e.s.d. reported); 1·257 (7) and 1·262 (11)–1·266 (11) (Saarinen & Korvenrantha, 1975*a*,*b*, respectively); 1·243 (7)–1·271 (6) (Bisi Castellani *et al.*, 1984); 1·18 (3)–1·26 (1) and 1·234 (6) (Bisi Castellani *et al.*, 1987, 1988, respectively). In fact, N coordination increases the double-bond character of the N—O bond, as is also shown by the N—O stretching frequencies found in the IR spectra (Baghlaf, Aly & Ganji, 1987).

A comparison of the bond lengths found in (*a*) $K(Clqo).\frac{1}{2}H_2O$, and in (*b*) the methanol adduct of $Cu(Clqo)_2$ (Bisi Castellani *et al.*, 1987) shows that the C—O and C—N bond distances are lengthened by chelation [(*a*): C—O, 1·254 (5); C—N, 1·348 (5) Å; (*b*): C—O, 1·278 (8); C—N, 1·358 (8) Å]. Such a difference in bond lengths is also observed between non-chelated (*anti*) and chelated (*syn*) *o*-quinone monooximes [*anti*-5-(2-chloroethoxy)-*o*-quinone 2-oxime (Van Oijen & Romers, 1966): C—O, 1·253 (7); C—N, 1·306 (7) Å; *syn*-5-*n*-propoxy-*o*-quinone 2-oxime (Romers, 1964): C—O, 1·270 (8); C—N, 1·319 (8) Å]. The other bond distances and angles within the organic ion compare well with the values reported for other *o*-quinone monooximes and their complexes.

The water molecule is hydrogen bonded to two N atoms through its two symmetry-equivalent H atoms: N···O(3*W*) = 2·888 (5) Å.

References

BAGHLAF, A. O., ALY, M. M. & GANJI, N. S. (1987). *Polyhedron*, **6**, 205–209.

BISI CASTELLANI, C., CARUGO, O. & CODA, A. (1987). *Inorg. Chem.* **26**, 671–675.

BISI CASTELLANI, C., CARUGO, O. & CODA, A. (1988). *Acta Cryst.* C**44**, 267–270.

BISI CASTELLANI, C., GATTI, G. & MILLINI, R. (1984). *Inorg. Chem.* **23**, 4004–4008.

BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1963). *ORFLS*. Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee, USA.

GRAZIANI, R., CASELLATO, U., VIGATO, P. A., TAMBURRINI, S. & VIDALI, M. (1983). *J. Chem. Soc. Dalton Trans.* pp. 697–705.

HAMILTON, W. C. (1959). *Acta Cryst.* **12**, 609–610.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99–101, 149–150. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

MCPARTLIN, M. (1973). *Inorg. Nucl. Chem. Lett.* **9**, 1207–1210.

NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

ROMERS, C. (1964). *Acta Cryst.* **17**, 1287–1294.

SAARINEN, H. & KORVENRANTA, J. (1975*a*). *Acta Chem. Scand. Ser. A*, **29**, 409–413.

SAARINEN, H. & KORVENRANTA, J. (1975*b*). *Acta Chem. Scand. Ser. A*, **29**, 861–865.

VAN OIJEN, J. W. L. & ROMERS, C. (1966). *Acta Cryst.* **20**, 169–176 and references cited therein.

ZACHARIASEN, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

Acta Cryst. (1988). C**44**, 267–270

Studies on Copper(II) Complexes of *o*-Quinone Monooximes. 5.* Bis(4-chloro-1,2-benzoquinone 2-oximato)(methanol)copper(II), $Cu(Clqo)_2.MeOH$

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(*Received* 15 *July* 1987; *accepted* 7 *October* 1987)

Abstract. $[Cu(C_6H_3ClNO_2)_2(CH_4O)]$, $M_r = 408{\cdot}7$, triclinic, $P\bar{1}$, $a = 12{\cdot}389\,(9)$, $b = 11{\cdot}730\,(9)$, $c = 5{\cdot}462\,(3)$ Å, $\alpha = 87{\cdot}21\,(5)$, $\beta = 78{\cdot}48\,(4)$, $\gamma = 74{\cdot}84\,(5)°$, $V = 750{\cdot}7\,(9)$ Å^3, $Z = 2$, $D_x = 1{\cdot}81$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71069$ Å, $\mu = 18{\cdot}82$ cm^{-1}, $F(000) = 410$, $T = 296$ K, final $R = 3{\cdot}3\%$ for 856 unique reflections. The copper(II) ion has distorted square-pyramidal coordination with the two bidentate ligands at the vertices of the square and the methanol O atom occupying the axial position.

Introduction. Copper(II) bis-chelated complexes of *o*-quinone monooximes are presently the focus of attention of many researchers because of their peculiar reactivity (McKillop & Sayer, 1976; Charalambous, Buckley & Brain, 1982; Charalambous, Kensett, Buckley, McPartlin, Mukerjee, Brain & Jenkins, 1983;

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* Parts 1, 2, 3 and 4 of this series: Bisi Castellani, Gatti & Millini (1984); Bisi Castellani & Millini (1984); Bisi Castellani, Carugo & Coda (1987); Bisi Castellani, Carugo, Tomba, Berbenni & Cinquetti (1987).

0108-2701/88/020267-04$03.00

Bisi Castellani & Millini, 1984; Bisi Castellani, Carugo, Tomba, Berbenni & Cinquetti, 1987). In fact they react with many potentially basic compounds (*e.g.* dimethylacetylenedicarboxylate, amines, triphenylphosphine, benzaldehyde, methanol) to give a wide variety of organic products. The mechanism of these reactions has been explained by assuming, as a preliminary step, the formation of adducts between the basic reagent and the Cu^{II} ion; in several cases a redox process is also involved, as evidenced by the Cu^{I}-containing residue recovered at the end of the reaction.

On these bases our work has been addressed (i) to the Lewis-acid behaviour of bis(4-chloro-1,2-benzoquinone 2-oximato)copper(II) [referred to as $Cu(Clqo)_2$] and (ii) to its redox properties (Bisi Castellani, Gatti & Millini, 1984; Bisi Castellani & Millini, 1984; Bisi Castellani, Buttafava, Carugo & Poggi, 1986; Bisi Castellani, Carugo & Coda, 1987).

(i) The Lewis acidity of $Cu(Clqo)_2$ toward halides and pseudohalides was studied carrying out the reactions in methanol. In several instances, instead or as well as the halide adduct, the methanol adduct was obtained, which was quite unexpected. No significant interaction between $Cu(Clqo)_2$ and methanol could be demonstrated spectrophotometrically, and all previous attempts to obtain this adduct by direct interaction between $Cu(Clqo)_2$ and CH_3OH had been unsuccessful. A detailed explanation of this behaviour is given elsewhere (Bisi Castellani, Carugo & Coda, 1987).

(ii) During the study of the redox behaviour of $Cu(Clqo)_2$ it was found that it is reduced to K[Cu(Clqo)(Clsqo)], [Clsqo = 4-chloro-1,2-benzosemiquinone 2-oximato (Fig. 1)], by the system KOCN/methanol, and it seems likely that the methanol adduct is an intermediate of this unusual redox reaction (Bisi Castellani *et al.*, 1986).

Therefore the determination of the crystal structure of the methanol adduct of $Cu(Clqo)_2$ seemed worthwhile; moreover, only one other crystal structure is reported for a pentacoordinated adduct of the (*o*-benzoquinone monooximato)copper(II) complex (McPartlin, 1973).

Experimental. Dark-brown crystals of the title compound suitable for X-ray structure determination were obtained by refluxing in methanol a suspension of $[Cu(Clqo)_2]$ with an excess of KBr (Bisi Castellani, Carugo & Coda, 1987) and allowing the resulting solution to evaporate slowly at room temperature.

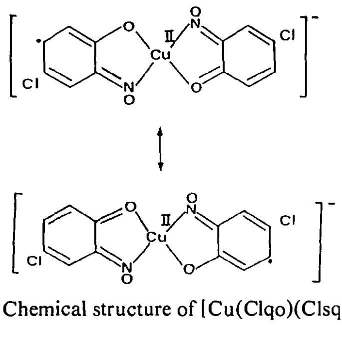

Fig. 1. Chemical structure of $[Cu(Clqo)(Clsqo)]^-$.

Dimensions of the prism used for the data collection: 0·77 × 0·13 × 0·19 mm. Philips PW1100 four-circle diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, ω–2θ scan. Lattice parameters from least-squares fit of 25 reflections in the 2θ range 10–30°, *via* the *LAT* Philips routine that measures accurate profiles of the reflections on both sides, positive and negative, of the 2θ circle. 2753 reflections measured in the whole reciprocal sphere with radius $(\sin\theta)/\lambda$ 0·4813 Å^{-1}, *i.e.* all the $\pm h$, $\pm k$, $\pm l$ set with $2 \leq \theta \leq 20°$, and with h, k, l maximum values 11, 10, 5, respectively. 1376 unique reflections, with $R_{\rm int} = 3\cdot6\%$ for the symmetry-equivalent reflections. 856 reflections with $I > 3\sigma_I$ used for refinement purposes. Three reflections (100; 020; 002) were monitored every 4 h, maximum intensity variation 5·5%; only random fluctuations detected. Lp correction. Analytical absorption correction for the sphere of the same volume as the crystal (radius = 0·01 cm) and North, Phillips & Mathews (1968) empirical correction by azimuthal scan of six reflections with $\chi \sim 90°$: 111, 323, 424 and centrosymmetric ones. Minimum and maximum absorption corrections applied to intensities, on a relative scale: 1·00 and 1·18. Space group $P\bar{1}$ from intensity statistics.

Structure solved by Patterson and Fourier methods. All non-H atoms refined anisotropically by full-matrix least squares, minimizing $\sum w(\Delta F)$, $w = 1/\sigma_F$; *ORFLS* program (Busing, Martin & Levy, 1963) and atomic scattering factors with anomalous-dispersion coefficients from *International Tables for X-ray Crystallography* (1974) were used. A secondary-extinction coefficient, according to Zachariasen (1963) was refined; final $g = 99\,(18) \times 10^{-7}$. All H atoms were located on difference maps; in the structure-factor calculation the idealized H coordinates were used, except for H(O3); $B = 3$ Å^2. Final maximum $\Delta/\sigma = 0\cdot002$; goodness of fit $S = 1\cdot33$; $\Delta\rho$ peaks in final ΔF between $-0\cdot49$ and $+0\cdot73$ e Å^{-3}. Final agreement factors for 856 unique F's: $R = 3\cdot3$; $wR = 2\cdot1\%$.

Discussion. Table 1 shows the atomic parameters, Table 2 the main bond distances and angles.[*]

As expected the copper complex has distorted square-pyramidal coordination with the two Clqo bidentate ligands at the vertices of the square and the methanol O atom occupying the axial position (Fig. 2).

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[*] Lists of structure factors, anisotropic thermal parameters, H-atom parameters and non-essential bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44438 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The Cu^{II} ion is not coplanar with the equatorial ligands, but is shifted *ca* 0·158 (2) Å toward the axial ligand [the maximum distance of N, O(1), N′, O(1′) from their least-squares plane concerns N′: 0·04 (1) Å].

Table 1. *Fractional atomic coordinates* ($\times 10^5$) *and equivalent isotropic thermal parameters*, B_{eq}, *with e.s.d.'s in parentheses*

B_{eq} according to Hamilton (1959), in Å^2.

	x	y	z	B_{eq}
Cu	8389 (1)	2256 (1)	414 (2)	3·80
Cl	2637 (2)	3524 (2)	2151 (3)	5·78
Cl′	14147 (2)	721 (2)	−2288 (3)	5·28
O(1)	7454 (4)	3147 (4)	−1890 (7)	3·75
O(2)	6723 (4)	1545 (4)	4324 (8)	4·68
O(1′)	9382 (3)	1119 (3)	2284 (7)	3·57
O(2′)	9969 (3)	2926 (4)	−3731 (7)	5·20
O(3)	8159 (3)	3809 (3)	2973 (6)	3·93
N	6882 (4)	2073 (4)	2329 (9)	3·14
N′	9856 (5)	2313 (5)	−1823 (9)	3·50
C(1)	6397 (6)	3214 (5)	−1039 (11)	3·14
C(2)	6009 (6)	2660 (5)	1229 (10)	2·92
C(3)	4831 (6)	2740 (5)	2293 (10)	3·26
C(4)	4063 (5)	3375 (6)	961 (12)	3·37
C(5)	4425 (6)	3905 (6)	−1328 (11)	4·41
C(6)	5531 (6)	3851 (5)	−2321 (10)	3·71
C(1′)	10429 (6)	1030 (5)	1339 (11)	3·12
C(2′)	10764 (6)	1644 (5)	−907 (11)	3·25
C(3′)	11891 (6)	1555 (5)	−2090 (10)	3·20
C(4′)	12722 (5)	879 (6)	−997 (11)	3·44
C(5′)	12437 (6)	268 (5)	1309 (11)	3·39
C(6′)	11345 (6)	313 (5)	2411 (10)	3·32
C(7)	8935 (6)	4545 (6)	2258 (11)	5·18

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

Cu–O(1)	1·960 (4)	Cu–O(1′)	1·961 (4)
Cu–N	2·009 (5)	Cu–N′	1·992 (5)
Cu–O(3)	2·274 (4)	C(7)–O(3)	1·440 (9)
N–O(2)	1·234 (6)	N′–O(2′)	1·242 (6)
N–C(2)	1·355 (8)	N′–C(2′)	1·362 (8)
O(1)–C(1)	1·282 (8)	O(1′)–C(1′)	1·275 (7)
O(1)–Cu–O(1′)	169·3 (2)	N–Cu–N′	172·7 (2)
O(1)–Cu–N	83·1 (2)	O(3)–Cu–N	87·2 (2)
O(1′)–Cu–N′	83·5 (2)	O(3)–Cu–N′	99·8 (2)
O(1)–Cu–N′	93·9 (2)	O(3)–Cu–O(1)	95·7 (2)
O(1′)–Cu–N	98·2 (2)	O(3)–Cu–O(1′)	94·9 (2)
Cu–N–O(2)	126·2 (5)	Cu–N′–O(2′)	126·2 (5)
Cu–N–C(2)	111·7 (4)	Cu–N′–C(2′)	111·2 (4)
Cu–O(1)–C(1)	110·3 (4)	Cu–O(1′)–C(1′)	110·8 (3)
N–Cu–O(3)–H(3″)	97·5 (4)	O(1)–Cu–O(3)–H(3″)	−179·7 (4)
N′–Cu–O(3)–H(3″)	−84·8 (4)	O(1′)–Cu–O(3)–H(3″)	−0·5 (4)

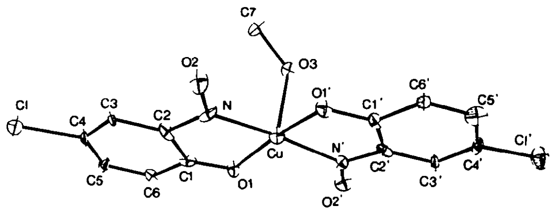

Fig. 2. *ORTEP* (Johnson, 1965) plot of the molecule with 50% probability ellipsoids.

Table 3. *Selected distances in* Å *from* Cu^{II} *to donor atom of the non-quinone monooximic ligand*

E.s.d.'s in parentheses. Meqo = 4-methyl-1-benzoquinone 2-oximato; βnqo = 1,2-naphthoquinone 2-oximato; αnqo = 1,2-naphthoquinone 1-oximato; py = pyridine; ac = acetone; bipy = 1,1′-bipyridine; sqp = square pyramidal; oct = octahedral.

Complex	Geometry	Cu–N	Cu–O	Reference
$Cu(Meqo)_2.py$	sqp	2·16 (1)		McPartlin (1973)
$Cu(Clqo)_2.MeOH$	sqp		2·274 (4)	This work
$Cu(\beta nqo)_2.H_2O$	sqp		2·195 (7)	Saarinen & Korvenranta (1975*a*)
$Cu(\alpha nqo)_2.2ac$	oct		2·651 (4)	Saarinen & Korvenranta (1975*b*)
$Cu(Clqo)_2.bipy$	oct	2·118 (6)		Bisi Castellani, Gatti & Millini (1984)

Bond lengths and angles in the chelate rings and Clqo ligands are in good agreement with the values reported for other adducts of copper(II) *o*-quinone monooximates (Saarinen & Korvenranta, 1975*a*,*b*).

The bond lengths between Cu^{II} and non-quinone monooximic ligands are reported in Table 3. It can be seen that methanol behaves as a weaker Lewis base than water and pyridine, but stronger than acetone.

It is known that in square-pyramidal copper(II) complexes the axial ligand is bonded at a distance of 0·2–0·6 Å longer than the in-plane ligands, because of the Jahn–Teller effect (Hathaway & Billing, 1970). In the case of the methanol adduct of $[Cu(Clqo)_2]$, the Cu^{II}—O axial bond is only 0·31 Å longer than the Cu^{II}—O in-plane bond length. This indicates a relatively strong interaction between Cu^{II} and methanol, and is consistent with a slight lengthening of the methanolic C—O distance from 1·428 (3) (Sutton, 1965) to 1·440 (9) Å. This is also in agreement with an enhanced acidity of the bonded methanol, supporting the view that the methanol adduct acts as an intermediate toward reduction of $[Cu(Clqo)_2]$ (Fig. 1), because it could behave as a Brönsted acid toward OCN^-, giving HOCN and methoxide.

The methanol O(3) atom forms a bifurcated hydrogen bond with another adduct molecule shifted one unit cell along **c**; $[(i) = x, y, z-1]$ $O(3^i)\cdots O(1) = 2·899$ (5) and $O(3^i)\cdots O(2') = 3·092$ (6) Å, where the second H bond is considerably weaker; the fork angle $O(1)\cdots H(O3^i)\cdots O(2')$ is *ca* 82°. In this way a sort of pentaatomic ring is formed: Cu—N′—O(2′)—$H(O3^i)$—O(1).

References

Bisi Castellani, C., Buttafava, A., Carugo, O. & Poggi, A. (1986). *J. Chem. Soc. Dalton Trans.* In the press.

Bisi Castellani, C., Carugo, O. & Coda, A. (1987). *Inorg. Chem.* **26**, 671–675.

Bisi Castellani, C., Carugo, O., Tomba, C., Berbenni, V. & Cinquetti, S. (1987). *Inorg. Chim. Acta.* In the press.

Bisi Castellani, C., Gatti, G. & Millini, R. (1984). *Inorg. Chem.* **23**, 4004–4008.

Bisi Castellani, C. & Millini, R. (1984). *J. Chem. Soc. Dalton Trans.* pp. 1461, 1462.

Busing, W. R., Martin, K. O. & Levy, H. A. (1963). *ORFLS*. Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee, USA.

Charalambous, J., Buckley, R. G. & Brain, E. G. (1982). *J. Chem. Soc. Perkin Trans.* 1, pp. 1075–1078.

Charalambous, J., Kensett, M., Buckley, R. G., McPartlin, M., Mukerjee, D., Brain, E. G. & Jenkins, J. M. (1983). *J. Chem. Soc. Perkin Trans.* 1, pp. 693–697.

Hamilton, W. C. (1959). *Acta Cryst.* **12**, 609–610.

Hathaway, B. J. & Billing, D. E. (1970). *Coord. Chem. Rev.* **5**, 143–207.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99–101, 149–150. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

McKillop, A. & Sayer, T. S. D. (1976). *J. Org. Chem.* **41**, 1079–1080.

McPartlin, M. (1973). *Inorg. Nucl. Chem. Lett.* **9**, 1207–1210.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Saarinen, H. & Korvenranta, J. (1975*a*). *Acta Chem. Scand. Ser. A*, **29**, 409–413.

Saarinen, H. & Korvenranta, J. (1975*b*). *Acta Chem. Scand. Ser. A*, **29**, 861–865.

Sutton, L. E. (1965). *Tables of Interatomic Distances and Configuration in Molecules and Ions. Supplement* 1956–1959. Spec. Publ. No. 18. London: The Chemical Society.

Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

Acta Cryst. (1988). C**44**, 270–272

Structure of Potassium Hexamethyldisilazide Toluene Solvate

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(*Received* 11 *November* 1985; *accepted* 2 *August* 1987)

Abstract. K[$C_6H_{18}NSi_2$].C_7H_8, $M_r = 291·62$, monoclinic, $C2/c$, $a = 13·213$ (5), $b = 17·077$ (4), $c = 15·796$ (4) Å, $\beta = 95·66$ (2)°, $V = 3546·8$ (1·2) Å^3, $Z = 8$, $D_x = 1·09$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 4·08$ cm^{-1}, $F(000) = 1264$, $T \sim 173$ K. Final $R = 0·045$ for 2062 unique observed reflections. The compound was found to be a dimeric aggregate, the core of which consists of a nearly planar K—N—K—N four-membered ring. The crystallization solvent, toluene, co-crystallizes with the dimer but is not coordinated. Bond lengths and angles are in agreement with those of the monomeric dioxane-solvated compound.

Introduction. The title compound is widely utilized as a base in the formation of kinetic enolate anions. Preparation of the material has been described by Wannagat & Niederprüim (1961). It is commercially available as a toluene solution from several sources (Callery Chemical Co. & Aldrich Chemical Co.). An X-ray crystal-structure determination of the dioxane solvate of this substance has been reported (Domingos & Sheldrick, 1974). Owing to our observation of a difference in reactivity of the differently solvated potassium amide base, it seemed reasonable to undertake the X-ray crystal-structure determination of the toluene solvate.

Experimental. Clear colorless crystal of the potassium amide toluene solvate, prepared by refluxing hexamethyldisilazane with potassium hydride in toluene followed by recrystallization from toluene (0·42 × 0·38 × 0·48 mm), aligned on Nicolet *R*3*m*/*E* diffractometer with Mo target, normal-focus tube, graphite monochromator and 0·5 mm pinhole collimator. Lattice parameters calculated from 25 reflections with $26 \leq 2\theta \leq 28°$. Systematic absences: $h + k = 2n + 1$ in entire reciprocal lattice; $l = 2n + 1$ for $h0l$ layer; $k = 2n + 1$ for $0k0$ axis. 2553 reflections measured at ~ 173 K (chilling under stream of N_2 with LT-1 device) over period of 30 h utilizing the $\theta/2\theta$ scan technique (variable scan speed from 6·0 to 29·3° min^{-1}; background/scan time = 0·1) within the ranges $3·7 \leq 2\theta \leq 45°$, $0 \leq h \leq 15$, $0 \leq k \leq 19$, $-20 \leq l \leq 20$. Intensities of three standard reflections ($39\bar{4}$; $\bar{1},\bar{1},10$; $\bar{6}2\bar{6}$) recorded after every 100 reflections. 78 standard reflections decreasing linearly by < 1·0%, used to correct the remaining 2062 unique observed reflections [$F_o > 2·5\sigma(F_o)$]. Data corrected for Lorentz and polarization effects. No absorption correction applied. All non-H atoms immediately located by direct-methods structure solution utilizing *SHELXTL* 4·0 (Sheldrick, 1983). All 26 H atoms located in subsequent difference Fourier synthesis. Structure refined by block cascade least squares with anisotropic thermal parameters for all atoms except H to minimize $\sum w(|F_o - F_c|^2)/\sum F_o^2$. H-atom thermal parameters taken as 1·2 times equivalent isotropic parameter of the bonded atom. H-atom position parameters fixed in calculated positions in the final stages of refinement. Final $R = 0·045$ and $wR = 0·052$ for 154 variables with $w = 1/[\sigma(F_o)^2 + 0·0002(F_o)^2]$ and $S = 1·29$. Maximum least-squares shift/e.s.d. = 0·016 in final refinement cycle. Maximum peak less than 0·3 e Å^{-3} in final Fourier map. Atomic scattering factors taken from *International Tables for X-ray Crystallography* (1974). All computer programs belong to the *SHELXTL* system.

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