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Structure of an $Ni(S_3C_4N_2)_2(NBu_4)_2$ Salt

BY D. MENTZAFOS

Physics Laboratory, Agricultural University of Athens, Iera Odos 75, 118 55 *Athens, Greece*

AND A. TERZIS*

NRCPS 'Democritos', Institute of Materials Science, 153 10 *Agia Paraskevi Attikis, Greece*

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Abstract. Tetrabutylammonium bis(5-cyano-3,4-dimercaptoisothiazole)nickelate, $[N(C_4H_9)_4]_2[Ni(C_4N_2S_3)_2]$, $M_r = 888{\cdot}15$, triclinic, $P\bar{1}$, $a = 10{\cdot}027\ (1)$, $b = 10{\cdot}177\ (1)$, $c = 13{\cdot}192\ (1)$ Å, $\alpha = 107{\cdot}098\ (8)$, $\beta = 108{\cdot}968\ (8)$, $\gamma = 75{\cdot}715\ (8)°$, $V = 1199{\cdot}6\ (2)$ Å^3, $Z = 1$, $D_m = 1{\cdot}24$, $D_x = 1{\cdot}229$ Mg m^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu = 3{\cdot}127$ mm^{-1}, $F(000) = 478$, $T = 295\ (2)$ K, final $R = 0{\cdot}0374$ for 3672 observed reflections with $F_o > 4{\cdot}5\sigma(F_o)$. The anions are stacked in the **a** direction but there is no metal–metal contact. There are no S···S or S···N intrastack or interstack contacts. All atoms show a significant thermal motion, which in NBu_4^+ progressively increases towards the end of the butyl chains.

Experimental. Crystals were provided by Dr Papavassiliou (Papavassiliou, Mousdis, Gionis, Zambounis & Yiannopoulos, 1987). Crystal dimensions $0{\cdot}12 \times 0{\cdot}45 \times 0{\cdot}22$ mm. Density measured by flotation. Syntex $P2_1$ diffractometer. Lattice parameters from 15 reflections ($51 < 2\theta < 54°$). Ni-filtered Cu radiation, $\theta/2\theta$ scan. Data in range $2\theta \leq 130°$. Range of hkl: $-11 \rightarrow 11$, $0 \rightarrow 11$, $-15 \rightarrow 15$. Scan speed 2·0–15·0 $(2\theta)°$ min^{-1}, scan width $1{\cdot}8°$ (2θ) plus α_1–α_2 divergence. Three standard reflections monitored every 67 reflections showed a systematic decrease in their intensities of 0·11% per hour of exposure. A correction for this, as well as Lp and numerical absorption correction ($T_{max}/T_{min} = 0{\cdot}7116/0{\cdot}4464$), was applied (Sheldrick, 1976). Data collected/unique/R_{int}, 4556/4082/0·0147. Positional coordinates for Ni were deduced from a Patterson synthesis. Subsequent difference Fourier synthesis revealed the positions of all the other non-hydrogen atoms. The structure was refined with *SHELX*76 (Sheldrick, 1976) in $P\bar{1}$ by full-matrix least squares, in which $\sum w\Delta F^2$ was minimized. Attempts to refine the structure in $P1$ were unsuccessful, leading to negative U's and very bad bond distances. All non-H atoms refined using anisotropic temperature factors. H atoms (calculated) isotropic riding at 0·98 Å. Unit weights. Final refinement $wR = 0{\cdot}0358$ for observed data. Eleven reflections showing poor agreement were given zero weight during final refinement cycles. R/wR: 0·0411/0·0477 for all data. $S = 0{\cdot}50$. $|\Delta/\sigma|_{max} = 0{\cdot}090$. Number of refined parameters 289. $(\Delta\rho)_{max}/(\Delta\rho)_{min}$: 0·530/−0·284 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The final atomic parameters of the non-H atoms are given in Table 1.†

Table 1. *Positional and equivalent isotropic thermal parameters* ($\times 10^4$) *of the non*-H *atoms with e.s.d.'s in parentheses*

$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$

	x	y	z	U_{eq}(Å^2)
Ni	0	0	0	549
S(1)	−293·0 (8)	1785·6 (6)	−703·2 (5)	652
S(2)	871·0 (8)	−1460·7 (6)	−1314·6 (5)	725
S(3)	1129·5 (8)	773·3 (7)	−3490·8 (6)	835
C(1)	390 (2)	1074 (2)	−1835 (2)	575
C(2)	894 (2)	−406 (2)	−2117 (2)	556
C(3)	1347 (3)	−724 (2)	−3051 (2)	646
C(4)	1986 (3)	−2036 (3)	−3592 (2)	737
N(1)	464 (2)	1804 (2)	−2482 (2)	746
N(2)	2525 (3)	−3070 (3)	−4031 (2)	962
N	3048 (2)	4139 (2)	2162 (2)	557
C(11)	2220 (2)	4419 (2)	1034 (2)	552
C(12)	3079 (3)	4625 (3)	354 (2)	680
C(13)	2122 (3)	4737 (3)	−783 (2)	678
C(14)	2904 (3)	4914 (3)	−1526 (2)	931
C(21)	1935 (2)	4018 (2)	2669 (2)	624
C(22)	2500 (3)	3666 (3)	3786 (2)	707
C(23)	1275 (3)	3591 (3)	4178 (2)	893
C(24)	1745 (3)	3245 (3)	5286 (2)	1037
C(31)	3889 (2)	5298 (2)	2878 (2)	611
C(32)	3013 (3)	6736 (2)	3102 (2)	679
C(33)	3958 (3)	7808 (3)	3818 (3)	904
C(34)	3106 (4)	9271 (3)	4018 (3)	1174
C(41)	4143 (2)	2809 (2)	2068 (2)	654
C(42)	3586 (3)	1476 (3)	1419 (3)	834
C(43)	4790 (3)	237 (3)	1613 (3)	1149
C(44)	4402 (4)	−1102 (4)	1084 (4)	1614

† Lists of observed and calculated structure factors, anisotropic thermal parameters of the non-H atoms and the bond lengths and angles of the cation have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51599 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

* To whom all correspondence should be addressed.

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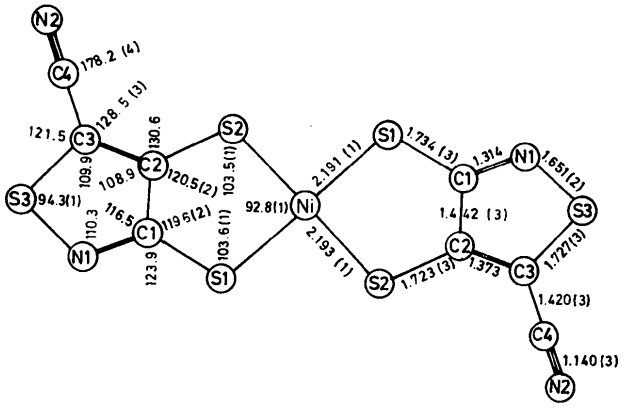

Fig. 1. The atom-numbering scheme of the anion with bond lengths and angles. Where no e.s.d.'s appear they are 0·004 Å for distances and 0·2° for angles.

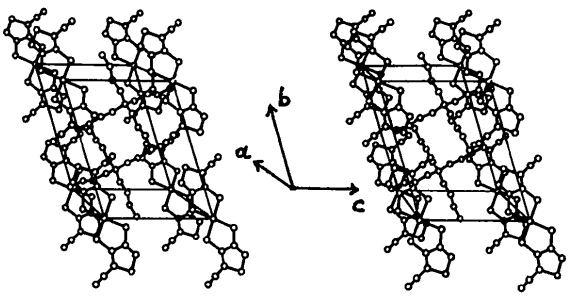

Fig. 2. An *ORTEP* stereoview of the molecular packing.

atom-numbering scheme of the anion with bond lengths and angles is given in Fig. 1 and an *ORTEP* (Johnson, 1976) stereoview of the molecular packing in Fig. 2.

Related literature. The Ni atom lies at the origin, as in the case of $Ni(S_5C_3)_2NBu_4$ (Mentzafos, Hountas & Terzis 1988), but in the present structure Ni is in formal oxidation state +2. As a result the Ni–S bonds are slightly longer in the present structure, but comparable to those observed in bis(isotrithione-dithiolate)-nickelate (Lindqvist, Sjölin, Sieler, Steimecke & Hoyer, 1982) where Ni is in the same formal oxidation state of +2.

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Bis(*N*-*n*-propylsalicylideneaminato)nickel(II)

BY DOYLE BRITTON AND LOUIS H. PIGNOLET

Department of Chemistry, University of Minnesota, Minneapolis, MN 55455, *USA*

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Abstract. $[Ni(C_{10}H_{12}NO)_2]$, $M_r = 383{\cdot}14$, monoclinic, $P2_1/c$, $a = 10{\cdot}026$ (2), $b = 10{\cdot}067$ (3), $c = 9{\cdot}167$ (2) Å, $\beta = 100{\cdot}26$ (3)°, $V/Z = 455{\cdot}3$ (4) Å^3, $Z = 2$, $D_x = 1{\cdot}397$ (1) g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71073$ Å, $\mu = 10{\cdot}8$ cm^{-1}, $F(000) = 404$, $T = 297$ (2) K, $R = 0{\cdot}030$ for 1048 reflections. The geometry around the Ni atom is square planar, and, except for the propyl groups, the entire molecule is approximately planar; the plane of the benzene ring in the ligand is tilted 5·4° with respect to the NiN_2O_2 plane. Bond lengths and angles are normal.

Experimental. The title compound was prepared by the method of Sacconi, Paoletti & Del Re (1957). It was characterized by its melting point (436–437 K), absorption spectra and magnetic properties (Holm, 1961). Plate-shaped crystals suitable for X-ray diffraction were present in the sample. The crystal used measured 0·08 × 0·25 × 0·30 mm. Data were collected on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator. 25 reflections with $10 < \theta < 15°$ were used to determine the cell parameters. Systematic extinctions ($0k0$, k odd; $h0l$, l odd) uniquely determined the space group. Data were collected, using ω–2θ scans, in the range $0 < \theta < 24°$ for one quadrant (ranges: h 0 to 11, k 0 to 11, l −10 to 10). Empirical absorption corrections were applied; correction factors ranged from 0·908 to 1·000. 1431 unique reflections

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