Synthesis and structural characterization of bis(tropolonatodimethylgallium), $[(C_7H_5O_2)Ga(CH_3)_2]_2$

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Details of the synthesis, physical properties, and solid-state structure of $[(C_7H_5O_2)Ga(CH_3)_2]_2$ are reported. Crystals of bis(tropolonatodimethylgallium) are monoclinic, a = 10.2669(9), b = 9.3237(3), c = 10.8671(10) Å, β = 112.896(4)°, Z = 2, space group $P2_1/n$ (22°C, λ = 0.70930 Å). The structure was solved by conventional heavy-atom techniques and was refined by full-matrix least-squares procedures to $R = 0.034$ and $R_w = 0.037$ for 1746 reflections with $I \geq 3\sigma(I)$. The centrosymmetric binuclear dimer features five-coordinate gallium atoms having irregular trigonal bipyramidal coordination geometry and a planar four-membered Ga_2O_2 ring. Important bond lengths (corrected for libration) are Ga—O(equatorial) = 1.972(2), Ga—O(axial) = 2.025(2) and 2.551(2) Å, and Ga—C = 1.951(4) and 1.954(4) Å.

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On rapporte les détails relatifs à la synthèse, aux propriétés physiques et à la structure à l'état solide du $[(C_7H_5O_2)Ga[CH_3)_2]_2$. Les cristaux du bis(tropolonatodiméthylgallium) sont monocliniques, a = 10,2669(9), b = 9,3237(3), c = 10,8671(10) Å, β = 112,896(4)°, Z = 2, groupe d'espace $P2_1/n$ (22°C, λ = 0,70930 Å). On a résolu la structure par les techniques conventionnelles des atomes lourds et on l'a affiné par la méthode des moindres carrés (matrice complète) jusqu'à des valeurs de $R = 0{,}034$ et $R_w = 0{,}037$ pour 1746 réflexions avec $I \geq 3\sigma(I)$. Le dimère binucléaire centrosymétrique comporte des atomes de gallium penta-coordonnés possédant une géométrie de coordination bipyramidale trigonale irrégulière et un cycle plan à quatre chaînons de Ga_2O_2. Les longueurs de liaisons importantes (corrigées pour la libration) sont: Ga—O (équatorial) = 1,972(2), Ga—O(axial) = 2,025(2) et 2,551(2) Å et Ga—C = 1,951(4) et 1,954(4) Å.

[Traduit par le journal]

Introduction

As part of our continuing study of compounds containing five-coordinate gallium atoms (1–6) we have synthesized and structurally characterized the compound bis(tropolonatodimethylgallium), $[(C_7H_5O_2)Ga(CH_3)_2]_2$. The choice of tropolone as the "active hydrogen" compound in this study was dictated by three considerations. Firstly, it was hoped to incorporate the tropolonato moiety in a mixed pyrazolyl gallate anionic ligand, secondly, we wished to extend our studies of five-coordinate gallium systems having three oxygen atoms in the coordination sphere about gallium and, finally, tropolone and its derivatives have been recently cited as antibiotic potentiators (7). Although the first objective was not fulfilled, the bis(tropolonatodimethylgallium) compound was isolated in high yield and shown to consist of dimer units in the solid state, displaying the familiar Ga_2O_2 central bridging unit and irregular trigonal bipyramidally coordinated gallium atoms. In dilute solution and in the gas phase, however, the compound exists solely as the monomeric species.

Experimental

Air-sensitive materials were handled in oxygen-free dry nitrogen. Tetrahydrofuran (THF) was dried by refluxing over sodium/benzophenone and used immediately following distillation. Tropolone (Aldrich) was used as supplied. Trimethylgallium was prepared as described earlier (8). 3,5-Dimethylpyrazole (pz″H) (Aldrich) and sodium hydride (Alfa) were used as supplied.

Attempted preparation of $Na^+[Me_2Gapz''(C_7H_5O_2)]^-$

$$Na^+[Me_3Gapz'']^- + C_7H_6O_2 \rightarrow Na^+[Me_2Gapz''(C_7H_5O_2)]^- + MeH$$

Trimethylgallium (2.156 g, 18.8 mmol) in THF was added to Napz″ (2.218 g, 18.8 mmol) (made from NaH + pz″H) in the same solvent. To the clear colorless solution of $Na^+[Me_3Gapz'']^-$ so formed was added tropolone (2.293 g, 18.8 mmol) in THF. An immediate formation of yellowish precipitate was observed. The mixture was refluxed for approximately 40 h and then filtered. The pale yellow solid precipitate was isolated and shown by elemental analysis to be sodium tropolonate, $Na^+C_7H_5O_2^-$. The soluble reaction product from the reaction was shown to be the $[Me_2Ga(\mu\text{-pz}'')]_2$ dimer (9).

Preparation of bis(tropolonatodimethylgallium), $[(C_7H_5O_2)Ga(CH_3)_2]_2$

Trimethylgallium (1.945 g, 16.96 mmol) in THF was added to tropolone (2.068 g, 16.95 mmol) in the same solvent. A vigorous evolution of methane occurred on mixing the two reactants. On cessation of gas evolution the solvent was removed under vacuum from the clear product solution. The solid obtained was recrystallized from benzene to yield colorless air-stable crystals of the product. *Anal.* calcd. for $[(C_7H_5O_2)Ga(CH_3)_2]_2$: C 48.93, H 4.98; found: C 48.61, H 5.16. ^1H nmr (C_6D_6, τ values refer to $\tau_{C_6H_6}$ = 2.84 ppm): 9.90 s ($GaMe_2$, 6), 6.32 t (J = 10 Hz, 1), 4.84 t (J = 10 Hz, 2), 2.84 d (J = 10 Hz, 2). The mass spectrum of the compound displayed weak signals due to the monomer parent ion $(C_7H_5O_2)Ga(CH_3)_2^+$ (= P^+) (4%), the strongest signals arising from the $P - Me^+$ ion (100%) and the $P - 2Me^+$ ion (13%). Mol. wt. calcd. for $(C_7H_5O_2)Ga(CH_3)_2$: 220.9; found (cryoscopy in benzene solution): 222.

X-ray crystallographic analysis of bis(tropolonatodimethylgallium)

A crystal bounded by the six faces (followed by their distances in mm from a common origin): ±(1 0 −1), 0.200, ±(0 1 1), 0.150, ±(1 −1 1), 0.138 was mounted in a general orientation. Unit-cell parameters were refined by least-squares on 2 sin θ/λ values for 25 reflections (2θ = 40–45°) measured on a diffractometer with Mo–$K\alpha$ radiation ($\lambda(K\alpha_1)$ = 0.70930, $\lambda(K\alpha_2)$ = 0.71359 Å). Crystal data at 22°C are:

$C_{18}H_{22}Ga_2O_4$ fw = 441.82
Monoclinic, a = 10.2669(9), b = 9.3237(3), c = 10.8671(10) Å, β = 112.896(4)°, V = 958.3(1) Å^3, Z = 2, ρ_c = 1.531 g cm^{-3}, $F(000)$ = 448, $\mu(Mo - K\alpha)$ = 27.3 cm^{-1}. Absent reflections: $h0l$, $h + l$ odd, and $0k0$, k odd, uniquely indicate the space group $P2_1/n$ [nonstandard setting of $P2_1/c$, C_{2h}^5, No. 14, equivalent positions: ±(x, y, z; 1/2 − x, 1/2 + y, 1/2 − z)].

Intensities were measured with graphite-monochromated Mo − $K\alpha$ radiation on an Enraf-Nonius CAD4-F diffractometer. An ω–2θ scan at 1.18–10.06° min^{-1} over a range of (0.60 + 0.35 tan θ) degrees in ω (extended by 25% on both sides for background measurement) was employed. Data were measured to 2θ = 65°. The intensities of three check reflections, measured every 3600 s throughout the data collection, showed uniform decreases to final values 10% less than the initial values, the intensities being scaled accordingly. After data reduction,[1] an absorption correction was applied using the Gaussian integration method (10, 11). Transmission factors ranged from 0.353 to 0.524 for 184 integration points. Of the 3445 independent reflections measured, 1746 (50.7%) had intensities greater than $3\sigma(I)$ above background where $\sigma^2(I) = S + 2B + (0.04(S - B))^2$ with S = scan count and B = normalized background count.

The coordinates of the Ga atom were determined from the Patterson function and those of the O and C atoms from a subsequent difference map. After full-matrix least-squares refinement of the non-hydrogen atoms with anisotropic thermal parameters to $R = 0.063$, a difference map gave the positions of all 11 independent hydrogen atoms which were refined with isotropic thermal parameters in subsequent cycles of refinement. The scattering factors of ref. 12 were used for non-hydrogen atoms and those of ref. 13 for hydrogen atoms. Anomalous scattering factors from ref. 14 were used for the Ga atoms. The weighting scheme, $w = 1/\sigma^2(F)$ where $\sigma^2(F)$ is derived from the previously defined $\sigma^2(I)$, gave uniform average values of $w(|F_o| - |F_c|)^2$ over ranges of both $|F_o|$ and $\sin\theta/\lambda$ and was employed in the final stages of refinement. Reflections with $I < 3\sigma(I)$ were not included in the refinement. An isotropic Type I extinction correction (Thornley-Nelmes definition of mosaic anisotropy with a Lorentzian distribution) was applied (15–17). The final value of g was $5.8(5) \times 10^4$. Convergence was reached at $R = 0.034$ and $R_w = 0.037$ for 1746 reflections with $I \geq 3\sigma(I)$. For all 3445 reflections $R = 0.097$. The function minimized was $\Sigma w(|F_o| - |F_c|)^2$, $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ and $R_w = (\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2)^{1/2}$.

On the final cycle of refinement the mean and maximum parameter shifts corresponded to 0.04 and 0.72σ, respectively. The largest shift was associated with the thermal parameter of H(2c). The mean error in an observation of unit weight was 1.575. A final difference map showed maximum fluctuations of $\pm 0.26(13)$ e Å^{-3}. The final positional and thermal parameters appear in Tables 1 and 5, respectively.[2] Measured and calculated structure factors have been placed in the Depository of Unpublished Data.[2]

The ellipsoids of thermal motion for the non-hydrogen atoms are shown in Fig. 1. The thermal motion has been analysed in terms of the rigid-body modes of translation, libration, and screw motion (18). The rms standard error in the temperature factors σU_{ij} (derived from the least-squares analysis) is 0.0015 Å^2. Analysis of the entire dimer indicated significant independent motion of the "GaMe$_2$" units. Independent analyses were carried out for two molecular fragments, the Ga coordination group and the tropolone moiety (rms ΔU_{ij} = 0.0016 and 0.0017 Å^2, respectively). The appropriate bond distances have been corrected for libration (18, 19), using shape parameters q^2 of 0.08 for all atoms involved. Corrected bond lengths appear in Table 2 along with the uncorreeted values; corrected bond angles do not differ by more than 1σ from the uncorrected values given in Table 3. Intraannular torsion angles defining the conformation of the tropolone ring are listed in Table 4. Bond lengths and angles involving hydrogen and a complete listing of torsion angles (tables 6–8) are included as supplementary material.[2]

TABLE 1. Final positional (fractional × 10^4, Ga × 10^5, H × 10^3) and isotropic thermal parameters ($U \times 10^3$ Å^2) with estimated standard deviations in parentheses

Atom	x	y	z	U_{eq}/U_{iso}
Ga	32135(3)	42110(3)	42483(3)	50
O(1)	4166(2)	5887(2)	5312(2)	53
O(2)	1561(2)	5226(2)	4432(2)	58
C(1)	2783(6)	4432(7)	2352(4)	82
C(2)	3485(5)	2536(4)	5390(5)	68
C(3)	3374(2)	6789(3)	5624(2)	42
C(4)	1872(2)	6387(3)	5107(2)	45
C(5)	800(3)	7164(3)	5310(3)	54
C(6)	856(4)	8435(4)	5963(3)	60
C(7)	1988(4)	9308(4)	6645(3)	61
C(8)	3378(4)	9100(3)	6840(3)	56
C(9)	3977(3)	7978(3)	6395(3)	50
H(5)	−2(4)	674(4)	497(4)	66(9)
H(6)	1(4)	864(4)	592(3)	64(9)
H(7)	189(4)	1006(5)	704(3)	75(10)
H(8)	408(4)	971(4)	738(3)	60(8)
H(9)	491(3)	800(3)	665(3)	59(9)
H(1a)	316(9)	520(11)	234(10)	186(39)
H(1b)	325(6)	386(6)	202(5)	110(17)
H(1c)	199(10)	460(9)	199(7)	183(35)
H(2a)	283(10)	224(8)	511(10)	146(38)
H(2b)	390(4)	185(5)	517(4)	86(12)
H(2c)	386(5)	274(5)	636(5)	161(16)

*U_{eq} = (1/3)trace (U_{diag}).

TABLE 2. Bond lengths (Å) with estimated standard deviations in parentheses

	Length			Length	
Bond	Uncorr.	Corr.	Bond	Uncorr.	Corr.
Ga —O(1)	1.963(2)	1.972	C(3)—C(4)	1.469(3)	1.478
Ga —O(2)	2.020(2)	2.025	C(3)—C(9)	1.382(4)	1.387
Ga —C(1)	1.943(4)	1.951	C(4)—C(5)	1.405(4)	1.411
Ga —C(2)	1.946(4)	1.954	C(5)—C(6)	1.371(4)	1.375
Ga —O(1)′	2.545(2)	2.551	C(6)—C(7)	1.376(5)	1.382
O(1)—C(3)	1.303(3)	1.308	C(7)—C(8)	1.372(5)	1.377
O(2)—C(4)	1.276(3)	1.279	C(8)—C(9)	1.392(4)	1.397

*Here and elsewhere primed atoms are related to those in Table 1 by inversion through the centre of symmetry at 1/2, 1/2, 1/2.

Results and discussion

The title compound, bis(tropolonatodimethylgallium), represents the second example of a complex having a five-coordinate gallium centre which does not include nitrogen in the pentacoordination sphere about the metal atom (the Ga atom is coordinated to three oxygen and two carbon atoms). As with the salicylaldehydatodimethylgallium dimer, (SalGaMe$_2$)$_2$, the first such example (3), the present compound is not associated in either the gas phase or in dilute solution. Thus the mass spectrum of the compound gives no signals at m/e values higher than those corresponding to the monomer ion or its ions arising from predictable fragmentation patterns. In dilute benzene solution, molecular weight measurements indicate the monomer species to be present. The ^{1}H nmr pattern in C_6D_6, consisting of a singlet for the "GaMe$_2$" protons and a triplet: triplet: doublet pattern for the protons of the tropolonato moiety, is also consistent with a monomeric species in solution.

The solid state structure consists of well-separated dimeric units in which each five-coordinate Ga atom is in an irregular trigonal bipyramidal environment. Space group symmetry re-

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[1] The computer programs used include locally written programs for data processing and locally modified versions of the following: ORFLS, full-matrix least-squares, and ORFFE, function and errors, by W. R. Busing, K. O. Martin and H. A. Levy; FORDAP, Patterson and Fourier syntheses, by A. Zalkin; ORTEP II, illustrations, by C. K. Johnson.

[2] The structure factor table, Table 5 (anisotropic thermal parameters) and other material mentioned in the text are available, at a nominal charge, from the Depository of Unpublished Data, CISTI, National Research Council of Canada, Ottawa, Canada K1A 0S2.

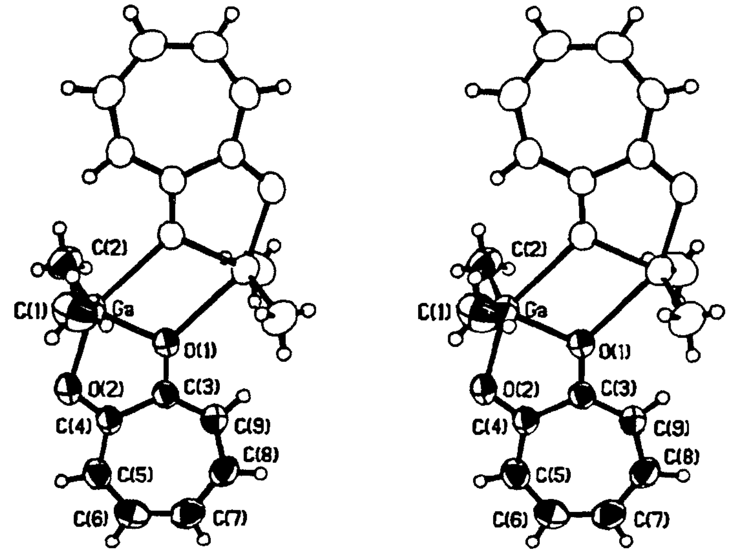

FIG. 1. Stereoscopic view of bis(tropolonatodimethylgallium). 50% probability thermal ellipsoids are shown for the non-hydrogen atoms. Hydrogen atoms have been assigned artificially small thermal parameters for the sake of clarity.

TABLE 3. Bond angles (deg) with estimated standard deviations in parentheses

Bonds	Angle (deg)	Bonds	Angle (deg)
O(1)—Ga —O(2)	79.27(8)	Ga —O(2)—C(4)	114.96(15)
O(1)—Ga —C(1)	113.3(2)	O(1)—C(3)—C(4)	113.9(2)
O(1)—Ga —C(2)	110.38(15)	O(1)—C(3)—C(9)	119.8(2)
O(1)—Ga —O(1)′	70.73(8)	C(4)—C(3)—C(9)	126.3(2)
O(2)—Ga —C(1)	101.6(2)	O(2)—C(4)—C(3)	115.3(2)
O(2)—Ga —C(2)	103.33(15)	O(2)—C(4)—C(5)	119.4(2)
O(2)—Ga —O(1)′	149.99(7)	C(3)—C(4)—C(5)	125.3(2)
C(1)—Ga —C(2)	132.7(2)	C(4)—C(5)—C(6)	130.5(3)
C(1)—Ga —O(1)′	89.5(2)	C(5)—C(6)—C(7)	130.3(3)
C(2)—Ga —O(1)′	88.43(14)	C(6)—C(7)—C(8)	127.6(3)
Ga —O(1)—C(3)	116.6(2)	C(7)—C(8)—C(9)	128.7(3)
Ga —O(1)—Ga′	109.27(8)	C(3)—C(9)—C(8)	131.2(3)
C(3)—O(1)—Ga′	134.10(15)		

TABLE 4. Intra-annular torsion angles (deg) standard deviations in parentheses

Atoms	Torsion angle (deg)
C(9)—C(3)—C(4)—C(5)	0.5(4)
C(3)—C(4)—C(5)—C(6)	2.1(5)
C(4)—C(5)—C(6)—C(7)	−2.0(6)
C(5)—C(6)—C(7)—C(8)	−0.2(6)
C(6)—C(7)—C(8)—C(9)	0.6(6)
C(7)—C(8)—C(9)—C(3)	1.5(6)
C(4)—C(3)—C(9)—C(8)	−2.6(5)

quires each dimer to possess a centre of symmetry. The dimerization occurs via the formation of a four-membered Ga_2O_2 ring in which each oxygen atom occupies an equatorial coordination site of one Ga atom and an axial site of the other. This type of dimerization has been noted for $(SalGaMe_2)_2$ (3), $(Me_2NCH_2CH_2OGaR_2)_2$ (R = H, Me) (5), and $[MeN(CH_2CH_2O)_2GaH]_2$ (6). The terminal tropolonato O atoms occupy the other axial positions while methyl groups complete the equatorial planes. The angle between the axial substituents, 149.99(8)°, is similar to the values of 150−152° found for the "ethanolamine" complexes (5, 6) in which five-membered chelate rings are also fused to the central Ga_2O_2 ring, but considerably smaller than the value of 164.4(1)° in $(SalGaMe_2)_2$ which contains six-membered chelate rings. The Ga atom is displaced by 0.2111(3) Å toward O(2) from the plane of the equatorial substituents. The system of five fused rings is planar to within 0.018(3) Å. The small but significant deviation from planarity arises in the tropolone ring which is significantly nonplanar ($\chi^2 = 84.6$, maximum deviation 0.015(3) Å), the four-membered ring being exactly planar and the five-membered chelate ring being planar within experimental error ($\chi^2 = 7.5$, maximum deviation 0.0035(21) Å).

The Ga—O distances[3] involving the bridging tropolonato O atom (1.972(2) and 2.551(2) Å for the equatorial and axial bonds, respectively) are significantly longer than those observed earlier for related molecules. In $(Me_2NCH_2CH_2OGaR_2)_2$ (R = H, Me) and $[MeN(CH_2CH_2O)_2GaH]_2$ which contain bridging alkoxide groups, the corresponding bond lengths are in the ranges 1.911(3)−1.960(8) Å and 2.018(2)−2.078(3) Å,

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[3] Libration-corrected bond lengths (esd's assumed equal to those associated with the uncorrected values) are employed throughout the discussion and are compared with similarly treated distances except in the case of $(Me_2NCH_2CH_2OGaH_2)_2$ (5).

respectively. The corresponding distances in $(SalGaMe_2)_2$ involving a bridging phenoxide moiety are 1.933(3) and 2.469(3) Å. The axial Ga—O bond to the terminal tropolonato O atom of 2.025(2) Å is similar to those quoted above for the bridging alkoxide groups and significantly shorter than the value of 2.127(3) Å for the aldehyde O atom in $(SalGaMe_2)_2$. A similar trend in B—O bond lengths has been noted for the tetrahedral tropolonato- (20), salicylaldehydato- (21), and aminoethanolatodiarylboron complexes (22–25). These findings are consistent with the known donor properties of these oxygen-containing ligands. The Ga—C distances are in good agreement with those reported earlier (1–6).

The observed geometry of the tropolonate ligand is intermediate between that in tropolone itself (26), in which there is partial localization of the double bonds, and the fully delocalized and symmetric structure such as that observed in the boron complex $(C_7H_5O_2)BPh_2$ (20). The structural inequivalence of the tropolonato oxygen atoms leads to a small alternation of bond lengths in the seven-membered ring (similar to that observed in the structure of $[(C_7H_5O_2)TlPh_2]_2$ (27)), although this can occur in the absence of structural differences as in the case of the square planar complex $(C_7H_5O_2)_2Cu$ (28) in which each ligand is bidentate and chelating. As expected, the two C—O distances differ significantly and both of these values (1.279(3) and 1.308(3) Å) are shorter than the mean C—O distance of 1.313(2) Å in the boron compound, consistent with relatively stronger O—B bonding than O—Ga bonding in these two tropolonate complexes. The shorter C—O bonds in the present structure are balanced by a significant lengthening of the unique C(3)—C(4) bond to 1.478(3) Å from the value of 1.446(2) Å in the boron compound. In tropolone itself, the C—O and unique C—C distances are 1.263(3), 1.333(3), and 1.454(4) Å, respectively. The average C—C distance for the remaining six tropolonato ring bonds is 1.388(13) Å compared to 1.385(26) Å in tropolone and 1.385(5) Å in the diphenylboron chelate. The mean short and long C—C bonds average 1.380(6) and 1.397(15) Å respectively, compared to corresponding values of 1.364(20) and 1.405(10) Å in tropolone (26).

Acknowledgments

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