

Structure of the Triclinic Form of Hexakis[di- μ -(ethanethiolato)nickel], and Redetermination of its Monoclinic Form

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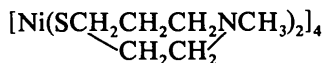
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Abstract. $[\text{Ni}(\text{SCH}_2\text{CH}_3)_2]_6$, $M_r = 1085.7$. Triclinic form: space group $P\bar{1}$, $a = 10.552$ (1), $b = 11.422$ (1), $c = 10.418$ (1) Å, $\alpha = 101.73$ (1), $\beta = 106.39$ (1), $\gamma = 105.39$ (1)°, $U = 1107.8$ (2) Å³, $D_x = 1.626$ Mg m⁻³, $Z = 1$, $F(000) = 564$, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 3.08$ mm⁻¹, $T = 298$ K, $R = 0.041$ for 3715 independent reflections. Monoclinic form: space group $P2_1/n$, $a = 12.312$ (1), $b = 16.048$ (6), $c = 11.690$ (1) Å, $\beta = 101.31$ (1)°, $U = 2264.8$ (9) Å³, $D_x = 1.591$ Mg m⁻³, $Z = 2$, $F(000) = 1128$, $\mu = 3.01$ mm⁻¹, $T = 298$ K, $R = 0.043$ for 4658 unique reflections. Each crystal is composed of $[\text{Ni}(\text{SCH}_2\text{CH}_3)_2]_6$ molecules with approximate D_{3d} symmetry. Six planar NiS_4 groups are linked by sharing an S...S edge to form a hexagonal prism. CH_3CH_2 - side chains occupy the axial and equatorial positions alternately with respect to the pseudo-hexagonal axis of the molecule. The major difference between the two crystal forms is that the rings stack parallel in the triclinic form, while they are packed with their planes at right angles to each other in the monoclinic form.

Introduction. From the viewpoint of structural inorganic chemistry, methyl-alkyl and aryl thiolates have been recognized as potential candidates for yielding a variety of multi-nuclear compounds due to the bridging disposition of thiolate ligands. The hexagonal cyclic molecule, $[\text{Ni}(\text{SEt})_2]_6$ (Et = $-\text{CH}_2\text{CH}_3$), was first characterized by Woodward, Dahl, Abel & Crosse (1965). The hexagonal homologue, $[\text{Ni}(\text{SCH}_2\text{CH}_2\text{OH})_2]_6$ (Gould & Harding, 1970) and the tetragonal and octagonal analogues,



(Gaete, Ros, Solans, Font-Altaba & Brianso, 1984) and $[\text{Ni}(\text{SCH}_2\text{CH}_2\text{COOEt})_2]_8$ (Dance, Scudder & Secomb,

1985), form a novel group on account of their crown-shaped ring structures. They are called 'tiara' compounds.

In spite of its starting point in the 'tiara' group, few details have been published of $[\text{Ni}(\text{SEt})_2]_6$, since the compound was found by accident (Abel, Brady & Crosse, 1966) or was obtained by a detouring (Abel & Crosse, 1966) synthesis. Only a short structural communication has been reported (Woodward *et al.*, 1965).

In the course of our studies on Ni-thiolate chemistry, we have published a new method for the controlled synthesis of this compound (Yamamura, Miyamae, Katayama & Sasaki, 1985). This paper presents the results of X-ray analyses of two crystal forms: monoclinic and triclinic.

Experimental. The preparation and other properties of the compound are given elsewhere (Yamamura, 1988). The title compound crystallizes in two forms, triclinic and monoclinic, at 253 K from an acetone solution of the reaction mixture $\{[\text{Ni}_3(\text{SEt})_8]^{2-} + \text{trace amount of water}\}$. X-ray data were collected for both forms at room temperature. Each crystal was sealed in a Lindemann-glass capillary under argon.

Triclinic form: Crystal dimensions ca $0.3 \times 0.2 \times 0.2$ mm. 6787 reflections ($2\theta \leq 60^\circ$, h 0→14, k -16→16, l -14→14) were measured, of which 3715 unique reflections with $|F| \geq 3\sigma(|F|)$ were considered 'observed'. θ - 2θ scan mode with scan range $(1.1 + 0.5 \tan \theta)^\circ$, Rigaku AFC-5 four-circle diffractometer, graphite-monochromated Mo $K\alpha$. Unit-cell dimensions refined from least-squares fit of 35 2θ values of independent reflections ($28.5 < 2\theta < 31.0^\circ$). Corrections applied for Lorentz and polarization effects, but not for absorption and extinction.

Monoclinic form: Crystal dimensions ca $0.3 \times 0.2 \times 0.2$ mm. 6877 reflections ($2\theta \leq 60^\circ$; h -17→17, k 0→22, l 0→16) were measured, of which 4658 unique

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reflections were considered 'observed'. Other conditions were the same as mentioned for the triclinic form. Unit-cell dimensions refined from 21 2θ values of independent reflections ($28.5 < 2\theta < 31.5^\circ$).

Three standard reflections monitored every 100 measurements, no significant decay ($\Delta < 0.7\%$ for both crystals).

The structures were solved by the heavy-atom method and refined by block-diagonal least squares using the UNICSIII program system (Sakurai & Kobayashi, 1979) on the FACOM M-360R and M-360 computer of Josai University. $\sum w(|F_o|^2 - |F_c|^2) / \sum |F_o|^2$ minimized with $w = 1.0$. H atoms located from difference maps. Complex neutral-atom scattering factors (*International Tables for X-ray Crystallography*, 1974). Final $R = 0.041$, $wR = 0.041$ with $S = 1.404$ for triclinic, $R = 0.043$, $wR = 0.044$ with $S = 3.657$ for monoclinic. Final Δ/σ values for non-H atoms are 0.27 and 0.54, $\Delta\rho_{\max} = 0.75$ and 0.63 e \AA^{-3} for triclinic and monoclinic forms, respectively.

The final atomic parameters with e.s.d.'s for non-H atoms are listed in Table 1. The atom-numbering scheme is given in Fig. 1.*

Discussion. The molecular structure is shown in Fig. 1. Bond distances and angles are compared in Table 2.

The center of the hexameric molecule occupies the crystallographic center of symmetry in both forms. The NiS_4 units, almost ideally coplanar, share S...S edges to form a hexagonal prism of approximate D_{6h} symmetry. Six Ni atoms in the prism lie on a plane within ± 0.0002 (7) \AA , and a pair of hexagons of S atoms forming the top and bottom surfaces of the prism lie in planes *ca* 1.44 (2) \AA above and below the Ni_6 hexagon. The Ni_6 hexagons of the triclinic and monoclinic forms are slightly different from each other in size; in the latter each Ni atom is 2.923 (1) \AA apart from its nearest neighbors and the center of the hexagon, while in the former the distance is 2.931 (1) \AA . Thus the sizes of the vacant holes of the 'tiaras' are 2.60 and 2.59 \AA in diameter for the triclinic and monoclinic forms, respectively, assuming a van der Waals radius of 1.63 \AA for Ni (Bondi, 1964).

The coordination geometry around the Ni atom is strongly distorted from square-planar to rectangular; the hexagonal prism is compressed along the pseudo-hexagonal axis. In the monoclinic crystal, the distance between adjacent S atoms in the S_6 planes is 3.318 (2) \AA , while the distance between the two S_6

Table 1. Atomic positional parameters ($\times 10^4$) and equivalent isotropic thermal parameters (\AA^2)

$$B_{\text{eq}} = \frac{1}{3} \sum_i \sum_j \beta_{ij} (\mathbf{a}_i \cdot \mathbf{a}_j).$$

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq}
Monoclinic form				
Ni(1)	-1411 (1)	1000 (1)	1198 (1)	3.54 (2)
Ni(2)	728 (1)	407 (1)	2475 (1)	3.80 (2)
Ni(3)	2144 (1)	-600 (1)	1260 (1)	3.62 (2)
S(1)	-2884 (1)	339 (1)	265 (1)	4.17 (4)
S(2)	-1749 (1)	1804 (1)	-360 (1)	4.28 (5)
S(3)	-978 (1)	143 (1)	2699 (1)	4.87 (5)
S(4)	56 (1)	1665 (1)	2133 (1)	4.24 (4)
S(5)	1404 (1)	-857 (1)	2786 (1)	4.43 (5)
S(6)	2375 (1)	647 (1)	2074 (1)	4.22 (4)
C(11)	-2927 (6)	-772 (5)	615 (6)	6.05 (23)
C(12)	-3770 (9)	-962 (7)	1295 (10)	10.88 (45)
C(21)	-3058 (6)	2321 (5)	-324 (7)	6.26 (24)
C(22)	-2952 (8)	2983 (6)	610 (8)	8.37 (34)
C(31)	-1234 (6)	737 (6)	3943 (5)	6.99 (27)
C(32)	-2451 (8)	965 (10)	3819 (8)	13.90 (60)
C(41)	769 (5)	2273 (5)	1204 (6)	5.58 (21)
C(42)	468 (7)	3186 (5)	1238 (9)	7.79 (31)
C(51)	334 (7)	-1661 (5)	2577 (7)	6.75 (26)
C(52)	718 (10)	-2416 (6)	3237 (11)	10.80 (47)
C(61)	3400 (5)	518 (5)	3432 (6)	5.68 (21)
C(62)	4523 (6)	401 (5)	3208 (7)	6.90 (27)
Triclinic form				
Ni(1)	532 (1)	-830 (1)	2566 (1)	3.06 (2)
Ni(2)	1962 (1)	1772 (1)	2739 (1)	2.94 (2)
Ni(3)	1436 (1)	2593 (1)	203 (1)	3.09 (2)
S(1)	490 (1)	-2636 (1)	1293 (1)	3.51 (4)
S(2)	-1758 (1)	-1834 (1)	1759 (1)	3.55 (4)
S(3)	2801 (1)	261 (1)	3214 (1)	3.57 (4)
S(4)	545 (1)	985 (1)	3779 (1)	3.20 (4)
S(5)	3362 (1)	2542 (1)	1671 (1)	3.68 (4)
S(6)	1025 (1)	3185 (1)	2157 (1)	3.62 (4)
C(11)	1912 (5)	-2452 (5)	611 (5)	4.27 (16)
C(12)	1986 (7)	-3739 (6)	-16 (7)	6.34 (20)
C(21)	-2113 (5)	-3182 (5)	2448 (5)	4.13 (16)
C(22)	-3598 (6)	-4091 (6)	1669 (6)	5.60 (27)
C(31)	3567 (6)	752 (5)	5138 (5)	4.55 (16)
C(32)	4833 (6)	1950 (6)	5710 (7)	6.31 (23)
C(41)	-1129 (5)	1237 (5)	3256 (5)	4.16 (18)
C(42)	-1132 (6)	2331 (6)	4359 (7)	5.60 (23)
C(51)	4022 (5)	1367 (6)	873 (6)	4.97 (20)
C(52)	5473 (9)	1554 (11)	1791 (9)	6.86 (39)
C(52')	5286 (23)	1932 (23)	598 (27)	5.16 (89)
C(61)	2357 (7)	4753 (5)	3206 (5)	4.97 (20)
C(62)	2725 (9)	4944 (6)	4743 (6)	7.17 (28)

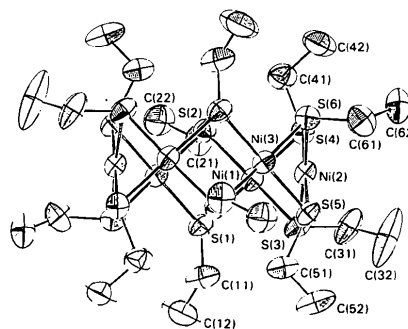


Fig. 1. ORTEP (Johnson, 1976) drawing of the molecule in the triclinic form with atom numbering; thermal ellipsoids are at 50% probability level.

planes is 2.892 (2) \AA . In the triclinic form these values are 3.327 (2) and 2.889 (2) \AA , respectively.

The $-\text{CH}_2\text{CH}_3$ side chains on the six coplanar S atoms occupy the axial and equatorial positions alternately. Thus, the symmetry falls from D_{6h} of the Ni-S framework to approximate D_{3d} . The axial-

* Observed and calculated structure amplitudes, anisotropic thermal parameters of non-H atoms, atomic parameters for H atoms, bond distances and angles, and the least-squares planes around the coordination plane of each Ni atom have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44572 (51 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond distances (\AA) and angles ($^\circ$) within the hexamer

	Monoclinic	Triclinic
Ni(1)...Ni(2)	2.920 (1)	2.897 (1)
...Ni(3)	2.910 (1)	2.958 (1)
Ni(2)...Ni(3)	2.940 (1)	2.938 (1)
S(1)...S(2)	2.900 (2)	2.884 (2)
S(3)...S(4)	2.890 (3)	2.886 (2)
S(5)...S(6)	2.887 (2)	2.897 (2)
Ni(1)-S(1)	2.198 (2)	2.196 (1)
-S(2)	2.203 (2)	2.204 (1)
-S(3)	2.210 (2)	2.216 (1)
-S(4)	2.195 (2)	2.192 (1)
Ni(2)-S(3)	2.208 (2)	2.218 (2)
-S(4)	2.188 (2)	2.192 (1)
-S(5)	2.197 (2)	2.198 (2)
-S(6)	2.202 (2)	2.205 (2)
Ni(3)-S(5)	2.195 (2)	2.197 (1)
-S(6)	2.210 (2)	2.210 (2)
-S(1)	2.196 (2)	2.207 (1)
-S(2)	2.207 (2)	2.207 (1)
S(1)-Ni(1)-S(2)	82.44 (6)	81.92 (5)
-S(3)	98.07 (7)	98.59 (5)
-S(4)	179.72 (7)	178.29 (6)
S(2)-S(3)	175.61 (7)	174.59 (6)
-S(4)	97.53 (7)	97.53 (5)
S(3)-S(4)	81.98 (7)	81.80 (5)
S(3)-Ni(2)-S(4)	82.18 (7)	81.75 (5)
-S(5)	98.15 (7)	98.24 (6)
-S(6)	174.51 (6)	176.55 (4)
S(4)-S(5)	178.99 (7)	179.25 (4)
-S(6)	97.57 (6)	97.67 (6)
S(5)-S(6)	82.02 (7)	82.29 (6)
S(5)-Ni(3)-S(6)	81.88 (7)	82.20 (5)
-S(1)	179.80 (7)	179.29 (6)
-S(2)	97.80 (7)	97.69 (6)
S(6)-S(1)	97.91 (7)	98.52 (6)
-S(2)	174.31 (6)	175.16 (5)
S(1)-S(2)	82.40 (7)	81.60 (5)
Ni(1)-S(1)-Ni(3)	82.40 (7)	84.40 (5)
-S(2)	82.58 (6)	84.24 (5)
Ni(1)-S(3)-Ni(2)	82.73 (6)	81.59 (5)
-S(4)	83.60 (6)	82.71 (5)
Ni(2)-S(5)-Ni(3)	84.07 (6)	83.91 (5)
-S(6)	83.60 (6)	83.45 (6)
Ni(1)-S(1)-C(11)	114.6 (2)	113.3 (2)
Ni(3)-S(1)	113.5 (3)	112.2 (2)
Ni(1)-S(2)-C(21)	105.8 (3)	108.4 (2)
Ni(3)-S(2)	107.3 (2)	104.5 (2)
Ni(1)-S(3)-C(31)	104.9 (3)	108.3 (2)
Ni(2)-S(3)	108.1 (2)	104.4 (2)
Ni(1)-S(4)-C(41)	114.3 (2)	114.3 (2)
Ni(2)-S(4)	113.2 (2)	114.2 (2)
Ni(2)-S(5)-C(51)	112.9 (3)	112.6 (2)
Ni(3)-S(5)	115.3 (3)	112.6 (2)
Ni(2)-S(6)-C(61)	107.5 (2)	105.5 (2)
Ni(3)-S(6)	106.1 (2)	107.0 (2)

Symmetry operation for both crystal forms: (i) $-x, -y, -z$.

equatorial alternation is the same in all mercapto-metal hexamers (Gould & Harding, 1970; Kunchur, 1968, 1971), tetramers (Gaete *et al.*, 1984) and octamers (Dance *et al.*, 1985).

The Ni-S-C bond angles for the equatorial conformation are in the range *ca* 104–109° [mean value: 106.3 (2)° for triclinic and 106.6 (2)° for monoclinic], while angles for the axial conformation are *ca* 112–115° [mean value: 113.2 (2)° for triclinic and 114.0 (3)° for monoclinic]. This implies that the lone

pair of the S atom which takes the equatorial conformation inclines towards the outside of the hexameric ring. A difference is also seen in the Ni-S bond lengths. The Ni-S bond distances of equatorial -SEt are longer than those observed for axial -SEt. The mean axial Ni-S bond distances are 2.197 (1) and 2.195 (2) \AA , for triclinic and monoclinic respectively. These are 2.207 (2) and 2.210 (2) \AA in the equatorial -SEt.

It should be significant to compare the structure of the Ni_2S_2 portion of this compound with the framework of $[\text{Ni}_2(\text{SEt})_6]^{2-}$ (Watson, Pulla Rao, Dorfman & Holm, 1985). The latter is formed by edge-sharing of two planar NiS_4 units, having in the center an Ni_2S_2 (*b*: bridged) planar rhomb. With respect to this plane a pair of bridging thiolates occupy 'anti' (Watson *et al.*, 1985; Cotton & Wilkinson, 1980) positions. In contrast, Ni_2S_2 of $[\text{Ni}(\text{SET})_2]_6$ is forced to bend along $\text{Ni}\cdots\text{Ni}$ so as to make a dihedral angle of 61.56 (6)° between the two NiS_4 planes. The anti configuration of $[\text{Ni}_2(\text{SEt})_6]^{2-}$ is transferred to the axial-equatorial S-C bond directions of the hexamer. As for a comparison of structural parameters, the Ni-S_b bond length of $[\text{Ni}_2(\text{SEt})_6]^{2-}$ [2.220 (2) \AA] is longer than any of those observed for the Ni-S of the hexamer. The bond angles of the dimer, Ni-S_b-Ni, are 98.2°, which are much larger than those of the hexamer [83.38 (5)° triclinic; 83.16 (6)° monoclinic]. On the other hand, the S-Ni-S bond angles of the hexamer, which are 81.93 (5) and 82.15 (7)° for the triclinic and monoclinic forms respectively, are comparable to the S_b-Ni-S_b angle of

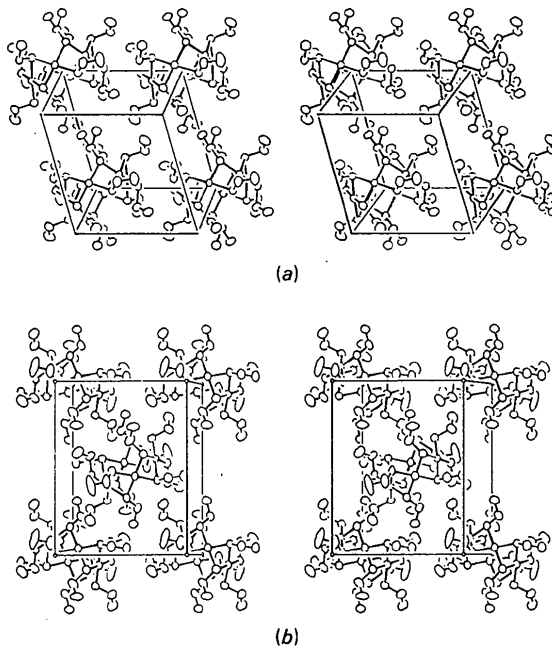


Fig. 2. Stereoscopic views of crystal packing. (a) Triclinic form viewed along $-b^*$; (b) monoclinic form viewed along $-c^*$.

the dimer $[81.8(1)^\circ]$. Consequently, the bent structure of the Ni_2S_2 unit in $[\text{Ni}(\text{SET})_2]_6$ is contracted and collapsed to its inside hole. The resulting short $\text{Ni}\cdots\text{Ni}$ distances, 2.931 (1) (triclinic) and 2.923 (1) Å (monoclinic), should be compared with the distance of 3.356 (2) Å in $[\text{Ni}_2(\text{SET})_6]^{2-}$.

The packing modes of the hexamers in the crystals are depicted in Fig. 2. The hexamer rings stack parallel in the triclinic form while they are packed so that the pseudo-hexagonal axis of the molecule is almost perpendicular with each in the monoclinic form. The molecular planes incline at *ca* 40° with respect to the 2_1 axis.

One of the axial methyl groups in the triclinic form, which is directed towards the center of the adjacent ring, is allowed to be situated in two sites (Fig. 2a). The methyl group restraint by the adjacent hexamer in the triclinic crystal does not seem to be enough to fix the alkyl configuration, presumably owing to the large vacancy of the tiara ring. Nevertheless, the density of the triclinic crystal is slightly larger than that of the monoclinic one.

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Structure of Potassium (Rubidium) Trihydrogen Diglutarate

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Abstract. $\text{KH}_3(\text{C}_5\text{H}_6\text{O}_4)_2$, $M_r = 302.3$, monoclinic, $P2_1/a$, $a = 9.392(2)$, $b = 12.782(1)$, $c = 11.147(2)$ Å, $\beta = 93.34(2)^\circ$, $V = 1335.9$ Å³, $Z = 4$, $D_m = 1.46$, $D_x = 1.50$ g cm⁻³, $\lambda(\text{Cu K}\alpha) = 1.5418$ Å, $\mu = 38.4$ cm⁻¹, $F(000) = 632$, $T = 291$ K, final $R = 0.049$ for 1866 independent observed reflections. $\text{RbH}_3(\text{C}_5\text{H}_6\text{O}_4)_2$, $M_r = 348.7$, monoclinic, $P2_1/a$, $a = 9.232$, $b = 13.306$, $c = 11.134$ Å, $\beta = 94.4^\circ$, $V = 1363.7$ Å³, $Z = 4$, $D_m = 1.64$, $D_x = 1.698$ g cm⁻³, $F(000) = 704$. The potassium and rubidium salts are isomorphous. Isomorphism of the two salts was established photographically and, as the structure of the K salt was easily determined, that of the Rb salt was not investigated.

HY^- anions ($\text{H}_2\text{Y} = \text{glutaric acid}$) are linked into infinite spiral chains by short asymmetric hydrogen bonds [$\text{O}\cdots\text{O} = 2.506(4)$ Å]. These chains are cross-linked through longer hydrogen bonds [$\text{O}\cdots\text{O} = 2.553(3)$ and $2.641(4)$ Å] to glutaric acid molecules. K^+ is eight-coordinate with $\text{K}^+\cdots\text{O}$ in the range $2.727\text{--}3.010(3)$ Å.

Introduction. In addition to their normal acid salts (MHY), dicarboxylic acids (H_2Y) form 'super-acid' salts of general formula (MHYH_2Y) where M is a monovalent cation (Marshall & Cameron, 1907). The crystal structures of two such salts of formula $\text{KH}_3(\text{CH}_2)_n(\text{CO}_2)_2$, $n = 1, 2$ have been determined using neutron diffraction data (Currie, 1972; Dunlop &

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