We have observed (unpublished results) that such a reaction occurs even more readily with tetrakis(piperidinodithiocarbamato)tellurium(IV).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 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 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 Te^{IV} featuring an oxo ligand formed by substitution reactions of TeL_4 under suitable conditions.

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

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

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(*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 $|Ni(en)_2(H_2O)_2|^{2+}$ cations and $(C_2Cl_3O_2)^-$ 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 several of these complexes (Alcalá Aranda, Fernández, Gómez-Beltrán & Larena, 1977; Gómez-Beltrán, Valero Capilla & Alcalá Aranda, 1978*a,b,* 1979; Valero Capilla, Alcalá Aranda & Gómez-Beltrán, 1980). The structures of several nickel complexes with ethylenediamine have been reported (Minacheva, Antsyshkina & Porai-Koshits, 1974); however, the present compound contains the trichloroacetate anion which, following our previous work (Gómez-Beltrán *et al.*, 1978*a*), might be expected to show disorder. The work reported here also aimed to gather more data concerning the behaviour of the trichloroacetate anion in complexes of this type.

Experimental. Blue crystal, 0·64 × 0·35 × 0·27 mm, unit-cell parameters and intensity data obtained on an Enraf–Nonius CAD-4F diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scans ($0 < \theta < 30°$), cell dimensions refined by least-squares fitting of θ values of 25 reflections; no appreciable drop in intensity of 3 standard reflections checked every hour; 2863 reflections collected, *hkl* range −10,−10,0 to 10,12,12, 2328 with $I > 3\sigma(I)$ used in subsequent calculations; measured intensities corrected for Lorentz and polarization effects, not for absorption ($\mu R = 1{\cdot}18$); scattering factors for neutral atoms and anomalous-dispersion corrections for Ni and Cl from *International Tables for X-ray Crystallography* (1974); a statistical test showed a centric intensity distribution. Structure solved, using *CRYSTAN* system (Burzlaff, Böhme & Gomm, 1977), by three-dimensional Patterson and standard Fourier techniques; H atoms located by ΔF synthesis. Full-matrix least-squares refinement on F, anisotropic thermal parameters for all non-hydrogen atoms, isotropic for H, final $R = 0{\cdot}051$ and $R_w = 0{\cdot}060$, $w = 1/\sigma^2(F_o)$; max. $\Delta/\sigma = 0{\cdot}1$, $\Delta\rho$ in final difference Fourier map $\pm 0{\cdot}4$ e Å^{-3}. Atomic parameters are listed in Table 1.* Fig. 1 shows the atom numbering.

Discussion. Interatomic distances and angles are listed in Table 2. All of them are normal for this kind of compound.

The [NiN$_4$O$_2$] chromophore has a pseudo-octahedral structure with ligand–central-ion distances of expected values. A listing of the geometrical features of the hydrogen bonds has been deposited.*

In the trichloroacetate ion, the average C—Cl distance [1·769 (5) Å] and the O—C—O angle [128·0 (5)°] are within the range found for this ion in

Table 1. *Atomic parameters and temperature factors* (Å^2 × 10^4, × 10^3 *for* H)

For non-hydrogen atoms, $U_{\text{eq}} = \frac{1}{3}\sum U_{ij}a^*_i a^*_j a_i a_j \cos(\mathbf{a}_i.\mathbf{a}_j)$.
For H atoms, U is the refined isotropic temperature factor.

	x	y	z	U_{eq} or U
Ni	0·00000	0·00000	0·00000	256 (3)
Cl(1)	−0·57413 (20)	−0·48897 (17)	−0·18976 (16)	494 (6)
Cl(2)	−0·70805 (20)	−0·16097 (19)	−0·38794 (18)	558 (7)
Cl(3)	−0·38215 (20)	−0·29998 (18)	−0·51140 (17)	519 (6)
O(1)	−0·2816 (4)	−0·0819 (4)	0·0757 (4)	371 (13)
O(2)	−0·4794 (5)	−0·1580 (5)	−0·1340 (4)	467 (15)
O(3)	0·2153 (4)	0·2189 (5)	0·2646 (5)	578 (18)
N(1)	0·0450 (5)	−0·0992 (4)	0·2527 (5)	346 (15)
N(2)	0·0692 (5)	−0·2446 (5)	0·0147 (5)	376 (16)
C(1)	0·1379 (7)	−0·2639 (6)	0·2889 (6)	450 (21)
C(2)	0·0536 (7)	−0·3559 (6)	0·1945 (7)	443 (21)
C(3)	−0·5053 (6)	−0·2838 (5)	−0·3294 (5)	309 (16)
C(4)	−0·3871 (6)	−0·2114 (5)	−0·2358 (6)	350 (18)
H(1)	−0·350 (9)	−0·098 (8)	−0·000 (8)	62 (21)
H(2)	0·124 (7)	−0·345 (6)	0·423 (7)	28 (15)
H(3)	−0·061 (8)	−0·116 (7)	0·310 (7)	28 (14)
H(4)	0·279 (8)	−0·244 (6)	0·253 (7)	37 (17)
H(5)	−0·353 (8)	−0·026 (6)	0·080 (7)	30 (15)
H(6)	0·116 (8)	−0·039 (6)	0·309 (6)	33 (16)
H(7)	0·120 (7)	−0·476 (5)	0·226 (6)	23 (10)
H(8)	0·184 (6)	−0·243 (5)	−0·020 (5)	13 (12)
H(9)	−0·060 (9)	−0·392 (7)	0·219 (7)	47 (18)
H(10)	−0·053 (8)	−0·276 (6)	−0·031 (6)	27 (15)

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

Ni–N(1)	2·093 (4)	Cl(2)–C(3)	1·770 (4)
Ni–N(2)	2·096 (4)	Cl(3)–C(3)	1·763 (5)
Ni–O(1)	2·123 (3)	O(2)–C(4)	1·250 (7)
N(1)–C(1)	1·473 (6)	O(3)–C(4)	1·236 (5)
N(2)–C(2)	1·484 (6)	C(1)–C(2)	1·528 (9)
Cl(1)–C(3)	1·773 (4)	C(3)–C(4)	1·546 (8)
N(2)–Ni–O(1)	87·4 (1)	Cl(1)–C(3)–Cl(3)	107·9 (3)
N(1)–Ni–O(1)	88·4 (1)	Cl(1)–C(3)–Cl(2)	108·9 (3)
N(1)–Ni–N(2)	83·0 (2)	Cl(3)–C(3)–C(4)	113·6 (3)
Ni–N(1)–C(1)	108·6 (3)	Cl(2)–C(3)–C(4)	111·5 (3)
Ni–N(2)–C(2)	107·7 (3)	Cl(1)–C(3)–C(4)	107·1 (3)
N(1)–C(1)–C(2)	108·7 (4)	O(3)–C(4)–C(3)	117·1 (4)
N(2)–C(2)–C(1)	108·0 (4)	O(2)–C(4)–C(3)	114·9 (4)
Cl(2)–C(3)–Cl(3)	107·8 (2)	O(2)–C(4)–O(3)	128·0 (5)

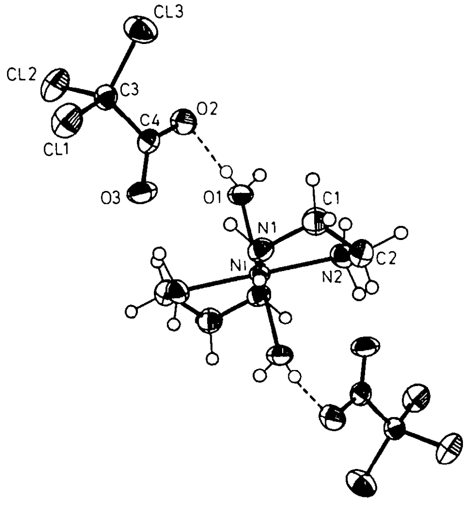

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule, showing the atom numbering.

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* Lists of observed and calculated structure amplitudes, anisotropic thermal parameters, bond lengths and angles involving H atoms, principal torsion angles and least-squares-planes' information have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39309 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

similar compounds (Golič & Lazarini, 1974*a,b*, 1975) and it was not necessary to invoke disorder of the Cl atoms to refine either their structures or the present structure. This fact, in contradiction to that found by Gómez-Beltrán *et al.* (1978*a*) in bis(tetramethylethylenediamine)Ni trichloroacetate, seems to indicate that disorder in the position of the Cl atoms in the trichloroacetate ion is far from being general behaviour.

Values of the angles between the best least-squares planes for various portions of the molecule and principal torsion angles, computed by *PARST* 5 (Nardelli, Musatti, Domiano & Andreetti, 1965), have been deposited.*

Most of the calculations were carried out on the HP-1000-F minicomputer of the Computing Center of the University of Oviedo.

The authors wish to thank Dr E. Gutiérrez-Puebla, Inorganic Chemistry Department , Universidad Complutense de Madrid, for measurement of the intensities.

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* See deposition footnote.

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Acta Cryst. (1984). C**40**, 1147–1150

Structural Studies of Tetraaquabis(saccharinato-*N*)zinc(II) Dihydrate, $[Zn(C_7H_4NO_3S)_2(H_2O)_4].2H_2O$, and Tetraaquabis(saccharinato-*N*)cadmium(II) Dihydrate, $[Cd(C_7H_4NO_3S)_2(H_2O)_4].2H_2O$*

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(*Received* 29 *December* 1983; *accepted* 5 *March* 1984)

Abstract. Zn compound: $M_r = 537·82$, monoclinic, $P2_1/c$, $a = 7·939$ (2), $b = 16·120$ (2), $c = 7·691$ (2) Å, $\beta = 99·87$ (2)°, $V = 969·7$ Å^3, $Z = 2$, $D_m = 1·83$, $D_x = 1·84$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 14·7$ cm^{-1}, $F(000) = 552$, $T = 294$ (2) K. Cd compound: $M_r = 584·85$, monoclinic, $P2_1/c$, $a = 8·036$ (2), $b = 16·145$ (3), $c = 7·870$ (1) Å, $\beta = 100·24$ (1)°, $V = 1004·8$ Å^3, $Z = 2$, $D_m = 1·92$, $D_x = 1·93$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 12·4$ cm^{-1}, $F(000) = 588$, $T = 294$ (2) K. $R = 0·028$ and 0·025 for 1581 and 1850 intensities, respectively. The isostructural pair of complexes have centrosymmetric *trans* octahedral geometry. Delocalization of the charge away from the N atom may explain the weakness of the M—N bond. The structures of the Cd^{II} and Zn^{II} compounds are compared with those involving Mn^{II}, Fe^{II}, Co^{II}, Ni^{II} and Cu^{II} and trends in the metal–ligand M—N, M—OH$_2$ bond lengths are discussed.

Introduction. We have described the syntheses and properties of a series of complexes with general formula $[M(C_7H_4NO_3S)_2(H_2O)_4].2H_2O$ (M = Mn^{II}, Fe^{II}, Co^{II}, Ni^{II}, Cu^{II} and Zn^{II}) (Haider, Malik & Ahmed, 1981;

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* Saccharin is 1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide.

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0108-2701/84/071147-04$01.50