

melt which became clear liquid at about 320°. It was believed to be impure hexaphenyldisilane. The benzene extract was concentrated to about 10 ml., and to this was added 20 ml. of petroleum ether (b.p. 60–70°). On cooling to room temperature 2.2 g. (53% based on diphenyldichlorosilane) of white solids melting at 238–244° was obtained by filtration. The crude product was recrystallized three times from a mixture of benzene and ethanol to give 1.2 g. (29%) of colorless needles melting at 260–262° to form a somewhat gelatinous mass which became water-clear liquid at 266° (uncor.).

Anal. Calcd. for $C_{48}H_{40}Si_2$: Si, 12.00. Found: Si, 11.98, 11.84.

Three check runs were made. In two of these runs a product melting at 302–303° was isolated. Analysis showed that it, too, had the composition of octaphenyltrisilane. From molecular model studies we suspected that this inconsistency in the melting points of these two products might possibly be due to the existence of isomeric forms. Further studies are now in progress.

Pentaphenylchlorosilane.—A triphenylsilylpotassium suspension prepared according to the procedure described above was added fairly rapidly to 4 g. (0.016 mole) of diphenyldichlorosilane dissolved in 20 ml. of ether. It was noticed that the first few drops of triphenylsilylpotassium was decolorized immediately when it was added to the diphenyldichlorosilane solution. When addition was completed, a gray suspension was formed. Color Test I¹¹ immediately after the addition was negative. The mixture was stirred 1 hour at room temperature and was filtered by suction. The solvent was distilled from the filtrate to give 7.1 g. of colorless solids melting at 120–140°. The crude product was recrystallized twice from petroleum ether (b.p. 60–70°) to give colorless granular crystals melting at 154–155° (uncor.). The yield of pure product was 3.7 g. (50%).

Anal. Calcd. for $C_{30}H_{26}Si_2Cl$: Si, 11.79; Cl, 7.43. Found: Si, 11.76, 11.81; Cl, 7.42, 7.36.

Pentaphenylchlorosilane is an interesting compound because even though it can be hydrolyzed in aqueous basic solution to give the corresponding hydroxy compound, still, unlike the triphenylchlorosilane, it is quite stable in air. A sample of this compound was put in a vial loosely plugged with cotton. After 3 months there was no depression in its melting point. Its preparation has been checked several times.

From some preliminary work it was found that pentaphenylchlorosilane can be treated with sodium in boiling xylene to give a white solid, presumably decaphenyltetrasilane. Further work is now in progress.

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Phenylphosphine¹

BY ROBERT J. HORVAT AND ARTHUR FURST

Phenylphosphine has been previously prepared by reducing dichlorophenylphosphine directly with anhydrous hydrogen iodide,^{2,3} or by first converting the dichlorophenylphosphine to diiodophenylphosphine hydrogen iodide and treating this with absolute alcohol.⁴ Yields were increased only after many hours of reaction.

It is possible to obtain phenylphosphine in much shorter time and in comparable over-all yields by simply reducing the dichlorophenylphosphine with lithium aluminum hydride.

Experimental

To an ice-cold solution of 4.18 g. (0.11 mole) of lithium aluminum hydride in 200 ml. of absolute ether was added

(1) A grant-in-aid from Research Corporation is gratefully acknowledged; also the technical assistance of O. Clark Chisim.

(2) A. Michaelis, *Ber.*, **7**, 6 (1874).

(3) H. Lecoq, *Bull. soc. chim. Belg.*, **43**, 199 (1933).

(4) H. Kohler and A. Michaelis, *Ber.*, **10**, 807 (1877).

drop by drop 35.5 g. (0.2 mole) of dichlorophenylphosphine (Victor Chemical Works) dissolved in 200 ml. of absolute ether. The reaction mixture turned yellow and soon began to reflux. After the addition was complete the mixture was refluxed for one hour, cooled and filtered through glass wool. In an atmosphere of nitrogen the ether was first removed; then the phenylphosphine was distilled and collected as a colorless liquid at 160–161°. The yield was 5.6 g. (25.4%); hydrogen iodide salt, m.p. 136° (uncor.).

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The Solubility of Aniline Hydrochloride in Water

BY EDWARD H. HOUSE AND JOHN H. WOLFENDEN

Three values for the solubility of aniline hydrochloride are recorded in the literature and regularly quoted in works of reference. Two of these relate to 25° and are in fair agreement; at this temperature C. J. Peddle and W. E. S. Turner¹ report that 100 g. of water dissolves 107.1 g. of the salt while N. V. Sidgwick, P. Pickford and B. H. Wilsdon² report that 100 g. of the saturated solution contains 52.1 g. of the salt. The third solubility determination relates to 15°, at which temperature S. v. Niementowski and J. v. Roszkowski³ report that 100 cc. of water dissolves 17.762 g. of aniline hydrochloride. This last figure is at variance with common observation in the purification of the salt by recrystallization, and suggests a remarkably high temperature coefficient of solubility. The molal heat of solution computed from the recorded solubilities at 15° and 25° implies an absorption of about 29 kcal. of heat, a value much larger than the value of 2.7 kcal. measured by Louguine,⁴ than the heats of solution of other amine hydrochlorides, which commonly range between 1 and 4 kcal. per mole, and than the heat of fusion of the salt as deduced from the cryoscopic data of Leopold⁵ (ca. 2 kcal. per mole).

The paucity of information about the solubility of this common organic compound and the implausibility of the only determination at any temperature other than 25° prompted us to measure the solubility over the temperature range from 0° to 100°. Saturated solutions of the pure recrystallized salt in water containing 0.2% of aniline to repress hydrolysis were analyzed by titration with

TABLE I
SOLUBILITY OF ANILINE HYDROCHLORIDE

Temp., °C.	g./100 g. H ₂ O	Mole fraction
0	63.50	0.08112
15	88.36	.1094
25	107.35	.1299
40	143.7	.1665
100	396	.355

(1) C. J. Peddle and W. E. S. Turner, *J. Chem. Soc.*, **103**, 1202 (1913).

(2) N. V. Sidgwick, P. Pickford and B. H. Wilsdon, *ibid.*, **99**, 1122 (1911).

(3) S. v. Niementowski and J. v. Roszkowski, *Z. physik. Chem.*, **22**, 145 (1897).

(4) Cited by M. Berthelot, *Ann. chim. Phys.*, 6e. ser., **21**, 355 (1890).

(5) G. H. Leopold, *Z. physik. Chem.*, **71**, 59 (1910).