Intercalation of Antimony Pentafluoride in the Lattice of Graphite

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Summary Antimony pentafluoride can be intercalated in the lattice of graphite by heating; the resulting product shows much improved resistance to hydrolysis and is easier to handle.

The intercalation of metals and salts in the lattice of graphite is well known.[1] Salts most easily intercalated are chlorides of transition metals such as $FeCl_3$ or $CoCl_2$. There are few instances of intercalation of fluorides, and fluorides of transition metals are reported as not being intercalated by standard techniques.[2] In contrast to this, we have found that SbF_5 can be intercalated easily in the lattice of graphite simply by heating a mixture of SbF_5 and graphite at 110° for a few days.

Differential thermal analysis (DTA) and *X*-ray powder diffraction show clearly that the fluoride is intercalated in the lattice. The DTA produces a sharp inflection at 492 °C and the *X*-ray pattern is typical of an expanded lattice, the strong band for pure graphite at 3·35 Å being almost completely eliminated by the intercalation and a new band appearing at 11·10 Å.

Elemental analysis of the intercalates gave a ratio of 5:1 for F:Sb, showing that SbF_5 itself is intercalated and that there is no appreciable reaction with the Pyrex flasks to give SbF_3 and the volatile SiF_4. After intercalation no antimony salt could be extracted from the intercalate, even with HCl–HNO_3. After degradation of the graphite lattice by H_2SO_4, the antimony salt could be recovered.

This technique gives a black glossy powder which remains very free-flowing and non-sticky with as much as 75% SbF_5 incorporated. The resistance of SbF_5 to hydrolysis is much increased by intercalation. A sample containing 35% SbF_5 was exposed to moist air for up to 15 min without decomposition. Under similar conditions, SbF_5 hydrolyses very rapidly. The 5% intercalate gave a detectable amount of HF only after 50 min exposure to moist air, while pure SbF_5 evolved HF immediately. Preliminary tests also indicate that the intercalated antimony pentafluoride is a milder agent for exchange of halogens with organic chlorides. This is in accordance with the pattern observed for the reactions of intercalated species such as CrO_3.[3] Consequently, intercalation of SbF_5 gives a much improved reagent for fluorination, in terms of control of the reaction and simplification of the handling of the fluorinating agent.

The graphite used for the intercalation (Fisher Reagent) was thoroughly dried by heating *in vacuo* at 150°, for at least 24 h. All operations were performed under dry nitrogen, in a dry box.

Various concentrations of SbF_5 in graphite were thus obtained: 5, 10, 20, 30, 50, and 75%. Up to 75% of SbF_5, the material remains dry in appearance and the SbF_5 appears entirely intercalated. With higher concentrations, some of the SbF_5 remains on the surface of the graphite and the material becomes sticky.

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