Table 2. *Interatomic distances* (Å) *and angles* (°)

| | | | |
|---|---|---|---|
| C5—Br | 1·899 (7) | C2—N1 | 1·344 (5) |
| C6—N1 | 1·339 (6) | N2—C2 | 1·355 (12) |
| C5—C6 | 1·374 (7) | | |
| C6—N1—C2 | 116·3 (5) | N1—C2—N1 | 126·3 (7) |
| N2—C2—N1 | 116·8 (4) | C5—C6—N1 | 120·8 (6) |
| C6—C5—C6 | 119·5 (7) | C6—C5—Br | 120·2 (3) |

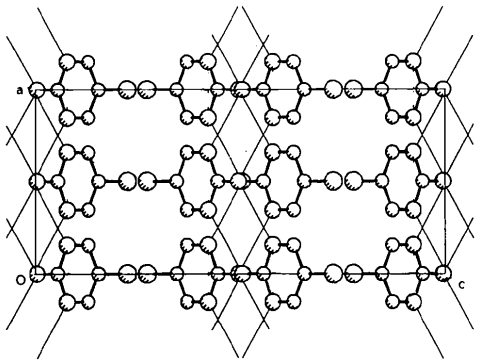


Fig. 2. The contents of the unit cell viewed down the *b* axis, showing hydrogen bonding between the bases.

computer. The atomic numbering is shown in the perspective drawing (Fig. 1), and tables of atomic parameters, bond lengths and angles are given (Tables 1 and 2). Fig. 2 shows a view of the molecular packing.*

**Related literature.** Base-pairing between nucleosides, nucleotides and nucleobases has been reviewed by Wilson & Tollin (1987).

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51167 (7 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 Intrasil Brilliant Yellow*

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**Abstract.** 3-(5-Chloro-2-benzoxazolyl)-7-(diethylamino)-2*H*-1-benzopyran-2-one, $C_{20}H_{17}ClN_2O_3$, $M_r$ = 369, monoclinic, $P2_1/a$, $a$ = 9·262 (2), $b$ = 13·282 (2), $c$ = 14·453 (2) Å, $\beta$ = 104·16 (2)°, $V$ = 1724 (1) Å$^3$, $Z$ = 4, $D_m$ (flotation in KI solution) = 1·43, $D_x$ = 1·42 Mg m$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·7107 Å, $\mu$ = 2·51 cm$^{-1}$, $F(000)$ = 768, $T$ = 293 K, $R$ = 0·056 for 1071 observed reflections. The dihedral angle between the benzoxazoline and the benzopyrone moieties is 5·9°, showing significant deviation from planarity. The two ring systems are planar.

**Experimental.** The title compound is a commercial dye. It was dissolved in chloroform and purified by column chromatography using 20% chloroform and 80% petroleum ether and recrystallized from acetone. Crystal approx. 0·20 × 0·05 × 1·00 mm. Nonius CAD-4F-11M diffractometer, graphite-monochromated radiation, $\omega/2\theta$ scan mode, scan speed 1° min$^{-1}$,

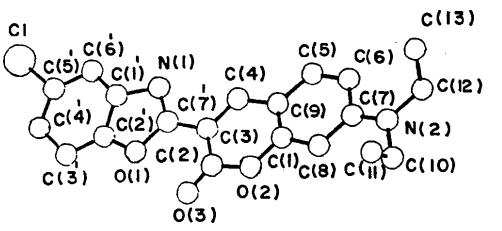


Fig. 1. A perspective view of the molecule with atomic numbering.

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0108-2701/88/101858-03$03.00

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters for non-hydrogen atoms with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}(B_{11}a^2 + B_{22}b^2 + B_{33}c^2 + B_{13}ac\cos\beta).$$

| | $x$ | $y$ | $z$ | $B_{eq}$(Å$^2$) |
|---|---|---|---|---|
| Cl | 7195 (2) | 4257 (2) | 211 (2) | 7·36 |
| O(1) | 2787 (5) | 1624 (4) | −2117 (3) | 3·93 |
| O(2) | −1052 (4) | 976 (3) | −4368 (3) | 3·25 |
| O(3) | 841 (5) | 359 (4) | −3313 (4) | 4·96 |
| C(1′) | 3775 (7) | 3154 (6) | −1679 (5) | 3·49 |
| C(2′) | 3913 (7) | 2117 (6) | −1498 (5) | 3·47 |
| C(3′) | 5048 (1) | 1745 (7) | −802 (6) | 5·28 |
| C(4′) | 6072 (8) | 2433 (7) | −265 (5) | 4·95 |
| C(5′) | 5894 (9) | 3439 (7) | −465 (6) | 4·97 |
| C(6′) | 4749 (9) | 3830 (6) | −1159 (6) | 4·65 |
| C(7′) | 1987 (7) | 2378 (5) | −2643 (5) | 3·19 |
| C(1) | −1895 (8) | 1744 (5) | −4822 (5) | 3·18 |
| C(2) | 213 (8) | 1103 (5) | −3642 (5) | 3·56 |
| C(3) | 639 (7) | 2137 (5) | −3386 (5) | 2·97 |
| C(4) | −210 (7) | 2897 (5) | −3859 (4) | 2·69 |
| C(5) | −2423 (8) | 3506 (5) | −5074 (5) | 3·40 |
| C(6) | −3663 (8) | 3274 (5) | −5810 (5) | 3·48 |
| C(7) | −4028 (9) | 2254 (6) | −6052 (5) | 3·75 |
| C(8) | −3132 (8) | 1492 (5) | −5538 (5) | 3·64 |
| C(9) | −1526 (8) | 2737 (6) | −4574 (5) | 3·08 |
| C(10) | −5669 (8) | 972 (6) | −7028 (5) | 4·65 |
| C(11) | −4776 (9) | 521 (7) | −7637 (6) | 5·49 |
| C(12) | −6157 (7) | 2814 (5) | −7345 (5) | 3·55 |
| C(13) | −5524 (9) | 3269 (6) | −8118 (6) | 4·88 |
| N(1) | 2463 (6) | 3280 (5) | −2449 (4) | 4·25 |
| N(2) | −5226 (6) | 2040 (4) | −6781 (4) | 3·96 |

$\theta < 23·5°$, $h$ 0 to 10, $k$ 0 to 14, $l$ −16 to 16, 2650 unique reflections collected, 1071 judged significant [$|F_o| > 3\sigma(|F_o|)$], lattice parameters from 25 reflections ($16 < 2\theta < 33°$), three standard reflections ($06\bar{3}$, $32\bar{5}$, $20\bar{4}$) every 3600 s, 4% variation in intensity. No correction for absorption. Structure solved by direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Full-matrix least-squares refinement (on $F$) using anisotropic thermal parameters (isotropic thermal parameters for H atoms, initial H positions calculated by stereochemistry and confirmed by difference Fourier synthesis); convergence at $R = 0·056$, $S = 1·64$, $w = 1·0$, $(\Delta/\sigma)_{max} = 0·2$, final $\Delta\rho$ excursions $< |0·3|$ e Å$^{-3}$. No correction for secondary extinction. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Correction for anomalous scattering used. Program *LALS* (Gantzel, Sparks & Trueblood, 1961) for refinement. The atomic parameters with their e.s.d.'s and equivalent isotropic thermal parameters are given in Table 1.* Bond lengths and bond angles involving the non-hydrogen atoms are given in Table 2. Fig. 1 gives a perspective view of the molecule along with the crystallographic numbering of atoms.

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

| | | | |
|---|---|---|---|
| Cl–C(5′) | 1·735 (9) | | |
| O(1)–C(2′) | 1·364 (9) | | |
| O(1)–C(7′) | 1·363 (8) | | |
| | | C(2′)–O(1)–C(7′) | 103·7 (5) |
| O(2)–C(1) | 1·353 (8) | | |
| O(2)–C(2) | 1·378 (8) | | |
| | | C(1)–O(2)–C(2) | 124·0 (5) |
| O(3)–C(2) | 1·185 (9) | | |
| C(1′)–C(2′) | 1·402 (11) | | |
| C(1′)–C(6′) | 1·361 (11) | | |
| C(1′)–N(1) | 1·443 (9) | | |
| | | C(2′)–C(1′)–C(6′) | 121·8 (7) |
| | | C(2′)–C(1′)–N(1) | 106·2 (6) |
| | | C(6′)–C(1′)–N(1) | 132·0 (7) |
| C(2′)–C(3′) | 1·358 (10) | | |
| | | O(1)–C(2′)–C(1′) | 109·2 (6) |
| | | O(1)–C(2′)–C(3′) | 129·8 (7) |
| | | C(1′)–C(2′)–C(3′) | 121·0 (7) |
| C(3′)–C(4′) | 1·406 (11) | | |
| | | C(2′)–C(3′)–C(4′) | 117·9 (7) |
| C(4′)–C(5′) | 1·368 (13) | | |
| | | C(3′)–C(4′)–C(5′) | 119·2 (7) |
| C(5′)–C(6′) | 1·371 (12) | | |
| | | Cl–C(5′)–C(4′) | 117·4 (7) |
| | | Cl–C(5′)–C(6′) | 118·7 (7) |
| | | C(4′)–C(5′)–C(6′) | 123·8 (8) |
| | | C(1′)–C(6′)–C(5′) | 116·3 (8) |
| C(7′)–C(3) | 1·469 (10) | | |
| C(7′)–N(1) | 1·283 (9) | | |
| | | O(1)–C(7′)–C(3) | 119·7 (6) |
| | | O(1)–C(7′)–N(1) | 117·0 (6) |
| | | C(3)–C(7′)–N(1) | 123·3 (6) |
| C(1)–C(8) | 1·385 (10) | | |
| C(1)–C(9) | 1·387 (10) | | |
| | | O(2)–C(1)–C(8) | 117·0 (6) |
| | | O(2)–C(1)–C(9) | 121·0 (6) |
| | | C(8)–C(1)–C(9) | 122·0 (7) |
| C(2)–C(3) | 1·451 (9) | | |
| | | O(2)–C(2)–O(3) | 116·5 (6) |
| | | O(2)–C(2)–C(3) | 115·9 (6) |
| | | O(3)–C(2)–C(3) | 127·6 (7) |
| C(3)–C(4) | 1·357 (9) | | |
| | | C(7′)–C(3)–C(2) | 121·5 (6) |
| | | C(7′)–C(3)–C(4) | 119·3 (6) |
| | | C(2)–C(3)–C(4) | 119·2 (6) |
| C(4)–C(9) | 1·408 (10) | | |
| | | C(3)–C(4)–C(9) | 123·2 (6) |
| C(5)–C(6) | 1·396 (10) | | |
| C(5)–C(9) | 1·401 (10) | | |
| | | C(6)–C(5)–C(9) | 120·4 (7) |
| C(6)–C(7) | 1·419 (10) | | |
| | | C(5)–C(6)–C(7) | 120·0 (7) |
| C(7)–C(8) | 1·401 (10) | | |
| C(7)–N(2) | 1·361 (10) | | |
| | | C(6)–C(7)–C(8) | 119·0 (7) |
| | | C(6)–C(7)–N(2) | 119·3 (7) |
| | | C(8)–C(7)–N(2) | 121·6 (7) |
| | | C(1)–C(8)–C(7) | 119·7 (7) |
| | | C(1)–C(9)–C(4) | 116·6 (7) |
| | | C(1)–C(9)–C(5) | 118·8 (7) |
| | | C(4)–C(9)–C(5) | 124·5 (7) |
| C(10)–C(11) | 1·475 (11) | | |
| C(10)–N(2) | 1·496 (10) | | |
| | | C(11)–C(10)–N(2) | 111·4 (6) |
| C(12)–C(13) | 1·509 (11) | | |
| C(12)–N(2) | 1·456 (9) | | |
| | | C(13)–C(12)–N(2) | 114·4 (6) |
| | | C(1′)–N(1)–C(7′) | 103·9 (6) |

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51129 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

**Related literature.** Related structures with planar benzopyran and benzoxazole rings: Cantrell & Stalzer (1982); Dreher, Bracht, Mobayed, Hütter, Winter & Rieker (1982).

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# Structure of a 1,4-Oxazepin-5-one Derivative

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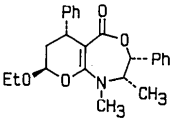
**Abstract.** (2*S*,3*R*,6*R*,8*R*)-8-Ethoxy-2,3,7,8-tetrahydro-1,2-dimethyl-3,6-diphenyl-1*H*,6*H*-pyrano[2,3-*e*][1,4]oxazepin-5-one–dichloromethane (1/1), $C_{24}H_{27}NO_4.CH_2Cl_2$, $M_r = 478{\cdot}42$, orthorhombic, $P2_12_12_1$, $a = 11{\cdot}885\ (1)$, $b = 12{\cdot}738\ (1)$, $c = 15{\cdot}817\ (2)$ Å, $V = 2394{\cdot}43$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}327$ Mg m$^{-3}$, $\lambda$(Mo $K\alpha$) $= 0{\cdot}71069$ Å, $\mu = 0{\cdot}30$ mm$^{-1}$, $F(000) = 1008$, $T = 298$ K, $R = 0{\cdot}049$ for 3497 observed reflections. The structure was investigated to determine the relative configuration, which could not be established unambiguously by NMR. The seven-membered and the six-membered rings both adopt a half-chair conformation.

**Experimental.** Title compound (I): crystal size 0·4 × 0·4 × 0·6 mm. Stoe–Siemens four-circle diffractometer, monochromated Mo $K\alpha$ radiation, profile-fitting




(I)

mode involving variable scan width and speed (Clegg, 1981). 4882 reflections measured, $2\theta_{max}$ 50°, $\pm h + k + l$, three check reflections with no significant intensity change. 4265 unique reflections ($R_{int} = 0{\cdot}022$), of which 3497 with $F > 4\sigma(F)$ were used for all calculations (*SHELXS*86, Sheldrick, 1985; *SHELX*76, Sheldrick, 1976). Cell constants refined from $\pm 2\theta$ values of 40 reflections in the range 20–25°. Absorption correction was not applied. Extinction correction was applied yielding a value of 0·0021 (3) for the secondary-extinction coefficient. Structure solution by direct methods. Refinement on $F$ to $R = 0{\cdot}049$, $wR = 0{\cdot}058$; all non-H atoms anisotropic, H atoms were included using a riding model [C—H 0·96 Å, $U$(H) = 0·08 Å$^2$, except for methyl protons $U$(H) = 0·1 Å$^2$]. We refined 290 parameters, $S = 1{\cdot}853$, weighting scheme $w^{-1} = \sigma^2(F) + 0{\cdot}0004F^2$ which led to a featureless analysis of variance in terms of sin$\theta$ and $F_o$, max. $\Delta/\sigma = 0{\cdot}006$, max. and min. heights in final $\Delta\rho$ map 0·37 and −0·35 e Å$^{-3}$ respectively. A Rogers (1981) $\eta$-refinement [$\eta = 1{\cdot}0\ (2)$] confirmed the known absolute configuration. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Atomic parameters are given in Table 1, selected bond distances and angles in Table 2.* Fig. 1 shows a thermal ellipsoid plot with the atom numbering, and Fig. 2 indicates the conformation of the molecule.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51152 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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