Crystal structure of 2,4-dimethyl-phenoxy-2-acetic acid, $C_{10}H_{12}O_3$

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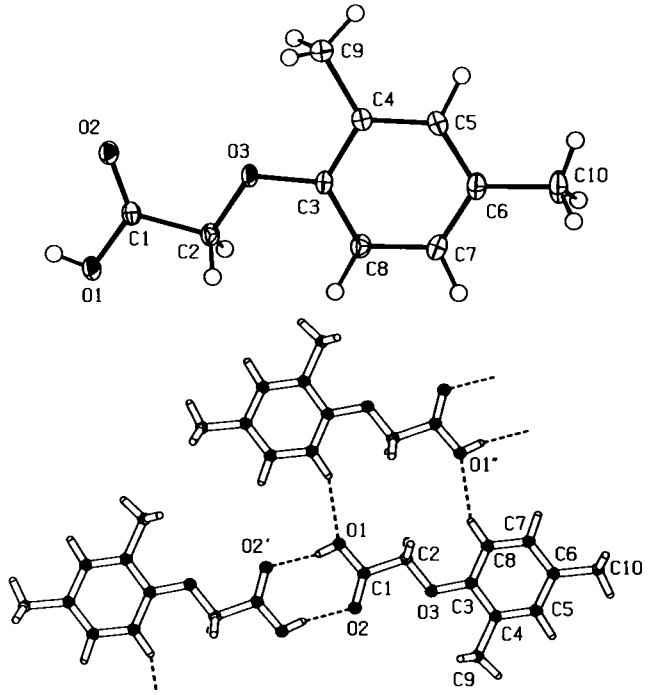

Abstract

$C_{10}H_{12}O_3$, triclinic, $P\bar{1}$ (no. 2), $a = 5.117(5)$ Å, $b = 8.054(5)$ Å, $c = 11.900(5)$ Å, $\alpha = 75.440(5)°$, $\beta = 80.553(5)°$, $\gamma = 78.135(5)°$, $V = 461.2$ Å^3, $Z = 2$, $R_{gt}(F) = 0.064$, $wR_{ref}(F^2) = 0.183$, $T = 100$ K.

Source of material

Equimolar quantities of chloroacetic 2,4-dimethyl phenol and chloroacetic acid with a mole of sodium hydroxide were dissolved in 15 ml of distilled water. The residue obtained after evaporation by heating, is again dissolved in 100 ml of hot water. The cooled solution at ambient temperature is then acidified with 1N hydrochloric acid. The heavy oil which results from it is extracted with sulphuric ether evaporated in Marie bath. One obtains the target acid sought in the form of yellow crystals.

Experimental details

Hydrogen atoms, except those of methyl groups, were located by Fourier difference analysis and were refined isotropically.

Discussion

The 2,4-dimethyl-phenoxy-2-acetic acid is a natural growth hormone produced by the banana tree. His synthesis was undertaken within the framework of the study of growth promoters on rejections scales of plantain banana tree in hydroponic culture. The crystallographic study of this compound was thus undertaken to be used as reference to a series of structural analogues under development.

Within phenyl ring, bonds lengths lie between 1.390(2) Å and 1.404(2) Å which highlight the aromatic character. The valence angle C6–C5–C4 [123.1(1)°] is larger than the standard value (120°). The opening of this angle is due to the presence of the methyl groups on the C4 and C6 atoms which involves a decrease of the ring angles of C4 [117.6(1)°] and C6 [117.7(1)°]. The C1—O1 distance [1.316(2) Å] is compatible with respective distances in related structures [1,2] and smaller than those usually observed in carboxylic acids [1.365 Å]. The torsion angle O2–C1–O1–H1 [–0.9(3)°] shows that the four atoms lie in the same plane. Molecular packing (figure, bottom) reveals that the molecules are linked by hydrogen bonds in two ways: (a) between H1 and O2^i atoms with d(H1···O2^i) = 1.89(4) Å, leading to the dimer formation as observed in the crystals of acetic acid [3-6]; (b) between O1 and H8^{ii} atoms with d(O1···H8^{ii}) = 2.48(2) Å, forming a supramolecular structure (symmetry codes: (i) $1-x$, $1-y$, $1-z$; (ii) $-x$, $-y$, $1-z$).

Table 1. Data collection and handling.

Crystal:	yellow plate, size 0.10 × 0.20 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	0.95 cm^{-1}
Diffractometer, scan mode:	Oxford Diffraction XCALIBUR, ω
$2\theta_{max}$:	62.34°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4829, 2324
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2017
$N(param)_{refined}$:	142
Programs:	SIR2002 [7], SHELXL-97 [8], PLATON [9]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	–0.443(8)	0.349(5)	0.549(3)	0.07(1)
H(21)	2*i*	0.172(4)	0.187(3)	0.490(2)	0.019(4)
H(22)	2*i*	0.031(5)	0.168(3)	0.386(2)	0.028(5)
H(5)	2*i*	0.958(4)	0.369(3)	0.070(2)	0.026(5)
H(7)	2*i*	0.851(6)	–0.119(3)	0.282(2)	0.043(7)
H(8)	2*i*	0.467(4)	0.004(3)	0.379(2)	0.019(5)
H(91)	2*i*	0.3533	0.5797	0.1317	0.05
H(92)	2*i*	0.6503	0.6108	0.0921	0.05
H(93)	2*i*	0.5214	0.6111	0.2210	0.05
H(101)	2*i*	1.2834	0.1132	0.0426	0.05
H(102)	2*i*	1.3370	–0.0329	0.1554	0.05
H(103)	2*i*	1.1689	–0.0599	0.0645	0.05

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(2)	2*i*	–0.2155(2)	0.5250(1)	0.41064(9)	0.0133(4)	0.0171(5)	0.0221(5)	–0.0025(4)	0.0022(4)	–0.0048(4)
O(1)	2*i*	–0.3202(2)	0.2813(1)	0.5364(1)	0.0142(4)	0.0185(5)	0.0237(6)	–0.0029(4)	0.0057(4)	–0.0049(4)
C(1)	2*i*	–0.1638(2)	0.3691(2)	0.4538(1)	0.0113(5)	0.0180(6)	0.0169(6)	–0.0033(4)	0.0001(4)	–0.0065(5)
C(2)	2*i*	0.0887(3)	0.2502(2)	0.4177(1)	0.0109(5)	0.0155(6)	0.0201(7)	–0.0029(4)	0.0021(4)	–0.0055(5)
O(3)	2*i*	0.2587(2)	0.3518(1)	0.33636(9)	0.0119(4)	0.0151(5)	0.0218(5)	–0.0016(3)	0.0059(4)	–0.0051(4)
C(3)	2*i*	0.4836(3)	0.2624(2)	0.2825(1)	0.0108(5)	0.0169(6)	0.0171(6)	–0.0001(4)	0.0005(4)	–0.0065(5)
C(4)	2*i*	0.6274(3)	0.3665(2)	0.1910(1)	0.0170(6)	0.0184(6)	0.0177(7)	–0.0010(5)	0.0024(5)	–0.0036(5)
C(5)	2*i*	0.8603(3)	0.2846(2)	0.1346(1)	0.0188(6)	0.0233(7)	0.0199(7)	–0.0011(5)	0.0054(5)	–0.0031(6)
C(6)	2*i*	0.9541(3)	0.1049(2)	0.1649(1)	0.0145(6)	0.0242(7)	0.0206(7)	0.0012(5)	0.0005(5)	–0.0091(6)
C(7)	2*i*	0.8040(3)	0.0053(2)	0.2549(1)	0.0146(6)	0.0177(6)	0.0230(7)	0.0009(5)	–0.0024(5)	–0.0081(5)
C(8)	2*i*	0.5691(3)	0.0827(2)	0.3146(1)	0.0131(5)	0.0165(6)	0.0207(7)	–0.0023(5)	0.0002(5)	–0.0057(5)
C(9)	2*i*	0.5292(3)	0.5596(2)	0.1557(2)	0.0287(8)	0.0183(7)	0.0273(8)	0.0010(6)	0.0074(6)	0.0011(6)
C(10)	2*i*	1.2089(3)	0.0243(2)	0.1010(2)	0.0180(6)	0.0353(9)	0.0293(8)	0.0033(6)	0.0049(6)	–0.0144(7)

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