Structures of Metal Diselenocarbamates. The Crystal Structure of Nickel Diethyldiselenocarbamate

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SYNTHESIS and spectral investigations of selenocompounds are at present being carried out by Furlani, *et al.*[1] in this Institute. In connection with these studies, the *X*-ray analysis of several diethyldiselenocarbamates was undertaken, namely of the K, Cr^{III}, Fe^{III}, Co^{III}, Ni, Cu^{II}, Zn, and Pd salts, as well as Fe^{III} monohalogenobis(diethyldiselenocarbamate).

These compounds are of special interest to us because of their similarity to the corresponding diethyldithiocarbamates. *X*-Ray investigations showed a distinct correlation between the cell constants, symmetry and structure for the nickel, copper, and zinc salts.[2] We describe here the crystal structure of nickel diethyldiselenocarbamate.

Crystals of $C_{10}H_{20}N_2NiSe_4$ are pleocroic (green and dark red) monoclinic prisms; $a = 6{\cdot}350 \pm 0{\cdot}010$, $b = 11{\cdot}618 \pm 0{\cdot}05$, $c = 11{\cdot}988 \pm 0{\cdot}010$ Å; $\beta = 93°59'$; $U = 882{\cdot}3$ Å^3; $D_m = 2{\cdot}07 \pm 0{\cdot}05$ g. cm^{-3} (by flotation); $Z = 2$; $D_c = 2{\cdot}044$ g. cm^{-3}; $F(000) = 516$. *Space group* $P2_1/c$ from systematic absences. Data are taken from Weissenberg photographs, using Cu-K_α ($\lambda = 1{\cdot}5418$ Å) radiation.

The *X*-ray intensities of 1139 independent reflections, visually estimated from equi-inclination Weissenberg photographs taken about *b* and *c* axes, were recorded.

Due to the great similarity of the cell dimensions and intensity distribution with nickel diethyldithiocarbamate, a structure-factor calculation was performed using co-ordinates of atoms of this salt except for the hydrogens (for selenium atoms the sulphur co-ordinates were used) and isotropic thermal motion $B = 3{\cdot}5$ Å^2 for all atoms). The reliability index R was 0·25. A Fourier synthesis did not show any other significant peak. Four isotropic least-squares cycles refined space and thermal parameters of the 9 heavy atoms to a set of values corresponding to $R = 0{\cdot}13$. Three anisotropic least-squares cycles gave the actual

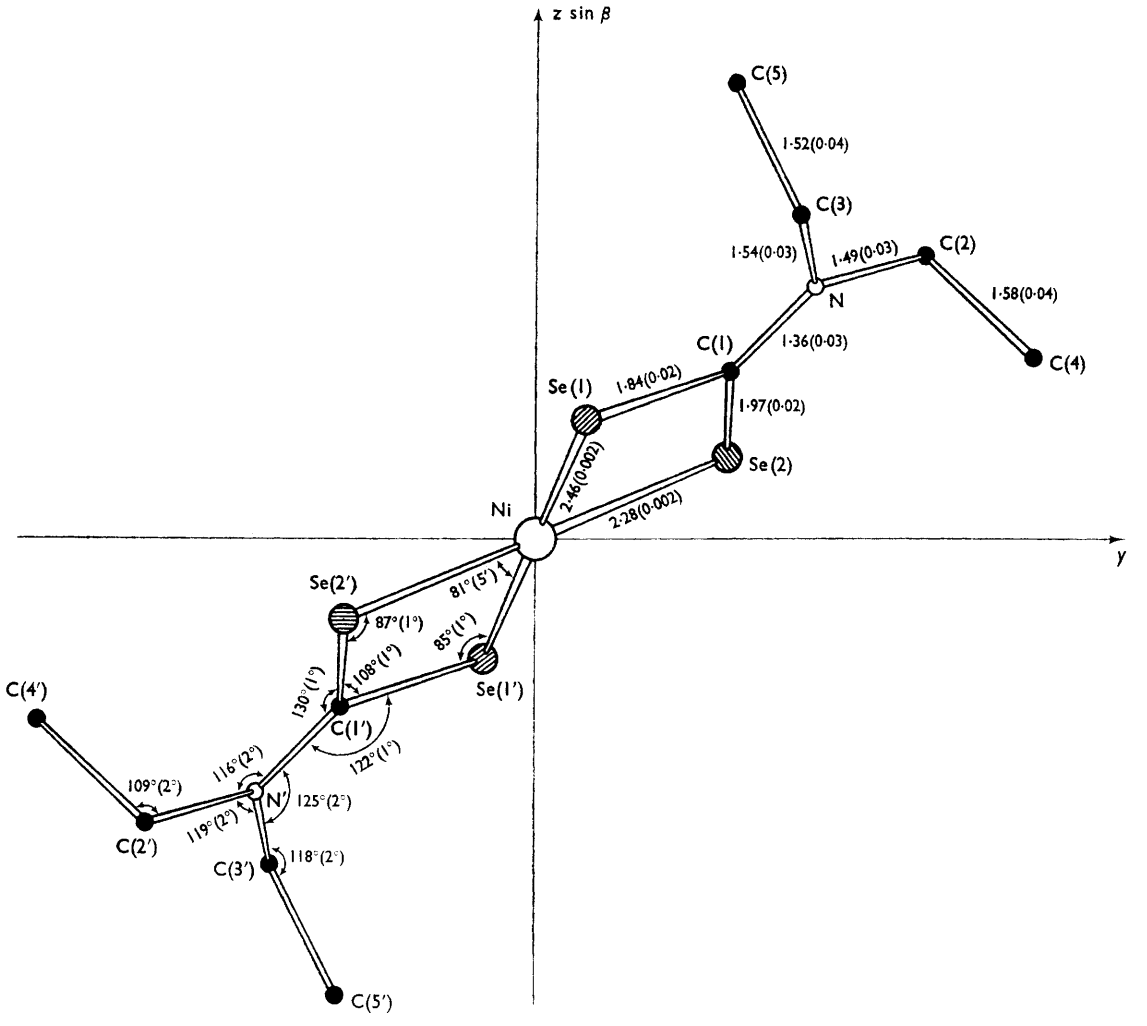
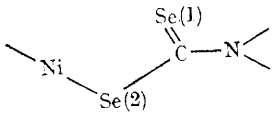


FIGURE. [100] *projection of the molecule, with bond lengths (in* Å*) and angles with standard deviations (in parenthesis).*

values ($R = 0{\cdot}103$), which define the bond lengths and angles given in the Figure.

The molecule is rather similar to that of nickel diethyldithiocarbamate. As in this molecule, planarity of all the atoms (apart from the terminal methyl groups) is good (the mean deviations from planarity being 0·05 Å). Distortions from exact square co-ordination for the nickel atom are also about the same. The two selenium atoms give significantly different bond lengths with Ni and C, whilst the two Ni–S and the two S–C bond lengths do not differ. From our point of view, this is the more interesting difference between the two structures. This fact could be interpreted in terms of a prevailing contribution of the form



to the structure.

(*Received, July* 31*st,* 1967; *Com.* 965.)

[1] C. Furlani, E. Cervone, and F. Diomedi Camassei, *Inorg. Chem.*, in the press.
[2] See M. Bonamico, G. Dessy, C. Mariani, G. Mazzone, A. Mugnoli, A. Vaciago, and L. Zambonelli, *Acta Cryst.*, 1965, **19**, 619; 886; 898.