Crystal Structure of π-Allyldi(thiourea)nickel Chloride

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COMPLEXES of general formula π-allylNi LL'X, with LL' = thiourea or alkylthiourea and X = Cl, Br, I,[1] are active catalysts for the synthesis of hexadienoic esters from allylic halides, acetylene, carbon monoxide, and alcohols.[2]

The structure of the complex π-allyldi(thiourea)nickel chloride has been examined by the single-crystal *X*-ray diffraction method. The space-group determination, as well as the collection of the intensity data, was performed by

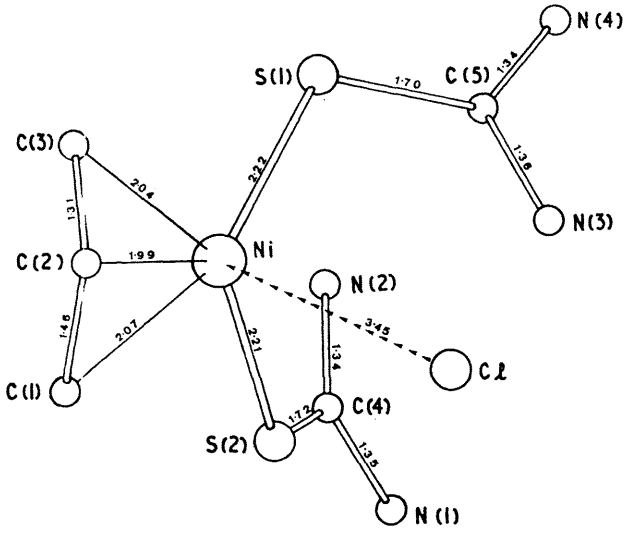

FIGURE.

the photographic Weissenberg method, with Cu-K_α radiation. The unit cell, of *Pbca* symmetry, contains 8 molecules and has *a* 25·24 ± 0·08, *b* 11·17 ± 0·04, c 8·63 ± 0·03 Å.

The structure was solved by three-dimensional Patterson synthesis, with refinement by block-diagonal least-squares with anisotropic thermal parameters for Ni, Cl, and S. $R = 0{\cdot}106$ for about 1100 observed reflections. The Figure shows a projection of the molecule along the crystallographic *c* axis.

The co-ordination around the nickel atom is square-planar with respect to the thiourea and allyl ligands. The chlorine atom completes a sort of pentaco-ordination around the metal atom occupying the vertex of a square pyramid. However, the value of the Ni–Cl distance is consistent with the ionic character of the complex inferred from solubility data.[1]

No particular significance should be attached to the difference between the carbon–carbon bond lengths of the allyl group, since the relative standard deviations are ~ 0·04 Å.

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[1] F. Guerrieri, *Chem. Comm.*, 1968, 983.
[2] G. P. Chiusoli and S. Merzoni, *Chimica e Industria*, 1961, **43**, 259; G. P. Chiusoli, M. Dubini, M. Ferraris, F. Guerrieri, S. Merzoni, and G. Mondelli, *J. Chem. Soc.* (*C*), 1968, 2889.