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Structure of 4-Chloro-5-dichloromethylene-2-furanone

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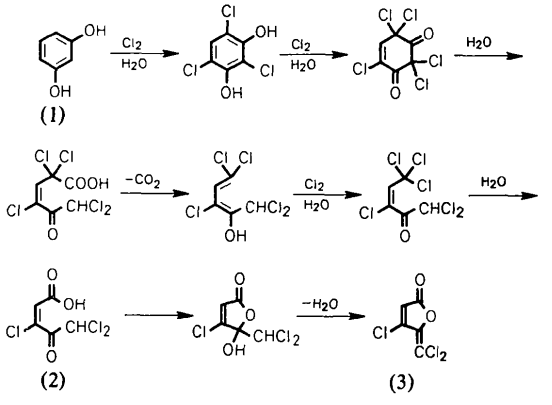
Abstract. $C_5HCl_3O_2$, $M_r = 199{\cdot}42$, monoclinic, $P2_1/n$, $a = 7{\cdot}3438\ (7)$, $b = 7{\cdot}1537\ (9)$, $c = 14{\cdot}058\ (1)$ Å, $\beta = 103{\cdot}683\ (8)°$, $V = 717{\cdot}6\ (1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}846$ g cm^{-3}, Mo $K\alpha_1$, $\lambda = 0{\cdot}70930$ Å, $\mu = 12{\cdot}1$ cm^{-1}, $F(000) = 392$, $T = 294$ K, $R = 0{\cdot}034$ ($wR = 0{\cdot}037$) for 670 unique reflections with $I \geq 3\sigma(I)$. The five-membered ring is planar within experimental error and the entire molecule is planar within 0·08 Å. Molecular dimensions are normal.

Introduction. During the investigation of chlorinated cyclopentenones in pulp mill bleach liquors, an attempt was made to prepare a trichlorocyclopentene-1,2-dione by the chlorination of resorcinol (Boyce & Hornig, 1983). Although spectral properties of the product obtained were similar to those reported and matched those of a minor component in the bleach liquor (McKague, Kolar & Kringstad, 1988), the compound appeared different as the melting point was 15 K higher than reported. This paper describes the X-ray structure of the compound which is a chlorinated enol lactone or furanone.



Experimental. Colourless crystals, 0·15 × 0·27 × 0·58 mm, faces {10$\bar{1}$}, {001}, {010}. Enraf–Nonius CAD-4F diffractometer, lattice parameters from 25 reflections with $\theta = 12$–18°, intensities for $\theta \leq 25°$, *hkl*: 0 to 8, −8 to 0, −16 to 16, ω–2θ scan, ω scan width $(0{\cdot}75 + 0{\cdot}35\tan\theta)°$ at 1·4–10·0° min^{-1}, extended 25% on each side for background measurement, three standard reflections (random variation, 4%), Lp and absorption corrections (numerical integration, 96 sampling points), transmission factors 0·695–0·845, 1255 unique reflections measured, 670 with $I \geq 3\sigma(I)$, where $\sigma^2(I) = S + 4(B_1 + B_2) + (0{\cdot}04S)^2$, S = scan, B_1 and B_2 = background counts. Structure by heavy-atom methods, refined by full-matrix least squares on F, H atom refined with an isotropic thermal parameter, scattering factors and anomalous-scattering corrections for Cl from *International Tables for X-ray*

Table 1. *Final positional (fractional* × 10^4, H × 10^3*) and isotropic thermal parameters* (U × 10^3 Å^2) *with e.s.d.'s in parentheses*

	x	*y*	*z*	U_{eq}/U_{iso}
Cl(1)	1980 (2)	3631 (2)	5043 (1)	56
Cl(2)	3363 (2)	3365 (2)	7129 (1)	62
Cl(3)	7746 (3)	1966 (2)	7708 (1)	63
O(1)	5549 (4)	2233 (5)	4889 (2)	39
O(2)	7733 (5)	1168 (5)	4149 (3)	55
C(1)	3824 (8)	3097 (7)	6006 (3)	40
C(2)	7302 (8)	1508 (8)	4892 (4)	41
C(3)	8313 (9)	1302 (8)	5903 (4)	43
C(4)	7196 (8)	1899 (7)	6462 (4)	40
C(5)	5414 (7)	2478 (7)	5840 (3)	32
H(3)	950 (7)	75 (7)	613 (3)	50 (16)

$U_{eq} = \frac{1}{3}$ × trace of diagonalized U tensor.

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

Cl(1)–C(1)	1·716 (5)	C(1)–C(5)	1·321 (7)
Cl(2)–C(1)	1·702 (5)	C(2)–C(3)	1·447 (7)
Cl(3)–C(4)	1·703 (5)	C(3)–C(4)	1·333 (8)
O(1)–C(2)	1·387 (6)	C(3)–H(3)	0·94 (5)
O(1)–C(5)	1·375 (5)	C(4)–C(5)	1·452 (7)
O(2)–C(2)	1·187 (6)		

C(2)–O(1)–C(5)	108·9 (4)	C(2)–C(3)–H(3)	127 (3)
Cl(1)–C(1)–Cl(2)	114·5 (3)	C(4)–C(3)–H(3)	125 (3)
Cl(1)–C(1)–C(5)	120·0 (4)	Cl(3)–C(4)–C(3)	125·9 (4)
Cl(2)–C(1)–C(5)	125·5 (4)	Cl(3)–C(4)–C(5)	124·9 (4)
O(1)–C(2)–O(2)	120·9 (5)	C(3)–C(4)–C(5)	109·2 (5)
O(1)–C(2)–C(3)	107·6 (5)	O(1)–C(5)–C(1)	119·0 (4)
O(2)–C(2)–C(3)	131·5 (5)	O(1)–C(5)–C(4)	106·7 (4)
C(2)–C(3)–C(4)	107·6 (5)	C(1)–C(5)–C(4)	134·3 (4)

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Crystallography (1974), locally written or locally modified versions of standard computer programs, final $R = 0.034$, $wR = 0.037$ for 670 reflections with $I \geq 3\sigma(I)$, $S = 1.319$, 96 parameters, isotropic type I extinction, $g = 0.7\,(1) \times 10^4$, $R = 0.098$ for all 1255 reflections, $\Delta/\sigma = 0.003$ (mean), 0.011 (maximum), maximum final difference density −0.25 to 0.29 e Å^{-3} (all large peaks near Cl atoms).

Discussion. Final positional and equivalent isotropic thermal parameters ($U_{eq} = \frac{1}{3}$ trace of diagonalized U) are given in Table 1, and geometrical data appear in Table 2.* A stereoview of the molecule is shown in Fig. 1.

The compound (3) is evidently formed through a complex series of reactions with resorcinol (1). A possible mechanism involves cyclization and dehydration of the intermediate chlorinated acid (2).

The five-membered ring is planar to within experimental error but the molecule as a whole deviates slightly from planarity, the maximum displacements from the weighted mean molecular plane being −0.08 (5) Å for H(3) and +0.063 (2) Å for Cl(3). The molecular geometry (Table 2) is normal, with mean distances: C(sp^2)—Cl = 1.707 (8), C=O = 1.187 (6), C(sp^2)—O = 1.381 (8), C=C = 1.327 (8), and C(sp^2)—C(sp^2) = 1.450 (4) Å. The shortest intermolecular distance between non-hydrogen atoms is Cl(2)···O(2) $(x-\frac{1}{2}, \frac{1}{2}-y, \frac{1}{2}+z) = 3.001$ (4) Å.

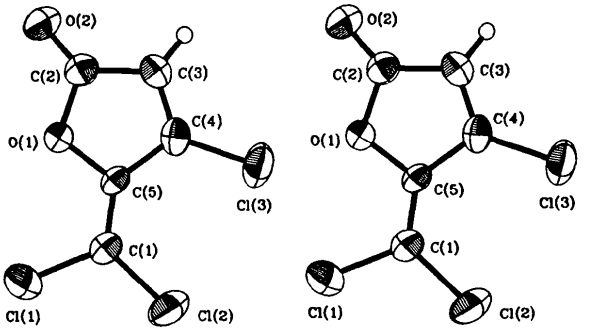

Fig. 1. Stereoscopic view of the molecule: 50% probability thermal ellipsoids are shown for the non-hydrogen atoms.

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References

BOYCE, S. D. & HORNIG, J. F. (1983). *Environ. Sci. Technol.* **17**, 202–211.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99–102 and 149. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

MCKAGUE, A. B., KOLAR, M.-C. & KRINGSTAD, K. P. (1988). *Environ. Sci. Technol.* **22**. In the press.

* Lists of anisotropic thermal parameters, torsion angles, and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44990 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of the (+)-Tartrate of the Selective 5-HT$_2$ Antagonist Irindalone

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Abstract. (+)-(1*R*,3*S*)-*trans*-1-[2-[4-[3-(4-Fluorophenyl)-1-indanyl]-1-piperazinyl]ethyl]-2-imidazolidinone, (+)-(2*R*,3*R*)-tartrate. $C_{28}H_{35}FN_4O_7$, $M_r = 558.6$, monoclinic, $P2_1$, $a = 24.716$ (9), $b = 8.457$ (10), $c = 6.290$ (3) Å, $\beta = 93.21$ (3)°, $V = 1313$ (3) Å^3, $Z = 2$, D_m(295 K) = 1.39 (1), D_x(105 K) = 1.413 Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, μ(Mo $K\alpha$) = 0.10 mm^{-1}, $F(000) = 592$, $T = 105$ (1) K. $R = 0.041$ for 3885 observed [$I \geq 3.0\sigma(I)$] reflections. The absolute configuration is 1*R*,3*S*, opposite to the expected configuration. The ions are connected into infinite chains *via* hydrogen bonds from piperazine N atoms to tartrate ions.

Introduction. The selective 5-HT$_2$* antagonist irindalone was developed by systematic variations of structural components (Bøgesø, 1988). The structure

* Abbreviations used: DA, dopamine; 5-HT$_2$, 5-hydroxotryptophan (serotonine); NE, norepinephrine.

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