Short Communications

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 *words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible. Publication will be quicker if the contributions are without illustrations.*

Acta Cryst. (1972). B**28**, 2298

The approximate structure of the tris-(2-aminomethylpyridine)nickel(II) complex ion.

By Allen A. Amaro and Karl Seff,* *Department of Chemistry, University of Hawaii, Honolulu, Hawaii* 96822, *U.S.A.*

(*Received* 2 *February* 1972)

Tris-(2-aminomethylpyridine)Ni(II) perchlorate crystallizes in space group $P\bar{4}3n$ with $a=16{\cdot}95$ (1) Å. The complexed Ni(II) ion must exhibit threefold symmetry and each pyridine ring must be *cis* to the other two.

$Ni(C_5H_5N{-}CH_2{-}NH_2)_3(ClO_4)_2$, tris(2-aminomethylpyridine)nickel(II) perchlorate, was prepared by mixing acetone–ethanol solutions of $Ni(ClO_4)_2$ and 2-aminomethylpyridine. Slow evaporation yielded magenta prismatic crystals.

From Weissenberg and precession X-ray photographs the crystals were found to be cubic with space group $P\bar{4}3n$

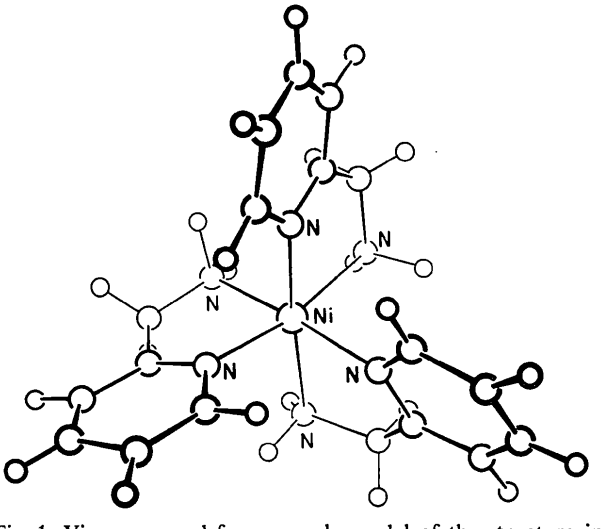

Fig. 1. View prepared from a scale model of the structure in which an approximate dihedral angle of 15°, natural to the model, was used for the exocyclic C–C bond.

* Author to whom correspondence should be addressed.

(*hkl*, no conditions; *hhl*, $l=2n$; and cyclicly). These photographs do not suggest disorder.

The cell constant, as determined by a least-squares refinement of fifteen intense reflections with 2θ values up to 23°, is $a=16{\cdot}95$ (1) Å at 19°C. For this, a Syntex computer-controlled diffractometer with Mo $K\alpha$ ($\lambda=0{\cdot}71069$ Å) radiation was used. Using a molecular weight of 585·06 amu and assuming eight formula-weights per unit cell, a density of 1·595 g.cm^{-3} can be calculated which agrees well with the observed value, 1·585 g.cm^{-3}, measured by flotation in CCl_4 ($\varrho=1{\cdot}585$ g.cm^{-3}).

Considering the symmetry of the ligand, and assuming that the complexed cation is not disordered, these conclusions follow: the Ni(II) ions are at Wyckoff positions 8(*e*) of $P\bar{4}3n$ on the threefold axes; the complexed Ni(II) ions contain a molecular threefold axis; and each of the three pyridine rings must be *cis* to each of the other two. It is felt that the coordination configuration is nearer to trigonal antiprismatic (pseudo-octahedral) (see, for instance, Swink & Atoji, 1960) than to trigonal prismatic, which has been found only where the ligands impose particularly severe constraints (see Parks, Wagner & Holm, 1970) on the coordination geometry. Since the five-membered NiNCC···N rings cannot deviate greatly from planarity due to ring strain, an approximate model (see Fig. 1) of the complexed cation was easily assembled. No further work is planned.

References

Parks, J. E., Wagner, B. E. & Holm, R. H. (1970). *J. Amer. Chem. Soc.* **92**, 3500.

Swink, L. N. & Atoji, M. (1960). *Acta Cryst.* **13**, 639.

Acta Cryst. (1972). B**28**, 2298

Structure cristalline du radical nitroxyde: subérate de di-tétraméthyl-2,2,6,6-pipéridinyl-4-oxyle 1.

Par Annie Capiomont, *Laboratoire de Spectrométrie Physique, Université Scientifique et Médicale de Grenoble, Cedex* 53, 38 - *Grenoble-Gare, France*

(*Reçu le* 6 *mars* 1972)

Di-(2,2,6,6-tetramethyl-4-piperidinyl-1-oxyl) suberate $C_{26}H_{46}N_2O_6$ is a nitroxide radical which crystallizes in the monoclinic system, space group $P2_1/c$. The dimensions of the unit cell are $a=6{\cdot}041$, $b=21{\cdot}52$, $c=13{\cdot}62$ Å, $\beta=126{\cdot}360°$. There are two molecules in a cell, each at a centre of symmetry.

Le subérate de di-tétraméthyl-2,2,6,6-piperidinyl-4-oxyle 1 a été synthétisé au Laboratoire de Chimie Organique Physique du CENG (Professor Rassat) (Rey, 1967).

Il fait, d'autre part, l'objet d'études de r.p.e., de chaleur spécifique et de susceptibilité magnétique à basse température. Ces dernières ont montré une anomalie de susceptibilité magnétique vers 0,5°K compatible avec une tendance à un ordre linéaire en-dessous de cette température.