April 1970

Brief 70-10105

NASA TECH BRIEF

Improved Process for Synthesizing Anilinosilane Compounds

A new process has been developed for producing good yields of anilinosilane compounds that can be readily isolated in a high state of purity. A variety of di- and tri-anilinosilanes, including diphenyl-, dimethyl-, methylphenyl-, methylvinyl-, phenylvinyl-, and methylallyl-dianilinosilanes and trianilinophenylsilane, were successfully prepared by this process. The key to the process is the use of s-collidine (2,4,6-trimethylpyridine) as an HCl acceptor. The silane compounds are of interest in that they can be melt-condensed with aromatic diols to provide a wide variety of high molecular weight polyaryloxysilane materials that are of potential importance in polymer technology.

Previously reported syntheses of anilinosilanes require the condensation of the corresponding chlorosilane with excess aniline, which functions as an HCl acceptor. These reactions are reversible and provide generally low, nonreproducible product yields which are difficult or impossible to isolate in the pure state. In the new process, it is believed that s-collidine plays a unique role as an HCl acceptor in that it is a stronger base than the aniline, with which it competes, and because it apparently forms sterically hindered HCl salts that cannot reversibly interact with the anilinosilanes. As a result, the reaction mixtures are relatively free from side products, greatly facilitating isolation of highly pure anilinosilanes by crystallization and/or vacuum distillation. These compounds have been prepared in yields ranging from 50 to 90 percent and in a high state of purity as determined from their melting and boiling points and elemental analysis.

The new process is carried out by condensing stoichiometric amounts of aniline with the appropriate chlorosilane in tetrahydrofuran in the presence of s-collidine. The insoluble s-collidine hydrochloride salts are removed from the reaction mixture by filtration, the filtrates are concentrated, and the pure anilinosilanes are isolated from the concentrates by either crystallization or vacuum distillation.

Note:

No further documentation is available. Inquiries may be directed to:

Technology Utilization Officer
Marshall Space Flight Center
Huntsville, Alabama 35812
Reference: B70-10105

Patent status:

Inquiries about obtaining rights for the commercial use of this invention may be made to NASA, Code GP, Washington, D.C. 20546.

Source: W. R. Dunnavant and R. A. Markle of
Battelle Memorial Institute
under contract to
Marshall Space Flight Center
(MFS-14948)

Category 04