

BRIEF
COMMUNICATIONS

Synthesis of High-Purity Tris(trimethylsilyl) Borate

M. G. Voronkov, S. V. Basenko, R. G. Mirskov, and S. N. Adamovich

*Favorskii Institute of Chemistry, Siberian Division, Russian Academy of Sciences,
Irkutsk, Russia*

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Abstract—A method was developed for synthesis of high-purity tris(trimethylsilyl) borate by reaction of trimethylacetoxysilane with powdered boric acid.

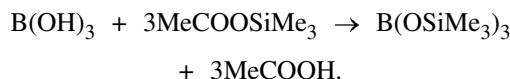
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Tris(trimethylsilyl) borate $B(OSiMe_3)_3$ (TMSB) is used as a monomer and comonomer in production of polyorganyl borosiloxane caoutchoucs for improving their technical parameters, as a catalyst for polymerization processes, and as an adsorbent of neutrons. It is recommended as a means of protection of surfaces subjected to action of an electric arc and as a half-product in synthesis of silicon-and-boron-containing compounds [1]. High-purity TMSB has been suggested by the authors for use in the microelectronic technology for plasmochemical deposition of thin layers of boron-doped silicon dioxide, with a boron content of 1.5 to 10 wt % [2].

Previously, TMSB has been synthesized in a 20% yield by heating hexamethyldisiloxane with B_2O_3 in an autoclave at 350°C or by boiling trimethylchlorosilane with H_3BO_3 (yield 26.5%) [3].

The yield of TMSB could be raised to 81% on heating hexamethyldisilthiane with boric acid to 120°C [4]. However, use of the difficultly accessible and foul-smelling hexamethyldisilthiane is unfeasible for industry. The reaction of hexamethyldisilazane with ammonium borate in a solvent [5] or with boric acid at 110–120°C in the course of 10 h [6] produced TMSB in 40 and 72% yields, respectively.

To be used in micro- and optoelectronics, TMSB should be of special purity. This forced the authors to develop a method for synthesis of high-purity TMSB by heating trimethylacetoxysilane with powdered boric acid of chemically pure grade at 110°C:



A mixture of 20 g (0.15 mol) of trimethylacetoxysilane and 3 g (0.05 mol) of finely ground boric acid of chemically pure grade was heated at 110°C for 1 h. After acetic acid was evaporated (8.4 g, 97%), the residue was distilled in a vacuum. The yield of TMSB was 7.95 g (95%), bp 70°C (19 mm Hg), $n_d^{20} = 1.3865$, in agreement with published data [3]: bp 48°C (5 mm Hg), $n_d^{20} = 1.3860$.

According to GLC data, TMSB purified by rectification in a vacuum on a quartz column is free of even trace amounts of organic impurities. According to the results of an atomic-absorption analysis, the content of metals (Na, K, Ca, Fe, Ni, Co, etc.) does not exceed 10^{-5} – 10^{-6} wt % for each of the metals.

CONCLUSION

A method for synthesis of high-purity tris(trimethylsilyl) borate for micro- and optoelectronics in a 92% yield was developed. The method is based on the reaction of trimethylacetoxysilane with powdered boric acid of chemically pure grade at 110°C for 1 h, with the subsequent purification of the product by rectification in a vacuum.

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