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Ethylenebis(diphenylphosphine)dihydrogensulfide-nickel(II)

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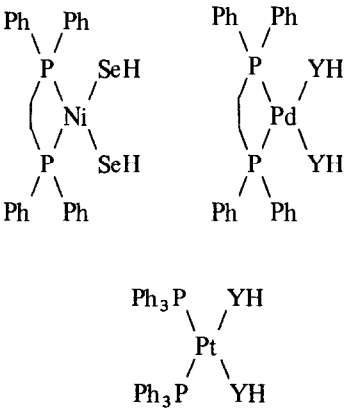
Recent studies have led to the synthesis of some hydrogensulfide and hydrogenselenide compounds of phosphine coordinated d^8 metals Ni, Pd, and Pt in the oxidation state + II [1–4]:

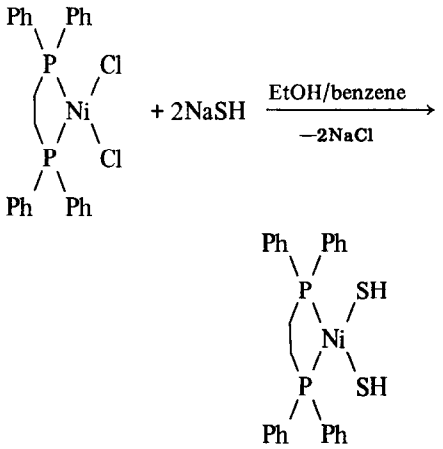


Y = S, Se

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)-nickel(II), (diphos)$NiCl_2$ 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)$NiCl_2$ 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.

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TABLE I. ^1H N.m.r. and I.r. Data of (diphos)$Ni(SH)_2$.

^1H *n.m.r. Chemical Shifts*

δ [ppm]	Multiplicity	Origin	Solvent/Standard
8.07–7.10	multiplet	arom. prot.	$CDCl_3/CH_2Cl_2$ int.
2.18	doublet	$=P-CH_2-$	
1.67	singlet	$-SH$	

I.r. Frequencies (nujol, CsI 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.

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

Compound	Decomp. Point [°C]	Yield [%]	%C	%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
$(PPh_3)_2Pt(SH)_2$	197	60	55.9 (55.0)	4.55 (4.11)	8.80 (8.16)	yellow

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)dihydrogensulfide-nickel(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)$NiCl_2$ under N_2 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)dihydrogensulfide-palladium(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), (PPh_3)$_2$Pt(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)$_2$-$PtCl_2$.

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