C21	0.5789 (4)	0.6643 (2)	−0.1409 (4)	0.0465 (7)
N3	0.7313 (3)	0.48445 (15)	−0.0650 (3)	0.0346 (5)
C4	0.7909 (4)	0.3954 (2)	−0.0537 (3)	0.0363 (6)
O4	0.7428 (3)	0.34416 (13)	−0.2055 (3)	0.0488 (6)
C41	0.6450 (5)	0.3919 (2)	−0.3714 (4)	0.0560 (9)
C5	0.8979 (4)	0.3527 (2)	0.1026 (4)	0.0408 (6)
C6	0.9429 (4)	0.4081 (2)	0.2578 (3)	0.0376 (6)
N6	1.0505 (4)	0.3744 (2)	0.4193 (3)	0.0569 (8)

Table 2. *Selected geometric parameters (Å, °)*

N1—C2	1.332 (3)	C4—O4	1.344 (3)
N1—C6	1.345 (3)	C4—C5	1.371 (4)
C2—N3	1.331 (3)	O4—C41	1.441 (4)
C2—S2	1.756 (3)	C5—C6	1.399 (4)
S2—C21	1.788 (3)	C6—N6	1.351 (3)
N3—C4	1.335 (3)		
C2—N1—C6	115.4 (2)	N3—C4—C5	124.3 (2)
N3—C2—N1	128.3 (2)	O4—C4—C5	117.8 (3)
N3—C2—S2	117.8 (2)	C4—O4—C41	117.4 (2)
N1—C2—S2	113.8 (2)	C4—C5—C6	116.0 (2)
C2—S2—C21	101.14 (12)	N1—C6—N6	116.8 (2)
C2—N3—C4	114.1 (2)	N1—C6—C5	121.7 (2)
N3—C4—O4	117.9 (2)	N6—C6—C5	121.4 (3)
C6—N1—C2—N3	0.7 (4)	N3—C4—O4—C41	5.5 (4)
C6—N1—C2—S2	−178.4 (2)	C5—C4—O4—C41	−174.6 (3)
N3—C2—S2—C21	1.4 (2)	N3—C4—C5—C6	0.7 (4)
N1—C2—S2—C21	−179.4 (2)	O4—C4—C5—C6	−179.2 (2)
N1—C2—N3—C4	1.0 (4)	C2—N1—C6—N6	178.1 (3)
S2—C2—N3—C4	−180.0 (2)	C2—N1—C6—C5	−1.8 (4)
C2—N3—C4—O4	178.2 (2)	C4—C5—C6—N1	1.1 (4)
C2—N3—C4—C5	−1.7 (4)	C4—C5—C6—N6	−178.7 (3)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N6—H6A···N1^{i}	0.86	2.20	3.060 (3)	175.2

Symmetry code: (i) $2 - x, 1 - y, 1 - z$.

The diagram was prepared using *ORTEP*II (Johnson, 1976) as implemented in *PLATON* (Spek, 1995*a*). Examination of the structure with *PLATON* showed that there were no solvent accessible voids in the crystal lattice.

Data collection: *CAD-4/PC Software* (Enraf–Nonius 1992). Cell refinement: *SET*4 and *CELDIM* in *CAD-4/PC Software*. Data reduction: *DATRD*2 in *NRCVAX*94 (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SOLVER* in *NRCVAX*. Program(s) used to refine structure: *NRCVAX*94; *SHELXL*93 (Sheldrick, 1993). Software used to prepare material for publication: *NRCVAX*94; *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1323). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tris(4-methoxyphenyl)methanol

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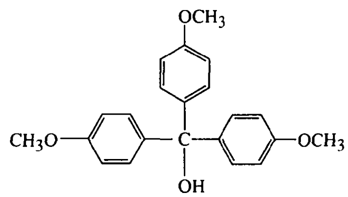
Abstract

Tris(4-methoxyphenyl)methanol, $C_{22}H_{22}O_4$, crystallizes in space group $P2_1$ with two molecules in the asymmetric unit. The molecules are linked into dimers by a weak O—H···O hydrogen bond [O···O 3.035 (3) Å].

Comment

Substituted diphenylmethanols, *R*CPh$_2$OH, exhibit a very wide range of intermolecular aggregation patterns in the solid state. When the *R* group carries no functionality, the aggregation usually depends upon O—H···O hydrogen-bond formation and examples are now known of cyclic (OH)$_2$ dimers [*R* = (C_5H_5)Fe(C_5H_4); Ferguson, Gallagher, Glidewell & Zakaria, 1993], cyclic (OH)$_3$ trimers (*R* = Me$_2$CH; Ferguson, Carroll, Glidewell, Zakaria & Lough, 1995), cyclic (OH)$_4$ tetramers (*R* = CH$_3$; Sultanov, Shnulin & Mamedov, 1985), tetrahedral tetramers (*R* = Ph; Ferguson, Gallagher, Glidewell, Low & Scrimgeour, 1992), cyclic (OH)$_6$ hexamers (Ferguson, Carroll, Glidewell, Zakaria & Lough, 1995) and extended chains (*R* = H; Ferguson, Carroll, Glidewell, Zakaria & Lough, 1995). In addition, the intermolecular aggregation can depend upon O—H$\cdots\pi_{arene}$, rather than upon O—H$\cdots$O, interactions, as when R = $PhCH_2$ (Ferguson, Gallagher, Glidewell & Zakaria, 1994) or when R = PhC≡C (Ferguson, Carroll, Glidewell, Zakaria & Lough, 1995). When a single nitrogen-containing functionality capable of acting as a hydrogen-bond acceptor is introduced into the substituent R, the aggregation pattern changes, giving either closed centrosymmetric dimers (Armesto, Barnes, Horspool & Langa, 1990) or extended chains (Lindner & von Gross, 1973; Glidewell & Ferguson, 1994), based in each case upon O—H$\cdots$N hydrogen bonds. Introduction of a single oxygen-containing functionality as hydrogen-bond acceptor can provide not only single chains, as when R = 2-hydroxyphenyl (Lewis, Duesler, Kress, Curtin & Paul, 1980), but also double chains for R = 4-hydroxy-3,5-dibromophenyl (Stora, 1971) and R = 4-hydroxy-3,5-dimethylphenyl (Lewis, Curtin & Paul, 1979), and infinite two-dimensional nets when R = 4-hydroxyphenyl (Lewis, Curtin & Paul, 1979). There are, in contrast, few structures known for compounds of the type Ar_3COH, in which all three aryl groups carry functionalities capable of acting as hydrogen-bond acceptors. The structure of tris(2-pyridyl)methanol (Keene, Snow & Tiekink, 1988) consists of centrosymmetric dimers, held together by O—H$\cdots$N hydrogen bonds in which only one of the N atoms participates; the other two N atoms are inactive. We report here the structure of a triarylmethanol compound having an oxygen-containing substituent on each ring, namely, tris(4-methoxyphenyl)-methanol, $(CH_3OC_6H_4)_3COH$, (I).



(I)

Compound (I) crystallizes in the monoclinic space group $P2_1$, with two molecules in the asymmetric unit (Fig. 1). The principal intramolecular differences between the two independent molecules are the conformations of the methoxy groups defined by C117 and C217 [compare torsion angles Cn13—Cn14—On1—Cn17; 174.3 (4) for n = 1 and 8.9 (6)° for n = 2] and the conformations of the hydroxy groups (Table 2; Fig. 1). The most significant differences in the overall environments within the crystal structure of the two independent molecules are those experienced by the two hydroxy groups. The hydroxy group of molecule 2 at (x, y, z) acts as a hydrogen-bond donor to the methoxy O11 atom in molecule 1 (at $1-x$, $\frac{1}{2}+y$, $1-z$), with an O$\cdots$O distance of 3.035 (3) Å, but the hydroxy O atom of molecule 1 has no intermolecular O$\cdots$O contacts of less than 3.6 Å. The shortest intermolecular contact of the hydroxy H atom on O1 with an adjacent C atom is with C222 (at $-x$, $-\frac{1}{2}+y$, $1+z$) at a distance of 2.83 Å.

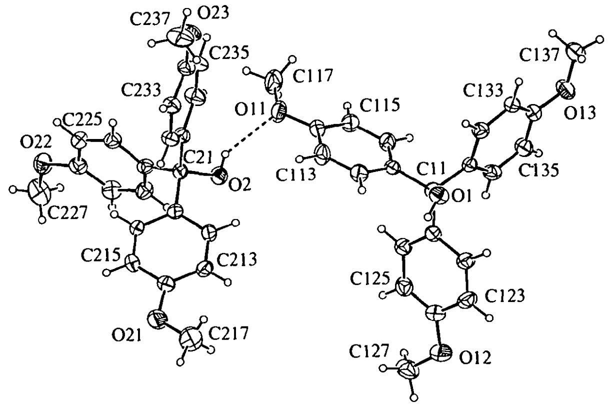


Fig. 1. A view of the two molecules of compound (I) linked by the O—H$\cdots$O hydrogen bond. Phenyl rings are numbered cyclically as Cnm1–Cnm6, n = 1 or 2, m = 1–6. For clarity only some of the atoms in the diagram are labelled. Displacement ellipsoids are drawn at the 30% probability level.

The overall conformations of the carbon skeletons in the two independent molecules, ignoring the methoxy and hydroxy groups, are almost mirror images (Fig. 1; Table 2). The numerical values of the *cis* torsion angles between the CH_3—O—C fragments and the aryl rings range from −2.8 (5) to 8.9 (6)°. In an electron diffraction study of anisole, CH_3—O—C_6H_5, it was found that the barrier to rotation about the exocyclic C—O bond was very low, no more than 10 kJ mol^{-1}, with the minimum-energy conformation having all the C and O atoms coplanar (Seip & Seip, 1973).

Other features of interest within the molecules of compound (I) include the geometry of the CH_3—O—C fragments and the C—OH bond lengths. The C—O—C angles are all significantly larger than the tetrahedral value [117.3 (3)–118.2 (3)°; mean 117.7 (4)°], while the exocyclic C—O bonds are displaced from the external bisectors of the C—C—C angles. The O—C—C angles *cisoid* to the methoxy substituents range from 123.9 (3) to 125.6 (3)° [mean 125.1 (6)°], while those *transoid* to the substituents range from 115.1 (3) to 116.4 (3)° [mean 115.8 (4)°]. The electron diffraction data for anisole (Seip & Seip, 1973) were analyzed by setting the angle between the exocyclic C—O bond and the C—C—C bisector at a series of fixed values and refining the rest of the geometry for each. The best fit was obtained for O—C—C angles fixed at 116 (*transoid*) and 124° (*cisoid*) and a refined C—O—C angle of 120 (2)°. The values found for the corresponding parameters in compound (I) are entirely consistent with, but more precise than, those found for anisole. The C—OH bond lengths (Table 2) are longer than those reported for Ph_3COH (Ferguson, Gallagher, Glidewell, Low & Scrimgeour, 1992), but are similar to the C—O distance in 4-$PhC_6H_4CPh_2OH$ (Ferguson, Carroll, Glidewell, Zakaria & Lough, 1995); the C—O bond distances in the series $RCPh_2OH$ have been correlated with the steric demands of the R group (Ferguson, Carroll, Glidewell, Zakaria & Lough, 1995). The other bond distances are all typical of their types (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987).

Because of the very considerable differences between the crystal structure of Ph_3COH (Ferguson, Gallagher, Glidewell, Low & Scrimgeour, 1992) and that of (I), we turned also to an investigation of the series (4-$CH_3C_6H_4)_3COH$, (II), (4-$CH_3C_6H_4)_2CPhCOH$, (III), and (4-$CH_3C_6H_4)CPh_2COH$, (IV). All three compounds, (II)–(IV), proved to be cubic, with space group $Pa3$ and $Z = 32$, so that $Z' = \frac{4}{3}$ (Wilson, 1993; Brock & Dunitz, 1994), and having cell dimensions 24.51 (2), 24.10 (2) and 23.47 (2) Å, respectively. These data require for each of compounds (II) to (IV), either four independent molecules, each lying upon a threefold axis (which seems implausible on grounds of packing efficiency), or one molecule lying in a general position and another lying on a threefold axis, precisely as was found in the crystal structure of Ph_3COH, which crystallizes, however, in the trigonal space group $R\bar{3}$, itself a subgroup of $Pa3$. The structure solution of compound (IV) confirmed the second of these possibilities (with disordered site occupancy of the phenyl and p-tolyl groups); presumably the same structure/disorder situation holds for (III) and also for (II) (although without the requirement of phenyl/tolyl disorder). The atomic resolution for the analysis of (IV) was poor, due to the small number of observed data and high thermal motion. Satisfactory refinement of the room-temperature diffraction data was not possible. We are presently investigating these structures at lower temperatures.

Experimental

Samples of compounds (I) and (II) were obtained from Lancaster Synthesis Ltd. Samples of compounds (III) and (IV) were prepared by the reactions of phenyllithium with 4,4′-dimethylbenzophenone and 4-methylbenzophenone, respectively, followed by acidic work-up. Crystals of compounds (I) to (IV) suitable for single-crystal X-ray diffraction were grown by slow evaporation of solutions in light petroleum (313–333 K).

Crystal data

$C_{22}H_{22}O_4$
$M_r = 350.40$
Monoclinic
$P2_1$
$a = 10.2784\,(14)$ Å
$b = 10.5411\,(14)$ Å
$c = 17.794\,(3)$ Å
$\beta = 101.332\,(11)°$
$V = 1890.3\,(5)$ Å^3
$Z = 4$
$D_x = 1.231$ Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 9.05–18.05°$
$\mu = 0.084$ mm^{-1}
$T = 294\,(1)$ K
Block
0.37 × 0.35 × 0.21 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
4554 measured reflections
4338 independent reflections
2494 observed reflections [$I > 2\sigma(I)$]
$R_{int} = 0.007$
$\theta_{max} = 26.90°$
$h = -13 \rightarrow 12$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 22$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.0407$
$wR(F^2) = 0.0872$
$S = 1.141$
4338 reflections
477 parameters
H atoms riding (*SHELXL* defaults; C—H 0.93–0.96, O—H 0.82 Å)
$w = 1/[\sigma^2(F_o^2) + (0.0529P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.007$
$\Delta\rho_{max} = 0.146$ e Å^{-3}
$\Delta\rho_{min} = -0.192$ e Å^{-3}
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983) parameter = −0.5 (12)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O1	0.2185 (2)	0.0000 (2)	0.84164 (12)	0.0563 (6)
C11	0.3159 (3)	−0.0711 (3)	0.8963 (2)	0.0474 (8)
C111	0.4558 (3)	−0.0284 (3)	0.8878 (2)	0.0438 (7)
C112	0.4820 (3)	0.0043 (4)	0.8172 (2)	0.0592 (9)
C113	0.6081 (3)	0.0358 (4)	0.8082 (2)	0.0670 (10)
C114	0.7120 (3)	0.0366 (3)	0.8702 (2)	0.0549 (9)
C115	0.6888 (3)	0.0035 (4)	0.9406 (2)	0.0600 (9)
C116	0.5615 (3)	−0.0280 (4)	0.9488 (2)	0.0570 (9)
O11	0.8334 (2)	0.0686 (3)	0.85487 (13)	0.0790 (8)
C117	0.9409 (4)	0.0822 (6)	0.9170 (2)	0.0972 (15)
C121	0.2937 (3)	−0.2127 (3)	0.8800 (2)	0.0457 (7)
C122	0.1671 (3)	−0.2626 (4)	0.8744 (2)	0.0635 (10)
C123	0.1408 (3)	−0.3894 (4)	0.8598 (2)	0.0687 (10)
C124	0.2428 (4)	−0.4697 (3)	0.8493 (2)	0.0575 (9)
C125	0.3676 (3)	−0.4215 (4)	0.8537 (2)	0.0601 (9)
C126	0.3920 (3)	−0.2935 (3)	0.8697 (2)	0.0530 (8)
O12	0.2073 (3)	−0.5953 (2)	0.8366 (2)	0.0792 (8)
C127	0.3042 (5)	−0.6790 (4)	0.8175 (2)	0.0895 (13)
C131	0.2912 (3)	−0.0328 (3)	0.9747 (2)	0.0455 (7)
C132	0.2732 (3)	0.0940 (3)	0.9907 (2)	0.0507 (8)
C133	0.2533 (3)	0.1348 (3)	1.0611 (2)	0.0528 (8)
C134	0.2538 (3)	0.0488 (3)	1.1192 (2)	0.0551 (9)
C135	0.2723 (4)	−0.0784 (3)	1.1050 (2)	0.0634 (10)
C136	0.2915 (3)	−0.1181 (3)	1.0341 (2)	0.0560 (8)
O13	0.2372 (3)	0.0785 (2)	1.19128 (13)	0.0763 (7)
C137	0.2238 (4)	0.2085 (4)	1.2090 (2)	0.0765 (11)
O2	0.1636 (2)	0.4663 (2)	0.30421 (11)	0.0589 (6)
C21	0.1057 (3)	0.5171 (3)	0.3653 (2)	0.0475 (8)
C211	0.1892 (3)	0.4628 (3)	0.4388 (2)	0.0445 (7)
C212	0.3246 (3)	0.4404 (3)	0.4452 (2)	0.0533 (8)
C213	0.4015 (3)	0.3961 (3)	0.5127 (2)	0.0577 (9)
C214	0.3457 (3)	0.3735 (3)	0.5752 (2)	0.0583 (8)
C215	0.2115 (3)	0.3954 (3)	0.5697 (2)	0.0568 (9)
C216	0.1356 (3)	0.4384 (3)	0.5028 (2)	0.0491 (8)

O21	0.4119 (3)	0.3292 (3)	0.6446 (2)	0.0875 (9)
C217	0.5528 (4)	0.3214 (7)	0.6574 (3)	0.130 (2)
C221	−0.0375 (3)	0.4718 (3)	0.3558 (2)	0.0463 (7)
C222	−0.0692 (3)	0.3460 (3)	0.3362 (2)	0.0555 (8)
C223	−0.1937 (3)	0.2970 (4)	0.3360 (2)	0.0612 (9)
C224	−0.2913 (3)	0.3720 (4)	0.3551 (2)	0.0575 (9)
C225	−0.2639 (3)	0.4987 (3)	0.3719 (2)	0.0571 (9)
C226	−0.1391 (3)	0.5471 (3)	0.3720 (2)	0.0513 (8)
O22	−0.4175 (2)	0.3327 (3)	0.3591 (2)	0.0793 (8)
C227	−0.4455 (4)	0.2004 (5)	0.3501 (3)	0.100 (2)
C231	0.1142 (3)	0.6620 (3)	0.3660 (2)	0.0445 (7)
C232	0.1621 (3)	0.7297 (3)	0.4319 (2)	0.0488 (8)
C233	0.1693 (3)	0.8612 (3)	0.4324 (2)	0.0506 (8)
C234	0.1287 (3)	0.9272 (3)	0.3654 (2)	0.0521 (8)
C235	0.0775 (4)	0.8623 (3)	0.2988 (2)	0.0646 (9)
C236	0.0705 (4)	0.7318 (3)	0.2998 (2)	0.0620 (9)
O23	0.1340 (3)	1.0568 (2)	0.35980 (14)	0.0755 (8)
C237	0.1932 (5)	1.1241 (4)	0.4260 (3)	0.0915 (15)

Table 2. *Selected geometric parameters (Å, °)*

O1—C11	1.459 (3)	O2—C21	1.441 (3)
C11—C111	1.543 (4)	C21—C211	1.527 (4)
C11—C121	1.529 (5)	C21—C221	1.525 (4)
C11—C131	1.521 (4)	C21—C231	1.530 (4)
O11—C114	1.371 (4)	O21—C214	1.370 (4)
O11—C117	1.409 (4)	O21—C217	1.423 (4)
O12—C124	1.380 (4)	O22—C224	1.376 (4)
O12—C127	1.421 (5)	O22—C227	1.427 (5)
O13—C134	1.364 (4)	O23—C234	1.372 (4)
O13—C137	1.418 (5)	O23—C237	1.407 (5)
O1—C11—C111	108.4 (2)	O2—C21—C211	105.2 (2)
O1—C11—C121	108.5 (2)	O2—C21—C221	109.4 (2)
O1—C11—C131	105.0 (2)	O2—C21—C231	110.2 (3)
C111—C11—C121	111.9 (2)	C211—C21—C221	110.2 (2)
C111—C11—C131	109.9 (2)	C211—C21—C231	110.2 (3)
C121—C11—C131	112.9 (3)	C221—C21—C231	111.4 (3)
O11—C114—C113	115.8 (3)	O21—C214—C213	125.4 (3)
O11—C114—C115	125.1 (3)	O21—C214—C215	115.6 (3)
C113—C114—C115	119.1 (3)	C213—C214—C215	119.0 (3)
C114—O11—C117	118.2 (3)	C214—O21—C217	118.2 (3)
O12—C124—C123	115.1 (3)	O22—C224—C223	125.6 (3)
O12—C124—C125	125.5 (3)	O22—C224—C225	115.6 (3)
C123—C124—C125	119.4 (3)	C223—C224—C225	118.8 (3)
C124—O12—C127	117.4 (3)	C224—O22—C227	117.4 (3)
O13—C134—C133	124.9 (3)	O23—C234—C233	123.9 (3)
O13—C134—C135	116.2 (3)	O23—C234—C235	116.4 (3)
C133—C134—C135	118.9 (3)	C233—C234—C235	119.6 (3)
C134—O13—C137	117.8 (3)	C234—O23—C237	117.3 (3)

C131—C11—O1—H1	−154
C211—C21—O2—H2	−172
O1—C11—C111—C112	−34.9 (4)
O1—C11—C111—C116	148.9 (3)
C113—C114—O11—C117	174.3 (4)
C115—C114—O11—C117	−7.3 (6)
O1—C11—C121—C122	−51.4 (4)
O1—C11—C121—C126	128.0 (3)
C123—C124—O12—C127	173.5 (3)
C125—C124—O12—C127	−8.0 (5)
O1—C11—C131—C132	−45.4 (4)
O1—C11—C131—C136	137.8 (3)
C133—C134—O13—C137	−2.8 (5)
C135—C134—O13—C137	177.2 (3)
O2—C21—C211—C212	33.4 (4)
O2—C21—C211—C216	−149.2 (3)
C213—C214—O21—C217	8.9 (6)
C215—C214—O21—C217	−171.5 (4)
O2—C21—C221—C222	43.1 (4)
O2—C21—C221—C226	−142.6 (3)
C223—C224—O22—C227	6.6 (5)
C225—C224—O22—C227	−173.6 (3)
O2—C21—C231—C232	−130.8 (3)
O2—C21—C231—C236	50.8 (4)
C233—C234—O23—C237	4.5 (5)
C235—C234—O23—C237	−176.3 (4)

The data did not allow us to determine the direction of the chiral axis. The figure was prepared using *ORTEP*II (Johnson, 1976) as implemented in *PLATON* (Spek, 1995). Examination of the structure with *PLATON* showed that there were no solvent accessible voids in the crystal lattice.

Data collection: *CAD*-4/*PC Software* (Enraf–Nonius, 1992). Cell refinement: *SET*4 (de Boer & Duisenberg, 1984) and *CELDIM* (Enraf–Nonius, 1992). Data reduction: *DATRD*2 in *NRCVAX*94 (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *NRCVAX*94 and *SHELXL*93 (Sheldrick, 1993). Software used to prepare material for publication: *NRCVAX*94 and *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1052). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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