Crystal structure of 2-α,α-acetoxymethylphenylethanolate, $C_{13}H_{14}O_6$

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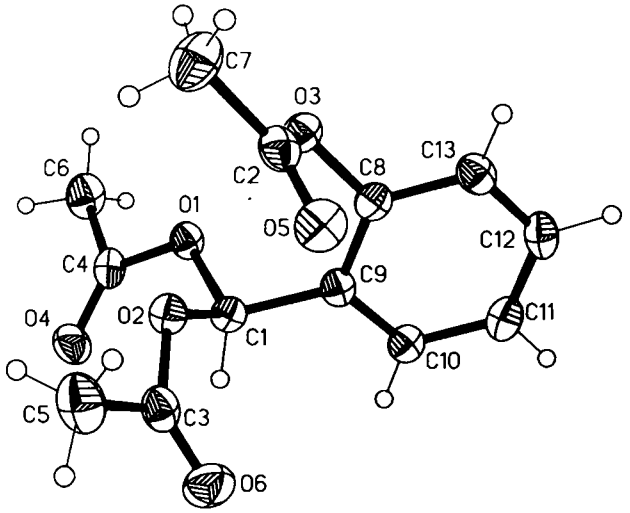

Source of material: The substance was obtained as a by-product in the synthesis of coumarin using the Perkin reaction. Probably no cyclisation due to water-impurities and weak base added in the reaction. The compound was recrystallised from glacical acetic acid.

All non-hydrogen atoms were refined with anisotropic displacement parameters. The H atoms of the three methylgroups were positioned geometrically and allowed to ride during the least-square refinements with the "AFIX 134" instruction of SHELXL where the distance is free to refine. The other hydrogen atoms were positioned geometrically but positions and an isotropic displacement parameter were let free to refine.

$C_{13}H_{14}O_6$, monoclinic, $P12_1/n1$ (No. 14), a =12.460(9) Å, b =8.182(5) Å, c =12.796(9) Å, β =99.43(6)°, V =1286.9 Å^3, Z =4, $R(F)$ =0.033, $R_w(F^2)$ =0.082.

Table 1. Parameters used for the X-ray data collection

Crystal:	colorless prism, size 0.34 x 0.36 x 0.54 mm
Wavelength:	Mo K_α radiation (0.71040 Å)
μ:	1.10 cm^{-1}
Diffractometer:	STOE AED4
Scan mode:	ω/2θ
$T_{measurement}$:	153 K
$2\theta_{max}$:	50°
$N(hkl)_{unique}$:	2027
Criterion for I_o:	$I_o > 2\,\sigma(I_o)$
$N(param)_{refined}$:	205
Program:	SHELXL-93

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

Atom	Site	x	y	z	U_{iso}
H(9)	4e	0.3081(2)	0.215(2)	0.3171(5)	0.081(9)
H(10)	4e	0.245(1)	0.3658(8)	0.2817(2)	0.10(1)
H(11)	4e	0.3596(8)	0.379(1)	0.3384(8)	0.087(9)
H(6)	4e	0.3623(4)	−0.2860(3)	−0.072(1)	0.089(9)
H(7)	4e	0.264(1)	−0.2863(3)	−0.0128(2)	0.077(9)
H(8)	4e	0.3740(5)	−0.357(1)	0.0414(7)	0.075(8)
H(12)	4e	−0.0191(4)	0.117(2)	0.0890(6)	0.10(1)
H(13)	4e	−0.061(1)	0.270(1)	0.1267(2)	0.14(2)
H(14)	4e	0.0345(7)	0.1907(5)	0.189(1)	0.13(1)
H(1)	4e	0.411(2)	0.185(2)	0.034(1)	0.019(4)
H(2)	4e	0.138(2)	0.525(2)	−0.296(1)	0.029(5)
H(3)	4e	0.037(2)	0.404(2)	−0.182(1)	0.028(5)
H(4)	4e	0.333(2)	0.496(3)	−0.264(2)	0.042(6)
H(5)	4e	0.414(2)	0.344(2)	−0.118(1)	0.022(4)

Table 3. Final 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(1)	4e	0.30103(9)	0.0067(1)	−0.00472(8)	0.0281(7)	0.0186(6)	0.0243(6)	0.0036(5)	0.0019(5)	−0.0010(4)
O(2)	4e	0.29640(9)	0.1978(1)	0.12347(8)	0.0295(7)	0.0237(6)	0.0215(6)	0.0013(5)	0.0059(5)	−0.0020(4)
O(3)	4e	0.09531(9)	0.2205(1)	−0.02120(9)	0.0263(7)	0.0248(6)	0.0296(6)	0.0001(5)	0.0079(5)	0.0010(5)
O(4)	4e	0.4512(1)	−0.0794(2)	0.1039(1)	0.0305(8)	0.0311(7)	0.0378(7)	0.0056(6)	−0.0032(6)	0.0036(5)
O(5)	4e	0.1079(1)	0.4418(2)	0.0854(1)	0.0368(8)	0.0309(7)	0.0376(7)	0.0040(6)	0.0115(6)	−0.0042(5)
O(6)	4e	0.4249(1)	0.3917(2)	0.1622(1)	0.052(1)	0.0363(7)	0.0377(7)	−0.0132(7)	0.0051(7)	−0.0066(6)
C(1)	4e	0.3309(2)	0.1716(2)	0.0231(1)	0.025(1)	0.0209(8)	0.0226(8)	0.0007(7)	0.0050(7)	−0.0019(6)
C(2)	4e	0.0724(1)	0.3071(2)	0.0636(1)	0.024(1)	0.0297(9)	0.0300(9)	0.0084(8)	0.0051(7)	0.0025(7)
C(3)	4e	0.3531(2)	0.3108(2)	0.1879(1)	0.037(1)	0.0210(8)	0.0253(8)	0.0055(8)	−0.0001(8)	−0.0006(7)
C(4)	4e	0.3724(2)	−0.1105(2)	0.0395(1)	0.030(1)	0.0242(8)	0.0232(8)	0.0054(7)	0.0075(8)	0.0039(7)
C(5)	4e	0.3126(2)	0.3182(2)	0.2913(1)	0.057(2)	0.038(1)	0.0270(9)	0.008(1)	0.0055(9)	−0.0060(8)
C(6)	4e	0.3401(2)	−0.2748(2)	−0.0050(2)	0.049(2)	0.0225(9)	0.039(1)	0.0061(8)	0.0043(9)	−0.0016(8)

Table 3. (Continued)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(7)	4*e*	–0.0006(2)	0.2117(3)	0.1229(2)	0.046(1)	0.040(1)	0.049(1)	0.003(1)	0.024(1)	0.0049(9)
C(8)	4*e*	0.1619(1)	0.2997(2)	–0.0849(1)	0.026(1)	0.0188(8)	0.0246(8)	0.0008(7)	0.0056(7)	–0.0027(6)
C(9)	4*e*	0.2741(1)	0.2798(2)	–0.0642(1)	0.027(1)	0.0173(8)	0.0215(8)	0.0033(7)	0.0032(7)	–0.0032(6)
C(10)	4*e*	0.3363(2)	0.3550(2)	–0.1318(1)	0.026(1)	0.0220(8)	0.0281(8)	0.0031(7)	0.0070(7)	–0.0017(7)
C(11)	4*e*	0.2872(2)	0.4462(2)	–0.2176(1)	0.039(1)	0.0235(9)	0.0267(8)	0.0026(8)	0.0117(8)	0.0012(7)
C(12)	4*e*	0.1752(2)	0.4628(2)	–0.2373(1)	0.040(1)	0.0245(8)	0.0241(8)	0.0078(8)	0.0021(8)	0.0019(7)
C(13)	4*e*	0.1121(2)	0.3900(2)	–0.1706(1)	0.027(1)	0.0254(9)	0.0290(9)	0.0053(8)	0.0015(8)	–0.0028(7)

References

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