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SHORT COMMUNICATION

Bis(trimethylgermyl)mercury

We describe here the preparation of bis(trimethylgermyl)mercury[1] by two methods: (*a*) the reaction of bromotrimethylgermane with sodium amalgam [analogous to the preparation of bis(trimethylsilyl)mercury[2]], and (*b*) the reaction of trimethylgermane with diethylmercury [analogous to the preparation of bis(triethylgermyl)mercury[3]]. We also report some physical properties of the compound.

Experimental

Preparation. (*a*) A mixture of bromotrimethylgermane (9.7 g), cyclohexane (20 ml), and 0.5% sodium amalgam (230 g) was shaken under dry nitrogen for 1 week after the first appearance of a yellow colour. The cyclohexane solution was filtered through a sintered glass disc under nitrogen, the solvent was removed by evaporation, and the residue sublimed ($83°/10^{-2}$ mm) to give bis(trimethylgermyl)mercury (7.5 g, 35%).

(*b*) Diethylmercury (2.6 g, 10 mmoles) and trimethylgermane (1.06 g, 8.9 mmoles) were placed in a reaction flask fitted with a cold finger and connected to a gas burette. The flask was flushed with argon, then kept at 60° for 11 h during which time the volume of gas produced was a little in excess of that of the expected amount

TABLE 1

MASS AND ABUNDANCE DATA FOR $HgGe_2$

Nominal mass	*multiplicity*	*Spread (ppm)*	*Peak mass (arith. mean)*	*Relative abundance*
336	singlet		335.814374	0.0379
337	no combination			
338	2	10	337.815191	2.7036
339	2	9.9	338.816787	4.4025
340	4	22	339.814630	13.1511
341	4	23	340.815200	17.1103
342	8	31	341.814415	41.1532
343	6	18	342.814052	39.6882
344	11	40	343.813873	76.2526
345	9	22	344.813543	58.3575
346	11	32	345.813527	100.0000
347	9	30	346.813039	55.4855
348	11	30	347.813391	87.3569
349	6	22	348.813488	34.0370
350	8	31	349.813501	51.5583
351	4	20	350.814533	9.8258
352	4	16	351.814168	18.6086
353	2	15	352.815668	0.9980
354	2	7.5	353.815154	3.4848
355	no combination			
356	singlet		355.816201	0.2534

of ethane. Unreacted diethylmercury and volatile products were removed under vacuum, and the residue was sublimed (60°/5 × 10^{-3} mm).

Properties. Bis(trimethylgermyl)mercury forms highly-refracting yellow crystals of m.p. 120–122° (*in vacuo*). The results of carbon and hydrogen analyses suggest that we have never obtained the compound wholly free of mercury, which sublimes with it (*e.g.* Found: C, 15.8; H, 4.20. $C_6H_{18}Ge_2Hg$ calcd.: C, 16.5; H, 4.16%.)

The compound decomposes rapidly to mercury and hexamethyldigermoxane on exposure to air. It is soluble in hydrocarbon solvents, in which it decomposes at rather lower temperatures than bis(trimethylsilyl)mercury[4].

The NMR spectrum at 60 Mc in benzene shows a singlet for the methyl protons at $\tau = 9.53$ ppm. (The benzene was used as reference).

The mass spectrum (A.E.I. M.S.9. mass spectrometer) at 25 eV and an inlet temperature of 34° showed well defined molecular ions $[(Me_3Ge)_2Hg]^+$ and ions arising from methyl loss, *i.e.* $Me_3GeHgGeMe_2^+$. For both sets the isotopic abundances were written 1% of the calculated[5] values. (Mass and abundance data for

TABLE 2

ISOTOPE PATTERN FOR MOLECULAR IONS, $[(Me_3Ge)_2Hg]^+$

Nominal mass	*Relative abundance*	*Nominal mass*	*Relative abundance*	*Nominal mass*	*Relative abundance*
426	0.0364	434	75.9080	442	18.6111
427	0.0025	435	61.0934	443	2.1875
428	2.5972	436	100.0000	444	3.4472
429	4.4050	437	59.9141	445	0.2285
430	12.9246	438	87.7123	446	0.2499
431	17.3001	439	38.4825	447	0.0165
432	40.6700	440	51.9048	448	0.0005
433	40.8340	441	12.8562		

$HgGe_2$ are shown in Table 1, and the calculated isotope pattern for $[(Me_3Ge)_2Hg]^+$, including ^{13}C and ^2H contributions, is shown in Table 2). The ions $Me_6Ge_2^+$ and fragments derived from it were also present, together with Hg^+ ions.

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