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# Structure of 3-Benzylidene-2-chloro-1-cyclohexenecarbaldehyde

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**Abstract.** $C_{14}H_{13}ClO$, $M_r = 232{\cdot}7$, orthorhombic, *Pbca*, $a = 11{\cdot}733\ (3)$, $b = 7{\cdot}470\ (2)$, $c = 26{\cdot}922\ (3)$ Å, $V = 2359{\cdot}6$ Å$^3$, $Z = 8$, $D_x = 1{\cdot}31$ g cm$^{-3}$, $\lambda$(Cu $K\alpha$) $= 1{\cdot}5418$ Å, $\mu = 26{\cdot}8$ cm$^{-1}$, $F(000) = 976$, $T = 296$ K, $R = 0{\cdot}05$ for 1304 observed reflections with $I > 4\sigma(I)$. The cyclohexane ring adopts a sofa conformation. The interplanar angles between the two rings is 36 (1)°. The benzylidene ring is planar. The Cl—C distance is 1·736 (4) Å.

**Experimental.** 3-Benzylidene-2-chloro-1-cyclohexenecarbaldehyde was prepared by the following procedure: a benzylidenecyclohexane was produced from a condensation reaction (Claisen–Schmidt) of cyclohexanone and benzaldehyde. The benzylidenecyclohexanone was then allowed to undergo a Vilsmeier–Hacck (Lotzbeyer & Bodendorf, 1967) reaction which gave the title compound and the product was purified through column chromatography.

A crystal of dimensions 0·32 × 0·38 × 0·48 mm was grown from alcohol solution, and mounted on an Enraf–Nonius CAD-4 diffractometer. The cell parameters and their e.s.d.'s were derived from a least-squares treatment of 25 reflections ($25 < \theta < 35°$). Intensities of reflections ($h$ 0→13, $k$ 0→8, $l$ 0→32) collected with $\omega$–$2\theta$ scan: $\theta_{max} = 75°$. Two reference reflections measured every 100 reflections showed no significant variation. 1983 unique reflections were collected, from which 1304 were considered observed, having $I > 4\sigma(I)$, $R_{int} = 0{\cdot}01$. Intensity data were corrected for Lp effects. No absorption correction. Structure solved by Patterson synthesis using *SHELXS*86 (Sheldrick, 1986) and refinement was carried out by full-matrix least-squares method using *SHELX*76 (Sheldrick, 1976). All H atoms located from difference Fourier map and were refined isotropically. In the final stage of refinement weights were introduced resulting in $R = 0{\cdot}05$, $wR = 0{\cdot}06$, $S = 1{\cdot}69$, $w = 1/[\sigma^2|F| + 0{\cdot}031021|F|^2]$, $(\Delta/\sigma)_{av} = 0{\cdot}005$, $(\Delta/\sigma)_{max} = 0{\cdot}08$ for 1304 reflections.

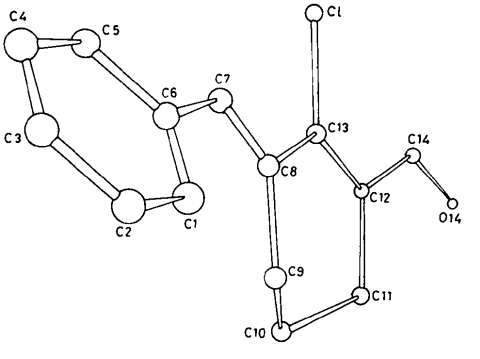


Fig. 1. A view of the molecule with the atom numbering.

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Table 1. *Positional parameters and equivalent isotropic thermal parameters of non-H atoms with e.s.d.'s in parentheses*

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$.

| | $x$ | $y$ | $z$ | $B_{eq}$ (Å$^2$) |
|---|---|---|---|---|
| Cl | 0·1704 (1) | 0·2234 (1) | 0·5867 (0) | 5·8 (0) |
| C(1) | 0·4527 (3) | 0·3117 (5) | 0·7460 (1) | 4·1 (1) |
| C(2) | 0·4793 (4) | 0·3142 (5) | 0·7963 (1) | 4·8 (1) |
| C(3) | 0·4105 (4) | 0·2293 (6) | 0·8303 (2) | 5·0 (1) |
| C(4) | 0·3126 (4) | 0·1449 (6) | 0·8145 (1) | 4·8 (1) |
| C(5) | 0·2854 (4) | 0·1421 (5) | 0·7648 (1) | 4·4 (1) |
| C(6) | 0·3558 (3) | 0·2242 (4) | 0·7291 (1) | 3·7 (1) |
| C(7) | 0·3158 (3) | 0·2187 (5) | 0·6774 (2) | 4·0 (1) |
| C(8) | 0·3768 (3) | 0·2314 (5) | 0·6345 (1) | 3·8 (1) |
| C(9) | 0·5046 (3) | 0·2471 (6) | 0·6326 (1) | 4·7 (1) |
| C(10) | 0·5535 (4) | 0·1636 (8) | 0·5856 (2) | 6·0 (1) |
| C(11) | 0·5014 (4) | 0·2465 (9) | 0·5401 (2) | 6·3 (2) |
| C(12) | 0·3744 (4) | 0·2490 (5) | 0·5423 (1) | 4·8 (1) |
| C(13) | 0·3182 (3) | 0·2332 (5) | 0·5862 (1) | 4·2 (1) |
| C(14) | 0·3138 (5) | 0·2709 (8) | 0·4951 (2) | 6·7 (2) |
| O(14) | 0·3615 (3) | 0·2909 (3) | 0·4560 (1) | 8·9 (1) |

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

| | | | |
|---|---|---|---|
| Cl—C(14) | 1·740 (3) | C(8)—C(9) | 1·505 (5) |
| C(1)—C(2) | 1·390 (4) | C(8)—C(13) | 1·471 (4) |
| C(1)—C(6) | 1·388 (5) | C(9)—C(10) | 1·523 (6) |
| C(2)—C(3) | 1·375 (6) | C(10)—C(11) | 1·503 (8) |
| C(3)—C(4) | 1·378 (6) | C(11)—C(12) | 1·491 (7) |
| C(4)—C(5) | 1·376 (4) | C(12)—C(13) | 1·358 (4) |
| C(5)—C(6) | 1·408 (5) | C(12)—C(14) | 1·465 (6) |
| C(6)—C(7) | 1·469 (6) | C(14)—O(14) | 1·201 (6) |
| C(7)—C(8) | 1·362 (6) | | |

| | | | |
|---|---|---|---|
| Cl—C(13)—C(8) | 117·3 (2) | C(5)—C(6)—C(7) | 116·6 (3) |
| Cl—C(13)—C(12) | 119·6 (2) | C(1)—C(6)—C(7) | 125·8 (3) |
| C(3)—C(1)—C(6) | 91·2 (2) | C(6)—C(7)—C(9) | 99·8 (2) |
| C(2)—C(1)—C(6) | 120·6 (3) | C(6)—C(7)—C(8) | 129·3 (4) |
| C(1)—C(2)—C(3) | 120·7 (3) | C(5)—C(7)—C(9) | 127·2 (2) |
| C(2)—C(3)—C(4) | 119·7 (4) | C(7)—C(8)—C(13) | 120·3 (3) |
| C(3)—C(4)—C(5) | 120·0 (4) | C(7)—C(8)—C(9) | 123·9 (3) |
| C(4)—C(5)—C(6) | 121·4 (4) | C(9)—C(8)—C(13) | 115·8 (3) |
| C(3)—C(5)—C(7) | 123·9 (2) | C(8)—C(9)—C(10) | 111·8 (3) |
| C(1)—C(6)—C(5) | 117·5 (2) | C(7)—C(9)—C(11) | 117·6 (2) |

| | | | |
|---|---|---|---|
| C(8)—C(9)—C(10)—C(11) | −51·7 (5) | C(11)—C(12)—C(13)—C(8) | 5·9 (6) |
| C(9)—C(10)—C(11)—C(12) | −46·2 (5) | C(12)—C(13)—C(8)—C(9) | −0·3 (5) |
| C(10)—C(11)—C(12)—C(13) | 18·1 (6) | C(13)—C(8)—C(9)—C(10) | −28·3 (4) |

Final difference Fourier map featureless with $\Delta\rho$ within $\pm 0{\cdot}19$ e Å$^{-3}$. The atomic scattering factors used for all atoms were as provided in the *SHELX*76 program. Computer programs: *PARST* (Nardelli, 1983) for geometrical calculations, *MOLDRAW* (Ugliengo, Borzani & Viterbo, 1988) for molecular illustrations. A view of the molecule with the adopted atom numbering is shown in Fig. 1. Table 1* lists the final atomic coordinates and equivalent isotropic thermal parameters of non-H atoms. The bond lengths, bond angles and selected torsion angles are shown in Table 2.

**Related literature.** The compound has antifungal activity. The X-ray crystal structure analysis of similar compounds has not so far been reported in the literature.

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* Lists of structure factors, anisotropic thermal parameters least-squares-planes calculations, bond lengths and angles involving H atoms and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53004 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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# Structure of 5,5′-Dibromo-2,2′-bipyridine

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**Abstract.** $C_{10}H_6Br_2N_2$, $M_r = 313{\cdot}99$, monoclinic, $P2_1/a$, $a = 21{\cdot}072$ (4), $b = 5{\cdot}956$ (1), $c = 3{\cdot}997$ (1) Å, $\beta = 91{\cdot}78$ (2)°, $V = 501{\cdot}4$ (2) Å$^3$, $Z = 2$, $D_x = 2{\cdot}08$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71068 Å, $\mu = 79{\cdot}65$ cm$^{-1}$, $F(000) = 300$, room temperature, $R = 0{\cdot}037$ for 830 reflections. The molecule has crystallo-

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