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Three Monofluorinated Small Molecules: *ortho*-Fluorophenylglycine, Methyl 4-(Fluorocarbonyl)benzoate and 16 α -Fluoro- α -estradiol 3-Methyl Ether

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Abstract

2-Amino-2-(2-fluorophenyl)acetic acid (*ortho*-fluorophenylglycine) consists of a planar acetate anion with a fluorophenyl group attached to the α C atom at a dihedral angle of 105.5°. All three amino H atoms form three hydrogen bonds to carboxylate O atoms of three adjacent molecules in the crystal. Methyl 4-(fluorocarbonyl)benzoate has a planar phenylfluorocarbonyl group and its planar carboxylate group is rotated by 6° from it. The estradiol 3-methyl ether, 16 α -fluoro-3-methoxyestra-1,3,5(10)-trien-17 α -ol, has the well known structure for the estradiol moiety. The alcoholic H atom is hydrogen bonded to the adjacent F atom of the molecule. There are no intermolecular hydrogen bonds.

Comment

The structures of these compounds were determined in order to assess the extent of ^{19}F spin diffusion in solid-state NMR experiments on fluorinated coals (Hagaman & Burns, 1993). This process depends on the strength of the ^{19}F dipole-dipole interaction, which has an inverse cubic dependence on intermolecular distance (Hagaman, Ho, Brown, Schell & Woody, 1990).

In *ortho*-fluorophenylglycine the non-H bond lengths are in close agreement with those of glycine (Jönsson & Kvick, 1972) found by neutron diffraction, but the C—H and N—H lengths are shorter, in agreement with the X-ray diffraction study by Marsh (1958). All H atoms of the NH_3 group are involved in hydrogen bonding: H6 to O1ⁱ at 2.75 Å, and H7 to O2ⁱⁱ at 2.85 Å and H8 to O2ⁱⁱⁱ at 2.83 Å. (A stereo drawing of the hydrogen-bonding network is deposited.*) This differs from glycine in which one of the hydrogen bonds is bifurcated. The plane of atoms C1, C2, O1, O2 makes a dihedral angle of 105.5° with the fluorophenyl plane.

* See deposition footnote.

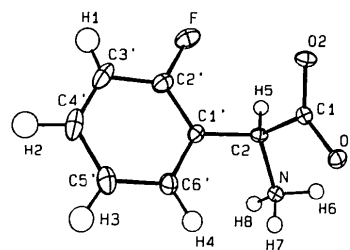


Fig. 1. The *ortho*-fluorophenylglycine molecule with non-H atoms represented by thermal ellipsoids and H atoms by spheres (each at 20% probability).

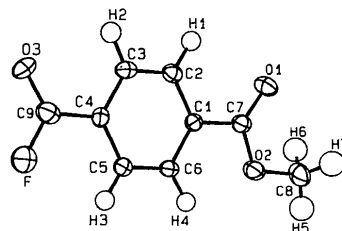


Fig. 2. The methyl 4-(fluorocarbonyl)benzoate molecule with non-H atoms represented by thermal ellipsoids and H atoms by spheres (each at 20% probability).

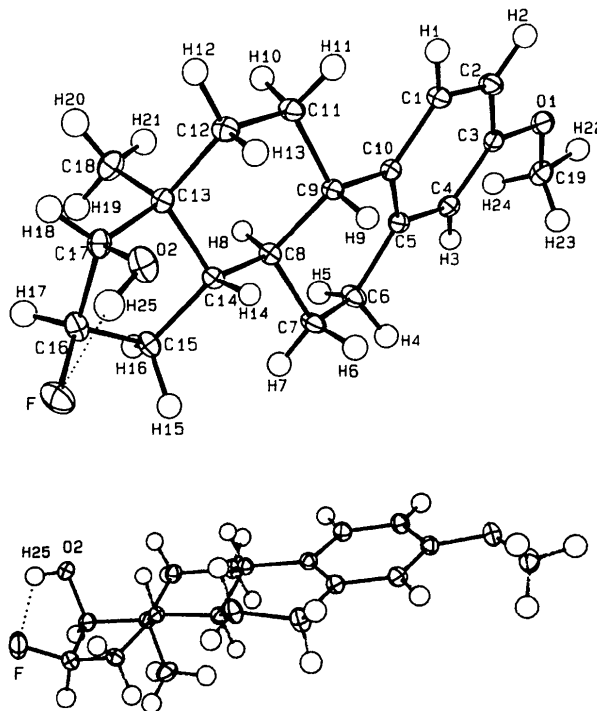


Fig. 3. A molecule of fluoroestradiol methyl ether shown in two perspective views. Non-H atoms are represented by thermal ellipsoids and H atoms by spheres (each at 15% probability). The hydrogen bond is indicated by a dotted line.

Methyl 4-(fluorocarbonyl)benzoate has a planar phenylfluorocarbonyl moiety, and the planar ester group is rotated about the C1—C7 bond by 6°. This is similar to dimethyl terephthalate (Brisse & Pérez, 1976) in which the twist is 4.7°, and to terephthalic acid (Bailey & Brown, 1967) where the angle is 5.3°. The different carboxylate bond lengths, C7—O1 (1.208 Å) and C7—O2 (1.328 Å), are the same as in dimethyl terephthalate and bis(2-hydroxyethyl) terephthalate (Kashino & Haisa, 1975).

The fluoroestradiol methyl ether has the same basic framework for the estradiol moiety as was found previously for 4-fluoroestradiol (Go & Kartha, 1981), 4-bromoestradiol (Norton, Kartha & Lu, 1964), estradiol hemihydrate (Busetta & Hospital, 1972), estradiol.urea (Duax, 1972) and estradiol.propanol (Busetta, Courseille, Geoffre & Hospital, 1972). Unlike these estradiols, which have hydrogen-bonding networks, the present compound has only one H atom available for hydrogen bonding; it is involved in an intramolecular bond to the adjacent F atom.

Experimental

o-Flourophenyglycine

Crystal data

C₈H₈FNO₂

$M_r = 169.16$

Monoclinic

$P2_1/c$

$a = 15.738 (7) \text{ \AA}$

$b = 4.854 (2) \text{ \AA}$

$c = 9.944 (5) \text{ \AA}$

$\beta = 96.58 (2)^\circ$

$V = 754.6 \text{ \AA}^3$

$Z = 4$

$D_x = 1.488 \text{ Mg m}^{-3}$

$D_m = 1.49 \text{ Mg m}^{-3}$

Density measured by flotation

Data collection

CAD-4 diffractometer

ω - 2θ scans, ω -scan width
($1.48 + 0.35 \tan \theta$)°

Absorption correction:

empirical

$T_{\min} = 0.625$, $T_{\max} = 0.997$

1400 measured reflections

1320 independent reflections

872 observed reflections

$[I > \sigma(I)]$

Refinement

Refinement on F

Final $R = 0.061$

$wR = 0.071$

Mo $K\alpha$ radiation

$\lambda = 0.71069 \text{ \AA}$

Cell parameters from 21 reflections

$\theta = 10.4$ – 19.6°

$\mu = 0.116 \text{ mm}^{-1}$

$T = 297 \text{ K}$

Platelet

$0.57 \times 0.47 \times 0.07 \text{ mm}$

Colorless

$R_{\text{int}} = 0.032$

$\theta_{\text{max}} = 25^\circ$

$h = 0 \rightarrow 18$

$k = 0 \rightarrow 5$

$l = -11 \rightarrow 11$

3 standard reflections

frequency: 168 min

intensity variation: -1.7%

$(\Delta/\sigma)_{\text{max}} = 0.13$

$\Delta\rho_{\text{max}} = 0.13 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.13 \text{ e \AA}^{-3}$

$S = 1.95$

872 reflections

133 parameters

Only coordinates of H atoms refined

$w = 4F_o^2 / \{[\sigma(I)]^2 + (0.05F_o^2)^2\}$

Methyl 4-(fluorocarbonyl)benzoate

Crystal data

C₉H₇FO₃

$M_r = 182.15$

Monoclinic

$P2_1/c$

$a = 7.394 (4) \text{ \AA}$

$b = 17.363 (4) \text{ \AA}$

$c = 7.288 (3) \text{ \AA}$

$\beta = 116.51 (2)^\circ$

$V = 837.2 \text{ \AA}^3$

$Z = 4$

$D_x = 1.445 \text{ Mg m}^{-3}$

$D_m = 1.49 \text{ Mg m}^{-3}$

Density measured by flotation

Data collection

CAD-4 diffractometer

ω - 2θ scans, ω -scan width
($0.6 + 0.35 \tan \theta$)°

Absorption correction:

empirical

$T_{\min} = 0.957$, $T_{\max} = 0.99$

1300 measured reflections

1160 independent reflections

518 observed reflections

$[I > \sigma(I)]$

Refinement

Refinement on F

Final $R = 0.050$

$wR = 0.050$

$S = 1.35$

518 reflections

139 parameters

Only coordinates of H atoms refined

$w = 4F_o^2 / \{[\sigma(I)]^2 + (0.05F_o^2)^2\}$

16 α -Fluoro- α -estradiol 3-methyl ether

Crystal data

C₁₉H₂₅FO₂

$M_r = 304.41$

Orthorhombic

$P2_12_12_1$

$a = 6.057 (2) \text{ \AA}$

$b = 8.968 (2) \text{ \AA}$

$c = 29.354 (5) \text{ \AA}$

$V = 1594.5 \text{ \AA}^3$

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2A)

Mo $K\alpha$ radiation

$\lambda = 0.71069 \text{ \AA}$

Cell parameters from 20 reflections

$\theta = 7.1$ – 13.2°

$\mu = 0.116 \text{ mm}^{-1}$

$T = 297 \text{ K}$

Platelet

$0.37 \times 0.22 \times 0.06 \text{ mm}$

Colorless

$R_{\text{int}} = 0.02$

$\theta_{\text{max}} = 23^\circ$

$h = 0 \rightarrow 8$

$k = 0 \rightarrow 18$

$l = -8 \rightarrow 7$

3 standard reflections

frequency: 168 min

intensity variation: -3.5%

$(\Delta/\sigma)_{\text{max}} = 0.04$

$\Delta\rho_{\text{max}} = 0.16 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2A)

Mo $K\alpha$ radiation

$\lambda = 0.71069 \text{ \AA}$

Cell parameters from 24 reflections

$\theta = 10.8$ – 17.6°

$\mu = 0.083 \text{ mm}^{-1}$

$T = 300 \text{ K}$

Prism

$Z = 4$
 $D_x = 1.268 \text{ Mg m}^{-3}$
 $D_m = 1.31 \text{ Mg m}^{-3}$
 Density measured by flotation

$0.40 \times 0.31 \times 0.22 \text{ mm}$
 Colorless

Table 3. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2) for 16 α -Fluoro- α -estradiol 3-methyl ether

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

Data collection

CAD-4 diffractometer $R_{\text{int}} = 0.034$
 ω -2 θ scans, ω -scan width $\theta_{\text{max}} = 25^\circ$
 $(0.7 + 0.35 \tan \theta)^\circ$ $h = 0 \rightarrow 7$
 Absorption correction: $k = 0 \rightarrow 10$
 empirical $l = 0 \rightarrow 34$
 $T_{\text{min}} = 0.97$, $T_{\text{max}} = 0.99$
 1750 measured reflections 3 standard reflections
 1660 independent reflections frequency: 168 min
 1228 observed reflections intensity variation: -1.4%
 $[I > \sigma(I)]$

Refinement

Refinement on F^2 $(\Delta/\sigma)_{\text{max}} = 0.13$
 Final $R = 0.033$ $\Delta\rho_{\text{max}} = 0.13 \text{ e \AA}^{-3}$
 $wR = 0.043$ $\Delta\rho_{\text{min}} = -0.13 \text{ e \AA}^{-3}$
 $S = 1.36$
 1228 reflections Atomic scattering factors
 275 parameters from *International Tables*
 Only coordinates of H atoms refined for *X-ray Crystallography* (1974, Vol. IV, Table 2.2A)
 $w = 4F_o^2 / \{[\sigma(I)]^2 + (0.05F_o^2)^2\}$

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2) for *o*-fluorophenylglycine

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
F	0.7380 (2)	-0.3851 (7)	1.0544 (3)	0.078 (2)
O1	0.9349 (1)	0.3119 (5)	1.1776 (2)	0.035 (1)
O2	0.8807 (2)	0.0502 (6)	1.0043 (2)	0.037 (1)
N	0.9001 (2)	-0.0516 (6)	1.3644 (3)	0.030 (1)
C1	0.8948 (2)	0.1075 (7)	1.1274 (3)	0.026 (2)
C1'	0.7606 (2)	-0.0370 (8)	1.2163 (4)	0.032 (2)
C2	0.8563 (2)	-0.0867 (7)	1.2238 (3)	0.027 (2)
C2'	0.7052 (3)	-0.185 (1)	1.1284 (4)	0.049 (2)
C3'	0.6175 (3)	-0.144 (1)	1.1104 (5)	0.070 (2)
C4'	0.5846 (3)	0.055 (1)	1.1860 (5)	0.067 (2)
C5'	0.6377 (3)	0.208 (1)	1.2765 (5)	0.062 (2)
C6'	0.7254 (2)	0.164 (1)	1.2912 (4)	0.044 (2)

Table 2. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2) for methyl 4-(fluorocarbonyl)benzoate

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
F	0.8531 (4)	0.1368 (2)	0.4852 (4)	0.113 (2)
O1	0.6936 (4)	-0.0952 (2)	-0.4104 (4)	0.078 (2)
O2	0.6641 (4)	-0.1682 (2)	-0.1742 (4)	0.076 (2)
O3	0.8443 (5)	0.2177 (2)	0.2549 (4)	0.090 (2)
C1	0.7270 (5)	-0.0369 (2)	-0.0998 (6)	0.047 (3)
C2	0.7391 (5)	0.0373 (3)	-0.1643 (6)	0.056 (3)
C3	0.7750 (5)	0.0981 (3)	-0.0311 (6)	0.057 (3)
C4	0.7953 (5)	0.0851 (2)	0.1655 (5)	0.047 (2)
C5	0.7805 (5)	0.0111 (2)	0.2280 (6)	0.052 (2)
C6	0.7468 (5)	-0.0494 (2)	0.0978 (6)	0.052 (2)
C7	0.6937 (6)	-0.1013 (3)	-0.2452 (6)	0.056 (2)
C8	0.6393 (7)	-0.2355 (3)	-0.3027 (7)	0.096 (3)
C9	0.8330 (6)	0.1509 (3)	0.3026 (6)	0.072 (3)

	x	y	z	U_{eq}
F	0.3026 (3)	0.7110 (2)	0.70003 (5)	0.077 (1)
O1	0.2985 (3)	1.1139 (2)	1.06514 (5)	0.050 (1)
O2	0.6426 (3)	0.6578 (2)	0.75237 (5)	0.058 (1)
C1	0.5327 (4)	0.8816 (3)	0.97687 (8)	0.043 (1)
C2	0.4950 (5)	0.9505 (3)	1.01818 (8)	0.046 (1)
C3	0.3213 (5)	1.0501 (3)	1.02279 (8)	0.040 (1)
C4	0.1880 (5)	1.0790 (3)	0.98600 (8)	0.042 (1)
C5	0.2264 (4)	1.0099 (3)	0.94407 (7)	0.039 (1)
C6	0.0743 (5)	1.0504 (3)	0.90496 (8)	0.054 (2)
C7	0.1644 (5)	1.0051 (3)	0.85875 (8)	0.049 (2)
C8	0.2515 (4)	0.8458 (3)	0.86069 (7)	0.038 (1)
C9	0.4521 (4)	0.8412 (3)	0.89226 (7)	0.037 (1)
C10	0.4002 (4)	0.9103 (3)	0.93876 (7)	0.037 (1)
C11	0.5520 (5)	0.6847 (3)	0.89547 (8)	0.044 (1)
C12	0.6041 (5)	0.6170 (3)	0.84869 (8)	0.044 (1)
C13	0.4033 (4)	0.6216 (3)	0.81734 (8)	0.040 (1)
C14	0.3221 (4)	0.7849 (3)	0.81436 (7)	0.036 (1)
C15	0.1616 (4)	0.7844 (3)	0.77408 (8)	0.046 (1)
C16	0.2417 (5)	0.6589 (3)	0.74343 (8)	0.052 (2)
C17	0.4444 (5)	0.5881 (3)	0.76692 (8)	0.047 (1)
C18	0.2242 (5)	0.5128 (3)	0.83374 (9)	0.057 (2)
C19	0.1149 (5)	1.2111 (3)	1.07127 (9)	0.055 (2)

Table 4. Geometric parameters (\AA , $^\circ$)

<i>o</i> -Fluorophenylglycine			
F—C2'	1.354 (6)	C2'—C3'	1.387 (6)
O1—C1	1.249 (4)	C3'—C4'	1.361 (8)
O2—C1	1.250 (4)	C4'—C5'	1.374 (7)
N—C2	1.496 (4)	C5'—C6'	1.387 (5)
C1—C2	1.520 (5)		
C1'—C2'	1.517 (5)	N...O1 ⁱ	2.758 (5)
C1'—C2'	1.367 (5)	N...O2 ⁱⁱ	2.837 (5)
C1'—C6'	1.382 (6)	N...O2 ⁱⁱⁱ	2.826 (5)
O1—C1—O2	126.4 (3)	F—C2'—C3'	118.0 (4)
O1—C1—C2	117.4 (3)	C1'—C2'—C3'	124.0 (4)
O2—C1—C2	116.1 (3)	C2'—C3'—C4'	117.8 (4)
C2—C1'—C2'	120.2 (3)	C3'—C4'—C5'	120.3 (4)
C2—C1'—C6'	122.9 (3)	C4'—C5'—C6'	120.6 (5)
C2'—C1'—C6'	116.8 (2)	C1'—C6'—C5'	120.4 (4)
N—C2—C1	110.1 (3)		
N—C2—C1'	112.1 (3)	N—H6...O1 ⁱ	165.9 (4)
C1—C2—C1'	109.6 (3)	C1—H7...O2 ⁱⁱ	170.0 (4)
F—C2'—C1'	118.0 (3)	N—H8...O2 ⁱⁱⁱ	151.0 (4)
Methyl 4-(fluorocarbonyl)benzoate			
F—C9	1.294 (6)	C1—C7	1.483 (6)
O1—C7	1.208 (6)	C2—C3	1.378 (6)
O3—C9	1.225 (6)	C3—C4	1.391 (6)
O2—C7	1.328 (5)	C4—C5	1.383 (6)
O2—C8	1.458 (6)	C4—C9	1.460 (6)
C1—C2	1.388 (6)	C5—C6	1.362 (6)
C1—C6	1.398 (6)		
C7—O2—C8	116.5 (4)	C4—C5—C6	120.2 (4)
C2—C1—C6	119.9 (4)	C1—C6—C5	120.1 (4)
C2—C1—C7	118.3 (4)	O1—C7—O2	122.5 (4)
C6—C1—C7	121.8 (4)	O1—C7—C1	124.9 (4)
C1—C2—C3	119.7 (4)	O2—C7—C1	112.6 (4)
C2—C3—C4	120.0 (4)	F—C9—O3	118.7 (4)
C3—C4—C5	120.1 (4)	F—C9—C4	117.1 (4)
C3—C4—C9	118.6 (4)	O3—C9—C4	124.2 (4)
C5—C4—C9	121.3 (4)		
16 α -Fluoro- α -estradiol 3-methyl ether			
F—C16	1.406 (3)	C8—C9	1.529 (3)
O1—C3	1.376 (3)	C8—C14	1.527 (3)
O1—C19	1.424 (4)	C9—C10	1.532 (3)
O2—C17	1.419 (3)	C9—C11	1.531 (3)
C1—C2	1.380 (3)	C11—C12	1.534 (3)

C1—C10	1.401 (3)	C12—C13	1.526 (4)
C2—C3	1.387 (4)	C13—C14	1.547 (3)
C3—C4	1.373 (3)	C13—C17	1.531 (3)
C4—C5	1.397 (3)	C13—C18	1.537 (4)
C5—C6	1.516 (4)	C14—C15	1.531 (3)
C5—C10	1.389 (4)	C15—C16	1.520 (4)
C6—C7	1.517 (4)	C16—C17	1.545 (4)
C7—C8	1.524 (3)	O2...F	2.613 (3)
C3—O1—C19	116.6 (2)	C5—C10—C9	121.0 (2)
C2—C1—C10	121.7 (2)	C9—C11—C12	112.9 (2)
C1—C2—C3	120.0 (2)	C11—C12—C13	111.4 (2)
O1—C3—C2	115.6 (2)	C12—C13—C14	108.3 (2)
O1—C3—C4	124.9 (2)	C12—C13—C17	116.6 (2)
C2—C3—C4	119.4 (2)	C12—C13—C18	110.9 (2)
C3—C4—C5	120.7 (2)	C14—C13—C17	100.5 (2)
C4—C5—C6	117.4 (2)	C14—C13—C18	113.2 (2)
C4—C5—C10	120.7 (2)	C17—C13—C18	107.0 (2)
C6—C5—C10	122.0 (2)	C8—C14—C13	112.2 (2)
C5—C6—C7	113.2 (2)	C8—C14—C15	120.7 (2)
C6—C7—C8	110.0 (2)	C13—C14—C15	104.1 (2)
C7—C8—C9	108.9 (2)	C14—C15—C16	104.9 (2)
C7—C8—C14	113.5 (2)	F—C16—C15	112.0 (2)
C9—C8—C14	107.9 (2)	F—C16—C17	109.4 (2)
C8—C9—C10	111.5 (2)	C15—C16—C17	107.1 (2)
C8—C9—C11	112.1 (2)	O2—C17—C13	110.0 (2)
C10—C9—C11	113.4 (2)	O2—C17—C16	110.9 (2)
C1—C10—C5	117.6 (2)	C13—C17—C16	102.8 (2)
C1—C10—C9	121.3 (2)	O2—H25...F	114.2 (3)

Symmetry code: (i) $2 - x, y - 0.5, 2.5 - z$; (ii) $x, 0.5 - y, 0.5 + z$;
(iii) $x, -0.5 - y, 0.5 + z$.

ortho-Fluorophenylglycine (Aldrich Chemical Co.) was recrystallized from H₂O/ethanol. Methyl 4-(fluorocarbonyl)benzoate was synthesized from terephthalic acid monomethyl ether by treatment with SF₄ (Hasek, Smith & Englehardt, 1960) and recrystallized from benzene/hexane. The 16 α -fluoro- α -estradiol 3-methyl ether was prepared from estrone 3-methyl ether (Sigma Chemical Co.) by fluorination of the trimethylsilyl derivative of the enol ether with fluoropyridinium trifluoromethanesulfonate (Umemoto, Tomita & Kawada, 1990), followed by reduction of the α -fluoroketone with NaBH₄ in methanol. Crystals were obtained from hexane. Calculations were performed with the *Enraf-Nonius Structure Determination Package* (Frenz, 1983). The structure was solved by *MULTAN11/82* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and difference Fourier maps.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry, as well as a stereo diagram showing the hydrogen-bond network of *o*-fluorophenylglycine, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71016 (35 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH1019]

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Structure of 4-[(3-Methoxyphenylimino)-methyl]phenol

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Abstract

3-Methoxy-*N*-(4-hydroxybenzylidene)aniline exhibits a non-planar conformation with a dihedral angle C7—N—C8—C13 of 45.9° between the planes of the two phenyl rings. The methoxy group is twisted from the phenyl ring by 3.0°.

Comment

The interaction of Schiff bases having a 2-hydroxy substituent on the benzylidene fragment with several metal ions has been described (Bullock, Ladd, Povey & Tajmir-Riahi, 1979; Kamwaya & Khoo, 1985;