X-Ray Crystallographic, ^{1}H, and ^{13}C Nuclear Magnetic Resonance Investigation of the Potentially Heptadentate Ligand Trensal, 2,2′,2″-Tris(salicylideneimino)triethylamine

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The potentially heptadentate ligand trensal, 2,2′2″-tris(salicylideneimino)triethylamine, has been investigated by n.m.r. spectroscopy and *X*-ray crystallography. The ^{1}H and ^{13}C n.m.r. spectra of this compound have been completely assigned. The single crystal *X*-ray investigation, $R = 0.073$, showed the compound to be monoclinic, space group $P2_1/c$, $a = 9.857(2)$, $b = 11.208(2)$, $c = 22.910(4)$ Å, $\beta = 98.81(1)$°. Strong intramolecular hydrogen bonding occurs between the hydroxy hydrogen atoms and the azomethine nitrogen atoms. The compound exhibits an approximate three-fold axis when looking down the unique nitrogen atom through the plane C(11)–C(21)–C(31).

Seven-co-ordinated compounds are now well established, but complexes containing heptadentate ligands are exceedingly rare.[1] Schiff's bases are well known ligands represented by many bi-, tri-, tetra-, and hexa-dentate examples.[2] Ligands with seven donor functions are relatively rare species. Two Schiff's bases, tren-py, tris-[4-(2-pyridyl)-3-azobutenyl]amine, and trensal, 2,2′,2″-tris(salicylideneimino)triethylamine, are examples of this class. Both compounds are derived from tren, 2,2′,2″-triaminotriethylamine, and are obtained by condensing this polyfunctional amine with pyridine-2-carbaldehyde or salicylaldehyde, respectively. Tren-py has not been isolated,[3] but trensal has been reported as a yellow crystalline solid.[4–6] Brief spectroscopic data have been given.[5] A few metal complexes of trensal[4] and of substituted trensals[5] have been reported. Trensal and related compounds were reported to inhibit the catalytic oxidation of organic products by metals.[6]

As part of a study of metal complexes of trensal and related ligands,[7] we have investigated the free ligand, trensal (1), in some detail by use of ^{1}H and ^{13}C n.m.r. spectroscopy and have also undertaken a single crystal *X*-ray structure analysis.

^{1}H *N.m.r. Spectroscopy*.—The ^{1}H n.m.r. spectrum of the title compound in [$^2H_4$]methanol at 199.50 MHz is relatively simple with the four aromatic proton signals at δ 7.27, 6.83, 6.54, and 6.23, the olefinic proton signal at δ 7.88, and the two methylene groups giving an AA′XX′ splitting pattern at δ 3.57 and 2.87. This spin system was analysed using the JEOL spin-simulation program from which the coupling constants were determined (Figure 1). The four aromatic and the two methylene proton signals were assigned using homonuclear proton decoupling techniques (Table 1).

The phenolic OH resonance was not observed with [$^2H_4$]-methanol as solvent, but was found to lie at δ 13.80 in [^{2}H]chloroform. This chemical shift was found to be invariant with concentration indicating the presence of strong intramolecular hydrogen bonding.[8] The *X*-ray crystallographic data presented in this paper show this to be the case. The trensal phenolic OH resonance, *viz.* δ 13.80, is further downfield than that of phenol itself (whose hydroxy proton resonance moves from δ 7.45 to 4.37 on increasing dilution in carbon tetrachloride),[9] and those of many other phenols.[10] The downfield signal is explained in terms of the combination of three factors, (1) hydrogen bonding; (2) the O–H ··· N group lying coplanar with the aromatic ring and hence susceptible to deshielding effects from the induced aromatic ring current giving a downfield shift; and (3) hydrogen bonding of the phenolic OH with an unsaturated azomethine nitrogen causing a decrease in the shielding at the hydroxy hydrogen. A series of simple *N*-alkylsalicylaldimines give similar low field (δ 13.26—14.00)

Table 1. ^{1}H N.m.r. chemical shifts for trensal

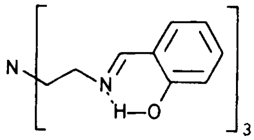
Proton	Chemical shift δ[a]	Proton	Chemical shift δ[a]
H(1)[b]	2.87	H(6)	7.27
H(2)	3.57	H(7)	6.54
H(3)	7.88	H(8)	6.23
H(5)	6.83	OH	13.80[c]

[a] All values relative to tetramethylsilane as internal standard with [$^2H_4$]methanol as solvent. [b] H(*n*1), H(*n*2) ··· H(*n*) (in Figure 2) = H(1), H(2) ··· H. [c] In $CDCl_3$.

Table 2. Proton–proton coupling constants

Protons	Coupling constant[a]	Protons	Coupling constant[a]
$^2J_{H(1)H(1')}$[b]	−13.5	$^3J_{H(6)H(7)}$	7.2
$^2J_{H(2)H(2')}$	−12.6	$^3J_{H(7)H(8)}$	7.8
$^3J_{H(1)H(2)}$	7.1	$^4J_{H(2)H(3)}$	1.0
$^3J_{H(1)H(2')}$	3.3	$^4J_{H(3)H(5)}$	<0.5
$^3J_{H(1')H(2')}$	7.1	$^4J_{H(5)H(7)}$	1.2
$^3J_{H(1')H(2)}$	3.3	$^4J_{H(6)H(8)}$	1.8
$^3J_{H(5)H(6)}$	8.4		

[a] Coupling constant values in Hz. [b] H(*n*1), H(*n*2) ··· H(*n*) (in Figure 2) = H(1), H(2) ··· H(8). The assignment of H(1), H(1′), H(2), and H(2′) in this Table is for the purposes of analysing the n.m.r. data and should not be confused with the labelling in the crystal structure diagrams.



(1)

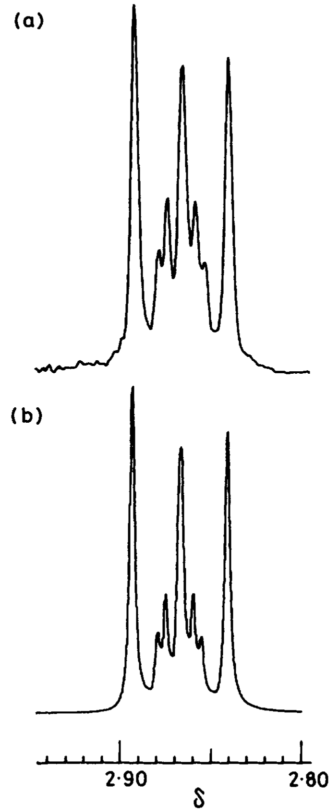


Figure 1. (a) ^{1}H N.m.r. spectrum (199.5 MHz) and (b) simulation of the XX′ part of the AA′XX′ spin system due to the methylene protons H(1) and H(1′)

Table 3. ^{13}C N.m.r. chemical shifts of trensal in [$^2H_4$]methanol at 50.1 MHz

Carbon	Chemical shift^a	Carbon	Chemical shift^a
C(1)^{b,c}	57.8	C(6)^b	134.0
C(2)^b	56.8	C(7)^b	119.1
C(3)^b	167.6	C(8)^b	133.5
C(4)^d	119.5	C(9)^d	164.9
C(5)^b	118.6		

^{a} All chemical shift values measured in p.p.m. relative to tetramethylsilane as internal standard. ^{b} Assigned using selective heteronuclear proton decoupling techniques. ^{c} C(n1), C(n2) ••• C(n9) (in Figure 2) = C(1), C(2) ••• C(9). ^{d} Assigned from chemical shifts of analogous compounds.

phenolic proton signals in [^{2}H]chloroform.[11] The proton–proton coupling constants are given in Table 2.

^{13}C *N.m.r. Spectroscopy*.—The ^{13}C n.m.r. spectrum of the title compound was obtained in [$^2H_4$]methanol at 50.1 MHz, and was assigned using selective heteronuclear proton decoupling methods (Table 3).

X-*Ray Crystallography*.—We also undertook a single crystal *X*-ray analysis of trensal. The final fractional atomic co-ordinates, bond lengths, and bond angles are given in Tables 4—6, respectively. Some of the more important bond lengths (averaged) are given in Figure 2. A salient feature of the molecule is the strong hydrogen bonding of the hydroxy hydrogen atoms to the azomethine nitrogen atoms (O ••• N, 2.573 Å, O–Ĥ ••• N = 149.5° mean values) giving an almost planar six-membered ring, which in turn is coplanar with the adjacent benzene ring (Figure 3).[12] These fused bicyclic systems

Table 4. Fractional atomic co-ordinates (× 10^4) for trensal

	x	*y*	*z*
N	1 589(3)	3 687(2)	9 476(1)
N(1)	2 059(3)	1 019(2)	9 752(1)
N(2)	520(2)	2 897(2)	8 204(1)
N(3)	4 441(3)	4 031(2)	9 188(1)
O(1)	430(2)	−644(2)	9 315(1)
O(2)	1 750(2)	3 739(2)	7 387(1)
O(3)	6 376(3)	2 673(2)	9 724(1)
C(11)	1 296(4)	2 995(3)	9 992(1)
C(12)	2 337(4)	2 026(3)	10 156(1)
C(13)	2 979(3)	593(3)	9 484(1)
C(14)	2 726(3)	−438(2)	9 102(1)
C(15)	3 741(3)	−878(3)	8 799(2)
C(16)	3 490(4)	−1 855(4)	8 425(2)
C(17)	2 223(5)	−2 405(3)	8 363(2)
C(18)	1 218(3)	−2 003(3)	8 660(1)
C(19)	1 451(3)	−1 027(2)	9 031(1)
C(21)	313(4)	4 052(3)	9 095(2)
C(22)	−277(3)	3 052(3)	8 688(2)
C(23)	862(3)	1 852(3)	8 069(1)
C(24)	1 648(3)	1 648(3)	7 594(1)
C(25)	2 028(4)	496(3)	7 452(2)
C(26)	2 815(4)	289(4)	7 017(2)
C(27)	3 208(4)	1 246(4)	6 696(2)
C(28)	2 848(3)	2 388(4)	6 822(1)
C(29)	2 072(3)	2 600(3)	7 267(1)
C(31)	2 505(4)	4 693(3)	9 652(2)
C(32)	3 407(3)	4 961(2)	9 187(2)
C(33)	4 480(3)	3 428(2)	8 724(1)
C(34)	5 467(2)	2 469(2)	8 700(1)
C(35)	5 519(3)	1 877(3)	8 173(1)
C(36)	6 452(3)	963(3)	8 135(1)
C(37)	7 349(3)	638(2)	8 636(1)
C(38)	7 310(3)	1 198(2)	9 162(1)
C(39)	6 384(3)	2 128(2)	9 201(1)
H(1)	729(48)	170(43)	9 492(19)
H(2)	1 121(49)	3 651(40)	7 746(20)
H(3)	5 690(34)	3 302(31)	9 640(14)

Table 5. Bond lengths (Å) for trensal

C(11)–N	1.479(3)	C(21)–N	1.475(4)
C(31)–N	1.461(4)	C(12)–N(1)	1.457(4)
C(13)–N(1)	1.264(3)	C(22)–N(2)	1.464(4)
C(23)–N(2)	1.270(3)	C(32)–N(3)	1.457(4)
C(33)–N(3)	1.265(3)		
C(19)–O(1)	1.348(3)	H(1)–O(1)	1.023(48)
C(29)–O(2)	1.354(4)	H(2)–O(2)	1.106(46)
C(39)–O(3)	1.344(3)	H(3)–O(3)	0.975(34)
C(12)–C(11)	1.502(5)	C(14)–C(13)	1.448(4)
C(15)–C(14)	1.392(4)	C(19)–C(14)	1.407(4)
C(16)–C(15)	1.389(5)	C(17)–C(16)	1.380(5)
C(18)–C(17)	1.361(5)	C(19)–C(18)	1.383(5)
C(22)–C(21)	1.516(5)	C(24)–C(23)	1.447(4)
C(25)–C(24)	1.397(4)	C(29)–C(24)	1.404(4)
C(26)–C(25)	1.374(5)	C(27)–C(26)	1.388(5)
C(28)–C(27)	1.371(5)	C(29)–C(28)	1.386(4)
C(32)–C(31)	1.518(4)	C(34)–C(33)	1.456(3)
C(35)–C(34)	1.385(3)	C(39)–C(34)	1.402(3)
C(36)–C(35)	1.389(4)	C(37)–C(36)	1.386(4)
C(38)–C(37)	1.364(4)	C(39)–C(38)	1.397(4)

Table 6. Bond angles (°) for trensal

C(21)–N–C(11)	111.5(2)	C(31)–N–C(11)	111.9(2)
C(31)–N–C(21)	112.6(2)	C(13)–N(1)–C(12)	121.4(3)
C(23)–N(2)–C(22)	119.2(3)	C(33)–N(3)–C(32)	119.5(3)
H(1)–O(1)–C(19)	106.5(25)	H(2)–O(2)–C(29)	104.0(23)
H(3)–O(3)–C(39)	104.6(19)		
C(12)–C(11)–N	111.7(2)	C(11)–C(12)–N(1)	110.3(3)
C(14)–C(13)–N(1)	121.7(3)	C(15)–C(14)–C(13)	120.9(3)
C(19)–C(14)–C(13)	120.8(3)	C(19)–C(14)–C(15)	118.3(3)
C(16)–C(15)–C(14)	120.8(3)	C(17)–C(16)–C(15)	119.2(4)
C(18)–C(17)–C(16)	121.1(4)	C(19)–C(18)–C(17)	120.3(3)
C(14)–C(19)–O(1)	120.9(3)	C(18)–C(19)–O(1)	118.8(3)
C(18)–C(19)–C(14)	120.2(3)		
C(22)–C(21)–N	111.8(2)	C(21)–C(22)–N(2)	110.7(2)
C(24)–C(23)–N(2)	121.5(2)	C(25)–C(24)–C(23)	121.0(3)
C(29)–C(24)–C(23)	121.2(3)	C(29)–C(24)–C(25)	117.8(3)
C(26)–C(25)–C(24)	121.7(3)	C(27)–C(26)–C(25)	119.3(4)
C(29)–C(28)–C(27)	120.3(3)	C(28)–C(27)–C(26)	120.5(3)
C(24)–C(29)–O(2)	120.6(3)	C(28)–C(29)–O(2)	119.0(3)
C(28)–C(29)–C(24)	120.3(3)		
C(32)–C(31)–N	111.3(2)	C(31)–C(32)–N(3)	110.0(2)
C(34)–C(33)–N(3)	122.3(2)	C(35)–C(34)–C(33)	120.1(2)
C(39)–C(34)–C(33)	121.4(1)	C(39)–C(34)–C(35)	118.5(2)
C(36)–C(35)–C(34)	121.5(1)	C(37)–C(36)–C(35)	119.1(3)
C(38)–C(37)–C(36)	120.7(2)	C(39)–C(38)–C(37)	120.5(2)
C(34)–C(39)–O(3)	121.0(2)	C(38)–C(39)–O(3)	119.3(2)
C(38)–C(39)–C(34)	119.8(2)		

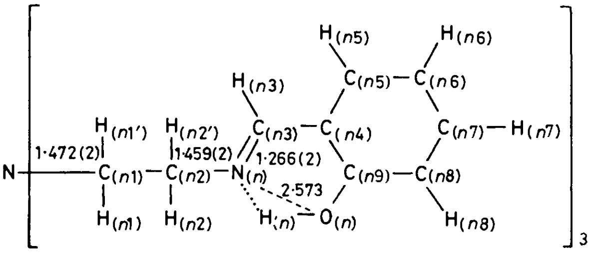


Figure 2. Some of the more important bond lengths (averaged) in Å of trensal

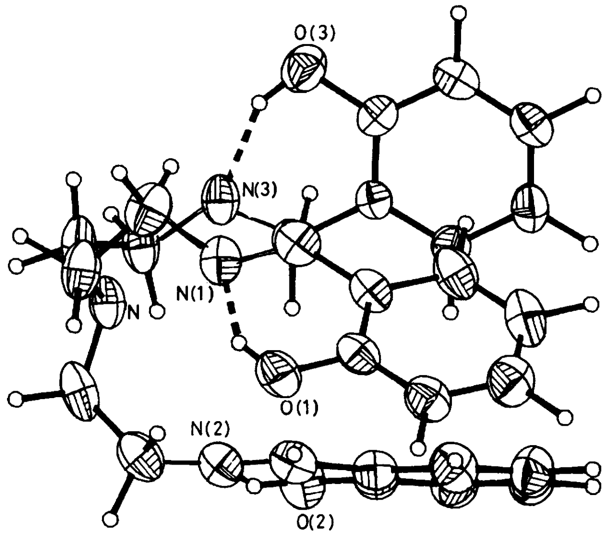


Figure 3. Molecular diagram of trensal showing the hydrogen bonding

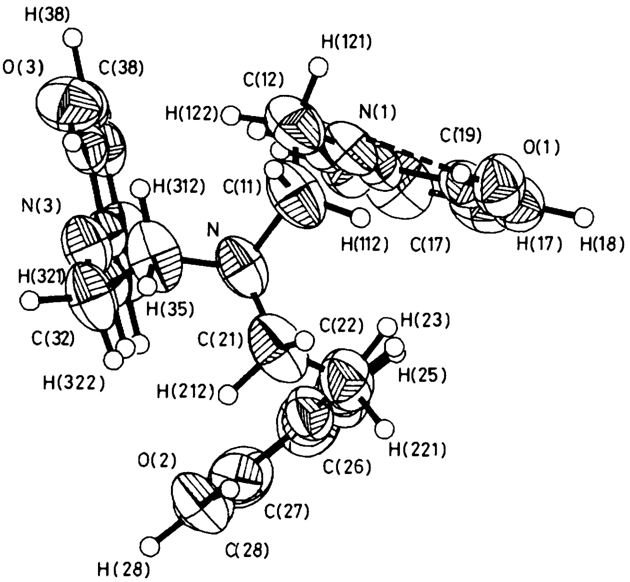


Figure 4. Molecular diagram of trensal. View down the threefold axis

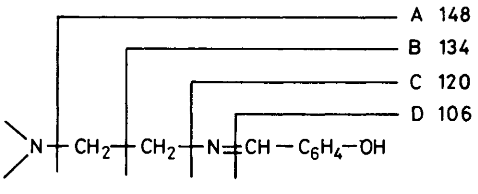


Figure 5. The mass spectrometric fragmentation pattern of trensal

are perpendicular to the plane through C(11)–C(21)–C(31). Looking down the unique nitrogen atom through this three carbon plane, one sees an approximate three-fold axis with the hydrogen bonds, O–H···N, uppermost in the bicyclic structural units (Figure 4).

The single bonds from the unique nitrogen atom to C(11), C(21), and C(31) at 1.472(2) Å (averaged) are significantly longer than those from C(12)–N(1), C(22)–N(2), and C(32)–N(3) at 1.459(2) Å (averaged), in line with the former nitrogen atom being sp^3-and the latter sp^2-hybridised. The methylene hydrogen atoms do not approach the benzene rings, but the aromatic protons *ortho* to the points of fusion may come under the shielding cone of the adjacent benzene rings. The CNC bond angles around the unique nitrogen atom are slightly larger at 111.6(2)° than those of a perfect tetrahedron.

Conformation.—The conformations of the substituted ethane groupings, NCH_2CH_2N, in the crystal are *gauche*, the mean NCCN dihedral angle being 75°. In solution the fact that we observe two coupling constants of 7.1 Hz, and two others of 3.3 Hz, rules out a single *gauche* rotamer, as well as an equal population of all three rotamers. Using the method of Abraham and Gatti[13] and values of $J(HCCH)$ of 2 and 10 Hz for dihedral angles of 60 and 180°, respectively, we deduce a rotamer population consisting of 66% of the *trans*-form and 34% of the two *gauche*-forms (in equal proportions). The calculated coupling constants for this mixture using the above data are 7.25 Hz (two) and 3.3 Hz (two).

U.v.–Visible Spectrum of Trensal.—Trensal shows three absorptions in the u.v.–visible region, at 255, 317, and 406 nm with the following molar extinction coefficients, 24 503, 11 138, and 339 dm^3 mol^{-1} cm^{-1}, respectively. The latter weak absorption in the blue end of the visible spectrum imparts the yellow colour to trensal.

I.r. Spectrum of Trensal.—The i.r. spectrum of trensal run as a KBr disc produces a profusion of sharp absorption bands below 1 700 cm^{-1}; therefore, a full analysis of the spectrum has not been attempted here. However, a few regions of this spectrum are of diagnostic value. The region between 2 200 and 3 000 cm^{-1} shows a broad absorption due to intramolecular hydrogen bonded OH, and the two groups of peaks above and below 3 000 cm^{-1} correspond to the aromatic, and aliphatic C–H stretching vibration respectively. Three peaks occur in the region of 1 600 cm^{-1} (*i.e.* 1 635, 1 615, and 1 588 cm^{-1}) and these are due to the azomethine (C=N) and aromatic (C=C) stretching modes. Aromatic C=C stretching absorptions are generally below 1 625 cm^1, and the C=N stretching vibrations are usually higher than this figure; therefore, the peak at 1 635 cm^{-1} can be assigned to the azomethine C=N stretch. The latter two peaks at 1 615 and 1 588 cm^{-1} can then be assigned to aromatic C=C stretching modes.

Mass Spectrometry.—The electron impact spectrum barely showed the molecular ion at 458, but $M + 1$ at 459 was an important peak. Nothing further was observed in the spectrum down to 339 with a major peak at 337. The fragmentation of the three branches from the central nitrogen are displayed in Figure 5. The base peak is represented by $M - \mathrm{B}$, with B also occurring as a major peak. The fragment $>\mathrm{N}{-}\overset{+}{\mathrm{C}}\mathrm{H}_2 \longleftrightarrow >\overset{+}{\mathrm{N}}{=}\mathrm{CH}_2$ is well known from solution studies. Similarly, important peaks occur at $M - \mathrm{C}$ and C, $M - 2\mathrm{C}$, $M - 2\mathrm{B}$, $M - \mathrm{C} - \mathrm{B}$ as well as A, but not $M - \mathrm{A}$. The latter ion is apparently too unstable; however, $M - \mathrm{A} - \mathrm{B}$ is observed.

Most of the fragments are associated with others showing the gain or the loss of one or more hydrogen atoms. Fragment D appears to be not a primary one, perhaps not surprisingly as the link to the adjacent atom is a double bond.

Experimental

Trensal was synthesised using methods described in the literature[4] giving a yellow crystalline solid, m.p. 93 °C (lit.,[4] 89—90 °C; lit.,[5] 82—83 °C) (Found: C, 70.8; H, 6.6; N, 12.2. $C_{27}H_{30}N_4O_3$ requires C, 70.7; H, 6.6; N, 12.2%).

N.m.r. spectra were obtained on a JEOL JNM FX-200 spectrometer operating at 199.5 and 50.1 MHz for proton and carbon-13, respectively. The i.r. spectrum was recorded for a KBr disc using a Perkin-Elmer 597 spectrophotometer operating between 4 000 and 200 cm^{-1}. The u.v.–visible spectra were recorded using a Perkin-Elmer 554 spectrophotometer operating between 900 and 200 nm with chloroform as solvent and reference.

The lattice parameters were determined by least-squares refinement of the setting angles for 25 reflections [$28 < \theta(\text{Cu-}K_\alpha) < 30$] automatically centred on a CAD-4 diffractometer. The intensity data were recorded on the same instrument using Cu-K_α radiation, in the range $\theta = 3$—70°, and following procedures previously described in detail.[14] Merging the equivalent reflections and omitting those with $|F_0| < 3\sigma(F_0)$ yielded 3 113 unique data ($R_{\text{int}} = 0.013$) (the more intense reflections showed extinction), out of 4 723 which were used in the structure analysis.

Crystal Data.—$C_{27}H_{30}N_4O_3$, $M = 458.56$, monoclinic, space group $P2_1/c$, $a = 9.857(2)$, $b = 11.208(2)$, $c = 22.910(4)$ Å, $\beta = 98.81(1)°$, $U = 2\,501.2$ Å^3, $Z = 4$, $D_c = 1.216$ g cm^{-3}, (Cu-K_α) = 1.541 78 Å, $\mu = 5.70$ cm^{-1}, $F(000) = 976$.

The structure was solved by direct methods by the application of MULTAN[15] and refined by least-squares with isotropic temperature factors for the hydrogen atoms and anisotropic factors for the heavier atoms. Although 26 hydrogen atoms out of 30 were located from a difference Fourier synthesis, only the three hydrogen atoms involved in hydrogen bonding were refined freely. All others were restricted to idealised positions (C–H = 1.08 Å) in the refinement. The weighting scheme was based on $\omega = 1/[\sigma^2(F_o) + 0.004\ F_o^2]$. The refinement converged at $R = 0.073$, $R_w = 0.069$ with maximum parameter shift/e.s.d. = 0.05. The absolute values of the peaks and troughs in the final difference map was $\leqslant 0.4$ e Å^{-3}.

The calculations were performed initially on the Queen Mary College ICL 2980 and finally, on a DEC VAX11/750 computer using the SHELX 76 program system.[16] Neutral scattering factors were taken from refs. 17 and 18 for the non-hydrogen and hydrogen atoms respectively.*

Acknowledgements

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