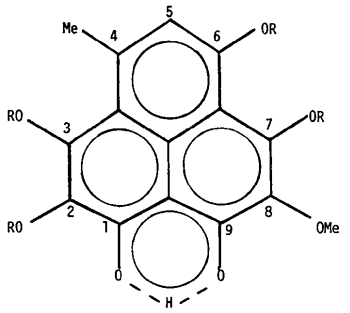
1957). The general structure of this type of molecule has been established by other studies (*e.g.* Barton, de Mayo, Morrison & Raistrick, 1959; Paul & Sim, 1965; Svensson, Abrahams, Bernstein & Haddon, 1979).



(I) $R = COCH_3$

The author thanks the Natural Sciences and Engineering Research Council of Canada for financial support, Professor Sir Derek Barton and Dr W. H. Schaeppi for crystals, and the late Professor J. Monteath Robertson for his guidance and support.

References

BARTON, D. H. R., DE MAYO, P., MORRISON, G. A. & RAISTRICK, H. (1959). *Tetrahedron*, **6**, 48–62.
HAMILTON, W. C. & IBERS, J. A. (1968). *Hydrogen Bonding in Solids*, pp. 99, 178–181. New York: Benjamin.
Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200A Research Forest Drive, The Woodlands, TX 77831, USA.
NEILL, K. G. & RAISTRICK, H. (1956). *Chem. Ind.* pp. 551–552.
PAUL, I. C. & SIM, G. A. (1965). *J. Chem. Soc.* pp. 1097–1112.
SVENSSON, C., ABRAHAMS, S. C., BERNSTEIN, J. L. & HADDON, R. C. (1979). *J. Am. Chem. Soc.* **101**, 5759–5764.
TROTTER, J. (1957). Unpublished.

Acta Cryst. (1992). C**48**, 942–943

Structure of $[Ni(OH_2)_2(NH_2CH_2CH_2CH_2NH_2)_2][picrate]_2$

BY WILLIAM CLEGG

Department of Chemistry, The University, Newcastle upon Tyne NE1 7RU, England

AND PARIMAL K. BHARADWAJ AND SUBRATA MANDAL

Department of Chemistry, Indian Institute of Technology Kanpur, PO–IIT, Kanpur-208016, *India*

(*Received* 16 *September* 1991; *accepted* 22 *October* 1991)

Abstract. Diaquabis(1,3-diaminopropane)nickel(II) picrate, $C_6H_{24}N_4NiO_2^{2+}.2C_6H_2N_3O_7^-$, $M_r = 699.2$, monoclinic, $I2/a$ (non-standard setting of $C2/c$), $a = 16.2880$ (6), $b = 7.5741$ (3), $c = 22.6836$ (8) Å, $\beta = 96.606$ (4)°, $V = 2779.8$ Å^3, $Z = 4$, $D_m = 1.69$ (1), $D_x = 1.670$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54184 Å, $\mu = 1.82$ mm^{-1}, $F(000) = 1448$, $T = 295$ K, $R = 0.0699$ for 2300 unique observed reflections. The structure contains discrete $[Ni(OH_2)_2(NH_2CH_2CH_2CH_2NH_2)_2]^{2+}$ cations and picrate anions, which are hydrogen bonded to the aqua ligands [O···O 2.670 (6) Å]. The Ni atom lies on a crystallographic centre of inversion; octahedral coordination around it is effected by two water and two bidentate 1,3-diaminopropane molecules to give a *trans*-N_4O_2 donor set. The Ni—O [2.126 (2) Å] and Ni—N [2.109 (2) and 2.101 (2) Å] distances are within normal ranges, and *cis* coordination angles range from 88.9 (1) to 91.2 (1)°. The six-membered chelate rings have a chair conformation.

Experimental. The compound was prepared by reaction of 0.51 g (1 mmol) nickel picrate (Black & McLean, 1971) with 0.15 g (2 mmol) of freshly distilled 1,3-diaminopropane in 25 ml acetonitrile. An orange solution was obtained, which upon slow evaporation at room temperature afforded well formed orange crystals. The density was measured by flotation. A crystal of size 0.5 × 0.5 × 0.5 mm, on a glass fibre, was examined on a Stoe–Siemens diffractometer. Unit-cell parameters were refined from 2θ values of 32 reflections (45–50°) measured at $\pm\omega$. Data collection employed ω/θ scans and on-line profile fitting (Clegg, 1981), to a maximum 2θ of 130°; index ranges were h −19→19, k 0→8, l 0→26, together with a partial set of equivalent reflections. No significant variation was observed in the intensities of three standard reflections. Semi-empirical absorption corrections were applied, with transmission factors in the range 0.261–0.392. The 3557 measured reflections yielded 2335 unique data, 2300 with $F > 4\sigma_c(F)$ (σ_c from counting statistics only); $R_{int} = 0.042$.

The structure was solved by direct methods, and blocked-cascade least-squares refinement on F, with weighting $w^{-1} = \sigma^2(F) = \sigma_c^2(F) + 54 - 229G + 857G^2$

0108-2701/92/050942-02$06.00

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^4$)

$U_{eq} = \frac{1}{3}$(trace of the orthogonalized U_{ij} matrix).

	x	y	z	U_{eq}
Ni	0	0	5000	370 (1)
O(1)	1096 (1)	1548 (2)	5085.6 (7)	645 (5)
N(1)	−295 (1)	992 (2)	5818.4 (7)	443 (4)
N(2)	−625 (1)	2103 (2)	4540.4 (7)	559 (6)
C(1)	−1077 (1)	1974 (3)	5848 (1)	570 (7)
C(2)	−1187 (2)	3460 (3)	5409 (1)	710 (8)
C(3)	−1347 (2)	2919 (3)	4765 (1)	722 (9)
C(4)	1720 (1)	1231 (2)	1583.2 (9)	406 (5)
C(5)	2615 (1)	1340 (2)	1670.3 (8)	402 (6)
C(6)	3091 (1)	675 (2)	2168.5 (8)	398 (5)
C(7)	2700 (1)	−172 (2)	2595.9 (7)	405 (5)
C(8)	1854 (1)	−364 (2)	2543.3 (8)	404 (6)
C(9)	1386 (1)	293 (2)	2044.7 (9)	439 (6)
O(2)	1290 (1)	1943 (2)	1156.3 (7)	680 (5)
N(3)	3055 (1)	2237 (2)	1245.8 (8)	473 (5)
O(31)	2748 (1)	2332 (3)	727.4 (7)	690 (5)
O(32)	3740 (1)	2868 (2)	1406.2 (9)	724 (6)
N(4)	3183 (1)	−842 (2)	3124.9 (8)	489 (5)
O(41)	3927 (1)	−751 (2)	3161.6 (8)	708 (6)
O(42)	2812 (1)	−1481 (3)	3515.1 (7)	807 (6)
N(5)	509 (1)	2 (3)	2016.5 (9)	572 (5)
O(51)	215 (1)	−609 (5)	2441 (1)	1188 (15)
O(52)	96 (1)	42 (5)	1532 (1)	1212 (9)

$-96H + 55H^2 - 45GH$ [$G = F_o/F_{max}$, $H = \sin\theta/\sin\theta_{max}$ (Wang Hong & Robertson, 1985)], including anisotropic thermal parameters from all non-H atoms, constrained H atoms [C—H and N—H 0.96 Å, H—C—H and H—N—H 109.5°, aromatic H on ring angle external bisectors, O—H 0.87 Å, U(H) $= 1.2U_{eq}(X)$], and an isotropic extinction parameter $x = 2.1\ (2) \times 10^{-5}$ [$F_c' = F_c/(1 + xF_c^2/\sin 2\theta)^{1/4}$]. At convergence, $R = 0.0699$, $wR = 0.0264$, $S = 2.37$ for 212 parameters. The mean and maximum Δ/σ were 0.007 and 0.027, repsectively, and all features in a final difference synthesis lay between +1.02 and −0.43 e $Å^{-3}$. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99, 149). *SHELXTL* (Sheldrick, 1985) and local computer programs were used.

Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1, bond lengths and angles in Table 2.* The structure of the centrosymmetric cation and two symmetry-related anions is shown in Fig. 1.

Related literature. The structure of the same cation has been reported in its nitrate (Pajunen, 1968) and perchlorate (Pajunen, 1969) salts, and also in the hydrated mixed salt $[Ni(NH_2CH_2CH_2CH_2NH_2)_3][Ni(NH_2CH_2CH_2CH_2NH_2)_2(OH_2)_2]Cl_4.H_2O$ (Andreetti, Cavalca & Sgarabotto, 1971), with essentially the same geometry as that found here.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54801 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL0511]

Table 2. *Bond lengths* (Å) *and angles* (°)

Ni—O(1)	2.126 (2)	Ni—N(1)	2.109 (2)
Ni—N(2)	2.101 (2)	N(1)—C(1)	1.482 (3)
N(2)—C(3)	1.470 (4)	C(1)—C(2)	1.500 (3)
C(2)—C(3)	1.512 (4)	C(4)—C(5)	1.451 (3)
C(4)—C(9)	1.424 (3)	C(4)—O(2)	1.250 (3)
C(5)—C(6)	1.389 (3)	C(5)—N(3)	1.436 (3)
C(6)—C(7)	1.378 (3)	C(7)—C(8)	1.377 (3)
C(7)—N(4)	1.449 (2)	C(8)—C(9)	1.381 (3)
C(9)—N(5)	1.440 (3)	N(3)—O(31)	1.226 (2)
N(3)—O(32)	1.230 (2)	N(4)—O(41)	1.207 (2)
N(4)—O(42)	1.228 (3)	N(5)—O(51)	1.214 (4)
N(5)—O(52)	1.221 (3)		

O(1)—Ni—N(1)	89.9 (1)	O(1)—Ni—N(2)	88.9 (1)
N(1)—Ni—N(2)	91.2 (1)	Ni—N(1)—C(1)	120.2 (1)
Ni—N(2)—C(3)	120.6 (1)	N(1)—C(1)—C(2)	112.6 (2)
C(1)—C(2)—C(3)	115.7 (2)	N(2)—C(3)—C(2)	113.1 (2)
C(5)—C(4)—C(9)	113.0 (2)	C(5)—C(4)—O(2)	123.1 (2)
C(9)—C(4)—O(2)	123.8 (2)	C(4)—C(5)—C(6)	123.3 (2)
C(4)—C(5)—N(3)	120.4 (2)	C(6)—C(5)—N(3)	116.2 (2)
C(5)—C(6)—C(7)	118.7 (2)	C(6)—C(7)—C(8)	121.8 (2)
C(6)—C(7)—N(4)	119.6 (2)	C(8)—C(7)—N(4)	118.5 (2)
C(7)—C(8)—C(9)	119.0 (2)	C(4)—C(9)—C(8)	124.1 (2)
C(4)—C(9)—N(5)	120.3 (2)	C(8)—C(9)—N(5)	115.6 (2)
C(5)—N(3)—O(31)	119.4 (2)	C(5)—N(3)—O(32)	119.4 (2)
O(31)—N(3)—O(32)	121.2 (2)	C(7)—N(4)—O(41)	118.9 (2)
C(7)—N(4)—O(42)	118.0 (2)	O(41)—N(4)—O(42)	123.1 (2)
C(9)—N(5)—O(51)	120.1 (2)	C(9)—N(5)—O(52)	118.5 (2)
O(51)—N(5)—O(52)	119.8 (2)		

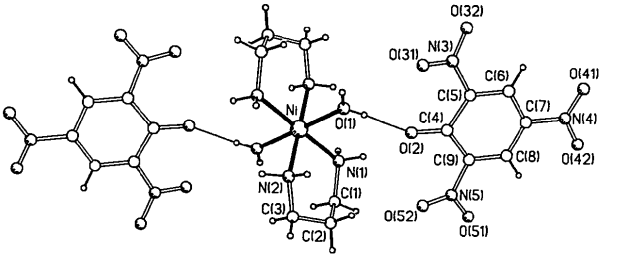


Fig. 1. Structure of the cation with two symmetry-related anions. Independent non-hydrogen atoms are labelled.

We thank SERC (UK) and the Department of Science and Technology, New Delhi (India), for financial support.

References

ANDREETTI, G. D., CAVALCA, L. & SGARABOTTO, P. (1971). *Gazz. Chim. Ital.* **101**, 494–501.

BLACK, S. C. & MCLEAN, I. A. (1971). *Aust. J. Chem.* **24**, 1401–1411.

CLEGG, W. (1981). *Acta Cryst.* A**37**, 22–28.

PAJUNEN, A. (1968). *Suom. Kemistil. B*, **41**, 232–236.

PAJUNEN, A. (1969). *Suom. Kemistil. B*, **42**, 397–400.

SHELDRICK, G. M. (1985). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Revision 5. Univ. of Göttingen, Germany.

WANG HONG & ROBERTSON, B. E. (1985). *Structure and Statistics in Crystallography*, edited by A. J. C. WILSON, pp. 125–136. New York: Adenine Press.