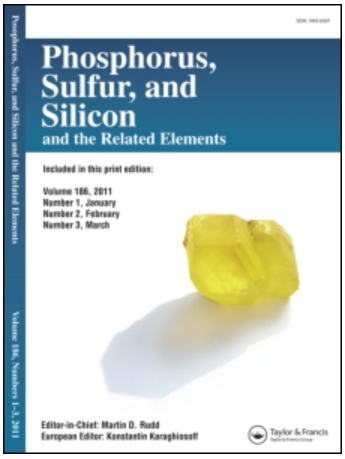

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SYNTHESIS OF $Hg(NSO)_2$ IN LIQUID AMMONIA

ROBERT J. REYNOLDS AND J.DEREK WOOLLINS.
Dept. of Chemistry, Loughborough University, Loughborough,
Leicestershire, LE11 3TU. England.

Abstract. The reaction of thionyl chloride with liquid ammonia produces the thionyl imide anion, NSO^- Addition of metal chloride bis-phosphine complexes such as $[PtCl_2(PMe_3)_2]$ the resulting NH_3(l) solution has been shown to produce compounds of the type $[Pt(NSO)_2(PMe_3)_2]$.[1] Here, we describe the synthesis of $Hg(NSO)_2$ in liquid ammonia, using $HgCl_2$ as the starting material.

EXPERIMENTAL.

NH_3 (l) was obtained by condensing NH_3 (g) into a Schlenk tube at -78 °C (dry ice/acetone cold bath) under N_2 (g). To this was added in slight excess (slightly greater than 2:1 molar ratio of $SOCl_2$:$HgCl_2$). The resulting solution was stirred for ten minutes to facilitate production of the NSO^- ion. The metal dichloride (0.5g, 1.84×10^{-3}mol.)was then added to the solution, an oil bubbler placed in the neck of the Schlenk tube and the N_2 (g) supply turned-off. The solution was stirred for a further 45 minutes and then left to warm-up slowly (ca 12 hours). Residual NH_3 (g) was removed in vaccuo (ca 2 hours), and the product recovered by heating to 30°C in THF for 30 minutes in a water bath. The solution was then filtered through a celite/glass wool plug and the solvent removed in vaccuo. The product was identified by i.r. spectroscopy and mass spectrometry.

RESULTS AND DISCUSSION.

The reaction scheme is illustrated in equations 1. And 2. below.

Equation 1. $SOCl_2$ (l) + NH_3 (l) ⟶ $NH_4Cl + NSO^-$

Equation 2. $2\,NSO^- + HgCl_2$ ⟶ $Hg(NSO)_2 + 2\,Cl^-$

(NB. Little is known about the stoichiometry of these reactions).

By i..r. and mass spectrometry, the $HgCl_2$ was found to have been converted to $Hg(NSO)_2$. The i.r. spectrum of the product showing bands characteristic of NSO compounds at 1261 cm^{-1} (vs)and 1070 cm^{-1}(s). EI mass spectrometry gave a spectrum identical to that which was theoretically predicted. It was not possible to obtain a Raman spectrum of the product due to it's very oily nature. The i.r. and mass spectra are shown in figures 1 and 2 respectively.

FIGURE 1..I.R.SPECTRUM OF $Hg(NSO)_2$.

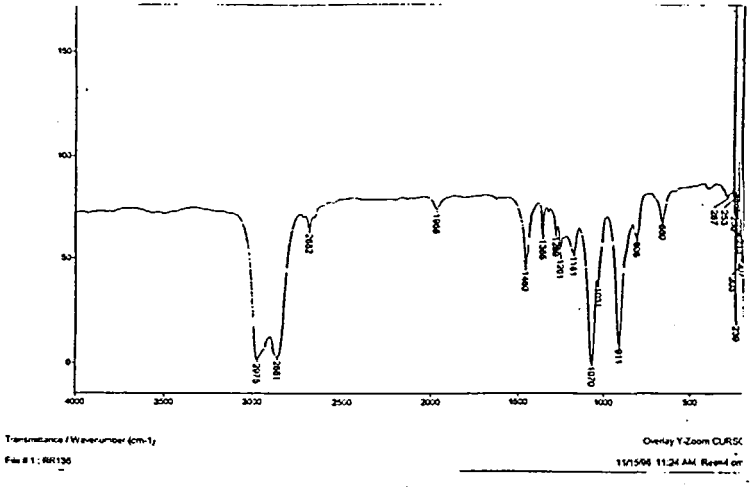
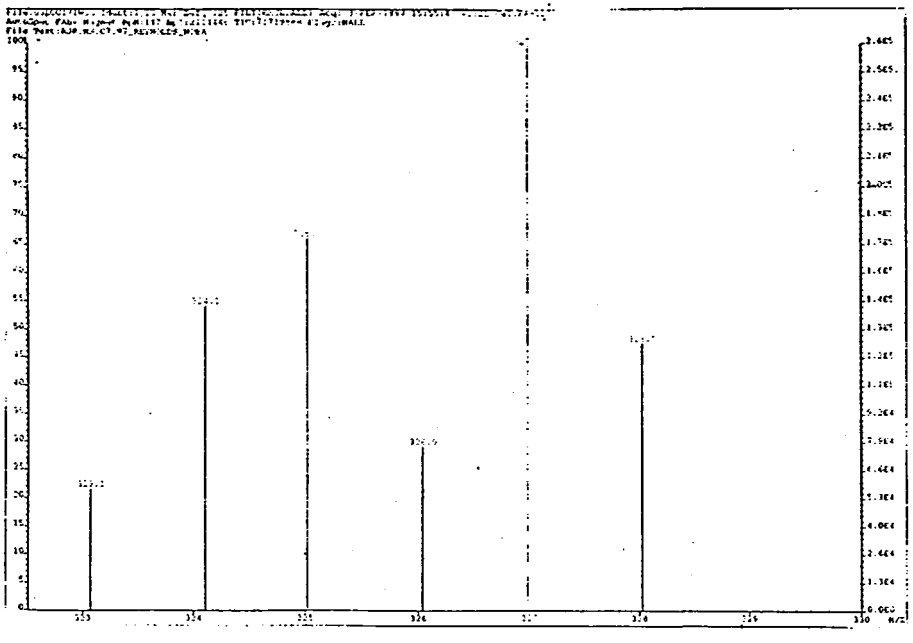


FIGURE 2. MASS SPECTRUM OF $Hg(NSO)_2$



Synthesis of $Hg(NSO)_2$ in liquid ammonia provides a simple alternative method of synthesis to the previously used "wet" chemistry procedure.

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