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Structure of Chloro-*N*,*N*,*N'*,*N'*-tetramethylformamidinium Bis[dichloromercury(II)] Chloride

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Abstract. $C_5H_{12}ClN_2^+.2HgCl_2.Cl^-$, $M_r = 714.06$, triclinic, $P\bar{1}$, $a = 10.840$ (3), $b = 10.409$ (3), $c = 7.422$ (2) Å, $\alpha = 96.59$ (5), $\beta = 100.87$ (5), $\gamma = 105.73$ (5)°, $V = 779.4$ (1) Å^3, $Z = 2$, $D_x = 3.05$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 199.8$ cm^{-1}, $F(000) = 636$, room temperature, $R = 0.040$, $wR = 0.042$ for 2904 observed reflections $[F_o \geq 3\sigma(F_o)]$. The chlorotetramethylformamidinium cation assumes a non planar conformation as a result of non-bonded intramolecular repulsion between methyl groups. There are two independent Hg atoms in the asymmetric unit. The coordination around Hg(1) can be described as a distorted octahedron of six chlorines with two short, two long and two very long Hg–Cl bonds. The coordination around Hg(2) is a distorted trigonal bipyramid with two short and one intermediate Hg–Cl bond in the equatorial plane, the coordination sphere being completed by two very long Hg–Cl contacts. The anionic framework can be described in terms of Hg$_2$Cl$_5^-$ units. If Hg–Cl contacts up to 3.30 Å are considered the structure can be described as being formed by layers of interconnected Hg$_2$Cl$_5^-$ units parallel to (010) with the cations located between the layers.

Introduction. Information concerning reactions of chloro-*N*,*N*,*N'*,*N'*-tetramethylformamidinium chloride

with metal complexes is very scarce (Petz, 1975; Hartshorn, Lappert & Turner, 1978). Some time ago the synthesis of chloro-*N*,*N*,*N'*,*N'*-tetramethylforma-midinium bis[dichloromercury(II)] chloride was reported (Altman & Welcman, 1980); we now report the crystal structure of this compound.

Experimental. Transparent plate (0.28 × 0.15 × 0.07 mm). Cell parameters refined using the setting angles of 25 strong reflections having $5.6 < 2\theta < 33.0°$. Intensity collected on a Philips PW 1100 four-circle diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, $\omega/2\theta$ scan technique, scan speed $3.0° \omega$ min^{-1}, scan width 1.15 in ω, $5° \leq 2\theta \leq 55°$, 3570 unique reflections (h −13→13, k −13→13, l 0→9) of which 2904 with $F_o \geq 3\sigma(F_o)$. No significant intensity variation for three standards ($2\bar{1}\bar{1}$, $\bar{2}21$, $21\bar{1}$) measured every two hours. Corrections for Lorentz, polarization and absorption (Busing & Levy, 1957). Structure solved by Patterson and refined using *SHELX*77 (Sheldrick, 1977) system of programs; hydrogen atoms were not located. Full-matrix least squares, 136 parameters refined (scale factor, atomic coordinates and anisotropic displacement factors for non-H atoms). Function minimized $\sum w(|F_o| - |F_c|)^2$. Final $R = 0.040$, $wR = 0.042$, weighting scheme $a/[\sigma^2(F_o) + b(F_o)^2]$ with $a = 1.658$ and $b = 0$, $(\Delta/\sigma)_{max} = 0.11$.

Max. and min. height in final $\Delta\rho$ map 1·34 and −1·80 e Å^{-3}. Final atomic coordinates and equivalent displacement factors are given in Table 1.* Atomic scattering factors from *SHELX*76.

Discussion. The structure consists of chlorotetramethylformamidinium cations and $Hg_2Cl_5^-$ anions. We describe first the chlorotetramethylformamidinium cation, then the coordination around the Hg atoms and finally the overall arrangement in the crystal.

(*a*) *The chloro-N,N,N',N'-tetramethylformamidinium cation.* The geometry of the chlorotetramethylformamidinium ion is given in Table 2. Least-squares planes through selected groups of atoms have been deposited. The cation exhibits the main features found for the formamidinium group in various compounds (Foss, Johnsen & Tvedten, 1958; Villa, Nardelli & Tani, 1970; Villa, Manfredotti, Nardelli & Tani, 1972; Sanz & Daly, 1972; Daly, 1972; Henke & Bärnighausen, 1972; Lis, 1979; Prick & Beurskens, 1979; Lis, 1980; Russo, Valle & Calogero, 1980; Valle, Russo & Calogero, 1980; Jameson, Blazsó, Seferiadis & Oswald, 1982; Lis & Starynowicz, 1985; Larsen, 1985; Demant, Conradi, Müller & Dehnicke, 1985; Chivers, Richardson & Smith, 1985).

The sums of angles around the central C atom C(1) and at the two N atoms are all close to 360° suggesting sp^2 hybridization. The C(1)—Cl bond length of 1·731 (10) Å is typical for the C(sp^2)—Cl single bond. Values of 1·735 (3) and 1·717 (10) Å have been reported for the related complexes of N,N'-(chlorosulfoniumylidene)bis(N^1,N^1-dimethylchloroformamidine) (Chivers *et al.*, 1985). The two C(1)—N bonds [1·318 (12) and 1·285 (12) Å] are not significantly different indicating π-electron delocalization over the region N(1)—C(1)—N(2) so that the positive charge is shared by the two N atoms. The bonds between the N atoms and the terminal C-methyl groups all have values close to the typical single bond (1·45–1·46 Å) (Cram & Hammond, 1959).

Within the cation three planar groups, each consisting of four atoms (maximum deviation 0·06 Å), can be distinguished. These are the planes of the sp^2 hybridized C(1), N(1) and N(2) atoms [planes (*A*), (*B*) and (*C*), respectively]. These three planes might be expected to be coplanar giving optimum π-overlap between the central C atom C(1), and the two N atoms. Examples of planarity of this type are to be found in urea and (to less than 0·1 Å) in thiourea (Sanz & Daly, 1972). However, this planar conformation is not found here. Thus, planes (*B*) and (*C*) are rotated in opposite
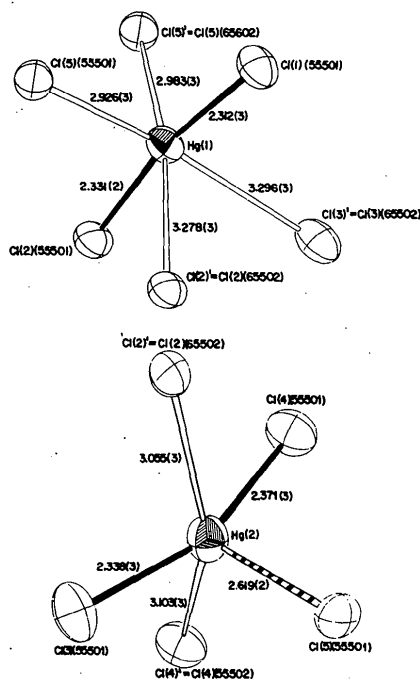
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* Anisotropic displacement factors of the non-H atoms, least-squares planes and a list of structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51362 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final atomic coordinates* ($\times 10^5$) *for Hg and* ($\times 10^4$) *for the rest of the atoms*: U_{eq} *values are* (Å$^2 \times 10^4$) *for Hg and Cl atoms and* (Å$^2 \times 10^3$) *for C and N; H atoms were not included in the refinement*

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

	x	y	z	U_{eq}
Hg(1)	51052 (4)	−9375 (4)	24843 (5)	390 (1)
Hg(2)	19993 (4)	3980 (4)	3023 (5)	382 (1)
Cl(1)	3945 (3)	−3108 (3)	2716 (4)	486 (6)
Cl(2)	6436 (3)	1026 (3)	1798 (3)	424 (5)
Cl(3)	3200 (3)	2487 (3)	−253 (4)	535 (7)
Cl(4)	263 (3)	−1671 (3)	−474 (4)	443 (6)
Cl(5)	3195 (3)	292 (3)	3670 (3)	422 (5)
Cl(6)	1273 (4)	5502 (3)	8339 (5)	647 (8)
C(1)	1433 (10)	4203 (9)	6809 (13)	35 (2)
C(2)	3799 (11)	5376 (11)	7428 (16)	50 (2)
C(3)	2636 (14)	3704 (11)	4515 (16)	53 (3)
C(4)	−968 (14)	3194 (15)	6263 (20)	67 (4)
C(5)	482 (14)	1782 (10)	5612 (14)	54 (3)
N(1)	371 (9)	3155 (8)	6170 (11)	41 (2)
N(2)	2531 (8)	4395 (7)	6306 (11)	37 (2)

Table 2. *Dimensions of the chloro-N,N,N',N'-tetramethylformamidinium cation*

Bonds (Å)		Angles (°)	
C(1)—Cl(6)	1·731 (10)	Cl(6)—C(1)—N(1)	115·8 (8)
C(1)—N(1)	1·318 (12)	Cl(6)—C(1)—N(2)	117·3 (7)
C(1)—N(2)	1·285 (12)	N(1)—C(1)—N(2)	126·6 (9)
N(1)—C(4)	1·477 (16)	C(1)—N(1)—C(4)	123·6 (10)
N(1)—C(5)	1·485 (13)	C(1)—N(1)—C(5)	120·7 (9)
N(2)—C(2)	1·496 (13)	C(4)—N(1)—C(5)	114·9 (10)
N(2)—C(3)	1·476 (13)	C(1)—N(2)—C(2)	123·5 (8)
		C(1)—N(2)—C(3)	122·5 (9)
		C(2)—N(2)—C(3)	114·0 (9)

directions about the C(1)—N bonds in order to relieve non-bonded intramolecular repulsions between methyl groups. The angles between the central plane (*A*) and planes (*B*) and (*C*) are 25(1) and 23(1)°, respectively; the angle between (*B*) and (*C*) being 45(1)°. Despite these rotations C(3) and C(5) are still quite close at 2·95 (1) Å. A similar behaviour has been reported for tetramethylformamidinium phosphate (Daly, 1972), and also for tetramethylformamidinium phosphate anhydride (Sanz & Daly, 1972).

(*b*) *Mercury coordination.* The Hg^{2+} ion has complete $4f$ and $5d$ electron shells, resulting in a spherical charge distribution. Due to the large volume and closed shells, Hg^{2+} can accommodate different numbers of ligands. A marked preference is observed for linear two-coordination forming an $HgCl_2$ unit which is similar to that found in the structure of $HgCl_2$ (Subramanian & Seff, 1980). Other common coordination numbers are three (trigonal), four (tetrahedral), five (trigonal bipyramidal) and six (octahedral) (Grdenič, 1965). The octahedral coordination is often encountered for chlorides and bromides. It is formed by four weak interactions with the central mercury of an almost linear HgX_2 unit.

Distances observed for chlorides range from 2·25–2·40 Å for the short covalent bonds, whereas the four long Hg—Cl bonds have distances between 2·7 and

3·5 Å as in $NH_4HgCl_3.H_2O$ (Sagisawa, Kitahama, Kiriyama & Kiriyama, 1974), in $(CH_3)_2NH_2HgCl_3$ and $(CH_3)_2NH_2Hg_2Cl_5$ (Ben Salah, Bats, Fuess & Daoud, 1982) and in $(CH_3)_3NHHgCl_3$ and $(CH_3)_3NHHg_2Cl_5$ (Ben Salah, Bats, Fuess & Daoud, 1983). In those

Cl(1)—Hg(1)—Cl(5)	94·1	Cl(3)—Hg(2)—Cl(2)'	90·2
Cl(1)—Hg(1)—Cl(2)	168·3	Cl(5)—Hg(2)—Cl(2)'	97·0
Cl(1)—Hg(1)—Cl(3)'	84·2	Cl(5)—Hg(2)—Cl(4)	102·1
Cl(5)—Hg(1)—Cl(2)	96·3	Cl(5)—Hg(2)—Cl(4)'	110·2
Cl(5)—Hg(1)—Cl(3)'	167·4	Cl(5)—Hg(2)—Cl(3)	103·7
Cl(2)—Hg(1)—Cl(3)'	84·5	Cl(4)—Hg(2)—Cl(4)'	83·5
Cl(1)—Hg(1)—Cl(2)'	96·1	Cl(4)—Hg(2)—Cl(3)	154·2
Cl(2)'—Hg(1)—Cl(3)'	81·3	Cl(4)—Hg(2)—Cl(2)'	87·8
Cl(2)'—Hg(1)—Cl(2)	79·2	Cl(4)'—Hg(2)—Cl(3)	86·6
Cl(2)'—Hg(1)—Cl(5)	86·5	Cl(4)'—Hg(2)—Cl(2)'	152·6
Cl(2)'—Hg(1)—Cl(5)'	168·5		
Cl(5)'—Hg(1)—Cl(1)	95·4		
Cl(5)'—Hg(1)—Cl(3)'	100·0		
Cl(5)'—Hg(1)—Cl(2)	89·5		
Cl(5)'—Hg(1)—Cl(5)	92·5		

Fig. 1. Geometric details of the coordination around the two Hg atoms of the reference asymmetric unit (*i.e.* atoms whose coordinates are given in Table 1). The codes of the chlorines are according to *ORTEP* (Johnson, 1965) notation. The sequence of symmetry elements is given in *International Tables for X-ray Crystallography* (1952). Atomic distances are given in Å and e.s.d.'s of the angles are 0·1°.

cases, where the weak Hg—Cl interactions are very long, the structure approaches the geometry found for the discrete (uncomplexed) $HgCl_2$ molecules such as reported for the structure of the 3:2 $HgCl_2$ and thiourea complex (Brotherton & White, 1973). In these compounds, such as crystalline $HgCl_2$ (Subramanian & Seff, 1980) and $(CH_3)_3NHHgCl_3$ (Ben Salah *et al.*, 1983), the Hg—Cl distances of 2·28–2·31 Å are only slightly longer than the value of 2·252 Å reported for gaseous $HgCl_2$ (Kashiwabara, Konaka & Kimura, 1973) and the Cl—Hg—Cl angle is equal to, or does not differ significantly from, the ideal value of 180°.

In trigonal bipyramidal coordination, three equatorial bonds of about 2·3–2·5 Å are found together with two long axial Hg—Cl bonds up to 3·1 Å. Such coordination has been observed in $(C_2H_5)_4NHgCl_3$ (Sandström & Liem, 1978), $(CH_3)_3SHgCl_3$ (Biscarini, Fusina, Nivellini & Pelizzi, 1977), $S_4N_3HgCl_3$ (Weidenhammer & Ziegler, 1977) and $(CH_3)_3NHHg_2Cl_3$ (Ben Salah *et al.*, 1983). A coordination number of five has been reported also for $C_6H_5CH_2NH_3Hg_2Cl_5$ (Bats, Fuess & Daoud, 1980) and for $CH_3NH_3HgCl_3$ and $CH_3NH_3Hg_2Cl_5$ (Ben Salah, Bats, Kalus, Fuess & Daoud, 1982).

The two crystallographically independent Hg atoms of the present complex show considerably different coordinations. The coordination spheres around the two Hg atoms and their corresponding geometries are shown in Fig. 1. Hg(1) has a distorted octahedral coordination of six Cl atoms. There are two short Hg—Cl bonds [2·312 (3) and 2·331 (2) Å], two long bonds [2·926 (3) and 2·983 (3) Å] and two very long bonds [3·278 (3) and 3·296 (3) Å]. It has been pointed out (Ben Salah *et al.*, 1983) that the distortion of the $HgCl_2$ pseudomolecule is dependent on the number of Hg—Cl long contacts even if these are longer than 3·0 Å. Thus, Hg—Cl contacts of the order of 3·0–3·3 Å appear to contribute to some extent to the bonding in these complexes. As expected, the $HgCl_2$ unit is distorted by the longer Hg—Cl contacts. Thus, the two short Hg—Cl bonds are slightly longer than the value of 2·252 Å reported for gaseous $HgCl_2$ (Kashiwabara *et al.*, 1973) and the angle between the two short bonds deviates significantly from the ideal 180° [168·3 (1)°]. A similar value [171·58 (8)°] has been reported for $(CH_3)_2NH_2Hg_2Cl_5$ (Ben Salah *et al.*, 1982) where all six
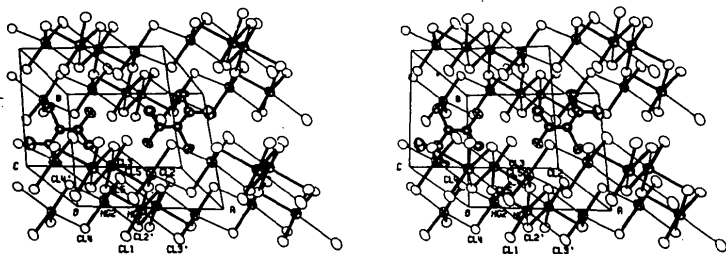

Fig. 2. Stereodiagram of the crystal structure. The thermal motion ellipsoids represent 50% probability distributions. Numbering is shown for the coordination spheres around the two Hg atoms of the reference asymmetric unit. The short and intermediate Hg—Cl bonds (up to 2·65 Å) are shown by thick lines, long contacts (up to 3·0 Å) by open bonds and very long contacts (up to 3·30 Å) by thin lines.

Hg—Cl bonds have values close to those found for the present complex. Larger deviations from 180° (values that range from 148° to 158°) have been reported for $(CH_3)_2NH_2HgCl_3$ (Ben Salah *et al.*, 1982) where the two long Hg—Cl contacts are shorter (2·67–2·84 Å) than those found here.

The second Hg atom, Hg(2), has a distorted trigonal bipyramidal coordination of five chlorines with the equatorial plane defined by Cl(3), Cl(4) and Cl(5). The Hg atom deviates by 0·018 Å from this plane. The three equatorial Hg—Cl bonds are not equal; two are short [2·338 (3) and 2·371 (3) Å] and one is of intermediate length [2·619 (2) Å]. This difference in the equatorial Hg—Cl bonds is clearly determined by the number of times each chlorine is bonded to the various symmetry-related Hg(1) atoms. Thus, Cl(3) and Cl(4) are terminal chlorines (if Hg—Cl contacts longer than 3·1 Å are ignored) whereas Cl(5) is involved also in two long bonds to Hg(1) [2·926 (3) and 2·983 (3) Å]. The two short Hg(2)—Cl bonds are again slightly longer than the value reported for gaseous $HgCl_2$. The Hg(2)—Cl(5) bond of intermediate length leads to the formation of a more distorted $HgCl_2$ unit. This is evident from a slight elongation in the Hg(2)—Cl short bonds [when compared to those found for Hg(1)] and by the distortion of the angle between the two short bonds [154·2 (1)°]. This angle value also represents the largest deviation from the ideal trigonal bipyramidal geometry which is completed by two very long axial Hg—Cl bonds [3·055 (3) and 3·103 (3) Å].

(c) *Overall arrangement.* A stereodiagram of the crystal structure is shown in Fig. 2. The anionic framework can be described in terms of $Hg_2Cl_5^-$ units; the reference asymmetric $Hg_2Cl_5^-$ unit with its numbering system is shown in Fig. 2. If only Hg—Cl bonds up to 2·4 Å are taken into account then the $Hg_2Cl_5^-$ anion might be considered to consist of two $HgCl_2$ molecules bridged by a chlorine anion, Cl(5), which is located closer to Hg(2) [2·619 (2) Å] than to Hg(1) [2·926 (3) Å]. Alternatively, the $Hg_2Cl_5^-$ anions might be regarded as consisting of an $Hg(1)Cl_2$ molecule linked to Cl(5) of an $Hg(2)Cl_3^-$ anion.

Pairs of $Hg_2Cl_5^-$ anions, related by a centre of symmetry at $\frac{1}{2},0,\frac{1}{2}$, are linked also through Cl(5) which is once again situated closer to Hg(2) [2·619 (2) Å] than to Hg(1) [2·983 (3) Å]. Such adjacent pairs are connected by Hg—Cl contacts longer than 3·0 Å [3·055 (3), 3·278 (3) and 3·296 (3) Å] *via* atoms Cl(2) and Cl(3). This arrangement leads to the formation of infinite zigzag chains extending along [001]. Neighbouring chains, related by a centre of inversion at 0,0,0 are connected by Hg—Cl contacts of 3·103 (3) Å *via* the Cl(4) atom to yield layers parallel to (010) with the cations located between such layers.

A somewhat similar arrangement of interconnected octahedral and trigonal bipyramidal Hg atoms has been reported for the structure of $(CH_3)_3NHHg_2Cl_5$ (Ben

Salah *et al.*, 1983). The trigonal bipyramidal Hg units form infinite zigzag chains which are linked by very long Hg—Cl contacts of 3·290 (2) Å to the octahedral environment of the second Hg atom.

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$C_5H_{12}ClN^+_2 \cdot 2HgCl_2 \cdot Cl^-$

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The Structure of Sodium Bis(tetraethylammonium) Tris(oxalato)aluminate(III) Monohydrate

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Abstract. $Na[N(C_2H_5)_4]_2[Al(C_2O_4)_3].H_2O$, $M_r = 591 \cdot 8$, monoclinic, Cc, $a = 16 \cdot 874$ (5), $b = 11 \cdot 347$ (2), $c = 17 \cdot 530$ (5) Å, $\beta = 115 \cdot 76$ (2)°, $V = 3023$ (3) Å^3, $Z = 4$, $D_x = 1 \cdot 299$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0 \cdot 133$ mm^{-1}, $F(000) = 1224$, $T = 293$ (1) K, $R = 0 \cdot 035$ for 2178 observed reflections. The Al atom is coordinated by six O atoms from three oxalate groups in octahedral configuration. The average Al–O bond distance is 1·897 (9) Å. The structure consists of sodium and tetraethylammonium cations, tris(oxalato)-aluminate(III) anions and disordered water molecules.

Introduction. As part of our study of metal(III) oxalate complexes we have undertaken the analysis of the influence of monovalent cations on the crystal structure and water content of these complexes (Bulc, Golič & Šiftar, 1982, 1983, 1985). We tried to prepare tetraethylammonium tris(oxalato)aluminate(III). However, due to impurities in tetraethylammonium hydroxide we obtained sodium bis(tetraethylammonium) tris(oxalato)aluminate(III) monohydrate. This compound falls into the scope of our work and therefore we determined its crystal structure.

Experimental. Crystals of the compound under investigation were grown from an aqueous solution of aluminium oxalate and sodium tetraethylammonium oxalate (1:3). Crystal size $0 \cdot 37 \times 0 \cdot 43 \times 0 \cdot 43$ mm, Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Space group Cc deduced from the systematic absences and distributions of $|E|$ and confirmed by the successful structure analysis. A trial refinement in the centrosymmetric space group $C2/c$ failed. Cell parameters by least-squares refinement of 25 reflections within $10 < \theta < 15°$, intensity measurement by ω–2θ scan, ω scan width (°) = $0 \cdot 7 + 0 \cdot 3$ tanθ, aperture (mm) = $2 \cdot 4 +$

Table 1. *Final fractional coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* U_{eq} (Å$^2 \times 10^3$)

	x	y	z	U_{eq}
Al	5000	1302 (1)	5000	40·5 (4)
Na	8218 (1)	3694 (1)	6471 (1)	48·8 (6)
O(1)	4554 (2)	180 (2)	4101 (1)	45 (1)
O(2)	3730 (2)	99 (2)	2707 (2)	53 (1)
O(3)	4568 (1)	2433 (2)	4107 (1)	47 (1)
O(4)	3917 (2)	2538 (2)	2696 (2)	51 (1)
C(1)	4141 (2)	629 (3)	3360 (2)	42 (1)
C(2)	4204 (2)	1992 (2)	3363 (2)	41 (1)
O(5)	5517 (1)	2496 (2)	5822 (1)	47 (1)
O(6)	6771 (2)	3504 (2)	6455 (2)	61 (1)
O(7)	6115 (1)	1238 (2)	4985 (1)	48 (1)
O(8)	7398 (2)	2184 (2)	5499 (2)	59 (1)
C(3)	6308 (2)	2762 (3)	5968 (2)	43 (1)
C(4)	6666 (2)	2010 (3)	5447 (2)	42 (1)
O(9)	3925 (1)	1389 (2)	5089 (1)	49 (1)
O(10)	3145 (2)	385 (3)	5615 (2)	62 (1)
O(11)	5262 (1)	51 (2)	5784 (1)	47 (1)
O(12)	4601 (2)	−1106 (2)	6364 (2)	64 (1)
C(5)	3810 (2)	563 (3)	5521 (2)	42 (1)
C(6)	4623 (2)	−276 (3)	5934 (2)	43 (1)
N(1)	1050 (2)	601 (3)	7890 (2)	63 (2)
C(11)	2033 (3)	324 (4)	8257 (3)	71 (2)
C(12)	2633 (3)	1310 (5)	8722 (4)	85 (3)
C(21)	790 (3)	940 (4)	8597 (3)	73 (2)
C(22)	934 (4)	7 (6)	9248 (3)	93 (3)
C(31)	574 (4)	−488 (4)	7424 (3)	82 (3)
C(32)	−412 (4)	−412 (7)	7045 (5)	110 (4)
C(41)	815 (3)	1652 (4)	7303 (3)	81 (3)
C(42)	1022 (5)	1528 (7)	6551 (4)	116 (4)
N(2)	7181 (2)	662 (3)	8679 (2)	65 (2)
C(51)	7343 (4)	1695 (5)	9284 (4)	98 (3)
C(52)	7091 (7)	1477 (8)	10004 (4)	137 (5)
C(61)	7687 (4)	−415 (5)	9144 (3)	88 (3)
C(62)	8682 (5)	−254 (9)	9564 (5)	129 (5)
C(71)	7473 (3)	1050 (5)	8019 (3)	90 (3)
C(72)	7379 (5)	170 (8)	7364 (3)	105 (4)
C(81)	6221 (3)	311 (4)	8286 (3)	71 (2)
C(82)	5587 (3)	1287 (5)	7824 (3)	86 (3)
O(20)	3293 (8)	−2193 (8)	2126 (9)	154 (10)
O(21)	4749 (18)	2378 (14)	4149 (26)	330 (33)

$0 \cdot 9$ tanθ, maximum scan time 60 s, hemisphere measured, with equality test (h +22/−22, k 0/13, l −23/+23), [(sinθ)/λ]$_{max}$ = 0·66 Å^{-1}, standard reflections $\overline{10},0,6$, $91\overline{5}$, $\overline{8}21$, remeasured every 1500 s of measuring time, showed an average increase in intensity of 6·1% during the data collection, 7258 reflections measured, merging produced 3622 unique reflections