Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	$U_{\rm eq}$
C1	0.2864 (2)	1.23216 (11)	0.2886 (2)	0.0680 (6)
C2	0.2286 (2)	1.18559 (11)	0.1892 (2)	0.0690 (6)
C3	0.3164 (2)	1.13997 (10)	0.1212 (2)	0.0611 (6)
C4	0.4633 (2)	1.14094 (9)	0.1501 (2)	0.0515 (5)
N5	0.5529 (2)	1.09245 (8)	0.08518 (15)	0.0564 (4)
C6	0.6304 (2)	1.04793 (10)	0.1816 (2)	0.0537 (5)
N7	0.6100 (2)	0.98021 (8)	0.1622 (2)	0.0631 (5)
C8	0.6777 (2)	0.93674 (11)	0.2541 (2)	0.0662 (6)
C9	0.7642 (2)	0.95668 (11)	0.3686 (2)	0.0629 (6)
C10	0.7889 (2)	1.02784 (11)	0.3826 (2)	0.0638 (6)
C11	0.7247 (2)	1.07516 (10)	0.2889 (2)	0.0553 (5)
N12	0.7701 (2)	1.14488 (8)	0.2929 (2)	0.0596 (5)
C13	0.6826 (2)	1.19583 (10)	0.2683 (2)	0.0554 (5)
C14	0.5234 (2)	1.19065 (9)	0.2446 (2)	0.0522 (5)
C15	0.4332 (2)	1.23465 (10)	0.3151 (2)	0.0607 (5)
N16	0.7359 (2)	1.26292 (8)	0.2752 (2)	0.0609 (5)
C17	0.6834 (2)	1.31582 (10)	0.1737 (2)	0.0636 (6)
C18	0.7088 (2)	1.38712 (10)	0.2354 (2)	0.0667 (6)
N19	0.8612 (2)	1.39806 (9)	0.2711 (2)	0.0693 (5)
C20	0.9124 (2)	1.34522 (11)	0.3719 (3)	0.0746 (6)
C21	0.8880 (2)	1.27274 (11)	0.3131 (3)	0.0733 (7)
C22	0.8875 (3)	1.46760 (11)	0.3304 (3)	0.0940 (8)
C23	0.8316 (3)	0.90336 (11)	0.4691 (3)	0.0923 (8)

Table 2. *Selected geometric parameters* (Å, °)

C4—C14	1.401 (2)	N7—C8	1.336 (2)
C4—N5	1.413 (2)	C11—N12	1.406 (2)
N5—C6	1.417 (2)	N12—C13	1.289 (2)
C6—N7	1.328 (2)	C13—N16	1.383 (2)
C6—C11	1.404 (2)	C13—C14	1.494 (3)
C14—C4—N5	119.8 (2)	C13—N12—C11	122.2 (2)
C4—N5—C6	114.66 (14)	N12—C13—N16	118.6 (2)
C11—C6—N5	120.9 (2)	N12—C13—C14	126.3 (2)
C6—N7—C8	117.8 (2)	N16—C13—C14	114.9 (2)
C6—C11—N12	123.5 (2)	C4—C14—C13	120.1 (2)
C3—C4—N5—C6	−118.4 (2)	C11—N12—C13—C14	5.4 (3)
C14—C4—N5—C6	61.7 (2)	N5—C4—C14—C13	4.6 (3)
C4—N5—C6—N7	122.6 (2)	N16—C13—C14—C15	−41.6 (3)
C4—N5—C6—C11	−58.5 (2)	N12—C13—C14—C4	−47.3 (3)
C9—C10—C11—N12	−169.8 (2)	N16—C13—C14—C4	138.3 (2)
N7—C6—C11—N12	166.4 (2)	N12—C13—N16—C17	141.4 (2)
N5—C6—C11—N12	−12.5 (3)	C14—C13—N16—C17	−43.8 (3)
C10—C11—N12—C13	−145.0 (2)	C13—N16—C17—C18	157.0 (2)
C6—C11—N12—C13	44.1 (3)		

H atoms were placed at standard calculated positions, except for atom HN5, which was obtained from a difference map. In the refinement, H atoms were constrained (included as riding atoms), except for HN5, which was kept fixed.

Data collection: *DIF*4 (Stoe & Cie, 1987*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1987*c*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1002). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1-Methoxy-5,6-methylenedioxy-3-(3,4,5-trimethoxyphenyl)-1,3-dihydroisobenzofuran

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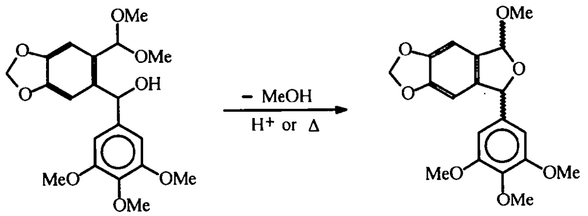
Abstract

The crystal structure of the title compound consists of discrete diastereoisomeric molecules of $C_{19}H_{20}O_7$ held together by van der Waals interactions. The two H atoms on the asymmetric C atoms of the hydrofuran ring are mutually *cis*. The orientations of the three methoxy groups on the phenyl ring are consistent with minimum steric interactions.

Comment

The title compound, (2), may be prepared either by the acid-catalysed (Keay, Plaumann, Rajapaksa & Rodrigo, 1983) or by the thermally induced (Ward, Pelter, Galletti & Qianrong, 1993) elimination of methanol from the corresponding hydroxyacetal (1).



(1) (2)

The molecular structure of compound (2) is shown in Fig. 1, which also indicates the crystallographic atom-numbering scheme. The two H atoms on the asymmetric C atoms of the hydrofuran ring are mutually *cis*. Of the three methoxy substituents of the phenyl ring, one MeO—C bond is nearly perpendicular to the plane of the ring, while the other two bonds are nearly parallel, as shown by the torsion angles C1—O1—C5—C10 = −4.2 (4), C2—O2—C6—C5 = 108.7 (3) and C3—O3—C7—C8 = 2.8 (4)°. Adoption of these orientations by the phenyl ring substituents appears to have been determined by the requirement of minimum steric interactions involving the methoxy groups. The phenyl ring and the six-membered ring of the dihydroisobenzofuran system are both planar within experimental error. The methylenedioxydihydroisobenzofuran system as a whole is non-planar, as shown by the deviations of atoms from the plane of the central six-membered ring: C11 0.029 (3), O5 −0.075 (3), C12 0.064 (3), O6 0.016 (3), O7 0.027 (3) and C16 −0.237 (4) Å. Bond lengths and angles have expected values.

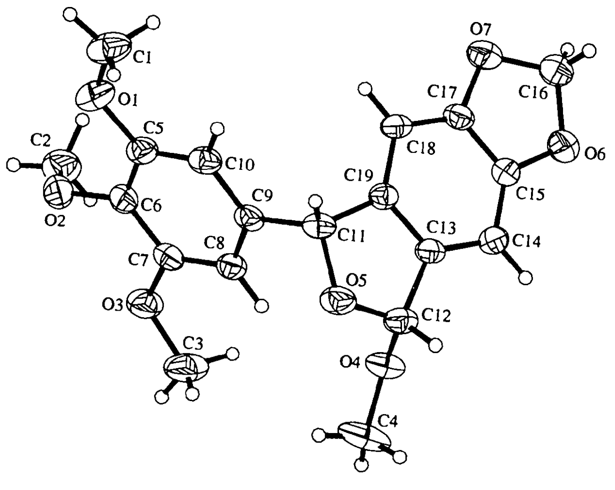


Fig. 1. The molecular structure of (2) showing the atom-labelling scheme. The displacement ellipsoids are plotted at the 40% probability level and H atoms are represented by small circles of arbitrary radii.

Experimental

Hydroxyacetal (1) (0.21 g, 0.535 mmol) was dissolved in dichloromethane (2 ml) and left to stand at room temperature for two days. The solution was evaporated to dryness and the residue purified by column chromatography on alumina using ether–benzene (2:1) as eluent, followed by crystallization from a benzene–petroleum spirit (313–333 K) solution. This produced a mixture of both *cis* and *trans* isomers of dihydroisobenzofuran (2) (m.p. 384–388 K, 51% yield), as evidenced by its ^{1}H NMR spectrum. After repeated crystallization from methanol, colourless crystals of the *cis* isomer were isolated in pure form (m.p. 402–404 K) and characterized by X-ray crystallography. ^{1}H NMR: δ($CDCl_3$) 6.82 (*s*, 1H), 6.66 (*s*, 2H), 6.49 (*s*, 1H), 6.09 (*s*, 1H), 5.97 (*AB* pattern, J = 1.25 Hz, 2H), 5.93 (*s*, 1H), 3.84 (*s*, 6H), 3.360 (*s*, 3H).

Crystal data

$C_{19}H_{20}O_7$	Mo $K\alpha$ radiation
$M_r = 360.35$	$\lambda = 0.71069$ Å
Monoclinic	Cell parameters from 250 reflections
$P2_1/c$	$\theta = 2.51\text{–}25.10°$
$a = 15.307 (3)$ Å	$\mu = 0.105\ \text{mm}^{-1}$
$b = 7.018 (1)$ Å	$T = 140.0 (2)$ K
$c = 17.014 (6)$ Å	Parallelepiped
$\beta = 107.61 (2)°$	0.28 × 0.16 × 0.12 mm
$V = 1742.0 (7)\ \text{Å}^3$	Colourless
$Z = 4$	
$D_x = 1.374\ \text{Mg m}^{-3}$	

Data collection

Delft Instruments FAST area detector diffractometer with rotating anode source	2514 independent reflections
Measurement method: Darr, Drake, Hursthouse & Malik (1993)	1515 observed reflections [$I > 2\sigma(I)$]
Absorption correction: none	$R_{\text{int}} = 0.0292$
4756 measured reflections	$\theta_{\text{max}} = 25.10°$
	$h = -13 \rightarrow 16$
	$k = -7 \rightarrow 6$
	$l = -14 \rightarrow 19$

Refinement

Refinement on F^2	$\Delta\rho_{\text{max}} = 0.128$ e Å^{-3}
$R(F) = 0.0363$	$\Delta\rho_{\text{min}} = -0.116$ e Å^{-3}
$wR(F^2) = 0.1137$	Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
$S = 0.520$	
2514 reflections	
259 parameters	
$w = 1/\sigma^2(F_o^2)$	
$(\Delta/\sigma)_{\text{max}} = 0.001$	

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\text{eq}} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O1	0.78664 (13)	0.5963 (2)	0.33497 (10)	0.0692 (5)
O2	0.92870 (11)	0.3733 (2)	0.40429 (9)	0.0618 (5)
O3	0.95451 (11)	0.0448 (2)	0.33399 (10)	0.0681 (5)
O4	0.76339 (11)	−0.2492 (2)	0.07333 (10)	0.0616 (5)
O5	0.72578 (11)	0.0713 (2)	0.04259 (9)	0.0579 (4)
O6	0.43117 (12)	−0.3958 (2)	0.10777 (11)	0.0699 (5)
O7	0.41813 (12)	−0.1178 (2)	0.17478 (11)	0.0699 (5)
C1	0.7179 (2)	0.7292 (3)	0.2955 (2)	0.0747 (8)
C2	0.9234 (2)	0.2739 (4)	0.4755 (2)	0.0739 (8)
C3	0.9749 (2)	−0.1211 (4)	0.2949 (2)	0.0842 (10)
C4	0.8385 (2)	−0.2684 (6)	0.0409 (2)	0.0961 (11)
C5	0.7948 (2)	0.4360 (3)	0.29250 (13)	0.0488 (6)
C6	0.8670 (2)	0.3165 (3)	0.33146 (13)	0.0486 (5)
C7	0.8800 (2)	0.1502 (3)	0.29188 (14)	0.0485 (6)
C8	0.82132 (15)	0.1022 (3)	0.21569 (13)	0.0477 (5)

C9	0.74940 (15)	0.2224 (3)	0.17747 (12)	0.0440 (5)
C10	0.7363 (2)	0.3888 (3)	0.21507 (13)	0.0491 (6)
C11	0.6822 (2)	0.1682 (3)	0.09534 (12)	0.0480 (5)
C12	0.6960 (2)	−0.1214 (3)	0.02941 (13)	0.0523 (6)
C13	0.61755 (15)	−0.1349 (3)	0.06285 (12)	0.0457 (5)
C14	0.5603 (2)	−0.2909 (3)	0.06026 (13)	0.0525 (6)
C15	0.4956 (2)	−0.2662 (3)	0.09938 (13)	0.0504 (6)
C16	0.3713 (2)	−0.2886 (4)	0.1406 (2)	0.0694 (7)
C17	0.4874 (2)	−0.1002 (3)	0.13952 (13)	0.0489 (6)
C18	0.5429 (2)	0.0539 (3)	0.14271 (13)	0.0480 (6)
C19	0.60919 (14)	0.0310 (3)	0.10225 (12)	0.0438 (5)

Table 2. *Selected geometric parameters* (Å, °)

O1—C5	1.363 (3)	C5—C10	1.390 (3)
O1—C1	1.415 (3)	C6—C7	1.392 (3)
O2—C6	1.371 (3)	C7—C8	1.377 (3)
O2—C2	1.421 (3)	C8—C9	1.383 (3)
O3—C7	1.366 (3)	C9—C10	1.374 (3)
O3—C3	1.422 (3)	C9—C11	1.512 (3)
O4—C12	1.400 (3)	C11—C19	1.506 (3)
O4—C4	1.424 (3)	C12—C13	1.479 (3)
O5—C12	1.423 (3)	C13—C19	1.369 (3)
O5—C11	1.441 (3)	C13—C14	1.395 (3)
O6—C15	1.380 (3)	C14—C15	1.360 (3)
O6—C16	1.424 (3)	C15—C17	1.375 (3)
O7—C17	1.373 (3)	C17—C18	1.366 (3)
O7—C16	1.427 (3)	C18—C19	1.398 (3)
C5—C6	1.386 (3)		
C5—O1—C1	117.8 (2)	O5—C11—C19	104.0 (2)
C6—O2—C2	115.3 (2)	O5—C11—C9	112.4 (2)
C7—O3—C3	117.6 (2)	C19—C11—C9	113.7 (2)
C12—O4—C4	113.9 (2)	O4—C12—O5	112.1 (2)
C12—O5—C11	111.1 (2)	O4—C12—C13	107.8 (2)
C15—O6—C16	104.9 (2)	O5—C12—C13	105.0 (2)
C17—O7—C16	104.9 (2)	C19—C13—C14	122.1 (2)
O1—C5—C6	115.7 (2)	C19—C13—C12	109.9 (2)
O1—C5—C10	124.3 (2)	C14—C13—C12	127.9 (2)
C6—C5—C10	120.0 (2)	C15—C14—C13	115.3 (2)
O2—C6—C5	119.3 (2)	C14—C15—C17	122.7 (2)
O2—C6—C7	121.4 (2)	C14—C15—O6	128.0 (2)
C5—C6—C7	119.1 (2)	C17—C15—O6	109.3 (2)
O3—C7—C8	124.4 (2)	O6—C16—O7	107.4 (2)
O3—C7—C6	114.8 (2)	C18—C17—O7	127.4 (2)
C8—C7—C6	120.8 (2)	C18—C17—C15	122.8 (2)
C7—C8—C9	119.5 (2)	O7—C17—C15	109.8 (2)
C10—C9—C8	120.4 (2)	C17—C18—C19	115.1 (2)
C10—C9—C11	119.3 (2)	C13—C19—C18	121.9 (2)
C8—C9—C11	120.2 (2)	C13—C19—C11	108.9 (2)
C9—C10—C5	120.1 (2)	C18—C19—C11	129.2 (2)

The unit-cell and intensity data were recorded at 140 K on a FAST area detector diffractometer using the routines *ENDEX*, *REFINE* and *MADONL* in the *MADNES* software (Pflugrath & Messerschmidt, 1989) and processed using *ABSURD* (Karaulov, 1992); detailed procedures are described by Darr, Drake, Hursthouse & Malik (1993). The structure was solved using *SHELXS*86 (Sheldrick, 1990) and refined using *SHELXL*93 (Sheldrick, 1993). The H atoms were included in idealized positions with $U_{\rm iso}$ values freely refined. The diagram was drawn with *SNOOPI* (Davies, 1983). Calculations were carried out on a 486DX2/66 personal computer. Material for publication was prepared using *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: BM1006). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Salicylaldehyde Benzoyl Hydrazone

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Abstract

The crystallographic characterization of *N*-(2-hydroxybenzylideneamino)benzamide, $C_{14}H_{12}N_2O_2$, reveals quasi coplanarity of the whole molecular skeleton, localization of the double bonds in the central —C=N—N—C=O chain, which has an *E* configuration with respect to the double bond of the hydrazone bridge, and an amide *trans,s-cis* configuration around the single N—N bond. The molecules are held together by hydrogen bonds linking them into layers along the [100] direction.

Comment

Aroyl hydrazones are currently being investigated as orally active iron chelators for the treatment of iron