Acta Cryst. (1984). C**40**, 1142–1145

Structure of Tetrakis[bis(2-hydroxyethyl)dithiocarbamato]tellurium(IV), $C_{20}H_{40}N_4O_8S_8Te$

By G. C. Rout and M. Seshasayee

Department of Physics, Indian Institute of Technology, Madras-600 036, *India*

and G. Aravamudan and K. Radha

Department of Chemistry, Indian Institute of Technology, Madras-600 036, *India*

(*Received* 5 *December* 1983; *accepted* 6 *February* 1984)

Abstract. $M_r = 848{\cdot}6$, triclinic, $P\bar{1}$, $a = 9{\cdot}008$ (2), $b = 17{\cdot}194$ (4), $c = 14{\cdot}501$ (2) Å, $\alpha = 128{\cdot}34$ (2), $\beta = 95{\cdot}60$ (2), $\gamma = 97{\cdot}13$ (2)°, $V = 1703{\cdot}44$ Å^3, $Z = 2$, $D_x = 1{\cdot}654$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 1{\cdot}295$ mm^{-1}, $F(000) = 864$, $T = 293$ K, $R = 0{\cdot}042$ and $R_w = 0{\cdot}045$ for 3482 unique observed reflections. In the molecule, the central tellurium atom is bonded to all eight sulphur atoms in a highly distorted dodecahedral configuration. The two planar trapezoids, each containing a TeS_4 group, make an angle of 86·50 (1)° with each other. The Te—S bond lengths are in the range 2·637 (4)–2·857 (4) Å, the average being 2·738 (4) Å. The extensive hydrogen bonding of the terminal hydroxyl groups in the range 2·678 (13)–2·763 (14) Å hinders the auto redox reaction in TeL_4 and also provides stability for the crystal packing.

Introduction. The ligand bis(2-hydroxyethyl)dithiocarbamate $(HOC_2H_4)_2NCSS^-$ (L) has afforded preparation and characterization (Sejekan, Janakiram & Aravamudan, 1978) and structural elucidation of novel mixed-ligand complexes of Te^{IV} featuring a dithiocarbamate group, namely $TeL_3Cl.2H_2O$ (Appa Rao, Seshasayee, Aravamudan & Radha, 1983*a*), $TeL_3SCN.H_2O$ (Rout, Seshasayee, Aravamudan & Radha, 1984), TeL_2I_2 (Appa Rao, Seshasayee, Aravamudan & Radha, 1983*b*) and also a tellurium(II) complex, TeL_2 (Rout, Seshasayee, Radha & Aravamudan, 1983). The title compound, TeL_4, had eluded preparation hitherto, as it very readily undergoes facile displacive reaction in contrast to tetrakis(diethyldithiocarbamato)tellurium(IV) [TeL'_4] (Husebye & Svaeren, 1973) and tetrakis(4-morpholinecarbodithioato)tellurium(IV) [TeL''_4] (Esperas & Husebye, 1975) analogues. However, conditions for the preparation of TeL_4 have now been standardized as detailed below. The structure of TeL_4 described here depicts yet another example of a rare highly coordinated Te^{IV} complex in addition to TeL'_4, TeL''_4 and tetrakis[(2-hydroxyethyl)methyldithiocarbamato]tellurium(IV) [TeL'''_4] (Husebye, 1979). Factors influencing the trends in reactivities of these tetrakis complexes are also discussed.

Experimental. Unlike TeL'_4 and TeL''_4, TeL_4 is not formed on addition of excess dithiocarbamate to solutions of Te^{IV} in strongly acid media. It is formed only under weakly acidic media and should be removed from the supernatant solution early to prevent gradual hydrolysis. However, when once separated it is indefinitely stable under anhydrous conditions. A solution of 10 mmol dm^{-3} of bis(2-hydroxyethyl)dithiocarbamic acid in 10 ml methanol, freshly prepared, is mixed with a solution of 1 mmol dm^{-3} TeO_2 in 10 ml 0·5 mol dm^{-3} NaOH at 283 K; 20 ml 1 mol dm^{-3} acetic acid is added; solution turns yellow and within minutes TeL_4 separates out as small bright yellow crystals; crystals were collected, washed repeatedly with cold ether, dissolved in acetone and recrystallized at 273–303 K. Analysis: found (expected) C 28·60 (28·29), H 4·92 (4·79), N 6·80 (6·60), S 31·20 (30·21), Te 14·78 (15·03)%.

Single crystal approximately 0·18 × 0·40 × 0·08 mm, lattice parameters from least-squares refinement of θ values for 25 high-angle reflections, Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan technique, scan $\Delta\omega = (0{\cdot}90 + 0{\cdot}35 \tan\theta)°$, background measured for one quarter of scan time at each end of peak scan interval; 6355 reflections (h 0–10, k −20–20, l −17–17) measured, $2 < \theta < 25°$, 2 strong reflections ($13\bar{3}$ and 201) recorded every hour during data collection, no significant variation in intensity, max. fluctuation 5%; 3482 unique reflections with $I > 3\sigma(I)$ considered observed, no absorption correction; structure solved with *SHELX* (Sheldrick, 1976); 3D Patterson map revealed position of Te atom, difference Fourier maps based on Te position showed locations of 8 S atoms and subsequently other non-H atoms, 3 cycles of full-matrix least-squares isotropic refinement of non-H atoms reduced R to 0·097 indicating very little anisotropic motion of the atoms in the molecule; H-atom positions located from difference Fourier map; all non-H atoms refined anisotropically, H atoms isotropically; $R = 0{\cdot}042$, $R_w = 0{\cdot}045$ for 3482 reflections, $\sum w(\Delta F)^2$ minimized, $w = 0{\cdot}012/[\sigma^2(F) + 0{\cdot}1908 \times |F|^2]$; max. $\Delta/\sigma = 0{\cdot}53$, av. $\Delta/\sigma = 0{\cdot}42$, 530 param-

0108-2701/84/071142-04$01.50

Table 1. *Fractional atomic coordinates* (×10^4) *and equivalent isotropic thermal parameters* (Å^2 ×10^3)

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Te	2079·2 (9)	4179·6 (5)	1742·1 (6)	19 (1)
S(1)	4732 (4)	3588 (2)	1213 (3)	30 (2)
S(2)	3825 (3)	4034 (2)	3362 (2)	27 (1)
S(3)	833 (4)	2230 (2)	814 (2)	32 (1)
S(4)	1170 (4)	2812 (2)	−698 (2)	30 (1)
S(5)	307 (3)	4680 (2)	3464 (2)	27 (1)
S(6)	−452 (4)	4695 (2)	1444 (2)	30 (2)
S(7)	3365 (4)	6199 (2)	3519 (2)	30 (1)
S(8)	3037 (4)	5181 (2)	940 (2)	30 (1)
C(1)	4891 (12)	3457 (7)	2306 (8)	22 (5)
N(1)	5854 (9)	2919 (6)	2306 (7)	21 (4)
C(11)	6710 (12)	2387 (8)	1351 (9)	24 (6)
C(12)	5675 (14)	1384 (8)	176 (10)	35 (6)
O(11)	6561 (9)	824 (5)	−696 (6)	32 (4)
C(13)	6098 (14)	2854 (8)	3281 (9)	31 (6)
C(14)	4947 (14)	1954 (8)	2994 (10)	33 (6)
O(12)	5433 (11)	1043 (6)	2153 (8)	44 (6)
C(2)	689 (11)	1865 (6)	−597 (7)	23 (4)
N(2)	225 (9)	893 (5)	−1582 (6)	22 (4)
C(21)	135 (12)	604 (7)	−2799 (7)	29 (5)
C(22)	1705 (13)	576 (8)	−3091 (8)	35 (6)
O(21)	1691 (11)	484 (5)	−4140 (6)	48 (5)
C(23)	−229 (11)	78 (7)	−1523 (9)	30 (5)
C(24)	1100 (14)	−308 (8)	−1331 (9)	36 (6)
O(22)	1672 (9)	−868 (5)	−2407 (6)	39 (4)
C(3)	−728 (10)	5083 (6)	2822 (7)	21 (4)
N(3)	−1693 (9)	5651 (5)	3330 (6)	21 (4)
C(31)	−2564 (12)	5973 (7)	2732 (8)	28 (5)
C(32)	−1627 (14)	6839 (8)	2864 (10)	44 (7)
O(31)	−1595 (10)	7821 (5)	3947 (7)	57 (5)
C(33)	−2033 (11)	5960 (7)	4475 (7)	26 (5)
C(34)	−822 (13)	6827 (7)	5579 (8)	36 (5)
O(32)	−715 (11)	7717 (5)	5693 (6)	62 (4)
C(4)	3621 (10)	6280 (6)	2420 (7)	22 (4)
N(4)	4260 (9)	7164 (5)	2706 (6)	26 (4)
C(41)	4513 (12)	7221 (7)	1757 (9)	34 (6)
C(42)	3215 (13)	7503 (8)	1336 (9)	39 (6)
O(41)	3407 (10)	8562 (6)	2021 (7)	51 (5)
C(43)	4811 (13)	8121 (7)	3989 (9)	36 (6)
C(44)	3561 (14)	8606 (8)	4587 (9)	39 (6)
O(42)	2975 (10)	9066 (6)	4128 (6)	54 (5)

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

Te–S(1)	2·707 (4)	N(2)–C(21)	1·50 (1)
Te–S(2)	2·857 (4)	N(2)–C(23)	1·47 (2)
Te–S(3)	2·736 (4)	N(3)–C(31)	1·50 (2)
Te–S(4)	2·712 (3)	N(3)–C(33)	1·48 (1)
Te–S(5)	2·824 (3)	N(4)–C(41)	1·48 (2)
Te–S(6)	2·637 (4)	N(4)–C(43)	1·50 (1)
Te–S(7)	2·712 (3)	C(11)–C(12)	1·55 (1)
Te–S(8)	2·716 (4)	C(13)–C(14)	1·54 (2)
S(1)–C(1)	1·73 (2)	C(21)–C(22)	1·51 (2)
S(2)–C(1)	1·70 (1)	C(23)–C(24)	1·52 (2)
S(3)–C(2)	1·71 (1)	C(31)–C(32)	1·51 (2)
S(4)–C(2)	1·73 (1)	C(33)–C(34)	1·51 (1)
S(5)–C(3)	1·73 (1)	C(41)–C(42)	1·53 (2)
S(6)–C(3)	1·73 (1)	C(43)–C(44)	1·50 (2)
S(7)–C(4)	1·72 (1)	C(12)–O(11)	1·43 (1)
S(8)–C(4)	1·72 (1)	C(14)–O(12)	1·43 (1)
C(1)–N(1)	1·34 (2)	C(22)–O(21)	1·43 (2)
C(2)–N(2)	1·33 (1)	C(24)–O(22)	1·43 (1)
C(3)–N(3)	1·30 (1)	C(32)–O(31)	1·42 (1)
C(4)–N(4)	1·33 (1)	C(34)–O(32)	1·42 (2)
N(1)–C(11)	1·47 (1)	C(42)–O(41)	1·41 (1)
N(1)–C(13)	1·48 (2)	C(44)–O(42)	1·43 (2)
S(1)–Te–S(2)	63·8 (2)	N(2)–C(21)–C(22)	110·7 (8)
S(3)–Te–S(4)	65·5 (2)	C(21)–C(22)–O(21)	111·3 (8)
S(5)–Te–S(6)	65·5 (2)	N(2)–C(23)–C(24)	114·3 (8)
S(7)–Te–S(8)	65·7 (2)	C(23)–C(24)–O(22)	108·7 (8)
S(2)–Te–S(5)	75·4 (2)	Te–S(5)–C(3)	84·2 (4)
S(4)–Te–S(8)	73·6 (2)	Te–S(6)–C(3)	90·3 (4)
S(1)–Te–S(6)	155·4 (2)	S(5)–C(3)–S(6)	117·5 (6)
S(3)–Te–S(7)	155·5 (2)	S(5)–C(3)–N(3)	122·9 (7)
Te–S(1)–C(1)	89·7 (5)	S(6)–C(3)–N(3)	119·6 (7)
Te–S(2)–C(1)	85·3 (5)	C(3)–N(3)–C(31)	120·7 (7)
S(1)–C(1)–S(2)	118·2 (8)	C(3)–N(3)–C(33)	121·9 (7)
S(1)–C(1)–N(1)	119·6 (8)	C(31)–N(3)–C(33)	117·4 (7)
S(2)–C(1)–N(1)	122·2 (8)	N(3)–C(31)–C(32)	113·6 (8)
C(1)–N(1)–C(11)	121·3 (8)	C(31)–C(32)–O(31)	114·2 (9)
C(1)–N(1)–C(13)	120·9 (8)	N(3)–C(33)–C(34)	114·1 (8)
C(11)–N(1)–C(13)	117·9 (8)	C(33)–C(34)–O(32)	109·6 (8)
N(1)–C(11)–C(12)	111·5 (9)	Te–S(7)–C(4)	88·3 (5)
C(11)–C(12)–O(11)	110·6 (9)	Te–S(8)–C(4)	88·1 (5)
N(1)–C(13)–C(14)	112·3 (9)	S(7)–C(4)–S(8)	117·9 (6)
C(13)–C(14)–O(12)	108·1 (9)	S(7)–C(4)–N(4)	121·0 (7)
Te–S(3)–C(2)	88·0 (5)	S(8)–C(4)–N(4)	121·0 (7)
Te–S(4)–C(2)	88·5 (5)	C(4)–N(4)–C(41)	120·7 (7)
S(3)–C(2)–S(4)	117·7 (6)	C(4)–N(4)–C(43)	121·7 (7)
S(3)–C(2)–N(2)	121·5 (7)	C(41)–N(4)–C(43)	117·5 (8)
S(4)–C(2)–N(2)	120·8 (7)	N(4)–C(41)–C(42)	114·3 (8)
C(2)–N(2)–C(21)	119·9 (7)	C(41)–C(42)–O(41)	113·1 (9)
C(2)–N(2)–C(23)	121·9 (7)	N(4)–C(43)–C(44)	114·3 (9)
C(21)–N(2)–C(23)	118·2 (7)	C(43)–C(44)–O(42)	110·0 (9)

eters refined; max. and min. heights in final ΔF map 1·82 and −1·23 e Å^{-3}; atomic scattering factors for non-hydrogen atoms from Cromer & Mann (1968), for hydrogen atoms from Stewart, Davidson & Simpson (1965); anomalous-dispersion corrections included (Cromer & Liberman, 1970).

Discussion. Fig. 1 depicts the configuration of the TeL_4 molecule and the thermal ellipsoid plots drawn by *ORTEP* (Johnson, 1965). Tables 1 and 2 contain the positional and bonding parameters.* As can be seen from Fig. 1, all the eight sulphur atoms of the four ligands are bonded to the central tellurium atoms in a distorted dodecahedral configuration as has been reported for TeL_4', TeL_4'' and TeL_4'''.

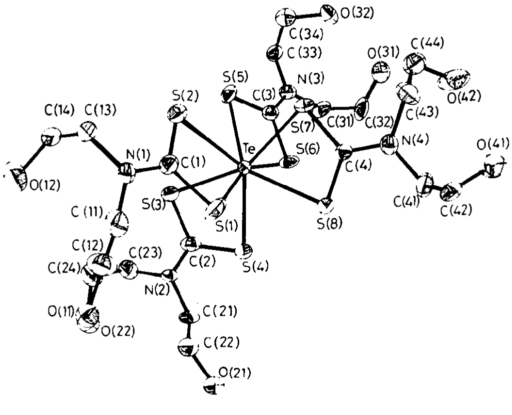

Fig. 1. Perspective view of the molecule TeL_4 with the thermal ellipsoids corresponding to 50% probability.

---

* Lists of structure factors, anisotropic thermal parameters, displacements from planes, coordinates and bonding parameters for hydrogen atoms and intramolecular short S–S contacts have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39260 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Dodecahedral-geometry parameters* (Å *and* deg)

	θ_A	θ_B	MA/MB	a	b	g	m
TeL_4	37·2	76·4	1·03	1·23	1·42	1·29	1·08
TeL_4'	35·0	79·9	1·02	1·16	1·42	1·31	1·07
TeL_4''	37·4	77·8	1·04	1·24	1·42	1·29	1·07
TeL_4'''	35·0	80·0	1·02	1·15	1·42	1·31	1·07
Hard-sphere model	36·85	69·46	1	1	1·499	1	1

The unit of length for a,b,g and m is in each case equal to the average central-atom to ligand bond length. θ_A (θ_B) is the angle that $MA(MB)$ makes with the unique axis passing through the central atom and the mid-point of the A–A contact. The parameter a is equal to the S_A–S_A distance within the two trapezoids of the dodecahedron, divided by the average M–S bond length.

In TeL_4 the distorted dodecahedral arrangement leads to TeS_8 coordination which could be thought of in terms of two intersecting TeS_4 trapezoids comprising Te, S(1), S(2), S(5), S(6) and Te, S(3), S(4), S(7), S(8), both of which are planar to within 0·116 (3) Å. Their interplanar angle is 86·50 (1)°, which can be compared with the corresponding angle in TeL_4' (88·5°), TeL_4'' (89·8°) and TeL_4''' (90°). This shows that, compared to the other three compounds, the present compound is more distorted from a regular dodecahedral D_{2d} symmetry which requires an interplanar angle of 90°.

A comparison table of the dodecahedral parameters for the above structures is presented in Table 3 according to notations given by Hoard & Silverton (1963). There are two sets of non-equivalent corners of the dodecahedron, four of type A and four of type B such that $MA > MB$, where M represents the central Te atom. In the present case the chelation is along edges m with atoms S(2), S(5), S(4) and S(8) being located at A sites while the other sulphur atoms are at B sites. The configurations of all the four complexes are distorted dodecahedra when compared to the ideal hard-sphere-model dodecahedral parameters (Kepert, 1978). TeL_4 is significantly different from TeL_4' and TeL_4''' with respect to parameters a and to some extent θ_A and θ_B. It closely resembles TeL_4''. The 5s lone pair of electrons on the Te atom is stereochemically inert as in other highly coordinated Te^{IV} complexes and it does not occupy a position in the coordination polyhedron.

The Te—S_A and Te—S_B bond lengths in TeL_4 range from 2·712 (3) to 2·857 (4) Å and 2·637 (4) to 2·736 (4) Å, respectively. The average Te—S bond length of 2·738 (4) Å is considerably longer than 2·36 Å (Alcock, 1972), the sum of the single covalent radii of Te and S and is very close to the corresponding bond lengths for TeL_4' (2·744 Å), TeL_4'' (2·737 Å) and TeL_4''' (2·737 Å). The average intraligand S—Te—S angle is 65·0 (2)° and the average interligand S—Te—S angles where two sulphur atoms are from different ligands in one trapezoid are 74·5 (2) and 155·4 (2)°, respectively. These seem to be normal. It is interesting to note that the core TeS_8 bond parameters for TeL_4 and TeL_4'' are very similar to each other in contrast to their widely differing reactivities (see later).

The four L ligands, except for their terminal CH_2OH groups, are planar to within 0·049 (9) Å. The C—S bond lengths vary between 1·70 (1) and 1·73 (1) Å, average 1·72 (1) Å. The C—N bond lengths range from 1·30 (1) to 1·35 (2) Å, average 1·33 (1) Å. The average C—S and C—N bond lengths are normal and exhibit partial double-bond character indicating the usual mesomeric shift of electrons from nitrogen to sulphur found in dithiocarbamate complexes. The bond angles at C(i) and N(i), $i = 1$ to 4, are consistent with sp^2 hybridization of these atoms. The average N—C, C—C, C—O, C—H and O—H bond lengths in the hydroxyethyl groups are 1·47 (2), 1·52 (2), 1·42 (1), 1·01 (11) and 0·90 (11) Å, respectively, and are in the expected range.

The packing of the molecules in the unit cell is shown in Fig. 2. The hydroxyl groups from the neighbouring molecules approach each other and each functions as both hydrogen-bond donor and acceptor so that all the oxygen atoms are involved in hydrogen bonding. The hydrogen-bond parameters and the sulphur–sulphur short contacts less than 3·7 Å are given in the deposited material. There are two intramolecular and six intermolecular contacts with O···O distances and O—H···O angles ranging from 2·678 (13) to 2·763 (14) Å and 151 (7) to 170 (7)°, respectively. The packing arrangement seems to be mainly dictated by the hydrogen-bonding network. Apart from normal van der Waals contacts among the molecules, there are several sulphur–sulphur intramolecular close approaches, the minimum being S(4)–S(8) = 3·252 (4) Å.

Structure-reactivity correlations amongst the different tetrakis(dithiocarbamato)tellurium(IV) complexes merit discussion. Two aspects of reactivity are discussed: (a) tendency to undergo auto-redox reaction; and (b) tendency to undergo ligand substitution reactions. The complexes TeL_4' and TeL_4'' have pronounced tendency to form TeL_2' and TeL_2'' and the respective thiuram disulphides L'-L' and L''-L'' in chloroform, dichloromethane and acetonitrile solutions.

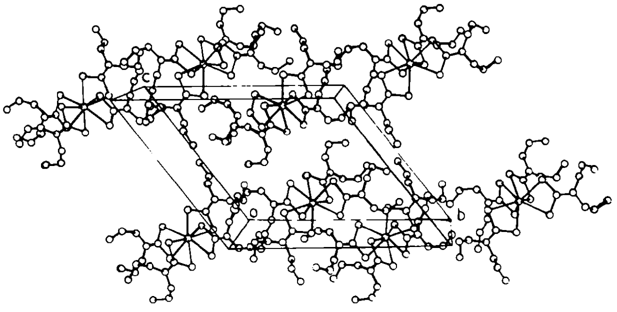

Fig. 2. Molecular packing in the unit cell of TeL_4.

We have observed (unpublished results) that such a reaction occurs even more readily with tetrakis(piperidinodithiocarbamato)tellurium(IV). This arises as a result of intramolecular auto-oxidation reaction. Esperas & Husebye (1975) have discussed these interactions in terms of close S–S approaches of two different ligands in the $\mathrm{TeS_8}$ core of TeL_4''. In TeL_4 and TeL_4''' these corresponding minimum approaches are 3·252 (2) and 3·159 (3) Å which are not very different from those observed in TeL_4' (3·16 Å) and TeL_4'' (3·30 Å). Yet, in these two complexes, namely TeL_4' and TeL_4'', there is no tendency for this type of self-redox reaction in acetonitrile. Examination of the molecular model of TeL_4 indicates that the C_2H_4OH groups in the structure do not have any steric influence to hinder the S–S approach and the auto-redox reaction. Therefore, it is tempting to suggest that this is hindered in TeL_4 and TeL_4''' primarily because of extensive hydrogen bonding of the C_2H_4OH groups. With respect to substitution reactions, TeL_4 and TeL_4''', in contrast to TeL_4', undergo very facile substitutions leading to mixed-ligand complexes of $\mathrm{Te^{IV}}$ (Rout, Seshasayee, Aravamudan & Radha, 1984; Appa Rao, Seshasayee, Aravamudan & Radha, 1983*a*, *b*). TeL_4' forms mixed-ligand complexes only under drastic conditions (Deuten, Schnabel & Klar, 1980; Schnabel, Deuten & Klar, 1982). TeL_4'' is stable towards substitution reactions. No mixed-ligand complex of TeL_4'' has been reported. This is ascribed to the solvation and also the better electron-withdrawing property of the C_2H_4OH groups favouring ready substitution in TeL_4 and TeL_4'''. We are currently studying the structure of TeL_2O, the first crystalline stable dithiocarbamate complex of $\mathrm{Te^{IV}}$ featuring an oxo ligand formed by substitution reactions of TeL_4 under suitable conditions.

References

Alcock, N. W. (1972). *Adv. Inorg. Chem. Radiochem.* **15**, 1–53.

Appa Rao, G. V. N., Seshasayee, M., Aravamudan, G. & Radha, K. (1983*a*). *Inorg. Chem.* **22**, 2590–2593.

Appa Rao, G. V. N., Seshasayee, M., Aravamudan, G. & Radha, K. (1983*b*). *Acta Cryst.* C**39**, 1018–1021.

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Deuten, K. V., Schnabel, W. & Klar, G. (1980). *Phosphorus Sulphur*, **9**, 93–98.

Esperas, S. & Husebye, S. (1975). *Acta Chem. Scand. Ser. A*, **29**, 185–194.

Hoard, J. L. & Silverton, J. V. (1963). *Inorg. Chem.* **2**, 235–243.

Husebye, S. (1979). *Acta Chem. Scand. Ser. A*, **33**, 485–490.

Husebye, S. & Svaeren, S. E. (1973). *Acta Chem. Scand.* **27**, 763–778.

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

Kepert, D. L. (1978). *Prog. Inorg. Chem.* **24**, 179–249.

Rout, G. C. Seshasayee, M., Aravamudan, G. & Radha, K. (1984). *J. Crystallogr. Spectrosc. Res.* **14**(2), 191–202.

Rout, G. C. Seshasayee, M., Radha, K. & Aravamudan, G. (1983). *Acta Cryst.* C**39**, 1021–1023.

Schnabel, W., Deuten, K. V. & Klar, G. (1982). *Phosphorus Sulphur*, **13**, 345–355.

Sejekan, B. G., Janakiram, C. & Aravamudan, G. (1978). *J. Inorg. Nucl. Chem.* **40**, 211–213.

Sheldrick, G. M. (1976). *SHELX*. Program for crystal structure determination. Univ. of Cambridge, England.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1984). C**40**, 1145–1147

Structure of *trans*-Diaquabis(ethylenediamine)nickel(II) Bis(trichloroacetate), $[\mathrm{Ni(C_2H_8N_2)_2(H_2O)_2}](\mathrm{C_2Cl_3O_2})_2$

By S. García-Granda and F. Gómez-Beltrán

Departamento de Química Física, Facultad de Química, Universidad de Oviedo, Calvo Sotelo S/N, Oviedo, Spain

(*Received* 26 *January* 1984; *accepted* 5 *March* 1984)

Abstract. $M_r = 539·7$, triclinic, $P\bar{1}$, $a = 7·2099$ (4), $b = 8·5966$ (3), $c = 8·6881$ (3) Å, $\alpha = 67·72$ (2), $\beta = 82·74$ (3), $\gamma = 87·08$ (3)°, $V = 494·29$ (9) Å^3, $Z = 1$, $D_x = 1·81$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 1·83$ mm^{-1}, $F(000) = 274$, $T = 298$ K, final $R = 0·051$ for 2328 observed reflections. The crystals are composed of centrosymmetric complex $|\mathrm{Ni(en)_2(H_2O)_2}|^{2+}$ cations and $(\mathrm{C_2Cl_3O_2})^-$ anions. The coordination of Ni is *trans* pseudo-octahedral. The bond lengths and angles of both cation and anion are normal for this type of compound. The Cl atoms of trichloroacetate are not disordered.

Introduction. In order to have enough data to understand the chemical and physicochemical behaviour of nickel and cobalt transition-metal complexes of ethylenediamine and C-substituted ethylenediamines, we have determined the crystal structures of

0108-2701/84/071145-03$01.50