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The reaction of copper hydride with phosphorus trichloride and silicon tetrachloride

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A report that Cu(I) hydride reacts with benzoyl chloride to give benzaldehyde[1] prompted our study of the reaction of some inorganic chlorides with this metal hydride.

The extremely vigorous reaction between these chlorides and Cu(I) hydride can be moderated by using carbon tetrachloride as a solvent for the halide.

EXPERIMENTAL

Cu(I) hydride was prepared by reduction of copper sulphate solution with hypophosphorous acid at 40°C. The hydride was dried by washing with actone followed by oven drying at 40°C for 1 hr. Phosphorus trichloride and silicon tetrachloride were freshly distilled. Carbon tetrachloride used as solvent was dried over magnesium sulphate followed by phosphorus pentoxide and then distilled.

Reactions were carried out under nitrogen in standard glass apparatus with ground glass joints. Gaseous products were examined by IR spectroscopy.

RESULTS AND DISCUSSION

Reaction of copper hydride with phosphorus trichloride

Phosphorus trichloride added to copper hydride in the presence of air reacted vigorously with flashes of yellow flame on each addition of the halide.

The vigorous nature of the reaction was moderated by dissolving the phosphorus trichloride in dry carbon tetrachloride.

In a typical experiment 1.7 g of copper hydride reacted under dry nitrogen with a solution of 1.3 g phosphorus trichloride in $6\,\mathrm{cm}^3$ of carbon tetrachloride. A steady evolution of gas resulted. Examination by IR spectroscopy indicated the presence of hydrogen chloride and smaller amounts of thionyl chloride. The hydrogen chloride appeared to be the major product. We did not detect phosphine.

Passing the gaseous products through dilute silver nitrate solution gave no coloration suggesting the presence of a reducing gas such as phosphine.

Reaction of copper hydride with silicon tetrachloride

Without a moderating solvent the reaction was very vigorous. In a typical experiment 2.3 g of copper hydride was reacted with silicon tetrachloride dropwise under nitrogen. Infra-red spectra showed the major gaseous product was hydrogen chloride together with a smaller amount of thionyl chloride.

In an experiment in which carbon tetrachloride $6\,\mathrm{cm}^3$, silicon tetrachloride 0.72 g and copper hydride 1.23 g reacted under nitrogen, the major product was hydrogen chloride and some chloroform was detected.

Copper, cuprous oxide and water have been detected in copper hydride made as above[2]. This work indicates that there must be appreciable quantities of a species containing sulphur and oxygen which can react with the halides used to give thionyl chloride.

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Anation of hexaaquochromium(III) by glycine

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The kinetics of substitution reactions of chromium have a special place from mechanistic point of view; two different pathways are involved—dissociative interchange (I_d) type[1–6] or associative interchange (I_a) type[7, 8]. We have undertaken a systematic study of the kinetics of anation of hexaaquochromium(III) with a variety of amino acids in aqueous solutions. This paper deals with the result of kinetic studies and the discussion of the reaction mechanism in the hexaaquochromium(III)–glycine system.

EXPERIMENTAL

Materials. All compounds were commercially obtained and were of reagent grade quality. Chromium nitrate solution was standardised by an ion-exchange method[5].

Kinetic measurements. The kinetics of the reaction was followed spectrophotometrically by recording changes in absorbance at 540 nm, using a Bausch & Lomb Spectronic 20 spectrophotometer. Pseudo first-order condition was maintained by keeping $[\text{glycine}]_T \geq [\text{hexaaquochromium(III)}]$. The pseudo-first-