The enhanced stability of this compound,[8] probably due to the additional π-type interaction, is clearly demonstrated by the temperature dependence of the 60-MHz nmr spectra shown in Figure 3. Rapid exchange on the nmr time scale does not occur until a temperature of $\sim 70°$ is reached, which implies that the exchange process in this system proceeds with a higher activation energy than that observed for any other system studied so far.

Further study of this system should lead to a better understanding of cyclopropyl π-type interactions and the nature of the electron-deficient bridge bond. To this end the exact analysis of the nmr spectrum is continuing and the crystal structure and optical spectra are currently under investigation.

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Comment on the Thermal Decomposition of Triphenylthallium

Sir:

In a recent interesting publication[1] concerned with thallium in organic synthesis is the statement: "Contrary to the suggestion of Gilman and Jones, we have shown that triphenylthallium does *not* disproportionate to biphenyl and thallium."[2] In the earlier experiments described by us[3] we obtained biphenyl when triphenylthallium dissolved in xylene was refluxed under nitrogen. Also, in two check experiments, a solution of triphenylthallium in xylene was refluxed while a slow stream of dry carbon dioxide was admitted over the surface of the boiling solution. Subsequent to cooling and hydrolysis, there was obtained biphenyl and benzoic acid. It may be relevant to remark that Birch[4] stated after heating what was presumably only a small quantity of solid triphenylthallium: "When heated it decomposes suddenly to give metallic thallium and a vapor possessing an odor of biphenyl."

We now confirm that biphenyl is indeed formed by thermal decomposition of triphenylthallium under our reported conditions which are more vigorous than those employed in ref 1. Thus, biphenyl is formed by thermal decomposition in xylene of freshly prepared triphenylthallium. These experiments were carried out with newly prepared materials. As in our earlier work the identity of the biphenyl was confirmed by the method of mixture melting points with an authentic specimen, and in the present work also by comparison of infrared spectra.

The following item may have some significance. An interesting and important aspect of the coupling reactions[1] is that they take place in the presence of excess TlBr (see footnote 4 of ref 1). In the reference to our experiment in which some biphenyl was obtained when phenyllithium was added to thallous chloride at $-15°$ (see ref 3) it is of interest that the TlCl was present in excess because the phenyllithium was added to it slowly. Ordinarily, in the preparation of organothallium compounds, the thallous chloride or bromide is added slowly to the RMgX or RLi compound so that thallous halide is not present in excess. This suggests that a key step in the coupling reaction may be expressed in eq 2 of ref 1.

$$RTl + 2TlBr \longrightarrow RTlBr_2 + 2Tl$$

There would then be little opportunity for this reaction to take place when TlBr is added to excess RMgX or RLi.

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[2] The authors[1] have informed us that this observation was made under the conditions of their coupling reaction, *i.e.*, in refluxing tetrahydrofuran–benzene (1:1), thus indicating that triphenylthallium was not an intermediate in the coupling.

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Synthesis of Novel Substituted (3)-1,7-Dicarbaundecaborate(12) Ions from 1,8-Dicarba-*closo*-undecaborane(11) and Carbanions

Sir:

Recently, the Lewis acid behavior of 1,8-dicarba-*closo*-undecaborane(11)[1-3] and the properties of some of its Lewis base adducts[4] have been reported. We now report the synthesis of a group of analogous ions, including two novel two-cage systems.

The previously described 1,8-$B_9C_2H_{11}$ carborane reacts with 1 mole of methyllithium in diethyl ether–hexane to produce a new $B_9C_2H_{11}CH_3^-$ ion I, which is isolated as its tetramethylammonium salt in 62% yield. Recrystallization from pure acetone yields white air-sensitive crystals. The infrared spectrum of a Nujol mull of I exhibits absorptions at 4.01 (s), 7.73 (w), 9.10 (m), 9.89 (w), 10.24 (m), 10.62 (s), 12.12 (w), and 13.79 μ (s). *Anal.* Calcd for $[(CH_3)_4N][B_9C_2H_{11}CH_3]$: C, 34.40; H, 12.51; B, 46.41; N, 6.63; equiv wt, 209.8. Found: C, 35.02; H, 11.29; B, 46.78; N, 6.57; mol wt (osmometric in acetonitrile), 207.

The 60-Mc/sec ^1H nmr spectrum of the tetramethylammonium salt of I in deuterioacetone exhibited a sharp singlet of intensity 12 at τ 6.64, a broad singlet of intensity 2 at τ 8.55, and a broad singlet of intensity 3 at τ 9.65. The intensity 12 resonance was assigned to the cation methyl protons, and the intensity 2 and 3 resonances were assigned to the carborane protons and to the cage methyl protons, respectively. The 32.1-Mc/sec ^{11}B nmr spectrum of I is complex, with a high-field doublet of intensity 2, a set of overlapping doublets of intensity 3, a singlet of intensity 1, and a low-field set of overlapping doublets of intensity 3. This spectrum, except for the aforementioned singlet, bears a striking resemblance to the spectrum of the previously reported (3)-1,7-$B_9C_2H_{12}^-$ ion,[5] which can now be obtained in

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