

around the Na atom are listed in Table 2.* The molecular structure is shown in Fig. 1. The molecular packing viewed along the *b* axis, showing atomic numbering and molecular conformation, is depicted in Fig. 2.

Related literature. The structural parameters can be compared with those of 2-sulfanilamido-5-methoxypyrimidine (Giuseppetti, Tadini, Bettinetti & Giordano, 1977), 4-amino-*N*-(4,6-dimethyl-2-pyrimidinyl)benzenesulfonamide (Basak, Mazumdar & Chaudhuri, 1983), tolbutamide (Donaldson, Leary, Ross, Thomas & Smith, 1981) and *p*-

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, bond lengths and bond angles involving H atoms, and deviations from least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55262 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0507]

aminobenzenesulfonacetamide sodium monohydrate (Ghosh, Basak & Mazumdar, 1987).

The authors thank Professor S. K. Mazumdar of Saha Institute of Nuclear Physics, Calcutta, India, for providing the crystal sample of the title compound and also for constant encouragement and helpful discussions.

References

- BASAK, A. K., MAZUMDAR, S. K. & CHAUDHURI, S. (1983). *Acta Cryst.* **C39**, 492–494.
 DONALDSON, J. D., LEARY, J. R., ROSS, S. D., THOMAS, J. K. & SMITH, C. H. (1981). *Acta Cryst.* **B37**, 2245–2248.
 GHOSH, M., BASAK, A. K. & MAZUMDAR, S. K. (1987). *J. Crystallogr. Spectrosc. Res.* **17**, 739–749.
 GIUSEPPE, G., TADINI, G., BETTINETTI, G. P. & GIORDANO, F. (1977). *Cryst. Struct. Commun.* **6**, 263–274.
 SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
 SHELDRICK, G. M. (1986). *SHELX86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.

Acta Cryst. (1992). **C48**, 2023–2025

Bis-Adducts of Trimethylaluminium and Trimethylgallium with *N,N,N',N'*-Tetramethylethylenediamine

BY JAMEICA J. BYERS, WILLIAM T. PENNINGTON* AND GREGORY H. ROBINSON*

Department of Chemistry, Clemson University, Clemson, SC 29634-1905, USA

(Received 17 September 1991; accepted 31 January 1992)

Abstract. μ -(*N,N,N',N'*-Tetramethylethylenediamine)-bis(trimethylaluminium) (*1a*), $[\text{Al}_2(\text{CH}_3)_6(\text{C}_6\text{H}_{16}\text{N}_2)]$, $M_r = 260.38$, monoclinic, $P2_1/n$, $a = 10.719$ (3), $b = 7.648$ (2), $c = 12.224$ (3) Å, $\beta = 110.32$ (2)°, $V = 939.7$ (5) Å³, $Z = 2$, $D_x = 0.92$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, graphite monochromator, $\mu = 1.4$ cm⁻¹, $F(000) = 292$, $T = 294$ K, final $R = 0.0487$ for 1132 observed [$I > 3\sigma(I)$] reflections. The molecule is situated about an inversion center coincident with the midpoint of the C—C bond. The Al—N bond distance is 2.074 (2) Å. μ -(*N,N,N',N'*-Tetramethylethylenediamine)-bis(trimethylgallium) (*1b*), $[\text{Ga}_2(\text{CH}_3)_6(\text{C}_6\text{H}_{16}\text{N}_2)]$, $M_r = 345.86$, monoclinic, $P2_1/n$, $a = 10.735$ (3), $b = 7.656$ (2), $c = 12.310$ (4) Å, $\beta = 110.70$ (2)°, $V = 946.4$ (4) Å³, $Z = 2$, $D_x = 1.21$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, graphite monochromator, $\mu = 28.2$ cm⁻¹, $F(000) = 364$, $T = 294$ K, final $R = 0.0800$ for 909 observed [$I > 3\sigma(I)$] reflections. Compound (*1b*) is

isomorphous with (*1a*). The Ga—N bond distance is 2.174 (8) Å.

Experimental. Data quality crystals of (*1a*) and (*1b*) were prepared by heating (393 K) stoichiometric amounts of the corresponding trimethylmetal derivative with *N,N,N',N'*-tetramethylethylenediamine in heptane, followed by slow cooling of the reaction mixture. All manipulations were carried out under an atmosphere of argon gas. Colorless parallelepiped crystals of (*1a*) (0.2 × 0.5 × 0.7 mm) and (*1b*) (0.1 × 0.3 × 0.5 mm), mounted in sealed glass capillaries under argon, were used for data collection on a Nicolet *R3m/V* diffractometer using $\omega/2\theta$ scans of 2–15° min⁻¹. Unit-cell dimensions were determined by least-squares fit to settings for a number of high-order reflections [34 with $27 < 2\theta < 40^\circ$ for (*1a*) and 46 with $27 < 2\theta < 36^\circ$ for (*1b*)]. Lorentz and polarization corrections were applied. Three standards monitored after every 100 measurements showed $\pm 2\%$ fluctuation for (*1a*) (18.3 h of X-ray exposure;

* Authors to whom correspondence should be addressed.

no correction) and 14% decline for (1b) (14.8 h of X-ray exposure; linear correction applied). Empirical absorption corrections were applied with transmission factors 0.91–1.00 for (1a) and 0.54–1.00 for (1b). For (1a) a total of 1696 reflections were measured ($2\theta_{\max} = 48^\circ$; $-13 \leq h \leq 0$, $-9 \leq k \leq 0$, $-14 \leq l \leq 14$), of which 1476 were unique ($R_{\text{int}} = 0.063$) and 1132 were observed [$I > 3\sigma(I)$]. For (1b) 1434 reflections were measured ($2\theta_{\max} = 45^\circ$; $-12 \leq h \leq 12$, $-9 \leq k \leq 0$, $-14 \leq l \leq 0$), of which 1249 were unique ($R_{\text{int}} = 0.095$) and 909 were observed [$I > 3\sigma(I)$]. The structures were solved by direct methods and refined by full-matrix least-squares methods on F , to convergence ($\Delta_{\max} \leq 0.01\sigma$). Positional and anisotropic thermal parameters for the non-H atoms were refined. H atoms were included in structure factor calculations with a riding model at optimized positions (C—H = 0.96 Å) with group thermal parameters [$U_{\text{iso}} = 0.114$ (3) Å² for (1a); $U_{\text{iso}} = 0.15$ (2) Å² for (1b)]. The function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = 1/[\sigma^2(F_o) + 0.0005|F_o|^2]$. For (1a) $R = 0.0487$, $wR = 0.0668$, $S = 1.76$, for 74 variables. For (1b) $R = 0.0800$, $wR = 0.0967$, $S = 2.56$, for 75 variables [secondary-extinction parameter = 0.0135 (2)]. Final difference Fourier peaks ranged from -0.14 to 0.23 e \AA^{-3} for (1a), and -0.88 to 1.09 e \AA^{-3} for (1b) [all in the vicinity of atom Ga(1)]. The somewhat higher agreement factors observed for (1b) are probably the result of poor crystal quality, crystal decay and higher absorption. Computations were performed using *SHELXTL* (Sheldrick, 1986). Scattering factors were those of Cromer & Waber (1974); real and imaginary anomalous-dispersion corrections were those of Cromer (1974). Final atomic coordinates are given in Table 1* and selected distances and angles for both compounds in Table 2; the molecule (1a) with general atom-labeling scheme is displayed in Fig. 1.

Related literature. A study of the factors affecting the formation and stability of compounds formed from trialkylaluminium and substituted ethylenediamine derivatives has been reported by Beachley & Racette (1976). The structure of *N,N'*-bis(trialkylaluminio)-ethylenediamine has been determined by Jiang & Interrante (1990).

We are grateful to the National Science Foundation for support of this work to GHR (RII-8520554).

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters, and a thermal ellipsoid plot of (1b) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55122 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0555]

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$)

Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
<i>(1a)</i>				
Al(1)	7541 (1)	1946 (1)	281 (1)	54 (1)
N(1)	8365 (2)	4393 (3)	253 (2)	50 (1)
C(1)	7356 (4)	940 (5)	-1260 (3)	94 (2)
C(2)	8862 (4)	729 (5)	1591 (4)	97 (2)
C(3)	5847 (3)	2422 (5)	495 (3)	80 (2)
C(4)	9638 (3)	4149 (3)	27 (3)	56 (1)
C(5)	7400 (3)	5450 (5)	-679 (3)	86 (2)
C(6)	8600 (4)	5236 (5)	1399 (3)	84 (2)
<i>(1b)</i>				
Ga(1)	7525 (1)	1843 (2)	306 (1)	62 (1)
N(1)	8376 (9)	4400 (10)	239 (6)	53 (3)
C(1)	7376 (16)	850 (19)	-1256 (11)	105 (7)
C(2)	8902 (14)	770 (19)	1679 (13)	106 (7)
C(3)	5813 (16)	2396 (17)	491 (14)	106 (8)
C(4)	9639 (10)	4169 (13)	37 (9)	59 (4)
C(5)	7388 (13)	5418 (18)	-696 (12)	98 (7)
C(6)	8531 (15)	5269 (16)	1356 (10)	94 (7)

Table 2. Distances (Å) and angles (°)

$M = \text{Al (1a), Ga (1b)}$.

	<i>(1a)</i>	<i>(1b)</i>
$M(1) - N(1)$	2.074 (2)	2.174 (8)
$M(1) - C(1)$	1.980 (4)	2.02 (1)
$M(1) - C(2)$	1.964 (4)	1.99 (1)
$M(1) - C(3)$	1.957 (4)	1.98 (2)
$N(1) - C(4)$	1.495 (4)	1.47 (2)
$N(1) - C(5)$	1.484 (4)	1.48 (1)
$N(1) - C(6)$	1.481 (4)	1.48 (1)
$C(4) - C(4a)$	1.529 (6)	1.51 (2)
$N(1) - M(1) - C(1)$	103.7 (1)	101.3 (5)
$N(1) - M(1) - C(2)$	104.5 (1)	102.0 (5)
$N(1) - M(1) - C(3)$	104.8 (1)	103.4 (5)
$C(1) - M(1) - C(2)$	113.4 (2)	116.0 (6)
$C(1) - M(1) - C(3)$	114.1 (2)	115.2 (6)
$C(2) - M(1) - C(3)$	114.7 (2)	115.7 (7)
$M(1) - N(1) - C(4)$	108.2 (2)	108.8 (6)
$M(1) - N(1) - C(5)$	108.5 (2)	107.9 (7)
$M(1) - N(1) - C(6)$	108.1 (2)	106.3 (7)
$C(4) - N(1) - C(5)$	111.3 (3)	112 (1)
$C(4) - N(1) - C(6)$	111.3 (2)	113.7 (9)
$C(5) - N(1) - C(6)$	109.4 (3)	107.4 (9)
$N(1) - C(4) - C(4a)$	114.3 (3)	116 (1)

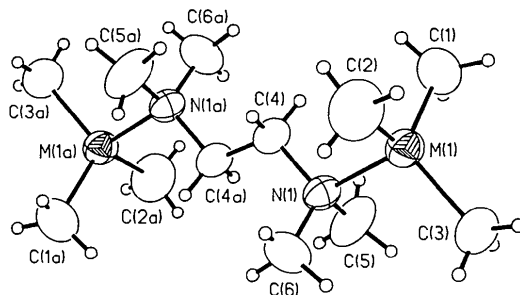


Fig. 1. Thermal ellipsoid plot (50% probability) of the molecule (1a) showing the general atom-labeling scheme. Compound (1b) is isomorphous with (1a).

References

- BEACHLEY, O. T. JR & RACETTE, K. C. (1976). *Inorg. Chem.* **15**, 2110–2115.
- CROMER, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- JIANG, Z. & INTERRANTE, L. V. (1990). *Chem. Mater.* **2**, 439–446.
- SHELDRIK, G. M. (1986). *SHELXTL. User's Manual*. Revision 5.1. Nicolet XRD Corporation, Madison, Wisconsin, USA.

Acta Cryst. (1992). **C48**, 2025–2026

Structure of Triphenyltin 2-(4-Pyridylthio)acetate

BY SEIK WENG NG

Institute of Advanced Studies, University of Malaya, 59100 Kuala Lumpur, Malaysia

AND V. G. KUMAR DAS

Department of Chemistry, University of Malaya, 59100 Kuala Lumpur, Malaysia

(Received 12 November 1991; accepted 26 February 1992)

Abstract. Triphenyl[2-(4-pyridylthio)acetato]tin, $C_{25}H_{21}NO_2SSn$, $M_r = 518.21$, monoclinic, $P2_1/a$, $a = 16.525$ (2), $b = 15.386$ (2), $c = 19.751$ (2) Å, $\beta = 113.82$ (1)°, $V = 4594$ (1) Å³, $Z = 8$, $D_x = 1.498$ g cm⁻³, $\lambda(Mo K\alpha) = 0.71073$ Å, $\mu = 12.22$ cm⁻¹, $F(000) = 2080$, $T = 298$ K, $R = 0.034$ for 4981 $I \geq 3\sigma(I)$ reflections. The geometry around the Sn atoms in the two independent molecules is a distorted *trans*-C₃SnNO trigonal bipyramid.

Experimental. The carboxylate was prepared by heating equimolar amounts of triphenyltin hydroxide and 2-(4-pyridylthio)acetic acid in ethanol. Crystals suitable for single-crystal analysis were obtained by cooling the solution slowly. A crystal measuring $0.18 \times 0.18 \times 0.22$ mm was used; the instrument was an Enraf–Nonius diffractometer. Cell dimensions were fixed from the 25 most intense reflections in the $14 \leq \theta \leq 16^\circ$ thin shell. Intensity data were collected up to a $2\theta_{max}$ of 50° (h 19, k 18, $\pm l$ 23) by using the ω - 2θ scan technique; 8395 data, 7755 unique data. Crystal decay was checked by monitoring three reflections hourly (loss in intensity = 15.7% for the 72h of collection), and a linear decay correction was applied to the data (min./max. correction = 1.0001/1.0889). The data were corrected for absorption effects by using the ψ -scan data (min./max. correction = 0.9454/0.9992). The structure was solved by vector methods from the 4981 reflections satisfying the $I \geq 3\sigma(I)$ criterion. All non-H atoms were refined anisotropically; H atoms were generated geometrically (C–H = 0.95 Å, $B = 5$ Å²) and were allowed to ride on their respective C atoms. Refinement based on F

for the 541 variables converged at $R = 0.034$, $wR = 0.038$; $S = 0.458$, $w = [\sigma(F)^2 + (0.02F)^2 + 1]^{-1}$ for the 541 variables, and the largest Δ/σ was less than 0.01. The highest and lowest ($\Delta\rho$) were 0.40 (5) and -0.13 (5) e Å⁻³. Scattering factors were taken from Tables 2.2B and 2.3.1. of *International Tables for X-ray Crystallography* (1974, Vol. IV). Computations were performed using the *MoIEN* structure determination package (Fair, 1990) on a DEC MicroVAX minicomputer. The atomic coordinates are listed in Table 1,* and selected bond distances and angles in Table 2. The two symmetry-independent molecules are depicted in Fig. 1.

Related literature. The two molecules of the title compound are bridged through their pyridyl groups to form polymeric helical chains parallel to b . The coordination polyhedron around the Sn atom is a distorted *trans*-C₃SnNO trigonal bipyramid; one apical position is occupied by the carboxyl O and the other by the N belonging to a symmetry-related ($\frac{1}{2} - x, y - \frac{1}{2}, z$; $1\frac{1}{2} - x, y - \frac{1}{2}, z$) anionic group. The distortion from ideal geometry is shown by the large difference between the axial Sn←:N and Sn—O distances. The Sn←:N distance is, however, somewhat shorter than that in the pyridyl-bridged tri-

* Lists of structure factors, anisotropic thermal parameters, calculated H positional parameters, and complete bond lengths and bond angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55257 (61 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0579]