

TABLE I  
SOLUBILITY OF SILICON TETRAFLUORIDE AT ROOM TEMPERATURE (27–33°) AND TOTAL PRESSURE OF 750–760 MM.

Solvent	G. SiF <sub>4</sub> /100 cc. solvent	Wt. % of SiF <sub>4</sub> in soln.
Methyl alcohol, absolute	..	32.8
Methyl alcohol, 94% by wt.	..	39.0
Ethyl alcohol, absolute	57.2	36.4
Ethyl alcohol, 96.1% by wt.	60.8	37.8
Ethyl alcohol, 95% (U. S. P.)	61.4	38.1
Ethyl alcohol, 94.3% by wt.	61.5	38.1
Ethyl alcohol, 92.6% by wt.	63.4	38.8
Ethyl alcohol, 91.0% by wt.	63.9	39.0
Isopropyl alcohol, 98%	39.4	28.2
n-Butyl alcohol, 98% (U. S. P.)	30.4	23.4
Amyl alcohols (fusel oil)	20.9	17.3
Glycol, c. p.	..	26.2
Diethylene glycol, c. p.	..	17.6
Glycerol, c. p.	..	5.7
Acetone, c. p. anhyd.	3.2	3.1
Acetic acid (glacial)	1.1	1.1
Pyruvic acid, tech. (38–45%)	..	4.4

In addition to these quantitative data, it was found that silicon tetrafluoride is sparingly soluble in benzene and trichloroethylene, and insoluble in carbon tetrachloride at room temperature.

On the addition of 9% by weight of water to the saturated solution in 95% alcohol (U. S. P.), a gelatinous precipitate of hydrated silica was formed. However, when the original solution was subjected to vacuum distillation at room temperature, silicon tetrafluoride was evolved until a solution containing approximately 26.5% of the gas remained, which corresponds to a mole ratio of alcohol to silicon tetrafluoride of approximately 6:1. On adding an equal volume of water to the residual solution from the vacuum distillation, no precipitate formed, possibly because the silicon tetrafluoride had combined chemically with the ethyl alcohol. Subsequent addition of a soluble barium salt precipitated 95% of the fluorine as pure barium fluosilicate. This reaction offers a convenient method for preparing barium fluosilicate of high purity.

When saturated solutions in glycol were distilled in a vacuum at room temperature, 50 and 100°, the residual solutions contained fluorine equivalent to 26.2, 24.5, and 2.8% silicon tetrafluoride, respectively; on addition of water to them, a precipitate was formed in each case.

The above information is of interest in that such solutions in organic solvents may furnish the basis for the synthesis of organic fluorine compounds as well as a means of preparing pure fluorine salts. However, the authors do not plan

to pursue the investigation further and, therefore, wish to relinquish the problem to others who may be interested in it.

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### Occurrence of the Syringyl Radical in Plant Products

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It has now been shown that treatment of jute fiber, rye straw and corn stalks with a 2% solution of hydrogen chloride in anhydrous ethanol, according to the method described for the ethanolysis of spruce and maple woods,<sup>1</sup> gives rise to typical ethanolysis products, namely, ethanol lignin and a mixture of volatile oils. The yields of ethanol lignin and volatile oils are of the same order of magnitude as those obtained from spruce and maple woods. The presence of the syringyl radical has been established in the volatile oils from each of these fibers. Thus, following the procedures previously described,<sup>2</sup>  $\alpha$ -ethoxypropiosyringone as the *p*-nitrobenzoate (m. p. 141–142°), syringoylacetalddehyde as the mono-semicarbazone (m. p. 207–208°), and syringaldehyde as the 2,4-dinitrophenylhydrazone (m. p. 235–235.5°), were isolated. Mixed melting points with authentic specimens showed no depression.

These, and previous results with hard woods,<sup>1,2</sup> would seem to point to the presence of syringyl derivatives in the lignin constituents of all angiosperms, for example as indicated in corn and rye (monocotyledons) and in jute and maple (dicotyledons).

(1) Brickman, Pyle, McCarthy and Hibbert, *THIS JOURNAL*, **61**, 868 (1939).

(2) Hunter, Cramer and Hibbert, *ibid.*, **61**, 516 (1939); Pyle, Brickman and Hibbert, *ibid.*, **61**, 2198 (1939).

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### Reaction of *p*-Fluorophenol with Benzene and Aluminum Chloride

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Recently it was found<sup>2</sup> that in the deethylation of *p*-fluorophenetole with aluminum chloride in benzene solution a by-product was obtained in

(1) Sharp and Dohme Post-Doctorate Fellow 1938–1939.

(2) Suter, Lawson and Smith, *THIS JOURNAL*, **61**, 163 (1939).