

Tableau 6. *Principaux contacts intermoléculaires*

Code de symétrie: (i) x, y, z ; (ii) $x, 1 + y, z$; (iii) $1 - x, 2 - y, 1 - z$; (iv) $2 - x, 1 + y, 1 - z$; (v) $1 - x, 1 + y, 1 - z$; (vi) $1 - x, y, 1 - z$.

Liaisons hydrogène (distances en Å, angles en deg)

| | | | |
|--|-----------|---|----------|
| N(10 ^a)...N(16 ^b) | 3,060 (4) | H(100 ^a)...N(16 ^b) | 2,06 (5) |
| N(10 ^a)...Cl(18 ^b) | 3,081 (3) | N(10 ^a)...H(100 ^a)...N(16 ^b) | 172 (4) |
| O(8 ^a)...N(16 ^b) | 3,043 (4) | H(101 ^a)...Cl(18 ^b) | 2,18 (5) |
| O(8 ^a)...Cl(18 ^b) | 3,124 (3) | N(10 ^a)...H(101 ^a)...Cl(18 ^b) | 163 (4) |
| | | O(8 ^a)...H(161 ^a) | 2,15 (5) |
| | | O(8 ^a)...H(161 ^a)...N(16 ^b) | 160 (5) |
| | | H(80 ^a)...Cl(18 ^b) | 2,23 (6) |
| | | O(8 ^a)...H(80 ^a)...Cl(18 ^b) | 150 (5) |

Contacts de van der Waals (<3,5 Å)

| | | | |
|--|-------------|--|-------------|
| O(8 ^a)...N(16 ^b) | 2,966 (4) Å | O(8 ^a)...Cl(17 ^a) | 3,498 (3) Å |
| O(8 ^a)...O(8 ^b) | 3,057 (4) | N(16 ^b)...Cl(18 ^b) | 3,463 (3) |

accord avec les valeurs trouvées par Leger (1979) dans un certain nombre de drogues β -adrénergiques.

Un réseau de liaisons hydrogène et de contacts de van der Waals est responsable de la cohésion cristalline (Tableau 6). La Fig. 2 représente la projection de la structure sur le plan (100).

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4a,5,8,8a-Tetrahydro-5,8-ethano-1,4-naphthoquinone

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Abstract. C₁₂H₁₂O₂, $M_r = 188.2$, orthorhombic, $P2_12_12_1$, $a = 6.725$ (1), $b = 11.636$ (2), $c = 12.616$ (1) Å, $Z = 4$, $D_x = 1.27$ Mg m⁻³, $F(000) = 400$; $R = 0.032$ for 1480 observed reflections. The conformation of the molecule is such that the bridge-head substituents are eclipsed and the six-membered rings not twisted with respect to each other, giving the molecule approximate m symmetry. The 2-ene-1,4-dione ring is close to planar, while the remaining six-membered rings are in boat conformations. The C=C bonds are essentially parallel with a mid-point separation of 3.53 Å.

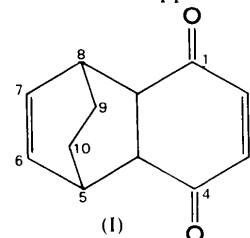
Introduction. Recrystallization of (I) from hexane afforded large poorly formed chunks from which a fragment of dimensions ca 0.25 × 0.40 × 0.30 mm was cut. A series of precession photographs provided an initial unit cell, the Laue symmetry mmm , and the space group $P2_12_12_1$. Accurate unit-cell parameters were determined by a least-squares analysis of the setting angles of 25 reflections automatically located and centred on an Enraf–Nonius CAD-4 diffractometer ($9^\circ < \theta < 14^\circ$, graphite-monochromated Mo

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Références

- BEALE, J. P. & GRAINGER, C. T. (1972). *Cryst. Struct. Commun.* **1**, 71–74.
- GADRET, M., LEGER, J. M., CARPY, A. & BERTHOD, H. (1978). *Eur. J. Med. Chem. Chim. Ther.* **13**, 367–372.
- GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1970). *Acta Cryst.* **B26**, 274–285.
- KAMBUROFF, P. L., PRIME, F. J. & SCHMIDT, O. P. (1977). *Br. J. Clin. Pharmacol.* **4**, 67–71.
- LEGER, J. M. (1979). Thèse Doctorat ès Sciences Physiques, Univ. de Bordeaux.
- LEGER, J. M., GADRET, M. & CARPY, A. (1978). *Acta Cryst.* **B34**, 3705–3709.
- LEGER, J. M., GADRET, M. & CARPY, A. (1980). *Mol. Pharmacol.* **17**, 339–343.
- LEGER, J. M., GOURSOLLE, M., GADRET, M. & CARPY, A. (1978). *Acta Cryst.* **B34**, 1203–1208.
- SCHOMAKER, V., WASER, J., MARSH, R. E. & BERGMAN, G. (1959). *Acta Cryst.* **12**, 600–604.

$K\alpha$ radiation). The intensities were collected with an ω - 2θ scan, $\Delta\omega = (0.8 + 0.35 \tan \theta)^\circ$, an aperture 4 mm high and $(1.5 + 1.0 \tan \theta)$ mm wide, a final acceptance limit of 30σ at $10.06^\circ \text{ min}^{-1}$ (in ω), and a maximum recording time of 70 s. 1939 intensities were recorded out to $\theta = 26^\circ$; measurements were made for hkl and $\bar{h}kl$ octants, to improve later anomalous-dispersion refinements. Of these, 1480 (76%) had $I/\sigma(I) > 3$ with $\sigma^2(I) = S + B + (0.04S)^2$ (S being the scan and B the time-corrected background count). During the data collection three standard reflections were checked periodically and their intensities all fell steadily to 87% of their initial value. This was corrected for during data processing where Lorentz and polarization corrections were applied.



All non-hydrogen atoms were located by direct methods using *MULTAN* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) using 484 $|E|$ values >0.9 derived from a *K*-curve method (Ladd, 1978). The $|E|$ statistics were consistent with the non-centrosymmetric space group. Following anisotropic refinement of C and O, the H atoms were located from a difference map and were assigned isotropic thermal parameters. Scattering factors for H were taken from Stewart, Davidson & Simpson (1965) and all others from Cromer & Mann (1968). The final refinements included all atomic parameters, an isotropic extinction parameter *g* (Becker & Coppens, 1974, 1975), and the anomalous-dispersion components for C and O (Cromer & Liberman, 1970). The final atomic coordinates, presented in Table 1, result from the final cycle of least squares which gave $R = 0.032$ ($R = \sum |ΔF| / \sum |F_o|$) for 1480 observed reflections (0.054 for all 1980 reflections). The function minimized was $\sum w(|F_o - |F_c||^2)$ with $w = 1/\sigma^2(F)$, giving $R_w = [\sum w(|F_o - |F_c||^2) / \sum w|F_o|^2]^{1/2} = 0.0432$ and σ_1 (goodness of fit) = 1.45.* The mean and maximum shifts on the final

* Lists of structure factors, thermal parameters, C–H distances and selected torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35485 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final positional parameters (fractional $\times 10^4$, for H $\times 10^3$) and isotropic thermal parameters (\AA^2) with estimated standard deviations in parentheses

| | x | y | z | Mean U^* |
|--------|-----------|-----------|----------|------------|
| C(1) | 9274 (3) | 368 (2) | 2959 (2) | 54 |
| C(2) | 9325 (4) | 1614 (2) | 2807 (2) | 64 |
| C(3) | 7725 (4) | 2258 (2) | 2887 (2) | 66 |
| C(4) | 5773 (3) | 1787 (2) | 3129 (2) | 57 |
| C(4a) | 5544 (3) | 555 (2) | 3455 (2) | 50 |
| C(5) | 4843 (4) | 516 (2) | 4627 (2) | 65 |
| C(6) | 6548 (5) | 835 (3) | 5324 (2) | 71 |
| C(7) | 8088 (4) | 159 (3) | 5227 (2) | 69 |
| C(8) | 7857 (5) | -777 (2) | 4440 (2) | 67 |
| C(8a) | 7413 (3) | -207 (2) | 3346 (2) | 49 |
| C(9) | 6045 (6) | -1510 (3) | 4735 (3) | 86 |
| C(10) | 4245 (5) | -733 (3) | 4868 (3) | 84 |
| O(1) | 10760 (3) | -191 (2) | 2771 (2) | 82 |
| O(4) | 4318 (3) | 2406 (2) | 3057 (2) | 96 |
| H(2) | 1064 (5) | 195 (3) | 258 (3) | 98 |
| H(3) | 777 (4) | 305 (2) | 276 (2) | 74 |
| H(4a) | 459 (4) | 28 (2) | 305 (2) | 65 |
| H(5) | 381 (4) | 102 (3) | 471 (3) | 78 |
| H(6) | 651 (5) | 149 (3) | 577 (3) | 93 |
| H(7) | 928 (5) | 30 (3) | 561 (3) | 90 |
| H(8) | 900 (5) | -123 (3) | 439 (3) | 87 |
| H(8a) | 714 (3) | -83 (2) | 285 (2) | 51 |
| H(91) | 650 (5) | -186 (3) | 542 (3) | 95 |
| H(92) | 574 (6) | -213 (3) | 420 (3) | 119 |
| H(101) | 317 (5) | -94 (3) | 437 (3) | 92 |
| H(102) | 370 (5) | -77 (3) | 556 (3) | 101 |

* $10^3 (U_{11} + U_{22} + U_{33})/3$; $\sigma = 2$ for non-hydrogen atoms, 10 for H atoms. The high ethano-bridge parameters may indicate slight disorder.

cycle were 0.04 and 0.20 standard deviations, respectively. Weighting analyses confirmed the suitability of the chosen weights. A difference Fourier synthesis after the final cycle showed random fluctuations of up to $\pm 0.2 e \text{\AA}^{-3}$. Refinement of the enantiomorph, the coordinates in Table 1 inverted through (0,0,0), produced only minor differences, with a slight increase in R_w to 0.0433. In view of the fact that this is calculated as a significant difference at the 2.5% level (Hamilton, 1965) the final refinements to the coordinates in Table 1, with both *hkl* and $\bar{h}k\bar{l}$ (three-dimensional) reflections included, were accepted. The final value of *g* was 5.5×10^4 .

Discussion. The X-ray study of the Diels–Alder adduct 4a,5,8,8a-tetrahydro-5,8-ethano-1,4-naphthoquinone (I) was undertaken as part of a study of the relationship between structure and photochemical reactivity (Scheffer, Bhandari, Gayler & Wostradowski, 1975; Scheffer, Jennings & Louwerens, 1976; Scheffer & Dzakpasu, 1978; Phillips & Trotter, 1977; Dzakpasu, Phillips, Scheffer & Trotter, 1976; Appel, Greenhough, Scheffer & Trotter, 1979; Appel, Greenhough, Scheffer, Trotter & Walsh, 1980). In both solution and the solid state, (I) undergoes an intramolecular 2 + 2 cycloadditive photoconversion (Cookson, Crundwell, Hill & Hudec, 1964). The photochemical study of *cis*-4a,5,8,8a-tetrahydro-1,4-naphthoquinone and several of its derivatives (Scheffer, Jennings & Louwerens, 1976; Dzakpasu, Phillips, Scheffer & Trotter, 1976) provided no examples of intramolecular 2 + 2 cycloaddition in solution or the solid state, while the analogous 1-naphthoquin-4 α -ol series all followed this reaction in solution (Appel, Greenhough, Scheffer, Trotter & Walsh, 1980). Structural studies of both series of complexes have shown that regardless of substitution all of the substrates possess the ‘twist’ conformation in the solid state (Phillips & Trotter, 1977; Greenhough & Trotter, 1980), producing well separated (*ca* 4.3–4.5 Å) and non-parallel C=C bonds. The ethano bridge of (I) constrains the two C=C bonds to be essentially parallel; the X-ray study was undertaken to establish the orientation of the ene-dione moiety with respect to the remainder of the cyclohexenedione ring, and hence establish the distances involved in the solid-state photoconversion.

The molecular structure of (I) is shown in Fig. 1. The discrete molecular units in the crystal structure have approximate *m* symmetry and exhibit a near-planar cyclohexenedione ring with the remaining six-membered rings in boat conformations. The deviations from planarity of the C(1) to C(4a), C(8a) ring are such that C(2), C(3), C(4a), and C(8a) are in plane with C(1) and C(4) displaced by 0.07 and 0.10 Å, respectively, on the same side of the plane as are the bridgehead H atoms. This conformation is thus a deviation from planarity towards the boat con-

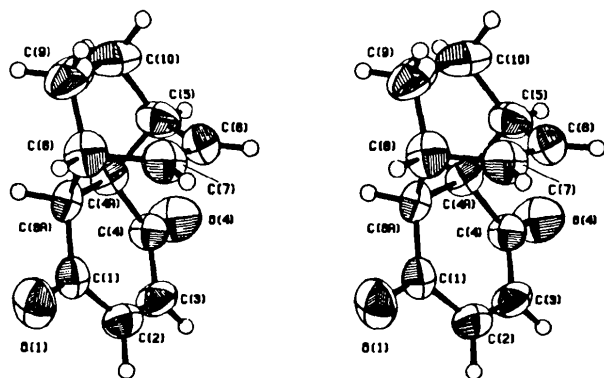


Fig. 1. Stereodiagram of 4a,5,8,8a-tetrahydro-5,8-ethano-1,4-naphthoquinone. Thermal ellipsoids are at the 50% probability level.

formation which gives the closest approach of the two C=C bonds in the molecule. The C=C bonds are virtually parallel with C(2)···C(7) and C(3)···C(6) intramolecular distances of 3.588 (4) and 3.580 (4) Å respectively, a mid-point separation of 3.584 Å, and C(2)···C(6) = 3.793 (4), C(3)···C(7) = 3.838 (4) Å. The two reacting C=C bonds thus conform to the geometrical requirements for *intermolecular* 2 + 2 photocycloaddition (parallel bonds separated by <4.1 Å, Schmidt, 1971), suggesting that a similar rule applies in unimolecular photoconversions of the same type.

Whereas *cis*-4a,5,8,8a-tetrahydro-1,4-naphthoquinone and 4a β ,5,8,8a β -tetrahydro-1-naphthoquin-4 α -ol and all their various substituted derivatives whose structures have been determined (Phillips & Trotter, 1977; Greenhough & Trotter, 1980) show the 'twist' conformation in the crystalline state, with H-C(4a)-C(8a)-H or Me-C(4a)-C(8a)-Me torsion angles of *ca* 60°, molecules of (I) are non-twisted with a bridgehead torsion angle H-C(4a)-C(8a)-H = 0 (2)°. The molecule is locked in this conformation by the CH₂-CH₂ bridge.

The remaining six-membered rings are in boat conformations showing torsional deviations of up to 7° from idealized boat values. C(5) and C(8) deviate by 0.68 and 0.67 Å from the C(6), C(7), C(9), C(10) plane, by -0.68 and -0.71 Å from the C(4a), C(8a), C(6), C(7) plane, and by -0.78 and -0.75 Å from the C(4a), C(8a), C(9), C(10) plane. The two carbonyl groups are planar to within 0.7 standard deviations and the C=C torsion angles are C(1)-C(2)=C(3)-C(4) = -0.3 (3) and C(5)-C(6)=C(7)-C(8) = 0.8 (3)°.

The bond lengths and angles given in Table 2 are generally close to expected values, with C(1)-C(8a)-C(4a) = 116.9 (2) and C(4)-C(4a)-C(8a) = 116.2 (2)° reflecting the planarity of the cyclohexenedione ring, and C(8)-C(7)=C(6) = 115.0 (3), C(5)-C(6)=C(7) = 113.8 (3)° indicating the strain in the boat-form cyclohexene rings.

Table 2. Bond distances (Å) and angles (°)

| | | | |
|------------------|-----------|------------------|-----------|
| C(1)-C(2) | 1.462 (3) | C(8)-C(8a) | 1.560 (3) |
| C(2)-C(3) | 1.315 (4) | C(1)-C(8a) | 1.501 (3) |
| C(3)-C(4) | 1.455 (4) | C(4a)-C(8a) | 1.544 (3) |
| C(4)-C(4a) | 1.500 (3) | C(1)-O(1) | 1.216 (3) |
| C(4a)-C(5) | 1.552 (4) | C(5)-C(10) | 1.539 (4) |
| C(5)-C(6) | 1.492 (4) | C(8)-C(9) | 1.533 (4) |
| C(6)-C(7) | 1.306 (4) | C(4)-O(4) | 1.218 (3) |
| C(7)-C(8) | 1.482 (4) | C(9)-C(10) | 1.521 (5) |
| C(1)-C(2)-C(3) | 122.4 (2) | C(4)-C(3)-C(2) | 122.6 (2) |
| C(8a)-C(1)-C(2) | 120.3 (2) | C(4a)-C(4)-C(3) | 120.7 (2) |
| C(8a)-C(1)-O(1) | 120.7 (2) | C(4a)-C(4)-O(4) | 120.2 (2) |
| C(2)-C(1)-O(1) | 119.0 (2) | C(3)-C(4)-O(4) | 119.1 (2) |
| C(1)-C(8a)-C(4a) | 116.9 (2) | C(4)-C(4a)-C(8a) | 116.2 (2) |
| C(1)-C(8a)-C(8) | 108.5 (2) | C(4)-C(4a)-C(5) | 108.7 (2) |
| C(4a)-C(8a)-C(8) | 108.8 (2) | C(8a)-C(4a)-C(5) | 108.4 (2) |
| C(8a)-C(8)-C(7) | 107.4 (2) | C(4a)-C(5)-C(6) | 108.7 (2) |
| C(8a)-C(8)-C(9) | 107.4 (2) | C(4a)-C(5)-C(10) | 107.2 (2) |
| C(7)-C(8)-C(9) | 109.2 (3) | C(6)-C(5)-C(10) | 108.6 (3) |
| C(8)-C(7)-C(6) | 115.0 (3) | C(5)-C(6)-C(7) | 113.8 (3) |
| C(5)-C(10)-C(9) | 109.4 (2) | C(8)-C(9)-C(10) | 109.2 (2) |

The eclipsed H bridgehead substituents at C(4a) and C(8a) are separated by H(4a)···H(8a) = 2.16 (3) Å but the external angles at these sites are close to tetrahedral values (105.3-109.4°). The eclipsed H substituents at C(9) and C(10) have contacts of H(92)···H(101) = 2.23 (5) and H(91)···H(102) = 2.28 (5) Å; the external angles at C(9) and C(10) do not indicate steric interference between the substituents. The only noteworthy intermolecular contact distances are O(1)···C(3)(2 - x, -½ + y, ½ - z) = 3.246 (3) and O(1)···H(3) = 2.37 (3) Å, with the angle C(3)-H(3)···O(1) = 156 (2)°.

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References

- APPEL, W. K., GREENHOUGH, T. J., SCHEFFER, J. R. & TROTTER, J. (1979). *J. Am. Chem. Soc.* **101**, 213-215.
- APPEL, W. K., GREENHOUGH, T. J., SCHEFFER, J. R., TROTTER, J. & WALSH, L. (1980). *J. Am. Chem. Soc.* **102**, 1158-1161.
- BECKER, P. J. & COPPENS, P. (1974). *Acta Cryst.* **A30**, 129-147.
- BECKER, P. J. & COPPENS, P. (1975). *Acta Cryst.* **A31**, 417-425.
- COOKSON, R. C., CRUNDWELL, E., HILL, R. R. & HUDEC, J. (1964). *J. Chem. Soc.* pp. 3062-3075.
- CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891-1898.
- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321-324.

- DZAKPASU, A. A., PHILLIPS, S. E. V., SCHEFFER, J. R. & TROTTER, J. (1976). *J. Am. Chem. Soc.* **98**, 6049–6051.
- GREENHOUGH, T. J. & TROTTER, J. (1980). *Acta Cryst.* **B36**, 1831–1835.
- HAMILTON, W. C. (1965). *Acta Cryst.* **18**, 502–510.
- LADD, M. F. C. (1978). Private communication.
- MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J. P. & WOOLFSON, M. M. (1978). *MULTAN 78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- PHILLIPS, S. E. V. & TROTTER, J. (1977). *Acta Cryst.* **B33**, 996–1003.
- SCHEFFER, J. R., BHANDARI, K. S., GAYLER, R. E. & WOSTRADOWSKI, R. A. (1975). *J. Am. Chem. Soc.* **97**, 2178–2189.
- SCHEFFER, J. R. & DZAKPASU, A. A. (1978). *J. Am. Chem. Soc.* **100**, 2163–2173.
- SCHEFFER, J. R., JENNINGS, B. M. & LOUWERENS, J. P. (1976). *J. Am. Chem. Soc.* **98**, 7040–7048.
- SCHMIDT, G. M. J. (1971). *Pure Appl. Chem.* **27**, 647–678.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

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2,3,4a β ,8a β -Tetramethyl-4a,5,8,8a-tetrahydro-1-naphthoquin-4 α -ol*

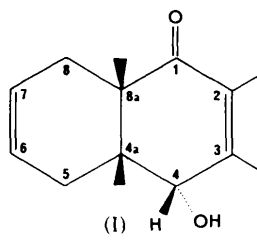
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Abstract. C₁₄H₂₀O₂, *M*_r = 220.3, triclinic, *P* $\bar{1}$, *a* = 6.9600 (5), *b* = 7.4762 (4), *c* = 12.4931 (11) Å, α = 83.875 (6), β = 78.039 (6), γ = 76.866 (5)°, *Z* = 2, *D*_x = 1.18 Mg m⁻³, *F*(000) = 240; *R* = 0.041 for 1677 observed reflections. The conformation of the molecule is twisted, with the bridgehead methyl groups staggered with a torsion angle of 63.0 (2)°, and the hydroxyl group pseudo-equatorial to the cyclohexenone moiety. Molecules in the crystal are linked by hydrogen bonds between symmetry-related molecules with O(1)⋯O(4) = 2.782 (2) Å.

Introduction. The photochemical study of various substituted 4a β ,5,8,8a β -tetrahydro-1-naphthoquin-4-ol systems has revealed reactivity differences with either no solid-state reaction or the formation of different products in solution and the solid state (Appel, Greenhough, Scheffer, Trotter & Walsh, 1980). The present paper continues these investigations, and describes the structure of 2,3,4a β ,8a β -tetramethyl-4a,5,8,8a-tetrahydro-1-naphthoquin-4 α -ol (I).



* IUPAC name: 4 α -hydroxy-2,3,4a β ,8a β -tetramethyl-4a,5,8,8a-tetrahydro-1(4*H*)-naphthalenone.

Recrystallization from dioxane afforded large colourless needles from which a fragment of dimensions *ca* 0.3 × 0.3 × 0.1 mm was cut. Accurate unit-cell parameters were determined by a least-squares analysis of the setting angles of 25 reflections automatically located and centred on an Enraf–Nonius CAD-4 diffractometer (35 < θ < 45°, graphite-monochromated Cu *K* α radiation). The intensities were collected with an ω -2 θ scan, $\Delta\omega$ = (0.7 + 0.14 × tan θ)°, an aperture 4 mm high and (1.75 + 1.0 tan θ) mm wide, a final acceptance limit of 30 σ at 10.06° min⁻¹ (in ω), and a maximum recording time of 75 s. Of 2336 recorded intensities out to θ = 70°, 1677 (72%) had *I*/ σ (*I*) > 3 with σ^2 (*I*) = *S* + *B* + (0.05*S*)² (*S* being the scan and *B* the time-corrected background count). During the data collection three standard reflections were checked periodically and their intensities remained constant to \pm 1%. Lorentz and polarization corrections were applied.

All non-hydrogen atoms were located by direct methods using *MULTAN* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) with 398 |*E*| values > 1.2 derived from a *K*-curve method (Ladd, 1978). The |*E*| statistics were consistent with the centrosymmetric space group. Following anisotropic refinement of C and O, the H atoms were located from a difference map and were assigned isotropic thermal parameters. Scattering factors for H were taken from Stewart, Davidson & Simpson (1965) and all others from Cromer & Mann (1968). The final refinements included all atomic parameters, and an isotropic extinction parameter *g* (Becker & Coppens, 1974,