SHORT COMMUNICATIONS

The Crystal Structure of Tetra-thiourea-palladium(II) Chloride, $[Pd(SCN_2H_4)_4]Cl_2$

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It is well known that thiourea is capable of forming a number of coordination compounds with metallic ions. Recently, we reexamined the preparation methods of some of them, the chemical analyses being carried out carefully, and obtained quite well-formed crystals of the palladium(II) complex salt, $Pd(SCN_2H_4)_4Cl_2$. We have therefore, attempted to investigate the crystal structure of this compound. There are only a few structural studies[1-4] of the thiourea coordination compounds.

The crystal was prepared by the method of Kurnakow[5]. The products are monoclinic, showing the predominant (100) face. The crystallographic data are: $a=16.89$, $b=11.18$ $c=8.89$ Å, $\beta=91.5$, $Z=4$, $\rho_{obs}=1.92$ and $\rho_{calc}=1.91$ g./cm^3. Space group $C2/c$ or Cc was given by the observation of the systematically absent reflections. However, the method of Howells

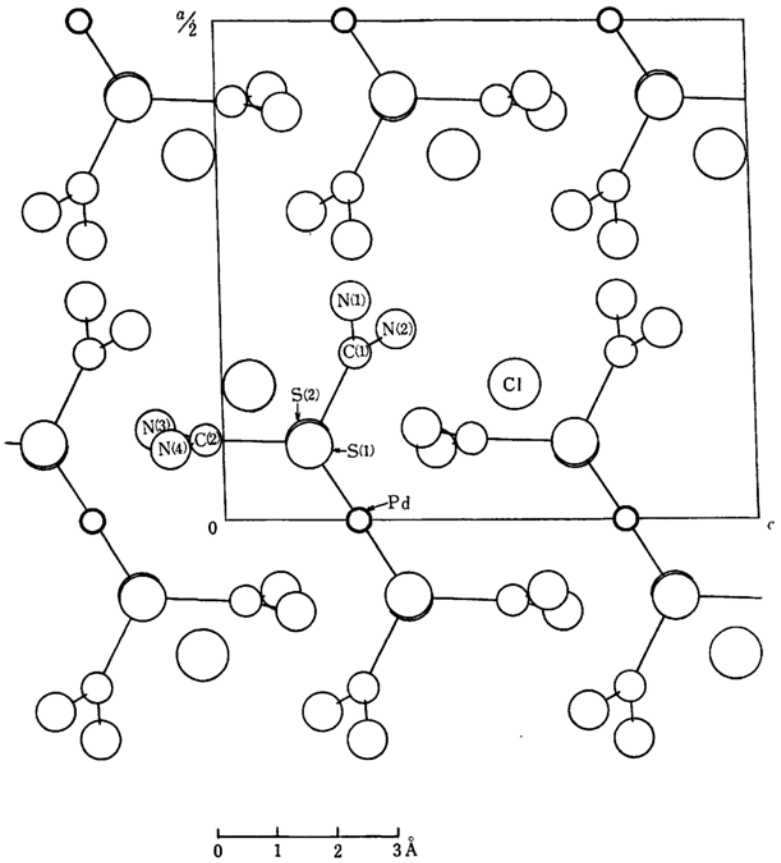

Fig. 1. Projection of the structure upon a plane normal to the *b*-axis.

1) M. Nardelli, L. Cavalca and A. Braibanti, *Gazz. chim. ital.*, **86**, 1037 (1956).
2) M. Nardelli, L. Cavalca and A. Braibanti, ibid., **87**, 137 (1957).
3) C. B. Knobler, Y. Okaya and R. Pepinsky, *Z. Krist.*, **111**, 385 (1959).
4) M. Nardelli and G. Fava, *Acta Cryst.*, **12**, 727 (1959).
5) Kurnakow, *J. prakt. Chem.*, [2] **50**, 496 (1894).

TABLE I. ATOMIC COORDINATES

	x/a	*y/b*	*z/c*
Pd	0	0.324	0.250
S (1)	0.073	0.482	0.158
S (2)	0.079	0.167	0.155
Cl	0.135	0.182	0.050
N (1)	0.220	0.561	0.242
N (2)	0.189	0.380	0.328
N (3)	0.092	0.078	−0.136
N (4)	0.071	0.288	−0.105
C (1)	0.167	0.476	0.247
C (2)	0.081	0.183	−0.037

et al.[6], applied to (*hk*0) reflections, was indicative of the presence of a center of symmetry, which was, in turn, favorite to $C2/c$. The structure analysis was carried out by using (*h*0*l*) and (*hk*0) data obtained from Weissenberg photographs which were taken with Cu$K\alpha$ radiation. From Patterson projections on (010) and (001), the positions of palladium, chlorine and sulfur atoms could be deduced. A usual Fourier method was, then, applied to further analysis. Atomic coordinates listed in Table I gave the reliability index $R=0.18$ and 0.16 for (*h*0*l*) and (*hk*0), respectively.

In Fig. 1, the structure projected on (010) is shown. The crystal is essentially ionic and consisted of $[Pd(SCN_2H_4)_4]^{2+}$ and Cl^-. A palladium atom is surrounded by four sulfur atoms in an approximately rectangular configuration with 3.00 and 3.15 Å for the lengths of shorter sides, and 3.50 Å for the longer ones. A complex ion has a two-fold rotation axis bisecting the two shorter sides and passing through the palladium atom. All S—C bonds projected on the plane formed with 4S's and Pd, are parallel to the shorter sides. Bond distances and angles found in a complex ion are: Pd—S=2.33 and 2.35 Å, ∠S—Pd—S=82°, 85° and 97°, ∠Pd—S—C=106 and 107°, S—C=1.72 and 1.75 Å, C—N=1.32, 1.37 and 1.47 Å, ∠S—C—N=120, 121, 123 and 126° and ∠N—C—N 117°. No significant discrepancies could be found in the dimensions between the thiourea molecule reported by Kunchur and Truter[7] on the thiourea crystal and the one in the present work. The bond angles formed at S's can be compared with those in $Cu(SCN_2H_4)_3Cl$[3] and in $Pb(SCN_2H_4)_2Cl_2$[4].

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6) E. R. Howells, D. C. Phillips and D. Rogers, *Acta Cryst.*, 3, 210 (1950).
7) N. R. Kunchur and M. Truter, *J. Chem. Soc.*, **1958**, 2551.