

A variety of experiments may shed light on the nature of these "pseudo-superoxides." These experiments will, however, involve a considerable length of time so we feel that the observation on the magnetism should be reported at this time.

Acknowledgments. We thank Dr. P. J. Bray, Dr. R. L. Carlin, and Mr. E. Terezakis for aid with the measurements. The work was partially supported by the Materials Science Program of the Department of Defense and by the U. S. Atomic Energy Commission (Contracts AT(30-1)-1983 and AT(30-1)-2024).

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Received February 23, 1965

The Structure of Five-Coordinated High-Spin Complexes of Nickel(II) and Cobalt(II) with N- β -Diethylethylamineethyl-5-chlorosalicylaldehyde¹

Sir:

Ring-substituted salicylaldehydes form with N,N-diethylethylenediamine Schiff bases of the general formula $X\text{C}_6\text{H}_3(\text{OH})\text{CH}=\text{NC}_2\text{H}_4\text{N}(\text{C}_2\text{H}_5)_2$. These ligands give complexes with nickel(II) of the general formula $[\text{X-SALen-N}(\text{C}_2\text{H}_5)_2]_2\text{Ni}$. Paramagnetic octahedral or diamagnetic planar complexes are formed depending on the nature of the X substituent in the benzene ring. When X = 3-Cl, 5-Cl, or 3,4-benzo, paramagnetic compounds ($\mu_{\text{eff}} = 3.3$ B.M.) of indeterminate structure are formed. Their reflectance spectra differ from those of the octahedral and planar forms. In solution, the absorption spectra show the existence of octahedral, planar, and unknown species.²

$[\text{5-Cl-SALen-N}(\text{C}_2\text{H}_5)_2]_2\text{Ni}$ and also $[\text{5-Cl-SALen-N}(\text{C}_2\text{H}_5)_2]_2\text{Co}$ are isomorphous with the following crystal data, respectively: $a = 12.68, 13.20 \text{ \AA.}$; $b = 21.77, 21.86 \text{ \AA.}$; $c = 12.05, 12.15 \text{ \AA.}$; $\beta = 122^\circ 48', 126^\circ 25'$; $Z = 4$; $d_c = 1.32, 1.31 \text{ g./cm.}$; $V_M = 699.4, 705.4 \text{ \AA.}$; space group $P2_1/c$.

Three-dimensional data have been collected on the nickel complex by the equi-inclination Weissenberg technique using Cu $K\alpha$ radiation. The structure has been solved by three-dimensional Patterson and Fourier syntheses and refined by several cycles of least squares.

- (1) Supported by the Italian Consiglio Nazionale delle Ricerche.
 (2) L. Sacconi, P. Nannelli, N. Nardi, and U. Campigli, *Inorg. Chem.*, in press.

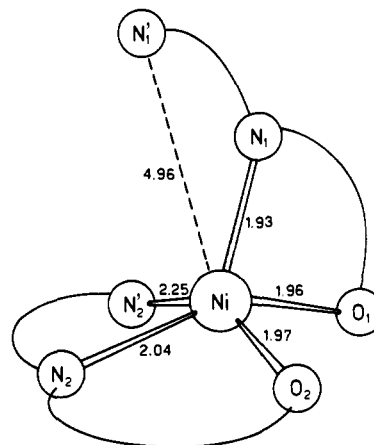


Figure 1. Sketch of the molecular structure of $[\text{5-Cl-SALen-N}(\text{C}_2\text{H}_5)_2]_2\text{Ni}$. Salicylaldimines residues are schematically represented by full lines.

At the present stage of refinement R is 0.15 over 1033 observed, independent reflections.

The structure consists of discrete molecules in which nickel(II) has a coordination number of five. The coordination polyhedron can be described as a distorted square pyramid (Figure 1). Bond lengths about the nickel atom are shown in the figure. The angles are: $\text{O}_1\text{-Ni-O}_2 = 94^\circ$, $\text{O}_2\text{-Ni-N}_2 = 89^\circ$, $\text{N}_2\text{-Ni-N}_2' = 80^\circ$, $\text{N}_2'\text{-Ni-O}_1 = 90^\circ$, $\text{O}_1\text{-Ni-N}_1 = 92^\circ$, $\text{O}_2\text{-Ni-N}_1 = 100^\circ$, $\text{N}_2\text{-Ni-N}_1 = 105^\circ$, $\text{N}_2'\text{-Ni-N}_1 = 105^\circ$. The basal set of four ligands lies in a plane within 0.1 \AA. The nickel atom is 0.36 \AA. above this plane. The nickel to nonbonded nitrogen atom distance is 4.96 \AA. The shortest intermolecular nickel to nonbonded nitrogen distance is 7.81 \AA.

This structure represents the first example of a high spin nickel(II) complex so far described with a five-coordinate geometry.

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Received March 1, 1965

The Structure of Villalstonine¹

Sir:

Alkaloid B (m.p. 235–270 dec. (*in vacuo*), $[\alpha]^{25\text{D}} + 79^\circ$ (c 1.0, pyridine)) isolated from *Alstonia muelleriana* Domin by Elderfield and co-workers² was characterized by them as an indole alkaloid of approximate composition $\text{C}_{40}\text{H}_{50}\text{O}_4\text{N}_4$. A tentative identification³ of Alkaloid B with villalstonine⁴ has been confirmed by a comparison of single crystal X-ray diffraction patterns.⁵ We wish to report the result of a crystallographic study which establishes the molecular structure

(1) This investigation was supported by Public Health Service Research Grants HE 04179 and HE 08612 from the National Heart Institute.

(2) R. C. Elderfield, R. E. Gilman, and A. Okano, to be published; R. E. Gilman, Ph.D. Thesis, University of Michigan, 1959.

(3) R. C. Elderfield, private communication.

(4) T. M. Sharp, *J. Chem. Soc.*, 1277 (1934); T. A. Henry, "The Plant Alkaloids," 4th Ed., The Blakiston Co., Philadelphia, Pa., 1949, p. 718.

(5) We are grateful to Dr. A. Chatterjee for sending us a sample of authentic villalstonine.