The Trans-Configuration in Dimethyltin and Dimethyllead Bis(acetylacetonates)

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Recently, a series of diorganotin bis(8-hydroxyquinolinates) and diorganotin (2,2′-bipyridyl)dichlorides have been characterized by the authors[1] and others[2]; all of these compounds seem to have a chelate structure containing a hexa-coordinated tin atom. It is not clear, however, whether they have a cis- or a trans-configuration with regard to the methyl groups. It seemed that it would be interesting to identify one of the isomers which is of either a cis- or a trans-configuration among similar compounds with a hexa-coordinated tin atom.

We have, therefore, prepared dimethyltin and dimethyllead bis(acetylacetonates). Dimethyltin bis(acetylacetonate). M. p. 177∼178°C. Found: C, 41.03; H, 5.83; Sn, 34.25. Calcd. for $C_{12}H_{20}O_4Sn$: C, 41.54; H, 5.81; Sn, 34.21%. Dimethyllead bis(acetylacetonate). M. p. 163∼163.5°C. Found: C, 33.10; H, 4.58; Pb, 47.53. Calcd. for $C_{12}H_{20}O_4Pb$: C, 33.10; H, 4.60; Pb, 47.58%.

The infrared spectra of these compounds were measured as mulls in Nujol and in hexachlorobutadiene, using a Hitachi EPI-2G grating infrared spectrophotometer for 2∼25 μ and a Leitz infrared spectrophotometer equipped with CsBr optics for 15∼33 μ. Furthermore, the Raman spectrum of dimethyltin bis(acetylacetonate) was measured in the solid state, using a Nippon Yūki-gōsei Raman spectrograph. The absorption bands and the Raman lines in the 300∼600 cm^{-1} region of these spectra are shown in Table I, together with the assignment of the M–C and M–O stretching vibrations. The assignment of the remaining bands will be discussed elsewhere.

The most interesting characteristic of the infrared spectrum of dimethyltin bis(acetylacetonate) is that the only strong antisymmetric stretching vibration of the Sn–C bond was observed at 556 cm^{-1}, while the symmetric one disappeared. On the other hand, the latter was observed at 508 cm^{-1} in the Raman spectrum, as Table I shows. In the infrared

TABLE I. INFRARED AND RAMAN SPECTRA OF DIMETHYLTIN AND DIMETHYLLEAD BIS(ACETYLACETONATES) IN THE REGION OF 300∼600 cm^{-1}

$(CH_3)_2Sn(acac)_2$		$(CH_3)_2\cdot Pb(acac)_2$	
IR	Raman	IR	
576 s	568 w	553 w	
556 s	—	541 s	M–C antisym. str.
—	508 s	—	M–C sym. str.
421 sh	—	—	
406 s	417 m	391 s	M–O str.

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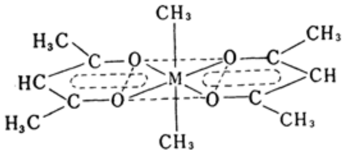
[1] T. Tanaka, M, Komura, Y. Kawasaki and R. Okawara, *J. Organometal. Chem.*, in press.

[2] D. Blake, G. E. Coates and J. M. Tate, *J. Chem. Soc.*, **1961**, 756; D. L. Alleston and A. G. Davies, ibid., **1962**, 2050.

spectrum of dimethyllead bis(acetylacetonate), similarly, the only antisymmetric stretching vibration of the Pb–C bond was found at 541 cm^{-1}. Considering the selection rules of the infrared and the Raman spectra, two methyl groups in these compounds will be in the trans-position with the central tin or lead atom. The absorption bands due to the ligand of the tin or lead chelate are similar to those of copper acetylacetonate,[3] in which four Cu–O bonds are considered to be equivalent. The four Sn–O or Pb–O bonds in these acetylacetonates seem, therefore, to be nearly equivalent. From these facts, the most probable structure of dimethyltin and dimethyllead bis(acetylacetonates) is considered to be:



Trans-dimethylmetal bis(acetylacetonates)
M=Sn, Pb

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[3] K. Nakamoto and A. E.Martell, *J. Chem. Phys.*, **32**, 588 (1960); K. Nakamoto, P. J. McCarthy and A. E. Martell, *J. Am. Chem. Soc.*, **83**, 1272 (1961); M. Mikami, I. Nakagawa and T. Shimanouchi, The 17th Annual Meeting of Japan Chem. Soc., 31X-21 (1964).