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The Molecular Structure (X-ray Analysis) of a Nickel(II) Compound of a Novel Quadridentate Ligand Derived from a Diamine and *o*-Aminobenzaldehyde

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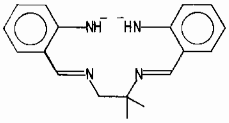
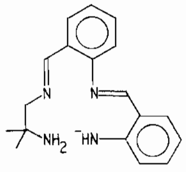
In a recent paper,[1] which included metal compounds of the ligand I,



I

we reported a novel cationic species of empirical formula [Ni(abbn H)] X (where X = acetate or perchlorate, abbn = I). This had been isolated from reaction mixtures designed to give the neutral nickel (II) compound of I. The available data were interpreted as evidence that the molecular cation was likely to be a dimer, $[Ni_2(abbn\ H)_2]^{2+}$. However, we now report the result of an X-ray analysis of the perchlorate of this cationic Ni^{II} species, which shows it to be a monomer in which the ligand is the novel condensation product II.



II

Results and discussion

Dark red plates of the perchlorate[1] were obtained from ethanol.

Crystal data

$C_{18}H_{21}N_4NiClO_4$, M = 451.6, Monoclinic, a = 19.25 (4), b = 13.91(1), c = 14.66(3) Å, β = 94.56(2)°, D_m (flotation) = 1.51 g cm^{-3}, Z = 8, D_c = 1.53 g cm^{-3}. Space group $B2_1/a$ {a non-standard setting of $P2_1/c$ [C_{2h}^5, No. 14] - chosen for the convenient β angle.}

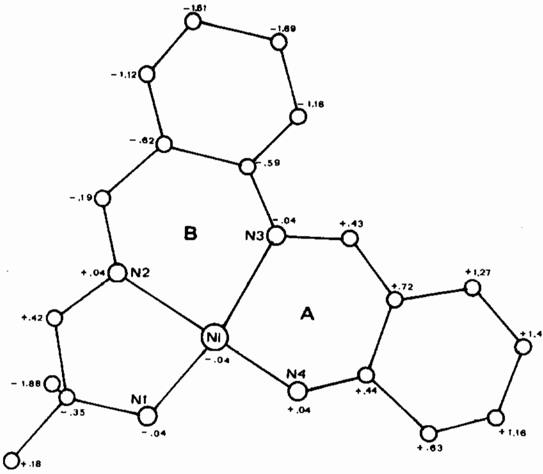


Figure. The molecular cation, showing the distances (in Å) of the various atoms above or below the least squares plane of the four nitrogens.

X-ray data were collected on a Stoe Stadi-2 diffractometer (monochromated Mo-K_α radiation) with stationary counter and moving crystal, giving 2244 independent reflections ($I > 3\sigma$).

The structure was solved by conventional Patterson and Fourier techniques and refined by block-diagonal least-squares to a current R of 0.089. The nickel atom has been allowed anisotropic vibrational parameters and all other non-hydrogen atoms have been given isotropic parameters.

The nickel polyhedron is essentially four-co-planar (Figure) with bond lengths reflecting the different natures of the nitrogen donors {Ni–N(1) = 1.94(1); Ni–N(2) = 1.88(1); Ni–N(3) = 1.89(1); and Ni–N(4) = 1.85(1) Å}. The small deviations from planarity of the atoms of the nickel polyhedron are shown in the Figure.

The ligand itself is far from planar: the dihedral angle between chelate rings A and B is 29°, and they are inclined to the $[N_4]$ plane at −20 and +19° respectively. The aliphatic chelate ring has an almost symmetric *gauche* conformation.

This appears to be the first recognition of a ligand in which there is self-condensation of two molecules of *o*-aminobenzaldehyde. Busch and co-workers[2] have studied in some detail the trimers and tetramers.

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1 E. D. McKenzie, R. D. Moore and J. M. Worthington, *Inorg. Chim. Acta, 14,* 37 (1975).
2 S. C. Cummings and D. H. Busch, *J. Am. Chem. Soc., 92,* 1924 (1970) and references therein.