

BEDT-TTF is 3.07 (2) Å for O(2)⋯S(22B) (see also Table 4).

One of the authors (MQ) expresses her appreciation to the Alexander von Humboldt Foundation for her scholarship in Germany.

References

- BENO, M. A., COX, D. D., WILLIAMS, J. M. & KWAK, J. F. (1984). *Acta Cryst.* **C40**, 1334–1337.
- GEISER, U., WANG, H. H., WEBB, K. S., FIRESTONE, M. A., BENO, M. A. & WILLIAMS, J. M. (1987). *Acta Cryst.* **C43**, 996–998.
- GLEIZES, A., MAURY, F. & GALY, J. (1980). *Inorg. Chem.* **19**, 2074–2078.
- HALL, S. R. & STEWART, J. M. (1987). Editors. *XTAL2.2 Users Manual*. Univs. of Western Australia, Australia, and Maryland, USA.
- JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- KAWAMOTO, A., TANAKA, J. & TANAKA, M. (1987). *Acta Cryst.* **C43**, 205–207.
- KELLER, E. (1986). *SCHAKAL86. A Fortran Program for the Graphical Representation of Molecular and Crystallographic Models*. Univ. of Freiburg, Germany.
- KOBAYASHI, H., KATO, R., KOBAYASHI, A. & SASAKI, Y. (1985). *Chem. Lett.* p. 191.
- KOBAYASHI, H., KATO, R., MORI, T., KOBAYASHI, A., SASAKI, Y., SAITO, G. & INOKUCHI, H. (1983). *Chem. Lett. Chem. Soc. Jpn.* pp. 759–762.
- KOBAYASHI, H., KOBAYASHI, A., SASAKI, Y., SAITO, G., ENOKI, T. & INOKUCHI, H. (1983). *J. Am. Chem. Soc.* **105**, 297–298.
- LEUNG, P. C. W., BENO, M. A., BLACKMAN, G. S., COUGHLIN, B. R., MIDERSKI, C. A., JOSS, W., CRABTREE, G. W. & WILLIAMS, J. M. (1984). *Acta Cryst.* **C40**, 1331–1334.
- LEUNG, P. C. W., EMGE, T. J., BENO, M. A., WANG, H. H. & WILLIAMS, J. M. (1984). *J. Am. Chem. Soc.* **106**, 7644–7646.
- PARKIN, S. S. P., ENGLER, E. M., SCHUMAKER, R. R., LAGIER, R., LEE, V. Y., SCOTT, J. C. & GREENE, R. L. (1983). *Phys. Rev. Lett.* **50**(4), 270–273.
- PORTER, L. C., WANG, H. H., MILLER, M. M. & WILLIAMS, J. M. (1987). *Acta Cryst.* **C43**, 2201–2203.
- QIAN, M., FU, H. & ZHU, D. (1984). *J. Struct. Chem.* **5**, 127.
- QIAN, M., WANG, X., ZHU, Y., ZHU, D., LI, L., MA, B., DUAN, H. & ZHANG, D. (1988). *Synth. Met.* **27**, A277.
- SHELDRIK, G. M. (1985). *SHELXS86*. In *Crystallographic Computing 3*, edited by G. M. SHELDRIK, C. KRUGER, & R. GODDARD, pp. 175–189. Oxford Univ. Press.
- SHIBAEVA, R. P., KAMINSKII, V. F. & YAGUBSKII, E. B. (1985). *Mol. Cryst. Liq. Cryst.* **119**, 361–373.
- YAGUBSKII, E. B., SHCHEGOLEV, I. F., LAUKHIN, V. N., KONONOVICH, P. A., KARTSOVNIK, M. V., ZVARYKINA, A. V. & BURAVOV, L. I. (1984). *JETP Lett.* **39**, 12.
- ZHU, D., WANG, P., WAN, M., YU, Z. & ZHU, N. (1986). *Solid State Commun.* **57**, 843.

Acta Cryst. (1991). **C47**, 2362–2365

Structure of Dicarbonyl(η^5 -cyclopentadienyl)[(diethyldithiocarbamato)-mercurio](trimethyl phosphite)molybdenum(II)(Mo—Hg)

BY MAREK M. KUBICKI* AND PHILIPPE RICHARD

*Laboratoire de Synthèse et d'Electrosynthèse Organométalliques associé au CNRS (URA 33),
Faculté des Sciences, 6 bd Gabriel 21000 Dijon, France*

AND MICHEL JANNIN

*Laboratoire de Physique du Solide associé au CNRS (URA 785), Faculté des Sciences Mirande,
21000 Dijon, France*

(Received 24 May 1991; accepted 21 June 1991)

Abstract. [(C₁₀H₁₄O₅P)MoHg(C₅H₁₀NS₂)], M_r = 689.99, monoclinic, $P2_1/n$, a = 8.853 (1), b = 9.873 (2), c = 25.231 (4) Å, β = 97.09 (1)°, V = 2188.2 Å³, Z = 4, D_x = 2.094 Mg m⁻³, $\lambda(\text{Mo } K\alpha)$ = 0.71073 Å, μ = 7.277 mm⁻¹, $F(000)$ = 1320, T = 296 (1) K, final R = 0.029 for 2676 observed reflections and 235 parameters. The structure is built of discrete molecules without the intermolecular Hg⋯S interactions which are generally observed in the

crystal structures of compounds containing Hg and S atoms. The Mo—Hg bond length is 2.680 (1) Å and the Mo—Hg—S unit deviates slightly from linearity [170.47 (6)°]. A large Hg—Mo—P angle [131.50 (5)°] is observed.

Introduction. For some years we have been interested in systematic qualitative NMR studies of metallic nuclei in carbonyl cyclopentadienyl complexes involving the molybdenum—mercury bond (Kubicki, Kergoat, Le Gall, Guerchais, Douglade & Mercier,

* To whom correspondence should be addressed.

1982; Kubicki, Le Gall, Pichon, Salaun, Cano & Campo, 1988). The complexes of the type $\text{Cp}'(\text{CO})_2\text{LMoHgX}$ are plausible for such a study by allowing the variation of the natures of the Cp' ligand by different substitutions, of the donor ligand L replacing one CO acceptor and of the ligands X bound to mercury. A number of structural studies have been performed on the RHgSR' compounds (Wong, Carty & Chieh, 1977; Taylor & Carty, 1977; Canty, Raston & White, 1979; Kuz'mina, Bokii, Struchkov, Kravtsov & Rokhlina, 1974) and on the mercury dithiocarbamates (Iwasaki, 1973; Iwasaki, Ito & Kobayashi, 1978; Ito & Iwasaki, 1979; Chieh & Leung, 1976; Tiekink, 1987; Kubicki, Kergoat, Guerchais, Mercier & Douglade, 1981; Kergoat, Kubicki, Guerchais, Norman & Orpen, 1982). The well developed systems of intermolecular $\text{Hg}\cdots\text{S}$ secondary interactions were recognized in these studies. In dithiocarbamate compounds the ligand is bound to mercury in an asymmetrical fashion with one short and one long $\text{Hg}-\text{S}$ intramolecular bond. The long intramolecular interaction varies from one compound to another, so it may be expected that the different chelating forces of the dithiocarbamate ligand can give rise to different electronic structures of the Hg atom. In order to give a more quantitative treatment of metallic NMR resonances we need the accurate geometrical parameters for the complexes studied. The crystal structure of an unsubstituted complex $\text{Cp}(\text{CO})_3\text{MoHg}(\text{dtc})$ (2) [dtc = diethyldithiocarbamate, $\text{S}_2\text{CN}(\text{C}_2\text{H}_5)_2$] has already been determined (Kubicki, Kergoat, Le Gall, Guerchais, Douglade & Mercier, 1982). Here we report the X-ray structure of its substituted derivative $\text{Cp}(\text{CO})_2\text{P}(\text{OMe})_3\text{MoHg}(\text{dtc})$ (1).

Experimental. Complex (1) was synthesized as previously described (Kubicki, Kergoat, Le Gall, Guerchais, Douglade & Mercier, 1982). Crystal was grown from CH_2Cl_2 /heptane solution. Crystal $0.3 \times 0.2 \times 0.2$ mm. CAD-4 diffractometer. Unit cell: 25 reflections, 2θ range $20-32^\circ$. Intensities measured by $\omega-2\theta$ technique, $(\sin\theta)/\lambda_{\text{max}} = 0.61 \text{ \AA}^{-1}$. Ranges of h, k, l : -1 to $11, 0$ to $12, -32$ to 32 , respectively. Three standard reflections monitored every hour: variation on $I = -4.6\%$, thus corrected for linear decay, 3992 unique reflections, 2676 observed with $I > 3\sigma(I)$, $w = [\sigma^2(F) + 0.0023F^2]^{-1}$, $R = 0.029$, $wR = 0.035$, $S = 0.930$. Structure solved by heavy-atom method. H atoms of cyclopentadienyl ring and of ethyl groups calculated after isotropic refinement, those of $\text{P}(\text{OMe})_3$ not located. All non-H atoms refined with anisotropic temperature factors (least squares on F). $(\Delta/\sigma)_{\text{max}} = 0.02$. Peaks in final ΔF map $+0.55$ to $-1.2 e \text{ \AA}^{-3}$ (mostly located in a bimetallic fragment). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974,

Vol. IV). Computing programs: *Structure Determination Package* from Enraf-Nonius (1979) and *SHELX76* (Sheldrick, 1976).

Discussion. Atomic positional parameters and equivalent isotropic temperature factors are listed in Table 1.* Selected bond lengths and angles are given in Table 2. The molecular structure of (1) is shown in Fig. 1 and the packing in the unit cell in Fig. 2.

The structure is built of discrete $\text{Cp}(\text{CO})_2\text{P}(\text{OMe})_3\text{MoHg}(\text{dtc})$ molecules without intermolecular $\text{Hg}\cdots\text{S}$ interactions. The shortest intermolecular distance between Hg and S atoms is $\text{Hg}\cdots\text{S1}'(1-x, 1-y, 1-z) = 6.12 \text{ \AA}$. However, the secondary $\text{Hg}\cdots\text{S}$ interactions seem to be a general property of the solid-state structures of thiolates, dithiocarbamates and xanthates of mercury, and to our knowledge (1) is the first example of such a structure where these interactions are lacking. We think that it may be due to the presence of the bulkier phosphite ligand in (1), which substitutes one of the carbonyl ligands in (2), and to its weaker electron-acceptor property.

The $\text{Cp}(\text{CO})_2\text{PMoHg}$ portion of the molecule has the usual irregular 'four-legged piano-stool' arrangement with the dihedral angle $\text{HgMoP}/\text{C1MoC2}$ equal to $90.7(2)^\circ$. The $\text{Hg}-\text{Mo}-\text{P}$ angle of $131.50(5)^\circ$ is greater than the *trans* $\text{Hg}-\text{Mo}-\text{C}(\text{CO})$ angle [$122.7(9)^\circ$ in (2)], whereas the $\text{C1}-\text{Mo}-\text{C2}$ angle of $105.5(2)^\circ$ is far less obtuse and smaller than in (2) [$113(1)^\circ$]. The $\text{Mo}-\text{Hg}$ bond length of $2.680(1) \text{ \AA}$ in (1) is shorter than in (2) [$2.714(3) \text{ \AA}$] and in the mercury-bridged $[\text{Cp}(\text{CO})_3\text{Mo}]_2\text{Hg}$ complex [$2.746(2) \text{ \AA}$] (Mickiewicz, Raston, White & Wild, 1977), but it is close to the values found in $\text{Cp}'(\text{CO})_2(\text{AsPhMe}_2)\text{MoHgI}$ [$2.685(3) \text{ \AA}$] ($\text{Cp}' = \text{C}_5\text{H}_4\text{Me}$) (Mickiewicz, Raston, White & Wild, 1977), in $\text{Cp}'(\text{CO})_3\text{MoHgCl}$ [$2.680(2) \text{ \AA}$] (Cano, Criado, Gutierrez-Puebla, Monge & Pardo, 1985) and in $\text{Cp}(\text{CO})_3\text{MoHgCl}$ [$2.683(1) \text{ \AA}$] (Bueno & Churchill, 1981). This indicates a higher electron density in the $\text{Mo}-\text{Hg}$ bond in (1) than in (2), as expected on the basis of the donor-acceptor properties of $\text{P}(\text{OMe})_3$ versus CO ligands. The $\text{Hg}-\text{S1}$ bond length of $2.440(2) \text{ \AA}$ is also short. Analogous bonds are $2.510(9) \text{ \AA}$ in (2), $2.50(2) \text{ \AA}$ in $\text{Cp}_2\text{Mo}(\text{Hgdtc})_2$ (Kubicki, Kergoat, Guerchais, Mercier & Douglade, 1981) and fall in the range of $2.51-2.53 \text{ \AA}$ in $\text{Cp}_2\text{Nb}(\text{Hgdtc})_3$ (Kergoat, Kubicki, Guerchais, Norman & Orpen, 1982). On the other hand, the secondary intra-

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, least-squares planes and a complete list of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54375 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic positional parameters and equivalent isotropic temperature factors for (1)

$$B_{iso} = (8\pi^2/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	B _{iso} (Å ²)
Hg	0.50652 (4)	0.26897 (3)	0.53385 (1)	2.906 (9)
Mo	0.61351 (7)	0.25933 (5)	0.43909 (2)	1.94 (2)
P	0.8616 (2)	0.2239 (2)	0.41642 (7)	2.53 (5)
S1	0.3715 (3)	0.2597 (2)	0.61245 (8)	4.20 (6)
S2	0.7086 (3)	0.2427 (2)	0.63641 (9)	3.88 (6)
O1	0.2913 (6)	0.0066 (5)	0.4982 (2)	4.0 (1)
O2	0.7745 (6)	0.5040 (5)	0.5004 (2)	3.9 (1)
O3	0.8756 (6)	0.0792 (5)	0.3896 (2)	3.6 (1)
O4	0.9231 (6)	0.3168 (5)	0.3712 (2)	3.4 (1)
O5	1.0080 (7)	0.2363 (5)	0.4605 (2)	3.9 (2)
N	0.516 (1)	0.2018 (7)	0.7074 (3)	4.8 (2)
C1	0.6754 (8)	0.0969 (6)	0.4800 (3)	2.8 (2)
C2	0.7169 (8)	0.4104 (6)	0.4793 (2)	2.7 (2)
C3	1.0202 (9)	0.0297 (8)	0.3736 (3)	4.5 (2)
C4	0.951 (1)	0.4569 (8)	0.3817 (3)	4.6 (2)
C5	1.013 (1)	0.2138 (8)	0.5145 (3)	3.9 (2)
C6	0.537 (1)	0.2323 (6)	0.6581 (3)	3.0 (2)
C7	0.647 (1)	0.1794 (8)	0.7481 (3)	5.0 (2)
C8	0.691 (1)	0.0326 (8)	0.7525 (3)	5.4 (2)
C9	0.357 (1)	0.169 (1)	0.7225 (4)	7.3 (4)
C10	0.293 (2)	0.303 (2)	0.7383 (5)	10.9 (6)
C11	0.352 (1)	0.2643 (7)	0.4068 (3)	3.6 (2)
C12	0.4181 (9)	0.1522 (8)	0.3841 (3)	3.9 (2)
C13	0.524 (1)	0.198 (1)	0.3524 (3)	4.5 (2)
C14	0.526 (1)	0.340 (1)	0.3550 (3)	4.8 (2)
C15	0.421 (1)	0.3798 (7)	0.3884 (3)	3.9 (2)
Cp†	0.4483	0.2666	0.3773	

† Cp is the gravity center of the C11–C15 cyclopentadienyl ring.

Table 2. Selected bond lengths (Å) and angles (°) for (1)

Hg—Mo	2.6798 (6)	Mo—Hg—S1	170.47 (6)
Hg—S1	2.440 (2)	Mo—Hg—S2	122.20 (5)
Hg—S2	2.970 (2)	Mo—Hg—P	131.50 (5)
Mo—CP	2.002	Hg—Mo—CP	112.9
Mo—C1	1.948 (6)	Hg—Mo—C1	70.3 (2)
Mo—C2	1.965 (6)	Hg—Mo—C2	72.9 (2)
Mo—P	2.363 (2)	P—Mo—CP	115.3
S1—C6	1.771 (9)	P—Mo—C1	78.3 (2)
S2—C6	1.678 (9)	P—Mo—C2	81.4 (2)
N—C6	1.32 (2)	C1—Mo—C2	105.5 (2)
N—C7	1.47 (1)	Hg—S1—C6	94.8 (3)
N—C9	1.54 (2)	S1—C6—S2	119.3 (4)
C7—C8	1.51 (1)	S1—C6—N	116.5 (7)
C9—C10	1.51 (2)	S2—C6—N	124.2 (7)
P—O3	1.592 (5)	C6—N—C7	120.2 (8)
P—O4	1.611 (5)	C6—N—C9	121.5 (7)
P—O5	1.606 (5)	C7—N—C9	117.8 (7)
C1—O1	1.181 (8)	Mo—C1—O1	175.4 (5)
C2—O2	1.154 (8)	Mo—C2—O2	175.9 (5)
O3—C3	1.47 (1)	Mo—P—O3	111.0 (3)
O4—C4	1.423 (9)	Mo—P—O4	119.5 (3)
O5—C5	1.374 (9)	Mo—P—O5	121.1 (2)

molecular weak bond Hg...S2 of 2.970 (2) Å is longer than the corresponding bonds in (2) [2.737 (7) Å], in Cp₂Nb(Hgdtc)₃ [mean value 2.831 (15) Å] and in Hg₂(dtc)₄ [2.662 (6) to 2.698 (6) Å] (Iwasaki, 1973) but similar to the values observed in Cp₂Mo(Hgdtc)₂ [2.940 (3) Å], in Hg(dtC)₂ [2.965 (4) Å] (Iwasaki, 1973; Iwasaki, Ito & Kobayashi, 1978; Ito & Iwasaki, 1979), in MeHg(dtC) [2.964 (7) Å] and in PhHg(dtC) (mean value 2.95 Å) (Tiekink, 1987). This indicates once more that in the presence of the

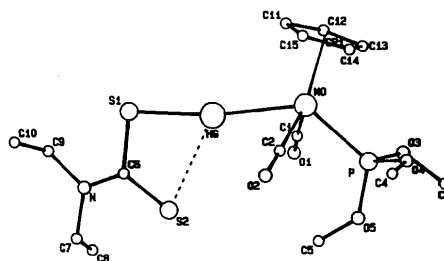


Fig. 1. Molecular structure of (1).

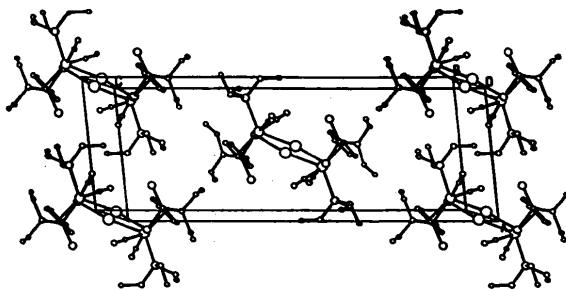


Fig. 2. Packing in the unit cell of (1).

weaker acceptor P(OMe)₃, and with the strong primary Hg—S1 bond the Hg atom does not look for supplementary electron density. The Mo—P bond length of 2.363 (2) Å in (1) is also short. It is shorter than the Mo—P distance of 2.549 (2) Å found in another complex with a molybdenum—main-group metal bond [Cp(NO)(CO)(PPh₃)—MoSnCl₃]⁺ (Ginzburg, Aleksandrov, Struchkov, Setkina & Kursanov, 1980). In this last complex the Sn—Mo—P angle is equal to 139.24 (6) *versus* 131.50 (5)° in (1). Both the Mo—P and M'—Mo—P parameters in (1) are smaller than in the triphenylphosphine molybdenum—tin bonded cation which should be primarily attributed to the smaller value of Tolman's cone angle of P(OMe)₃ (107°) than that of PPh₃ (145°) (Tolman, 1977). The dithiocarbamate ligand is roughly planar with the largest deviations from the best least-squares plane observed for C9 [0.10 (1) Å] and for S2 [0.081 (2) Å].

References

- BUENO, C. & CHURCHILL, M. R. (1981). *Inorg. Chem.* **20**, 2197–2202.
- CANO, M., CRIADO, R., GUTIERREZ-PUEBLA, E., MONGE, A. & PARDO, M. P. (1985). *J. Organomet. Chem.* **292**, 375–383.
- CANTY, A. J., RASTON, C. L. & WHITE, A. H. (1979). *Aust. J. Chem.* **32**, 311–320, 1165–1166.
- CHIEH, C. & LEUNG, L. P. C. (1976). *Can. J. Chem.* **54**, 3077–3084.
- Enraf-Nonius (1979). *Structure Determination Package*. Enraf-Nonius, Delft, The Netherlands.
- GINZBURG, A. G., ALEKSANDROV, G. G., STRUCHKOV, YU. T., SETKINA, V. N. & KURSANOV, D. N. (1980). *J. Organomet. Chem.* **199**, 229–242.
- ITO, M. & IWASAKI, H. (1979). *Acta Cryst.* **B35**, 2720–2721.
- IWASAKI, H. (1973). *Acta Cryst.* **B29**, 2115–2124.

- IWASAKI, H., ITO, M. & KOBAYASHI, K. (1978). *Chem. Lett.* pp. 1399–1402.
- KERGOAT, R., KUBICKI, M. M., GUERCHAI, J. E., NORMAN, N. C. & ORPEN, A. G. (1982). *J. Chem. Soc. Dalton Trans.* pp. 633–638.
- KUBICKI, M. M., KERGOAT, R., GUERCHAI, J. E., MERCIER, R. & DOUGLADE, J. (1981). *J. Cryst. Mol. Struct.* **11**, 43–54.
- KUBICKI, M. M., KERGOAT, R., LE GALL, J. Y., GUERCHAI, J. E., DOUGLADE, J. & MERCIER, R. (1982). *Aust. J. Chem.* **35**, 1543–1554.
- KUBICKI, M. M., LE GALL, J. Y., PICHON, R., SALAUN, J. Y., CANO, M. & CAMPO, J. A. (1988). *J. Organomet. Chem.* **348**, 349–356.
- KUZ'MINA, L. G., BOKII, N. G., STRUCHKOV, YU. T., KRAVTSOV, D. N. & ROKHLINA, E. M. (1974). *Zh. Strukt. Khim.* **15**, 491–496.
- MICKIEWICZ, M. M., RASTON, C. L., WHITE, A. H. & WILD, S. B. (1977). *Aust. J. Chem.* **30**, 1685–1691.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- TAYLOR, N. J. & CARTY, A. J. (1977). *J. Am. Chem. Soc.* **99**, 6143–6145.
- TIEKINK, E. R. T. (1987). *J. Organomet. Chem.* **322**, 1–10.
- TOLMAN, C. A. (1977). *Chem. Rev.* **77**, 313–348.
- WONG, Y. S., CARTY, A. J. & CHIEH, C. (1977). *J. Chem. Soc. Dalton Trans.* pp. 1801–1808.

Acta Cryst. (1991). **C47**, 2365–2367

Structure of 2,2-Bis[3-(dimethylamino)propyl]-1,1,1,3,3,3-hexaphenyltristannane-(2Sn–Sn)

BY D. SCHOLLMAYER, H. HARTUNG,* C. MREFTANI-KLAUS AND K. JURKSCHAT

Fachbereich Chemie, Martin-Luther-Universität Halle-Wittenberg, Postfach, D-0-4010 Halle (Saale), Germany

(Received 14 January 1991; accepted 17 May 1991)

Abstract. $[\text{Sn}_3(\text{C}_6\text{H}_5)_6(\text{C}_5\text{H}_{12}\text{N})_2]$, $\text{C}_{46}\text{H}_{54}\text{N}_2\text{Sn}_3$, $M_r = 991.1$, orthorhombic, *Pbca*, $a = 16.835$ (5), $b = 21.834$ (7), $c = 24.720$ (8) Å, $V = 9086$ (4) Å³, $Z = 8$, $D_x = 1.45$ g cm⁻³, $\lambda(\text{Mo K}\alpha) = 0.71069$ Å, $\mu = 16.8$ cm⁻¹, $F(000) = 3952$, $T = 293$ K, final $R = 0.050$ ($wR = 0.051$) for 3392 observed reflections. The Sn–Sn distances are 2.772 (1) and 2.777 (1) Å and the Sn1–Sn2–Sn3 angle is 111.31 (3)°. There are no intra- or intermolecular Sn–N interactions; all three tin atoms are tetrahedrally coordinated.

Introduction. Since the synthesis of $[(\text{Me}_3\text{Si})_2\text{CH}]_2\text{Sn}$ as the first stable carbon bonded stannylene with coordination number two (Davidson & Lappert, 1973) the chemistry of this class of compounds has attracted more and more interest. In recent papers it has been shown that not only bulky substituents stabilize monomeric tin(II) compounds but also intramolecular donor–acceptor interactions or a combination of both (Angermund, Jonas, Krüger, Latten & Tsay, 1988; Jastrzebski, van der Schaaf, Boersma, van Koten, Zoutberg & Heijdenrijk, 1989; Engelhardt, Jolly, Lappert, Raston & White, 1988; Jurkschat, Klaus, Dargatz, Tzschach, Meunier-Piret & Mahieu, 1989). Within our studies on this subject (Jurkschat *et al.*, 1989) we have prepared $(\text{Me}_2\text{NCH}_2\text{CH}_2\text{CH}_2)_2\text{SnCl}_2$ (1) and performed its reduction with sodium/naphthalene. However, this

reaction did not give, as might be expected, the monomeric stannylene $(\text{Me}_2\text{NCH}_2\text{CH}_2\text{CH}_2)_2\text{Sn}$ (2) but only a mixture of oligomers which have not been isolated and identified. Neumann and co-workers have shown that stannylenes $R_2\text{Sn}$ can also be generated by thermolysis or photolysis of polystannanes (Neumann & Schwarz, 1975). However, analogous investigations on the title compound $(\text{Me}_2\text{NCH}_2\text{CH}_2\text{CH}_2)_2\text{Sn}(\text{SnPh}_3)_2$ (3) did not provide evidence for a stable stannylene (2) or related definite oligomers (Klaus & Jurkschat, 1988). To our knowledge, (3) is only the second known oligostannane containing a functional substituent in the form of the dimethylaminopropyl group (*cf.* the survey of literature referring to this given by Adams & Dräger, 1988). We report here the X-ray analysis of (3) which does not indicate any intramolecular Sn–N interaction. This might explain the above mentioned failure of (3) to give the stannylene (1) by thermolysis or photolysis.

Experimental. The title compound (3) was prepared by reaction of $(\text{Me}_2\text{NCH}_2\text{CH}_2\text{CH}_2)_2\text{Sn}(\text{NEt}_2)_2$ with Ph_3SnH (Klaus & Jurkschat, 1988). The colorless crystals obtained by recrystallization from toluene decomposed slowly by X-ray treatment; therefore, the crystal selected for the diffraction experiments with dimensions $0.28 \times 0.39 \times 0.41$ mm was covered with a thin protective film of shellac and then mounted on a Huber four-circle diffractometer; graphite-

* To whom correspondence should be addressed.