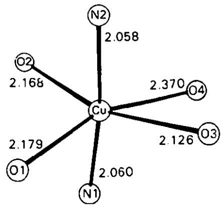

Fig. 3. The environment of the Cu atom viewed perpendicular to the plane defined by N(1),O(2),O(4).

structures, of which about thirty have been determined, the ligand acts as an anionic 2-dialkylaminoethanolato chelate.

The Cu atom is six coordinated, and the structure of the $CuN_2O_4$ core is shown in Fig. 3. The environment of the Cu atom is not typical for six-coordinate $Cu^{II}$ chromophores (Hathaway, 1984) and represents a further example of the stereochemical variety found in $Cu^{II}$ structures. On the basis of bond lengths and angles the environment of the Cu atom is perhaps best considered as a compressed octahedral stereochemistry. It should be added, however, that this classification is not unambiguous, because axial equatorial bond directions could be selected in a different way.

Except for the O(4)—C(9) bond of 1·212 (11) Å, the distances and angles within coordinated and uncoordinated benzoate groups are very similar and do not show a greater variation than would be expected on the basis of standard deviations. Distance and angular parameters of the 2-dimethylaminoethanol ligand are about as expected and do not warrant further comment.

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*Acta Cryst.* (1985). C**41**, 1730–1733

# (4,7,13,16,21,24-Hexaoxa-1,10-diazabicyclo[8.8.8]hexacosane)sodium Tetratelluride(2—)

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(*Received* 4 *June* 1985; *accepted* 22 *July* 1985)

**Abstract.** $[Na(C_{18}H_{36}N_2O_6)]_2[Te_4]$, $M_r = 1309{\cdot}3$, monoclinic, $P2_1/c$, $a = 10{\cdot}710\,(5)$, $b = 12{\cdot}038\,(9)$, $c = 39{\cdot}14\,(2)$ Å, $\beta = 95{\cdot}07\,(4)°$, $U = 5027$ Å$^3$, $D_x = 1{\cdot}73$ Mg m$^{-3}$, $Z = 4$, Mo $K\bar{\alpha}$, $\lambda = 0{\cdot}71069$ Å, $\mu = 2{\cdot}52$ mm$^{-1}$, $T = 295$ K, $F(000) = 2568$, $R_1 = 0{\cdot}082$ for 2660 observed $[I > 3\sigma(I)]$ reflections. Lattice contains cryptated $Na^+$ ions and tetratelluride anions, $Te_4^{2-}$, with terminal bond lengths 2·702 (3) and 2·702 (3) Å, a longer central bond of length 2·746 (3) Å, Te—Te—Te bond angles 109·7 (1) and 110·0 (1)° and a Te—Te—Te—Te dihedral angle of 105·3 (1)°.

**Introduction.** Elements of Group VI form anionic chains having the general formula $Ch_x^{2-}$. A large number of these anions have been structurally characterized, with $x = 3$–7 for S, 3–6 for Se, and 3–5 for Te (Huffman & Haushalter, 1984, and references therein). It is only recently that the structure of the third Te anion, $Te_4^{2-}$, has been determined in $[Ph_4P]_2[Te_4]$.-$2CH_3OH$ (Huffman & Haushalter, 1984). Each end of the tetratelluride(2—) anion is hydrogen bonded to a $CH_3OH$ molecule. The present structure determination of $Te_4^{2-}$ in $[(2,2,2\text{-crypt})Na]_2[Te_4]$ represents a $Te_4^{2-}$ anion in an unsolvated environment. Significant differences in the gross structural features of the anions

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derived from the two environments are compared and discussed.

**Experimental.** Stoichiometric amount of 2,2,2-crypt (Merck) corresponding to the alkali metal thoroughly mixed with 150–200 mg of powdered $NaTlTe_2$ alloy in a glass ampoule. Approximately 10 mL of ethylenediamine (en), previously dried over $CaH_2$, vacuum distilled onto the mixture and tube and contents allowed to warm to room temperature. Extraction of the alloy within one week. Over ensuing two-week period, $[(2,2,2\text{-crypt})Na]_2[Te_4]$ deposited as deep-red-violet crystals from the extract solution. Crystals isolated by decantation of the supernatant liquid followed by vacuum drying to remove residual en were cleaved into smaller irregular fragments and sealed in Lindemann capillaries in a dry-nitrogen-filled glove box equipped with a microscope. Crystals are of poor quality as indicated by preliminary precession photographs. Further work on an Enraf–Nonius CAD-4 diffractometer using graphite-monochromatized Mo $K\bar{\alpha}$ radiation and an approximately rectangular-shaped plate of dimensions 0·10 × 0·25 × 0·30 mm. Unit-cell dimensions by least-squares fit for diffracting positions of 25 reflections ($5·3 < \theta < 14·4°$). Peak profiles broad. Intensity data collected using $\omega$–$2\theta$ scans over $\omega$-scan ranges $(0·9 + 0·35 \tan\theta)°$. Scan rates conditional on information collected in prescans were selected to give an $I/\sigma(I)$ ratio of 25 within a max. scan time of 55 s. Gradual decrease in the intensities of 3 standard reflections monitored every 9000 s of exposure time corrected for after data reduction [max. rescale factor (on $F$) = 1·26]. Backgrounds by extending scan by 25% on either side of peak were measured for half the time taken to collect the peak. 9095 reflections in quadrant $h,k,\pm l$ with $2\theta \leq 48°$ measured. Lorentz, polarization and crystal-decay corrections applied to all reflections. No correction for absorption or extinction. 2660 observed reflections [$I > 3\sigma(I)$; 33%] in final data set. Structure solved by the use of *MULTAN*11/82 (Main *et al.*, 1982) to locate four Te positions: other atoms from Fourier syntheses. Te and Na atoms refined anisotropically. H atoms placed in calculated positions (C—H = 1·08 Å) with common temperature factor for each crypt. Blocked least-squares refinements minimizing $\sum w||F_o| - |F_c||^2$ converged (max. $\Delta/\sigma = 0·03$) to final agreement indices $R = 0·082$ ($wR = 0·086$) for 2660 observed data with $I > 3\sigma(I)$. High $R$ factors are a feature of structures containing crypt moieties. Weights given by $w = [\sigma^2(F) + 0·008F^2]^{-1}$. 5 reflections with $\Delta F/\sigma > 5·5$ rejected from final cycle. Most significant features in final difference Fourier map are some peaks up to 1·1 e Å$^{-3}$ in height close to the Te atoms. Programs: Enraf–Nonius *SDP* and *SHELX*76 (Sheldrick, 1976) on PDP 11/23 and Gould 9705 computers.

**Discussion.** The final atomic positional and thermal parameters are included in Table 1; the coordination geometry of the sodium ions is in Table 2.*

The alloy KTlTe has been shown to react with ethylamine/ethylenediamine in the presence of 2,2,2-crypt (4,7,13,16,21,24-hexaoxa-1,10-diazabicyclo-[8.8.8.]hexacosane) to give the heteropolyatomic anion

Table 1. *Final atomic positional* ($\times 10^4$) *and thermal* ($\times 10^3$) *parameters*

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

| | $x$ | $y$ | $z$ | $U$ or $U_{eq}$(Å$^2$) |
|---|---|---|---|---|
| Te(1) | −908 (2) | 3621 (2) | 9127 (1) | 66·3 (15) |
| Te(2) | 100 (2) | 1563 (2) | 9152 (1) | 59·1 (13) |
| Te(3) | −954 (2) | 316 (2) | 8614 (1) | 59·4 (13) |
| Te(4) | 676 (2) | 94 (2) | 8133 (1) | 68·4 (15) |
| Na(1) | 6018 (10) | 2505 (9) | 427 (3) | 43 (6) |
| N(11) | 3413 (19) | 2437 (17) | 404 (5) | 33 (6) |
| C(12) | 3079 (29) | 3324 (24) | 647 (8) | 56 (9) |
| C(13) | 3743 (24) | 4425 (21) | 604 (7) | 33 (7) |
| O(14) | 5035 (16) | 4267 (14) | 649 (4) | 36 (5) |
| C(15) | 5733 (26) | 5205 (24) | 615 (7) | 47 (8) |
| C(16) | 7099 (26) | 4976 (23) | 589 (7) | 43 (8) |
| O(17) | 7169 (16) | 4400 (13) | 277 (4) | 34 (5) |
| C(18) | 8416 (27) | 4397 (24) | 190 (7) | 47 (8) |
| C(19) | 9315 (29) | 3711 (24) | 407 (8) | 57 (9) |
| N(110) | 8876 (19) | 2540 (16) | 424 (5) | 27 (5) |
| C(111) | 9211 (27) | 1923 (23) | 130 (7) | 46 (8) |
| C(112) | 8350 (27) | 859 (24) | 78 (8) | 52 (9) |
| O(113) | 7083 (18) | 1150 (15) | 23 (5) | 46 (5) |
| C(114) | 6836 (28) | 1668 (26) | −299 (8) | 57 (9) |
| C(115) | 5417 (27) | 1587 (25) | −407 (8) | 54 (9) |
| O(116) | 4842 (17) | 2233 (15) | −142 (4) | 41 (5) |
| C(117) | 3485 (26) | 2010 (24) | −188 (7) | 45 (8) |
| C(118) | 2939 (28) | 2684 (24) | 53 (7) | 50 (9) |
| C(119) | 3028 (30) | 1377 (26) | 532 (8) | 63 (10) |
| C(120) | 3772 (27) | 972 (26) | 849 (7) | 52 (9) |
| O(121) | 5085 (18) | 1035 (15) | 801 (5) | 47 (5) |
| C(122) | 5871 (32) | 627 (31) | 1083 (9) | 82 (12) |
| C(123) | 7188 (30) | 856 (26) | 1002 (8) | 60 (10) |
| O(124) | 7355 (18) | 1981 (16) | 984 (4) | 46 (5) |
| C(125) | 8648 (26) | 2384 (24) | 1055 (7) | 44 (8) |
| C(126) | 9412 (27) | 2020 (24) | 765 (7) | 42 (8) |
| Na(2) | 5899 (10) | 5784 (9) | 2052 (3) | 45 (6) |
| N(21) | 3306 (22) | 5726 (20) | 2086 (6) | 49 (7) |
| C(22) | 2781 (33) | 6755 (27) | 1935 (9) | 75 (11) |
| C(23) | 3628 (36) | 7801 (32) | 2034 (11) | 96 (13) |
| O(24) | 4850 (18) | 7538 (15) | 1885 (5) | 48 (5) |
| C(25) | 4825 (27) | 7796 (25) | 1524 (7) | 53 (9) |
| C(26) | 6230 (27) | 7659 (25) | 1452 (8) | 53 (9) |
| O(27) | 6461 (18) | 6537 (16) | 1483 (5) | 54 (6) |
| C(28) | 7808 (28) | 6301 (26) | 1417 (8) | 55 (9) |
| C(29) | 8751 (31) | 6523 (27) | 1685 (8) | 69 (10) |
| N(210) | 8474 (22) | 5879 (20) | 2021 (6) | 50 (7) |
| C(211) | 9011 (30) | 6360 (26) | 2325 (8) | 64 (10) |
| C(212) | 8151 (26) | 7328 (23) | 2454 (7) | 48 (8) |
| O(213) | 6944 (18) | 6839 (16) | 2528 (5) | 55 (6) |
| C(214) | 7062 (29) | 6323 (26) | 2858 (8) | 59 (9) |
| C(215) | 5697 (32) | 5762 (29) | 2876 (9) | 76 (11) |
| O(216) | 5301 (21) | 5166 (18) | 2616 (5) | 15 (5) |
| C(217) | 4055 (30) | 4814 (27) | 2627 (8) | 68 (10) |
| C(218) | 3111 (32) | 5496 (27) | 2449 (8) | 70 (11) |
| C(219) | 2759 (30) | 4758 (25) | 1875 (8) | 59 (9) |
| C(220) | 3471 (32) | 4554 (29) | 1568 (8) | 69 (10) |
| O(221) | 4706 (35) | 4358 (31) | 1682 (9) | 148 (13) |
| C(222) | 5221 (42) | 3467 (38) | 1641 (12) | 114 (15) |
| C(223) | 6544 (32) | 3279 (29) | 1741 (9) | 74 (11) |
| O(224) | 6908 (25) | 3918 (21) | 2028 (7) | 94 (8) |
| C(225) | 8100 (36) | 3869 (32) | 2172 (10) | 94 (13) |
| C(226) | 8966 (30) | 4686 (25) | 1975 (8) | 62 (9) |

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* Lists of structure factor amplitudes, anisotropic thermal parameters, H-atom positions and bond lengths and bond angles for the crypt groups have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42394 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Coordination geometries of the cryptated* $Na^+$ *ions*

Bond lengths (Å)

| | $n = 1$ | $n = 2$ | | $n = 1$ | $n = 2$ |
|---|---|---|---|---|---|
| Na(*n*)–N(*n*1) | 2·79 (2) | 2·79 (3) | Na(*n*)–O(*n*13) | 2·61 (2) | 2·45 (2) |
| O(*n*4) | 2·55 (2) | 2·45 (2) | O(*n*16) | 2·48 (2) | 2·47 (2) |
| O(*n*7) | 2·68 (2) | 2·53 (2) | O(*n*21) | 2·56 (2) | 2·52 (4) |
| N(*n*10) | 3·06 (2) | 2·77 (3) | O(*n*24) | 2·58 (2) | 2·50 (3) |

Bond angles (°)

| | $n = 1$ | $n = 2$ |
|---|---|---|
| N(*n*1)–Na(*n*)–O(*n*4) | 66·0 (6) | 66·4 (7) |
| –O(*n*7) | 119·8 (7) | 111·5 (7) |
| –N(*n*10) | 177·8 (7) | 179·0 (8) |
| –O(*n*13) | 116·9 (7) | 111·5 (8) |
| –O(*n*16) | 62·6 (6) | 67·2 (7) |
| –O(*n*21) | 63·7 (6) | 63·6 (10) |
| –O(*n*24) | 120·0 (7) | 114·5 (9) |
| O(*n*4)–Na(*n*)–O(*n*7) | 65·1 (6) | 66·2 (7) |
| –N(*n*10) | 115·6 (6) | 112·7 (8) |
| –O(*n*13) | 160·5 (7) | 85·6 (7) |
| –O(*n*16) | 102·9 (7) | 110·4 (8) |
| –O(*n*21) | 100·7 (7) | 103·6 (10) |
| –O(*n*24) | 97·5 (7) | 162·5 (9) |
| O(*n*7)–Na(*n*)–N(*n*10) | 60·6 (6) | 68·4 (7) |
| –O(*n*13) | 99·5 (7) | 110·9 (8) |
| –O(*n*16) | 97·1 (6) | 176·4 (8) |
| –O(*n*21) | 157·6 (7) | 83·4 (10) |
| –O(*n*24) | 99·3 (6) | 98·8 (9) |
| N(*n*10)–Na(*n*)–O(*n*13) | 61·0 (6) | 67·7 (7) |
| –O(*n*16) | 115·3 (7) | 112·9 (8) |
| –O(*n*21) | 116·9 (7) | 117·3 (11) |
| –O(*n*24) | 61·7 (6) | 66·4 (8) |
| O(*n*13)–Na(*n*)–O(*n*16) | 65·6 (6) | 67·1 (7) |
| –O(*n*21) | 97·4 (7) | 165·3 (11) |
| –O(*n*24) | 96·9 (7) | 109·0 (8) |
| O(*n*16)–Na(*n*)–O(*n*21) | 103·2 (7) | 98·7 (10) |
| –O(*n*24) | 157·9 (7) | 84·8 (9) |
| O(*n*21)–Na(*n*)–O(*n*24) | 64·0 (6) | 64·3 (10) |

$Tl_2Te_2^{2-}$ (Burns & Corbett, 1981). The present analysis indicates, however, that the reaction of $NaTlTe_2$ alloy with 2,2,2-crypt results in a tetratelluride anion, $Te_4^{2-}$, along with cryptated $Na^+$ ions. Similarly the Zintl phase $K_4SnTe_4$ has been shown to react with tetraphenylphosphonium bromide in methanol to give $[Ph_4P]_2$-$[Te_4].2CH_3OH$ also containing the tetratelluride anion (Huffman & Haushalter, 1984) and the reaction of $BaBiSe_3$ with 2,2,2-crypt and en also gives a tetraselenide salt $[(2,2,2\text{-crypt})Ba][Se_4]$.en (König, Eisenmann & Schäfer, 1983). In both tetratelluride anions, as well as corresponding tetraselenide(2–) and tetrasulfide(2–) anions (König *et al.*, 1983; Tegman, 1973),* the end bonds are shorter than the middle bond. In the present salt, the anion has no crystallographic symmetry, the Te—Te distances are 2·702 (3) and 2·702 (3) Å (end) and 2·746 (3) Å (middle), and the Te—Te—Te bond angles are 109·7 (1) and 110·0 (1)° with a Te—Te—Te—Te dihedral angle of 105·3 (1)°.

These Te—Te bond lengths are significantly shorter than those in the tetraphenylphosphonium salt [2·724 (1) ×2 (end) and 2·762 (1) Å (middle)] (Huffman & Haushalter, 1984) but are comparable to the bond lengths observed for the pentatelluride anion in $[Bu_4N]_2[Te_5]$ [2·704 (1) ×2 (end) and 2·746 (1) Å ×2 (middle)] (Teller, Krause & Haushalter, 1983). For comparison, the Te—Te bond lengths in a tritelluride salt in a crypt structure are 2·692 (5) and 2·720 (4) Å (Cisar & Corbett, 1977). In both $[Ph_4P]_2$-$[Te_4].2CH_3OH$ and $[(2,2,2\text{-crypt})K]_2[Te_3]$.en, hydrogen bonding is considered to be responsible for some of the bond differences observed, although it is somewhat surprising that this should account for such significant lengthenings (*ca* 0·02 Å) in each case. Perhaps more significant is that the Te—Te—Te—Te dihedral angle of 99·9 (1)° in the $Ph_4P^+$ structure is considerably smaller than the present value of 105·3 (1)°. As noted by Hordvik (1966) there is a correlation between disulfide bond lengths and dihedral angles (presumably due to eclipsing of lone pairs on the chalogen atoms involved) and this may be responsible for the longer bond lengths in the $Ph_4P^+$ salt. Unfortunately the dihedral angles in the $Te_5^{2-}$ salt are not readily available.

The geometries of the cryptated $Na^+$ ions are normal with Na···O and Na···N distances [in the range 2·45 (2)–3·06 (2) Å] comparable to those observed in related salts including $[(2,2,2\text{-crypt})Na]_4[Sn_9]$ (2·47–3·30 Å; Corbett & Edwards, 1977), $[(2,2,2\text{-crypt})Na]_3$-$[Sb_7]$ [2·40 (2)–2·94 (3) Å; Adolphson, Corbett & Merryman, 1976], $[(2,2,2\text{-crypt})Na]_2[M_5]$ ($M$ = Sn or Pb) [2·43 (2)–3·43 (7) Å; Edwards & Corbett, 1977] and $[(2,2,2\text{-crypt})Na]I$ [2·57 (2)–2·78 (2) Å; Moras & Weiss, 1973]. There is some variability in the geometries of these cryptated Na moieties due to the fact that the $Na^+$ ion is somewhat undersized for the 2,2,2-crypt ligand (Moras & Weiss, 1973). Presumably this could also account for the different chemistry observed between the Na and K alloys, $NaTlTe_2$ and KTlTe. A view of the crystal packing is given in Fig. 1. The closest Te···H contacts are ≥2·96 Å in length (*cf.* the proposed Te···H hydrogen-bond distance of *ca* 2·8 Å in $[Ph_4P]_2[Te_4].2CH_3OH$).

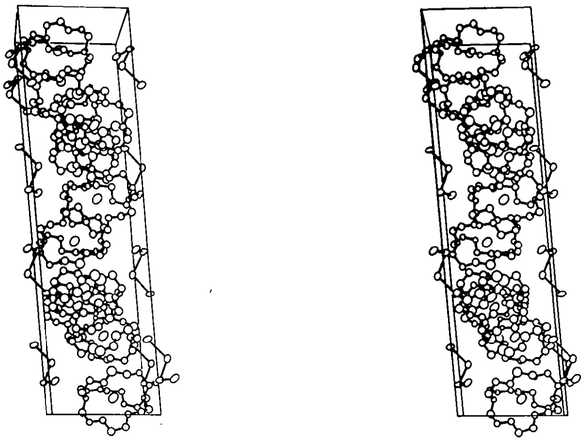

Fig. 1. *ORTEP* view (Johnson, 1965) of the crystal packing in $[(2,2,2\text{-crypt})Na]_2[Te_4]$.

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* Note, however, that in $BaS_4.H_2O$ the S—S bond lengths are 2·079 (3)–2·062 (3) Å with the bond alternation more significant in one $S_4^{2-}$ anion than in the other (Abrahams & Bernstein, 1969).

The Natural Sciences and Engineering Research Council of Canada is thanked for providing operating grants to GJS and an equipment grant for the diffractometer.

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*Acta Cryst.* (1985). C**41**, 1733–1736

# Structure of Diaquachloro(2,2′:6′,2″-terpyridyl)nickel(II) Chloride Monohydrate

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(*Received* 22 *May* 1985; *accepted* 28 *August* 1985)

**Abstract.** $[NiCl(C_{15}H_{11}N_3)(H_2O)_2]Cl.H_2O$, $M_r = 416{\cdot}9$, triclinic, $P\bar{1}$, $a = 6{\cdot}945\,(1)$, $b = 9{\cdot}6684\,(9)$, $c = 13{\cdot}632\,(2)$ Å, $\alpha = 81{\cdot}74\,(1)$, $\beta = 79{\cdot}37\,(1)$, $\gamma = 75{\cdot}73\,(1)°$, $V = 867{\cdot}2$ Å$^3$, $Z = 2$, $D_m = 1{\cdot}60\,(2)$, $D_x = 1{\cdot}60$ g cm$^{-3}$, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 14{\cdot}45$ cm$^{-1}$, $F(000) = 428$, room temperature, $R = 0{\cdot}026$ ($wR = 0{\cdot}030$) for 2309 observed reflections. The crystal consists of discrete monomeric molecules, linked by hydrogen bonds involving the water molecules and the Cl atoms. The Ni atom is hexacoordinated. Bond lengths and angles for the essentially planar terdentate ligand conform to the values given in the literature. Inter- and intramolecular hydrogen bonds are present in the compound. Distortions from an octahedron to a trigonal prism for different terpyridine metal complexes are calculated. The $\mu_{\text{eff}}$ (room temperature) and $g$ values (3·21 BM; 2·15) are in good agreement with octahedral stereochemistry for the Ni atom.

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0108-2701/85/121733-04$01.50