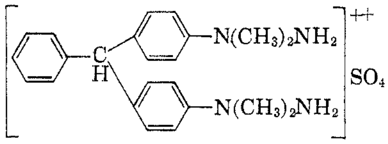
trates were evaporated to dryness and the residue shaken with a 50/50 (by volume) mixture of water and ether. The aqueous layer was treated with Norit, and evaporated to dryness. The residue was taken up in isopropyl alcohol and reprecipitated with ethyl acetate. Weight of product: 2 g. (24% yield) m.p. (after recrystallization) 193–194°.

Anal.[4] Calcd. for



C, 60.24%; H, 6.59%; N, 12.22%; S, 6.99%. Found: C, 56.69%; H, 6.20%; N, 11.76%; S, 6.97%.

The chloroplatinate salt was prepared.

Anal.[4] Calcd. for $[C_{23}H_{30}N_4PtCl_6]$: C, 35.85%; H, 3.92%; N, 7.27%; Cl, 27.61%; Pt, 25.33%. Found: C, 36.27%; H, 4.17%; N, 7.01%; Cl, 24.19%; Pt, 25.24%.

Reaction of sodium hydroxylamine-O-sulfonate with dimethylaniline. To a methanolic solution of 5.6 g. hydroxylamine-*O*-sulfonic acid was added 6.2 g. sodium carbonate monohydrate. Carbon dioxide was immediately evolved. Dimethylaniline (6 g.) was added to the suspension of the sodium salt. The mixture was heated for 30 min. and filtered hot to remove inorganic salts. Addition of chloroform to the methanolic solution caused gradual precipitation of a dark colored product. Recrystallization of the dark material from ethanol–diethyl ether acidified with sulfuric acid produced 4.5 g. of pink platelets identical to those obtained by reaction of the free acid with the amine (38.5% yield).

General characteristics of the hydrazinium sulfates. The quaternized hydrazinium sulfates are colorless crystalline compounds, very soluble in water and generally insoluble in organic solvents. Those prepared from aminoalcohols are very hygroscopic and difficult to crystallize. The products from *N*-(2-hydroxyethyl) pyrrolidine and triethanolamine resisted all attempts at crystallization and were obtained only as viscous oils.

It was shown that hydroxylamine-*O*-sulfonic acid also reacts with *N,N*-diethyl cyclohexylamine, 3-dimethylamino propylamine, tri-*n*-heptylamine, *N,N*-dimethyl dodecylamine, but the hydrazinium salts were not isolated.

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New Simple Preparation of Hexaphenylcyclotrisiloxane

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Hexaphenylcyclotrisiloxane (1) has usually been prepared by the method of Burkhard[1] which involves condensation of diphenylsilanediol in the presence of hydrochloric acid as a catalyst.

In separate papers the author has reported two methods[2] for the preparation of I directly from diphenyldichlorosilane. It is found now that I can be obtained easily from the reaction of diphenyldichlorosilane with ammonium thiocyanate dissolved in acetone.

Stoichiometry supports the following equation:

$$(C_6H_5)_2SiCl_2 + 2NH_4CNS + H_2O \longrightarrow {}^1/_3[(C_6H_5)_2SiO]_3 + 2NH_4Cl + 2HCNS$$

Although the details of reaction mechanism are not yet clear, the acidity of by-products including ammonium chloride would cause the formation of trimer in good yields.

EXPERIMENTAL

Reagents. Ammonium thiocyanate was purified by recrystallization from methanol followed by drying in vacuum. Highest purity diphenyldichlorosilane was received from the Shin-etsu Chemical Industrial Company. Reagent grade acetone was used without further purification.

Preparation of trimer. In a 2 liter, 3-necked flask equipped with reflux condenser and dropping funnel in which 150 g. of diphenyldichlorosilane dissolved in 200 ml. of acetone had been placed, was placed 95 g. of powdered ammonium thiocyanate and 1000 ml. of acetone. When ammonium thiocyanate was dissolved by heating on a water bath, addition of silane solution portionwise immediately precipitated ammonium chloride as a fine white powder. After the addition was complete, the mixture was heated 10 min. further under reflux. After cooling, the product was filtered rapidly under suction and the precipitate was washed with a small amount of fresh acetone; 62 g. (99%) of ammonium chloride was obtained after drying. The filtrate then was transferred to a separate flask and was refluxed gently for about 1 hr. after the addition of 20 ml. of distilled water. The yellowish transparent product was transferred in a clean crystallizing dish and was concentrated to about 100 ml. on a water bath; a yellowish crystalline mass mixed with oily product having a pungent odor was obtained upon cooling. The crystalline mass was rinsed several times with a small amount of methanol and was recrystallized from ethanol-benzene (1:1); crude trimer was obtained as white shiny platelets melting at 187–188°. Further purification was effected by recrystallization from purified ethyl acetate; 108 g. (92%) of pure trimer was obtained as elongated hexagonal plates melting at 189.6°.

Anal. Calcd. for $C_{36}H_{30}Si_3O_3$: Si, 14.16; C, 72.68; H, 5.09; mol. wt., 594. Found: Si, 14.1; C, 72.5; H, 4.9; mol. wt., 588 (benzene), 579 (camphor).

The x-ray powder pattern data were in complete agreement with those given by Hyde and coworkers.[3]

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