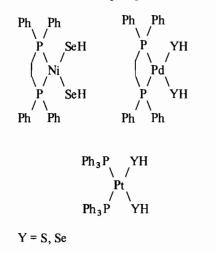
Ethylenebis(diphenylphosphine)dihydrogensulfidenickel(II)

M. SCHMIDT*, G. G. HOFFMANN and R. HÖLLER Institut für Anorganische Chemie der Universität Würzburg, Am Hubland, D-8700 Würzburg, F.R.G. Received October 11, 1978

Recent studies have led to the synthesis of some hydrogensulfide and hydrogenselenide compounds of phosphine coordinated d^8 metals Ni, Pd, and Pt in the oxidation state + II [1-4]:



In spite of the vicinal YH-groups, those compounds exhibit a remarkable stability towards air and moisture in the solid state. However, it should be pointed out that the choice of the phosphine ligand is of great importance for the stability [5]: with nickel and palladium, bidentate phosphines are necessary, whereas with platinum monodentate ligands are sufficient.

In the above-mentioned series the corresponding nickel-sulfur compound was missing. All experiments to react dichloroethylenebis(diphenylphosphine)-

*To whom correspondence should be addressed.

TABLE II. Analytical Data of the SH-Metal Compounds Formed by NaSH.

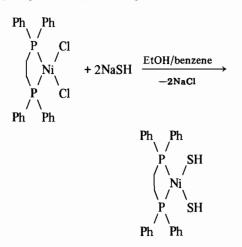
Compound	Decomp. Point [°C]	Yield [%]	%С	%H	%S	Colour
			[found (calculated)]			-
(diphos)Ni(SH) ₂	100	62	60.3 (59.7)	5.30 (5.01)	12.4 (12.3)	orange-red
(diphos)Pd(SH) ₂	139	76	55.5 (54.7)	4.22 (4.59)	11.6 (11.2)	ochre yellow
(PPh ₃) ₂ Pt(SH) ₂	197	60	55.9 (55.0)	4.55 (4.11)	8.80 (8.16)	yellow

¹ H n.m.r. Chemical Shifts							
δ [ppm] 8.07-7.10 2.18 1.67	Multiplicity multiplet doublet singlet	Origin arom. prot. =P-CH ₂ - -SH	Solvent/Standard CDCl ₃ /CH ₂ Cl ₂ int.				

I.r. Frequencies (nujol, Csl discs): 2535w (SH), 998s, 883s, 850w, 820s, 753s, 743s, 712s, 696s, 685s, 653s, 615m, 534s, 528sh, 484s, 472m, 450sh, 439m (NiS), 399m, 338b (NiS), 277w.

nickel(II), (diphos)NiCl₂ with hydrogen sulfide (which works with palladium and platinum) led only to dark brown products [1].

This experimental difficulty could be overcome by a rather simple experimental procedure: sodium hydrogensulfide, according to



does form with (diphos)NiCl₂ in 62% yield the title compound (diphos)Ni(SH)₂. Thus, the above-mentioned series of compounds could be completed.

The afore-mentioned experimental method is also suitable for the synthesis of the already known palladium- and platinum-dihydrogenchalcogeno compounds, prepared from H_2S [2-4]. The products, prepared by this method, are identical with the already described compounds. The tetra-coordinated new nickel compound, an orange-red solid, decomposes at 100 °C. At room temperature it is stable in air. Solutions in chlorinated hydrocarbons (*e.g.*, CH_2Cl_2 and $CHCl_3$) decompose rather fast with formation of black solids; the compound is slightly soluble in alcohol and insoluble in hydrocarbons.

¹H n.m.r. and i.r. spectra are recorded in Table I; analytical data are given in Table II.

Experimental

Ethylenebis(diphenylphosphine)dihydrogensulfidenickel(II), (diphos)Ni(SH)₂

To a solution of 0.28 g (5.00 mmol) sodium hydrogensulfide (prepared from 5.00 mmol NaOEt and hydrogensulfide [6]) in 25 ml ethanol and 10 ml benzene are added 1.30 g (2.50 mmol) (diphos)NiCl₂ under N₂ atmosphere; the suspension slowly turned orange-red. After stirring for 24 h, filtration, washing with water, ethanol and n-hexane, the product was dried.

Ethylenebis(diphenylphosphine)dihydrogensulfidepalladium(II), (diphos)Pd(SH)₂

Synthesis and treatment like $(diphos)Ni(SH)_2$: 0.28 g (5.00 mmol) sodium hydrogensulfide, 1.44 g (2.50 mmol) $(diphos)PdCl_2$.

Bis(triphenylphosphine)dihydrogensulfideplatinum (II), (PPh₃)₂Pt(SH)₂

Synthesis and treatment like $(diphos)Ni(SH)_2$, in addition, boiling under reflux: 0.28 g (5.00 mmol) sodium hydrogensulfide, 1.98 g (2.50 mmol) $(PPh_3)_2$ -PtCl₂.

References

- 1 M. Schmidt and G. G. Hoffmann, Angew. Chem., 90, 630 (1978); Angew. Chem. Int. Ed. Engl., 17, 598 (1978).
- 2 M. Schmidt and G. G. Hoffmann, J. Organometal. Chem., 124, C5 (1977).
- 3 M. Schmidt and G. G. Hoffmann, Z. Naturforsch., in press.
- 4 B. Kreutzer, P. Kreutzer and W. Beck, Z. Naturforsch., 27b, 461 (1972).
- 5 M. Schmidt and G. G. Hoffmann, Chem. Ber., in press.
- 6 G. Brauer, "Handbuch der präparativen anorganischen Chemie", Ferdinand Enke Verlag, Stuttgart (1960).