
H13	0.6663	−0.1898	0.1123	0.067*
C14	0.2886 (5)	−0.0675 (6)	0.1493 (3)	0.0823 (16)
C15	0.1874 (5)	0.0050 (6)	0.1787 (3)	0.1032 (19)
H15A	0.1311	−0.0468	0.2018	0.155*
H15B	0.1438	0.0466	0.1443	0.155*
H15C	0.2217	0.0622	0.2091	0.155*
C16	0.7680 (6)	0.0677 (5)	−0.0739 (2)	0.0792 (15)
C17	0.8086 (7)	0.1835 (6)	−0.1032 (3)	0.120 (2)
H17A	0.8882	0.1734	−0.1234	0.180*
H17B	0.8140	0.2432	−0.0692	0.180*
H17C	0.7498	0.2086	−0.1359	0.180*
C18	0.9585 (5)	−0.0393 (6)	0.0418 (3)	0.0821 (15)
C19	1.0539 (6)	−0.1225 (6)	0.0159 (3)	0.124 (2)
H19A	1.0194	−0.1692	−0.0194	0.187*
H19B	1.0809	−0.1750	0.0507	0.187*
H19C	1.1231	−0.0773	−0.0003	0.187*
C20	0.7666 (5)	−0.1294 (4)	0.1910 (2)	0.0670 (13)
H20A	0.7209	−0.1721	0.2249	0.080*
H20B	0.8388	−0.1766	0.1794	0.080*
C21	0.8859 (5)	−0.0145 (5)	0.2653 (2)	0.0731 (14)
C22	0.9208 (6)	0.1090 (5)	0.2857 (3)	0.0973 (18)
H22A	0.8510	0.1476	0.3059	0.146*
H22B	0.9463	0.1540	0.2476	0.146*
H22C	0.9878	0.1053	0.3168	0.146*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.062 (2)	0.055 (2)	0.0534 (19)	0.0008 (19)	−0.0100 (17)	−0.0002 (18)
O1	0.069 (2)	0.0494 (16)	0.077 (2)	0.0002 (16)	−0.0246 (18)	0.0102 (14)
O2	0.083 (2)	0.061 (2)	0.095 (2)	−0.0107 (18)	−0.037 (2)	0.0081 (17)
O3	0.060 (2)	0.0645 (18)	0.0515 (17)	0.0054 (15)	−0.0011 (16)	0.0003 (13)
O4	0.0571 (19)	0.075 (2)	0.0714 (18)	0.0045 (19)	0.0075 (17)	0.0013 (17)
O5	0.099 (3)	0.096 (3)	0.170 (4)	−0.015 (3)	0.035 (3)	0.016 (3)
O6	0.074 (2)	0.0671 (18)	0.0549 (17)	−0.0070 (17)	−0.0021 (17)	0.0039 (15)
O7	0.175 (5)	0.109 (3)	0.083 (2)	−0.002 (3)	0.043 (3)	−0.015 (2)
O8	0.057 (2)	0.0658 (18)	0.0736 (19)	0.0114 (17)	0.0010 (16)	−0.0063 (16)
O9	0.073 (3)	0.095 (3)	0.153 (4)	−0.014 (2)	0.009 (3)	−0.006 (3)
O10	0.080 (2)	0.0673 (19)	0.0651 (17)	0.0067 (19)	−0.0188 (18)	−0.0055 (15)
O11	0.113 (4)	0.111 (3)	0.116 (3)	0.022 (3)	−0.055 (3)	0.003 (3)

C1	0.067 (3)	0.049 (2)	0.054 (2)	0.004 (2)	−0.008 (2)	0.000 (2)
C2	0.057 (3)	0.050 (2)	0.049 (2)	0.001 (2)	−0.005 (2)	0.0066 (19)
C3	0.062 (3)	0.052 (2)	0.059 (2)	0.002 (2)	−0.011 (2)	0.005 (2)
C4	0.072 (3)	0.064 (3)	0.070 (3)	0.009 (3)	−0.019 (3)	0.003 (2)
C5	0.107 (5)	0.051 (3)	0.091 (3)	0.006 (3)	−0.042 (4)	0.013 (2)
C6	0.094 (4)	0.047 (2)	0.088 (3)	−0.004 (3)	−0.030 (3)	−0.001 (2)
C7	0.073 (4)	0.079 (3)	0.085 (3)	−0.017 (3)	−0.026 (3)	0.014 (3)
C8	0.060 (3)	0.054 (2)	0.064 (3)	−0.002 (2)	−0.004 (2)	−0.005 (2)
C9	0.054 (3)	0.066 (3)	0.057 (3)	0.004 (2)	−0.002 (2)	−0.001 (2)
C10	0.056 (3)	0.051 (2)	0.057 (2)	−0.003 (2)	−0.007 (2)	−0.004 (2)
C11	0.059 (3)	0.047 (2)	0.055 (2)	−0.003 (2)	−0.001 (2)	−0.004 (2)
C12	0.054 (3)	0.050 (2)	0.053 (2)	0.007 (2)	−0.001 (2)	−0.0019 (18)
C13	0.062 (3)	0.053 (2)	0.054 (2)	0.002 (2)	−0.006 (2)	−0.002 (2)
C14	0.062 (4)	0.103 (4)	0.082 (3)	0.000 (3)	0.007 (3)	0.019 (3)
C15	0.051 (3)	0.151 (5)	0.108 (4)	0.009 (4)	0.012 (3)	0.021 (4)
C16	0.088 (4)	0.092 (4)	0.058 (3)	−0.012 (3)	0.000 (3)	0.003 (3)
C17	0.147 (7)	0.131 (5)	0.083 (4)	−0.049 (5)	0.010 (4)	0.017 (4)
C18	0.060 (4)	0.101 (4)	0.086 (4)	0.012 (3)	0.007 (3)	0.004 (3)
C19	0.083 (4)	0.158 (6)	0.132 (5)	0.035 (5)	0.023 (4)	−0.031 (5)
C20	0.072 (3)	0.063 (3)	0.066 (3)	0.006 (3)	−0.009 (3)	0.000 (2)
C21	0.067 (3)	0.088 (4)	0.064 (3)	0.006 (3)	−0.013 (3)	−0.006 (3)
C22	0.079 (4)	0.119 (5)	0.094 (4)	−0.002 (4)	−0.017 (3)	−0.028 (4)

Geometric parameters (Å, °)

N1—C8	1.271 (5)	C7—H7B	0.9600
N1—C10	1.461 (5)	C7—H7C	0.9600
O1—C2	1.351 (4)	C8—H8	0.9300
O1—H1	0.8200	C9—C10	1.526 (6)
O2—C3	1.368 (5)	C9—H9	0.9800
O2—C7	1.433 (5)	C10—C11	1.519 (6)
O3—C9	1.406 (5)	C10—H10	0.9800
O3—C13	1.435 (5)	C11—C12	1.512 (5)
O4—C14	1.359 (6)	C11—H11	0.9800
O4—C9	1.417 (5)	C12—C13	1.525 (6)
O5—C14	1.172 (6)	C12—H12	0.9800
O6—C16	1.350 (6)	C13—C20	1.501 (6)
O6—C11	1.448 (5)	C13—H13	0.9800
O7—C16	1.184 (6)	C14—C15	1.485 (7)
O8—C18	1.356 (6)	C15—H15A	0.9600
O8—C12	1.438 (5)	C15—H15B	0.9600
O9—C18	1.182 (6)	C15—H15C	0.9600
O10—C21	1.343 (5)	C16—C17	1.486 (7)
O10—C20	1.435 (5)	C17—H17A	0.9600
O11—C21	1.176 (5)	C17—H17B	0.9600
C1—C6	1.393 (6)	C17—H17C	0.9600
C1—C2	1.394 (5)	C18—C19	1.483 (7)
C1—C8	1.446 (6)	C19—H19A	0.9600
C2—C3	1.395 (5)	C19—H19B	0.9600

C3—C4	1.370 (6)	C19—H19C	0.9600
C4—C5	1.380 (6)	C20—H20A	0.9700
C4—H4	0.9300	C20—H20B	0.9700
C5—C6	1.354 (6)	C21—C22	1.487 (7)
C5—H5	0.9300	C22—H22A	0.9600
C6—H6	0.9300	C22—H22B	0.9600
C7—H7A	0.9600	C22—H22C	0.9600
C8—N1—C10	119.4 (3)	O8—C12—C13	106.2 (3)
C2—O1—H1	109.5	C11—C12—C13	111.4 (3)
C3—O2—C7	117.9 (3)	O8—C12—H12	110.1
C9—O3—C13	110.4 (3)	C11—C12—H12	110.1
C14—O4—C9	117.0 (4)	C13—C12—H12	110.1
C16—O6—C11	119.2 (4)	O3—C13—C20	108.0 (3)
C18—O8—C12	118.5 (3)	O3—C13—C12	108.6 (3)
C21—O10—C20	116.2 (4)	C20—C13—C12	113.3 (4)
C6—C1—C2	118.3 (4)	O3—C13—H13	109.0
C6—C1—C8	120.2 (4)	C20—C13—H13	109.0
C2—C1—C8	121.5 (4)	C12—C13—H13	109.0
O1—C2—C1	122.0 (3)	O5—C14—O4	122.6 (6)
O1—C2—C3	118.3 (4)	O5—C14—C15	126.5 (6)
C1—C2—C3	119.7 (4)	O4—C14—C15	110.8 (5)
O2—C3—C4	125.4 (4)	C14—C15—H15A	109.5
O2—C3—C2	114.4 (3)	C14—C15—H15B	109.5
C4—C3—C2	120.2 (4)	H15A—C15—H15B	109.5
C3—C4—C5	120.1 (4)	C14—C15—H15C	109.5
C3—C4—H4	120.0	H15A—C15—H15C	109.5
C5—C4—H4	120.0	H15B—C15—H15C	109.5
C6—C5—C4	120.1 (4)	O7—C16—O6	123.2 (5)
C6—C5—H5	120.0	O7—C16—C17	127.0 (5)
C4—C5—H5	120.0	O6—C16—C17	109.7 (5)
C5—C6—C1	121.6 (4)	C16—C17—H17A	109.5
C5—C6—H6	119.2	C16—C17—H17B	109.5
C1—C6—H6	119.2	H17A—C17—H17B	109.5
O2—C7—H7A	109.5	C16—C17—H17C	109.5
O2—C7—H7B	109.5	H17A—C17—H17C	109.5
H7A—C7—H7B	109.5	H17B—C17—H17C	109.5
O2—C7—H7C	109.5	O9—C18—O8	122.9 (5)
H7A—C7—H7C	109.5	O9—C18—C19	127.1 (6)
H7B—C7—H7C	109.5	O8—C18—C19	110.0 (5)
N1—C8—C1	122.8 (4)	C18—C19—H19A	109.5
N1—C8—H8	118.6	C18—C19—H19B	109.5
C1—C8—H8	118.6	H19A—C19—H19B	109.5
O3—C9—O4	105.2 (3)	C18—C19—H19C	109.5
O3—C9—C10	110.7 (3)	H19A—C19—H19C	109.5
O4—C9—C10	106.5 (3)	H19B—C19—H19C	109.5
O3—C9—H9	111.4	O10—C20—C13	108.6 (3)
O4—C9—H9	111.4	O10—C20—H20A	110.0
C10—C9—H9	111.4	C13—C20—H20A	110.0
N1—C10—C11	109.8 (3)	O10—C20—H20B	110.0

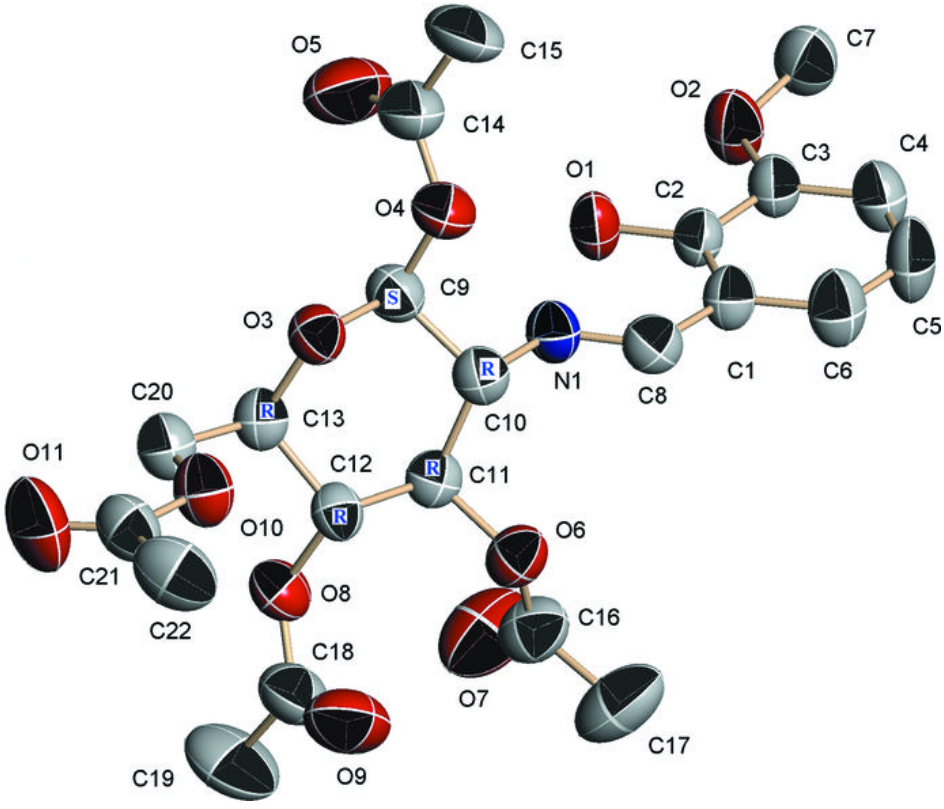
N1—C10—C9	107.9 (3)	C13—C20—H20B	110.0
C11—C10—C9	111.4 (3)	H20A—C20—H20B	108.3
N1—C10—H10	109.3	O11—C21—O10	122.1 (5)
C11—C10—H10	109.3	O11—C21—C22	126.0 (5)
C9—C10—H10	109.3	O10—C21—C22	111.9 (5)
O6—C11—C12	109.3 (3)	C21—C22—H22A	109.5
O6—C11—C10	104.8 (3)	C21—C22—H22B	109.5
C12—C11—C10	112.2 (3)	H22A—C22—H22B	109.5
O6—C11—H11	110.1	C21—C22—H22C	109.5
C12—C11—H11	110.1	H22A—C22—H22C	109.5
C10—C11—H11	110.1	H22B—C22—H22C	109.5
O8—C12—C11	108.9 (3)		

C6—C1—C2—O1	178.2 (4)	C16—O6—C11—C12	100.4 (4)
C8—C1—C2—O1	−5.2 (6)	C16—O6—C11—C10	−139.2 (4)
C6—C1—C2—C3	−2.7 (6)	N1—C10—C11—O6	77.7 (4)
C8—C1—C2—C3	173.9 (4)	C9—C10—C11—O6	−162.9 (3)
C7—O2—C3—C4	−8.2 (7)	N1—C10—C11—C12	−163.8 (3)
C7—O2—C3—C2	172.3 (4)	C9—C10—C11—C12	−44.4 (4)
O1—C2—C3—O2	0.3 (6)	C18—O8—C12—C11	99.4 (4)
C1—C2—C3—O2	−178.7 (4)	C18—O8—C12—C13	−140.6 (4)
O1—C2—C3—C4	−179.1 (4)	O6—C11—C12—O8	−80.6 (4)
C1—C2—C3—C4	1.8 (6)	C10—C11—C12—O8	163.6 (3)
O2—C3—C4—C5	−178.4 (5)	O6—C11—C12—C13	162.6 (3)
C2—C3—C4—C5	1.0 (7)	C10—C11—C12—C13	46.8 (4)
C3—C4—C5—C6	−2.8 (8)	C9—O3—C13—C20	−169.9 (3)
C4—C5—C6—C1	1.8 (8)	C9—O3—C13—C12	66.9 (4)
C2—C1—C6—C5	0.9 (7)	O8—C12—C13—O3	−175.1 (3)
C8—C1—C6—C5	−175.7 (5)	C11—C12—C13—O3	−56.7 (4)
C10—N1—C8—C1	−175.9 (4)	O8—C12—C13—C20	64.9 (4)
C6—C1—C8—N1	175.8 (4)	C11—C12—C13—C20	−176.7 (3)
C2—C1—C8—N1	−0.8 (7)	C9—O4—C14—O5	−2.3 (8)
C13—O3—C9—O4	179.7 (3)	C9—O4—C14—C15	176.9 (4)
C13—O3—C9—C10	−65.7 (4)	C11—O6—C16—O7	1.7 (8)
C14—O4—C9—O3	−98.4 (4)	C11—O6—C16—C17	−177.7 (4)
C14—O4—C9—C10	144.0 (4)	C12—O8—C18—O9	3.0 (8)
C8—N1—C10—C11	−100.9 (4)	C12—O8—C18—C19	−177.5 (4)
C8—N1—C10—C9	137.6 (4)	C21—O10—C20—C13	−174.1 (4)
O3—C9—C10—N1	174.0 (3)	O3—C13—C20—O10	−66.5 (4)
O4—C9—C10—N1	−72.1 (4)	C12—C13—C20—O10	53.8 (5)
O3—C9—C10—C11	53.4 (4)	C20—O10—C21—O11	−0.8 (7)
O4—C9—C10—C11	167.3 (3)	C20—O10—C21—C22	178.7 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.90	2.625 (4)	147.

Fig. 1


O5
C15
C14
O4
C9
S
O3
C20
R
C13
O11
C12
R
C11
R
O10
R
O6
C21
C22
O8
C18
C16
O7
C19
O9
C17
O1
C2
O2
C7
C3
C4
C5
C1
C6
N1
C8
R
C10