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Short Communication

A new method for the preparation of silicon tetrafluoride

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Silicon tetrafluoride is gaining importance in industrial applications such as "Ocration", *i.e.* the treatment of concrete products with this gas[1]. The conventional method of preparing this gas is by heating a mixture of a metallic fluoride and silica or a silicofluoride with sulphuric acid[2,3]. Attempts have also been made to prepare it by heating silicon tetrachloride and calcium fluoride[4] at 400–500 °C or by heating antimony trifluoride[5]. Silicon tetrafluoride is separated by fractional distillation from fluorochlorosilanes, which are also formed in the reaction.

A suspension of sodium fluoride in acetonitrile has been found to be a good medium for the preparation of several fluorine compounds such as sulphur tetrafluoride[6] or fluorocyclophosphazenes[7]. A new method has now been developed by fluorinating silicon tetrachloride with sodium fluoride in an acetonitrile medium. Silicon tetrachloride does not react with acetonitrile[8].

40 g of sodium fluoride was suspended in 200 cm^3 of dry acetonitrile in a three-necked flask fitted with a mechanical stirrer and an ice-cooled reflux condenser. The preparation unit was well protected from moisture by connecting appropriate guard tubes or cold traps. All operations were carried out in a slow stream of nitrogen. To the gently refluxing acetonitrile was added 10 cm^3 of silicon tetrachloride over a period of 30 min by means of a dropping funnel whose end dipped into the suspension. Bubbles of gas (silicon tetrafluoride) were evolved and were swept into two traps maintained at −40 °C and −187 °C, respectively. Unreacted silicon tetrachloride was removed in the first trap, while silicon tetrafluoride was collected in the second. The solvent was allowed to reflux for a further period of 30 min following which time the second trap was warmed to −60 °C when silicon tetrafluoride sublimed into a dry glass evacuated globe. The amount of gas collected was 1600 cm^3 at N.T.P., equivalent to an 80% yield. The purity was examined by IR spectroscopic analysis and shown to be 99.9%. Strictly anhydrous conditions were maintained throughout.

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