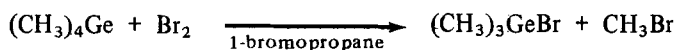


Chapter Six

GERMANIUM HYDRIDE DERIVATIVES

27. BROMOTRIMETHYLGEMANE



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There is a variety of reported routes to the halotrimethylgermanes.¹⁻⁴ These procedures have caused considerable difficulty in that they are often irreproducible, require long reaction times, involve sealed-tube reactions that are difficult to perform on a large scale, or produce only fair yields of products. The following procedure, a modification of one used by Mironov and Kravchenko,⁵ circumvents these problems.

Procedure

■ **Caution.** *All operations should be carried out in a well-ventilated area because of the toxicity of bromine and the germane derivatives.*

Bromine (35 g, 11.3 mL, 0.22 mole) is added dropwise over a period of 1 hour to 26.4 g (0.20 mole) of tetramethylgermane⁶ mixed with 15 mL of 1-bromopropane in a flame-dried 250-mL, three-necked flask, equipped with a magnetic stirrer,

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a stoppered pressure-equalizing dropping funnel, thermometer, and water-cooled reflux condenser with CaCl_2 drying tube at the top. The resulting mixture is heated to reflux for 16 hours, during which time the pot temperature rises to $80\text{--}90^\circ$. After cooling to room temperature, approximately 5 mL of mercury is added, with stirring, to remove the excess bromine. The clear solution is decanted into a 100-mL flask. Fractional distillation through a Vigreux column yields 25-31 g (65-80%) of pure bromotrimethylgermane, bp $112\text{--}113^\circ$.

Properties

Bromotrimethylgermane is a water-sensitive, clear liquid, the density of which is 1.544 g/mL at 18° .¹ It is generally soluble in organic liquids and its ^1H NMR spectrum in CCl_4 has a singlet at $\delta 0.84$.

References

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28. DIMETHYLGERMANE AND MONOHALODIMETHYLGERMANES

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The germanium-halogen bond is particularly labile and has played an important role in germanium chemistry. Synthetic routes to the fully substituted halo-

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