

Synthesis and Characterization of Bulky Aryl Derivatives of the Heavier Main Group 3 Elements

Mark A. Petrie, P. P. Power,* H. V. Rasika Dias, Karin Ruhlandt-Senge, Krista M. Waggoner, and Rudolf J. Wehmschulte

Department of Chemistry, University of California, Davis, California 95616

Received July 21, 1992

The synthesis and structural characterization of several bulky aryl derivatives of aluminum, gallium, and indium are described. With the exception of the previously synthesized species $\text{Al}(\text{Mes})\text{Cl}_2\cdot\text{THF}$ (1) and $[\text{Ga}(\text{Mes})_2\text{Cl}]_2$ (6) (Mes = 2,4,6-Me₃C₆H₂) the compounds are derivatives of the very crowding 2,4,6-*i*-Pr₃C₆H₂⁻ (Trip) or 2,4,6-*t*-BuC₆H₂⁻ (Mes*) substituents, as indicated by the formulas $\text{Al}(\text{Trip})\text{Br}_2(\text{Et}_2\text{O})$ (2), $[\text{Al}(\text{Trip})_2\text{Br}]_2$ (3), $\text{Ga}(\text{Trip})\text{Cl}_2\cdot\text{THF}$ (4), $[(\text{Et}_2\text{O})\text{LiCl}_3\text{-Ga}(\text{Trip})_2]$ (5), $\text{Ga}(\text{Trip})_2\text{Cl}$ (X = Cl (7), Br (8)), $\text{Ga}(\text{Trip})_3$ (9), $\text{Al}(\text{Mes}^*)\text{Br}_2$ (10), and $\text{M}(\text{Mes}^*)\text{-Cl}_2$ (M = Ga (11), In (12)). The most interesting feature of these compounds is that, in several instances (7, 8, 10-12), previously unknown unassociated species can be obtained in the crystal phase with only partial substitution of the halides. In the case of compounds 10-12, uncomplexed monoaryl dihalide derivatives can be obtained even in the presence of ether. Thus, these compounds are useful precursors for the synthesis of other derivatives in which the presence of three-coordinate aluminum, gallium, or indium is desirable.

The use of bulky groups in the kinetic stabilization of compounds with multiple bonds between heavier main-group elements has been a major development in inorganic chemistry over the past two decades. Most of this work has been concentrated in main groups 4 and 5, and at present virtually nothing is known regarding multiple bonding among the heavier main group 3 elements.^{1,2} Recent synthetic work has suggested that this may soon change. Compounds such as R_2BBR_2 ^{3,4} (R = CH₂(*t*-Bu)) and the more recently reported species R_2MMR_2 (M = Al,⁵ Ga,⁶ In;⁷ R = -CH(SiMe₃)₂), in addition to being of interest in their own right, are possible precursors to compounds that contain formal multiple bonds between main group 3 elements via the reduction in eq 1. In this



process two adjacent empty p orbitals are combined and become occupied with either one or two electrons to form a π bond. Examples of both one-^{8,9} and two-electron reductions¹⁰ are now known for boron. For the tetraalkyl-diboron species the process apparently stops at the one-electron stage,^{8,9} whereas for a tetraaryl species a two-electron reduction can be readily accomplished.¹⁰ The apparent greater facility with which the aryl derivatives

undergo smooth reduction has led us to focus our attention on the synthesis and characterization of bulky aryl derivatives of aluminum, gallium, and indium as possible precursors to these compounds. The synthesis and characterization of these precursors and some related species, many of which possess unique structural features, are described in this paper.

Experimental Section

General Procedures. All work performed under anaerobic and anhydrous conditions by using Schlenk techniques or a Vacuum Atmospheres HE-43 Dry Box. AlCl_3 and AlBr_3 were obtained from commercial suppliers and purified by sublimation; MesBr, GaCl_3 , and InCl_3 were used as received. TripBr,¹¹ Mes*Br,¹² TripMgBr,¹³ Mes*Li,¹⁴ $\text{Al}(\text{Mes})\text{Cl}_2\cdot\text{THF}$,¹⁵ and $[\text{Ga}(\text{Mes})_2\text{Cl}]_2$ ¹⁶ were synthesized by literature procedures. Solvents were freshly distilled under N₂ from Na/K alloy and degassed three times immediately before use.

Physical Measurements. ¹H NMR spectra were obtained on a General Electric QE-300 spectrometer. With the exception of 5, which desolvates rapidly, all compounds gave satisfactory C and H analysis.

$\text{Al}(\text{Trip})\text{Br}_2(\text{Et}_2\text{O})$ (2). A solution of TripBr (5.66 g, 20 mmol) in Et₂O (40 mL) was treated dropwise with *n*-BuLi (12.5 mL of a 1.6 M solution in hexane), with stirring in an ice bath. The ice bath was removed, and stirring was continued for 12 h. This solution was then added dropwise to AlBr_3 (2.64 g, 10 mmol) in hexane (20 mL) to give a white precipitate and a pale yellow solution. This solution was stirred for 12 h, and the solvents were removed under reduced pressure. The residue was extracted with hexane (70 mL), and the extracts were filtered and concentrated to about 30 mL. Cooling in a -20 °C freezer for 20

(1) Sn-Sn double bonds: Goldberg, D. E.; Harris, D. H.; Lappert, M. F.; Thomas, K. M. *J. Chem. Soc., Chem. Commun.* 1976, 261. Si-Si double bonds: West, R.; Fink, M. J.; Michl, J. *Science (Washington, D.C.)* 1981, 1343. P-P double bonds: Yoshifuji, M.; Shima, I.; Inamoto, N.; Hirotsu, K.; Higuchi, T. *J. Am. Chem. Soc.* 1983, 103, 4587.

(2) West, R. *Angew. Chem., Int. Ed. Engl.* 1987, 26, 449. Cowley, A. H.; Norman, N. C. *Prog. Inorg. Chem.* 1986, 34, 1.

(3) Biffar, W.; Nöth, H.; Pommerening. *Angew. Chem., Int. Ed. Engl.* 1980, 19, 56.

(4) Schlüter, K.; Berndt, A. *Angew. Chem., Int. Ed. Engl.* 1980, 19, 57.

(5) Uhl, W. Z. *Naturforsch.* 1988, 43B, 1113.

(6) Uhl, W.; Layh, M.; Hildenbrand, T. *J. Organomet. Chem.* 1989, 364, 289.

(7) Uhl, W.; Layh, M.; Hiller, W. *J. Organomet. Chem.* 1989, 368, 139.

(8) Klusik, H.; Berndt, A. *Angew. Chem., Int. Ed. Engl.* 1981, 20, 870.

(9) Klusik, H.; Berndt, A. *J. Organomet. Chem.* 1981, 222, C25.

(10) Moezzi, A.; Olmstead, M. M.; Power, P. P. *J. Am. Chem. Soc.* 1992, 114, 2715. Moezzi, A.; Bartlett, R. A.; Power, P. P. *Angew. Chem., Int. Ed. Engl.* 1992, 31, 1082.

(11) Whitesides, G. M.; Eisenhut, M.; Banting, W. M. *J. Am. Chem. Soc.* 1974, 96, 5398.

(12) Pearson, D. E.; Frazer, M. G.; Frazer, V. S.; Washburn, L. C. *Synthesis* 1976, 621.

(13) Bartlett, R. A.; Dias, H. V. R.; Olmstead, M. M.; Power, P. P.; Weese, K. *J. Organometallics* 1990, 9, 146. The preparation described here is based upon that of A. Pelter and co-workers in 1987.

(14) Yoshifuji, M.; Shima, I.; Inamoto, N. *Tetrahedron Lett.* 1979, 396, 3.

(15) Seidel, W. Z. *Anorg. Allg. Chem.* 1985, 524, 101.

(16) Beachley, O. T.; Churchill, M. R.; Pazik, J. C.; Ziller, J. W. *Organometallics* 1987, 6, 2088.

Table I. Abridged Summary of Data Collection,^a Structure Solution, and Refinement for 1–12

	1	2	3	4	5	6	7	8	9	10	11	12
formula	C ₁₃ H ₁₉ AlCl ₂ O	C ₁₉ H ₃₃ AlBr ₂ O	C ₆₀ H ₉₂ Al ₂ Br ₂	C ₁₉ H ₃₁ ClGaO	C ₁₉ H ₃₃ Cl ₃ GaLiO	C ₁₈ H ₂₂ ClGa	C ₃₀ H ₄₆ ClGa	C ₃₀ H ₄₀ BrGa	C ₄₅ H ₆₉ Ga	C ₁₈ H ₂₉ AlBr ₂	C ₁₈ H ₂₉ Cl ₂ Ga	C ₂₈ H _{31.5} Cl ₂ In
fw	289.2	464.3	1027.1	416.1	460.5	343.5	511.8	556.3	679.7	432.2	386.0	553.8
space group	P1	P1	P1	P2 ₁ /n	P2 ₁ /n	P2 ₁ /c	P2 ₁ /c	P2 ₁ /c	P2 ₁ /c	C2/c	C2/c	P2 ₁ /n
a, Å	8.697(3)	7.721(7)	12.557(3)	12.784(5)	9.834(4)	12.245(3)	15.486(10)	15.292(3)	11.695(8)	14.227(6)	14.331(7)	12.672(5)
b, Å	9.317(2)	8.837(5)	13.579(3)	9.407(2)	24.755(9)	15.581(5)	13.787(4)	13.672(5)	10.684(5)	12.374(4)	12.170(6)	15.181(5)
c, Å	9.801(2)	17.427(8)	19.183(4)	17.839(5)	10.677(4)	9.037(2)	14.952(6)	15.292(3)	35.17(2)	12.120(3)	11.841(5)	21.692(7)
α, deg	69.40(2)	83.36(2)	100.49(2)			108.83(2)	111.79(2)	111.80(2)	94.11(4)	111.47(2)	109.79(2)	106.31(2)
β, deg	74.04(2)	88.55(2)	108.02(2)	99.9(2)	114.11(3)							
γ, deg	86.32(2)	72.50(2)	97.21(2)									
V, Å ³	714.4(3)	1126.4(1.1)	3000.3(1.2)	2113(1)	2372(2)	1631.8(9)	2964(3)	2968(1)	4383(4)	1985.6(1.2)	1941.3(1.6)	4005(2)
Z	2	2	2	4	4	4	4	4	4	4	4	8
D(calc), g/cm ³	1.344	1.369	1.137	1.308	1.289	1.398	1.147	1.245	1.030	1.446	1.321	1.837
linear abs coeff, cm ⁻¹	0.498	3.64	1.414	1.557	1.502	1.838	1.033	2.287	0.653	4.081	1.683	1.464
2θ range, deg	0–55	0–45	0–50	0–55	0–45	0–55	0–48	0–55	0–50	0–55	0–46	0–50
no. of obs rflns	2894	2121	6514	4062	2065	3013	1679	3219	2173	1465	828	2275
no. of variables	154	208	557	208	226	144	181	148	195	60	59	163
R, R _w	0.035, 0.041	0.091, 0.093	0.082, 0.081	0.061, 0.053	0.059, 0.061	0.041, 0.051	0.084, 0.079	0.069, 0.069	0.075, 0.072	0.073, 0.109	0.083, 0.109	0.10, 0.14

^a Data collected at 130 K with Mo K α radiation ($\lambda = 0.71069$ Å). ^b $I > 3\sigma(I)$ for 1, 7–12, $I > 2\sigma(I)$ for 2–6.

h afforded the product 2 as colorless crystals: yield 2.8 g (60% based on Al); mp 118–120 °C; ¹H NMR (C₆D₆) δ 0.92 (t, CH₃, Et₂O), 1.25 (d, *p*-CHMe₂), 1.37 (d, *o*-CHMe₂), 2.78 (sept, *p*-CHMe₂), 3.67 (q, CH₂, Et₂O), 3.83 (sept, *o*-CHMe₂), 7.18 (s, *m*-H).

[Al(Trip)₂Br]₂ (3). An Et₂O solution (40 mL) of recrystallized [Li(Et₂O)Trip]₂ (1.71 g, 3 mmol) was added dropwise to a pentane solution (20 mL) of AlBr₃ (0.8 g, 3 mmol) with cooling in an ice bath. After the mixture was stirred for 12 h at room temperature, the solvents were removed under reduced pressure. The residue was taken up in pentane (40 mL), and the solution was filtered. Concentration of the solution to ca. 12–15 mL and cooling in a dry ice–acetone bath afforded crystals of the product 3: yield 20%, 0.6 g; mp 174–176 °C; ¹H NMR (C₆D₆) δ 1.19 (d, *p*-CHMe₂, 12H), 1.32 (d, *o*-CHMe₂, 24 H), 2.77 (sept, *p*-CHMe₂, 2H), 3.11 (sept, *o*-CHMe₂, 7.05 (s, *m*-H, 4H).

Ga(Trip)Cl₂(THF) (4). A THF solution of Mg(Trip)₂(THF)₂ (2.87 g, 5 mmol) was added to GaCl₃ (1.76 g, 20 mmol) in THF (100 mL) and refluxed for 20 h. Removal of THF under reduced pressure, followed by extraction with hexane (3 × 100 mL) and filtration, gave a pale yellow solution. Concentration to 30 mL under reduced pressure and cooling of the mixture overnight in a –20 °C freezer gave the product 4 as colorless crystals: yield 2.06 g, 60%; mp 133 °C; ¹H NMR (C₆D₆) δ 1.23 (d, *o*-CHMe₂), 1.32 (d, *p*-CHMe₂), 1.36 (broad, THF), 2.80 (sept, *o*-CHMe₂), 3.07 (sept, *p*-CHMe₂), 3.59 (broad THF), 7.08 (s, aryl H). In addition, small amounts of a species that gave signals at δ 1.17 (d) and at δ 6.99 (s) were observed. The assignment of these signals is given in the Results and Discussion.

[Et₂O]LiCl₃Ga(Trip)₂ (5). A rapidly stirred solution of TripBr (2.83 g, 10 mmol) in Et₂O (20 mL) was treated dropwise with *n*-BuLi (6.25 mL of a 1.6 M solution in hexane) with cooling in an ice bath. The solution was stirred for 1 h and warmed to room temperature, whereupon stirring was continued for 16 h.

This solution was then added dropwise to a stirred solution of GaCl₃ (1.76 g, 10 mmol) in Et₂O (20 mL) and cooled in an ice bath. Little precipitation was observed upon stirring for another 3 h at room temperature. The solution was filtered through Celite, and the volume was reduced under low pressure to ca. 15 mL. Cooling overnight in a –20 °C freezer afforded the product 5 as colorless crystals: yield 2.4 g, 52%; mp >100 °C dec.

Ga(Trip)₂Cl (7). A THF solution (100 mL) of Mg(Trip)₂(THF)₂ (5.74 g, 10 mmol) was added dropwise to GaCl₃ (1.76 g, 10 mmol) in THF (100 mL). The solution was refluxed for 12 h, and the THF was removed under reduced pressure. The residue was extracted with hexane (3 × 100 mL) and filtered. Removal of the hexane yielded a white solid, which crystallized from ca. 40 mL of hexane at –20 °C: yield 3.3 g, 65%; mp 161–163 °C; ¹H NMR δ 1.20 (d, *p*-CHMe₂), 1.30 (d, *o*-CHMe₂), 2.77 (sept, *p*-CHMe₂), 3.01 (sept, *o*-CHMe₂), 7.05 (s, aryl H).

Ga(Trip)₂Br (8). A THF solution of TripMgBr (76 mL, 0.33 M) was added dropwise to a THF solution (150 mL) of GaCl₃ (1.76, 10 mmol) over a period of 1 h. After the addition was complete, the mixture was refluxed overnight. The THF was removed under reduced pressure, and the residue was extracted with hexane (3 × 100 mL). Filtration and complete removal of the hexane yielded a clear, yellow oil which crystallized at room temperature upon standing for several hours: yield 2.9 g, 53%; mp 167–169 °C. Further purification of the compound by recrystallization from ethers, hexane, or pentane resulted in greatly reduced yields. However, the ¹H NMR (C₆D₆) of the crude crystals revealed that the product had >95% purity: δ 1.18 (d, *p*-CHMe₂), 1.31 (d, *o*-CHMe₂), 2.75 (sept, *p*-CHMe₂), 3.05 (sept, *o*-CHMe₂), 7.07 (s, aryl H).

Ga(Trip)₃ (9). A THF solution of TripMgBr was prepared according to the literature procedure. The Grignard solution (120 mL, 0.33 M, 39.6 mmol) was added dropwise to a THF solution (200 mL) of GaCl₃ (1.76 g, 10 mmol) over a period of 1

Table II. Selected Atomic Coordinates ($\times 10^4$) and Isotropic Thermal Parameters ($\text{\AA}^2 \times 10^3$) for 1-12

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ^a		<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ^a
Compound 1									
Al	1112(1)	-1926(1)	8300(1)	17(1)	C(1)	1666(2)	208(2)	6923(2)	18(1)
Cl(1)	1875(1)	-2490(1)	10333(1)	25(1)	C(2)	589(2)	1267(2)	6306(2)	19(1)
Cl(2)	1821(1)	-3644(1)	7284(1)	26(1)	C(6)	3274(2)	714(2)	6508(2)	20(1)
O	-1060(1)	-2387(1)	9168(1)	19(1)					
Compound 2									
Al	2934(5)	10864(5)	6788(2)	18(2)	C(1)	1466(17)	12102(16)	7578(8)	18(5)
Br(1)	4688(2)	8213(2)	7022(1)	32(1)	C(2)	1265(18)	11497(17)	8337(8)	24(6)
Br(2)	1341(2)	10922(2)	5682(1)	30(1)	C(6)	438(19)	13716(18)	7349(8)	37(6)
O(1)	4757(12)	11789(11)	6485(5)	25(4)					
Compound 3									
Al(1)	3828(2)	8480(2)	7381(1)	20(1)	C(17)	4664(6)	7108(6)	6447(4)	21(3)
Al(2)	992(2)	7791(2)	7453(1)	19(1)	C(21)	4971(7)	6652(7)	7638(5)	25(3)
Br(1)	2962(1)	8454(1)	8389(1)	22(1)	C(31)	36(7)	8832(6)	7307(4)	18(3)
Br(2)	1866(1)	7843(1)	6465(1)	24(1)	C(32)	-859(7)	8582(6)	6609(5)	18(3)
C(1)	4406(7)	9958(6)	7562(5)	21(3)	C(36)	158(6)	9797(6)	7792(4)	17(3)
C(2)	3908(7)	10673(7)	7169(5)	24(3)	C(46)	660(1)	6426(1)	7648(1)	23(1)
C(6)	5328(7)	10348(7)	8243(5)	21(3)	C(47)	710(1)	5463(1)	7228(1)	23(1)
C(16)	4770(7)	7318(6)	7178(4)	19(3)	C(51)	564(1)	6461(1)	8359(1)	21(1)
Compound 4									
Ga	498(1)	3683(1)	2282(1)	32(1)	C(1)	3469(3)	3428(5)	1917(3)	30(2)
Cl(1)	5727(1)	5665(1)	1961(1)	34(1)	C(2)	3088(4)	3368(5)	1132(3)	38(2)
Cl(2)	5865(1)	3067(2)	3410(1)	43(1)	C(6)	2729(4)	3258(5)	2414(3)	33(2)
O	5699(3)	2219(3)	1724(2)	36(1)					
Compound 5									
Ga	186(3)	925(1)	6367(1)	25(1)	C(1)	3577(10)	1399(4)	7162(9)	24(4)
Cl(1)	-232(2)	1293(1)	4732(2)	15(1)	O	-1526(9)	740(3)	1523(8)	53(4)
Cl(2)	1889(3)	143(1)	5251(2)	31(1)	Li	-737(19)	395(7)	3361(16)	34(7)
Cl(3)	1092(3)	549(1)	7893(2)	34(1)					
Compound 6									
Ga	3749(1)	5134(1)	571(1)	19(1)	C(6)	4528(3)	4072(2)	3497(3)	22(1)
Cl	4452(1)	4138(1)	-911(1)	22(1)	C(10)	2616(3)	5950(2)	-739(4)	20(1)
C(1)	3602(3)	4473(2)	2360(4)	20(1)	C(11)	2590(3)	6765(2)	-65(4)	21(1)
C(2)	2476(3)	4317(2)	2410(4)	22(1)	C(15)	1780(3)	5761(2)	-2189(5)	23(1)
Compound 7									
Ga	2032(1)	779(1)	3643(1)	23(1)	C(6)	1879(12)	-323(12)	1939(12)	32(5)
Cl	831(3)	1498(3)	3838(3)	17(2)	C(16)	2988(11)	441(11)	4889(11)	25(4)
C(1)	1914(10)	594(11)	2307(10)	22(4)	C(17)	2796(11)	-239(11)	5514(11)	22(4)
C(2)	1810(10)	1433(11)	1701(10)	19(4)	C(21)	3811(12)	960(13)	5234(12)	35(5)
Compound 8									
Ga	7917(1)	792(1)	1380(1)	198(1)	C(6)	8165(6)	1444(6)	3285(5)	20(2)
Br	9229(1)	1504(1)	1219(1)	39(1)	C(16)	6960(6)	471(5)	140(5)	19(2)
C(1)	8032(5)	630(5)	2680(5)	19(2)	C(17)	6128(6)	1001(6)	-184(6)	28(2)
C(2)	8029(6)	-311(6)	3054(6)	27(2)	C(21)	7148(6)	-222(6)	-416(5)	23(2)
Compound 9									
Ga	2335(1)	1307(1)	1476(1)	24(1)	C(17)	1445(11)	3361(11)	984(4)	27(4)
C(1)	3322(11)	401(11)	1143(3)	27(3)	C(21)	-20(12)	2144(13)	1258(4)	38(4)
C(2)	4494(12)	525(13)	1184(4)	42(4)	C(31)	2485(10)	1077(12)	2037(3)	32(4)
C(6)	2839(11)	-398(12)	858(4)	32(4)	C(32)	2704(12)	2113(13)	2280(4)	43(4)
C(16)	1161(10)	2411(11)	1223(4)	29(3)	C(36)	2372(11)	-83(12)	2196(4)	32(4)
Compound 10									
Al	0	2929(2)	2500	24(1)	C(2)	-706(5)	5082(4)	1559(6)	16(1)
Br(1)	1077(1)	1886(1)	1951(1)	47(1)	C(6)	-1229(6)	3388(5)	307(7)	32(2)
C(1)	0	4507(6)	2500	16(2)					
Compound 11									
Ga(1)	0	2952(1)	2500	43(1)	C(2)	-701(6)	5123(5)	1572(8)	19(2)
Cl(1)	-1039(2)	1885(2)	3066(4)	40(2)	C(6)	-1702(12)	5185(11)	-665(12)	59(4)
C(1)	0	4557(10)	2500	20(4)					
Compound 12									
In(1)	3068(2)	1775(1)	1379(1)	22(1)	In(2)	1925(2)	-472(1)	-1302(1)	24(1)
Cl(1)	2767(6)	818(4)	463(2)	23(2)	Cl(3)	2238(6)	-1540(3)	-450(2)	15(2)
Cl(2)	3346(6)	754(4)	2260(14)	23(2)	Cl(4)	1610(6)	-1406(4)	-2233(2)	24(2)
C(1)	3077(20)	3163(16)	1331(14)	32(2)	C(19)	1951(20)	926(17)	-1275(14)	32(2)
C(2)	2120(22)	3642(15)	1304(15)	32(2)	C(20)	1002(23)	1406(15)	-1285(15)	32(2)
C(6)	4041(22)	3624(15)	1347(15)	32(2)	C(25)	-123(28)	971(21)	-1325(15)	32(2)

^a Equivalent isotropic *U* defined as one-third of the trace of the orthogonalized U_{ij} tensor.

h. After the addition was complete, the mixture was refluxed overnight. The solvent was then removed under reduced pressure,

and the resulting off-white, pasty solid was extracted three times with 100-mL portions of hexane. The combined extracts were

Table III. Summary of Important Bond Distances (Å) and Angles (deg) for 1-12

compd	M-C	M-X	M-O	C-M-X	X-M-X	C-M-C
Al(Mes)Cl ₂ ·THF (1)	1.969(1)	2.148(5)	1.852(1)	113.5(1) 115.3(1)	110.3(1)	
Al(Trip)Br ₂ ·Et ₂ O (2)	1.976(14)	2.311(6)	1.865(11)	124.2(4) 114.0(4)	103.4(2)	
[Al(Trip) ₂ Br] ₂ (3)	1.973(10)	2.500(3) 2.475(4)			93.3(6)	130.0(4)
Ga(Trip)Cl ₂ ·THF (4)	1.946(4)	2.211(3)	2.011(4)	117.7(1) 126.6(1)	106.3(1)	
[(Et ₂ O)LiCl ₃ GaTrip] ₂ (5)	1.944(8)	2.270(8)		116.4(2) 123.5(3) 114.7(3)	98.0(1) 104.7(1) 96.1(1)	
[Ga(Mes) ₂ Cl] ₂ (6)	1.972(3)	2.407(1) 2.384(1)		105.4(1) 113.2(1)	84.1(1)	126.6(2)
Ga(Trip) ₂ Cl (7)	1.954(16)	2.220(5)		113.1(2.2)		133.8(7)
Ga(Trip) ₂ Br (8)	1.955(12)	2.324(2)		112.2(15)		135.5(4)
Ga(Trip) ₃ (9)	1.972(11)					116.6(5) 121.4(5) 121.9(5)
Al(Mes*)Br ₂ (10)	1.953(8)	2.281(2)		124.4(1)	111.1(1)	
Ga(Mes*)Cl ₂ (11)	1.953(13)	2.212(4)		125.4(1)	109.2(2)	
In(Mes*)Cl ₂ (12)	2.110(24)	2.408(6)		124.3(8)	102.7(2)	

then filtered and concentrated to ca. 40 mL. Cooling in a -20 °C freezer afforded 9 as colorless crystals: yield 5.3 g, 78%; ¹H NMR (C₆D₆) δ 1.23 (d, *o*-CHMe₂), 1.39 (d, *p*-CHMe₂), 2.80 (sept, *o*-CHMe₂), 3.00 (sept, *p*-CHMe₂), 7.04 (s, aryl H).

Al(Mes*)Br₂ (10). With rapid stirring Mes*Br (8.2 g, 25 mmol) in hexane (120 mL) and Et₂O (80 mL), cooled to ca. -10 °C in an ice/salt mixture, were treated dropwise with *n*-BuLi (15.6 mL of 1.6 M solution in hexane). The addition resulted in a pale yellow solution which was stirred at room temperature for a further 16 h. AlBr₃ (2.67 g, 10 mmol) in *n*-hexane (50 mL) cooled in an ice/salt bath were added dropwise by a double-tipped needle. The solution was then refluxed for 12 h. After it was cooled to ambient temperature, the solution was filtered and all volatile materials were removed under reduced pressure. Extraction with *n*-hexane (100 mL) followed by filtration and reduction of the solution volume to ca. 30 mL resulted in the appearance of colorless crystals on the walls of the Schlenk tube. Cooling in a -20 °C freezer overnight gave the product 10 as colorless crystals: mp 143-145 °C; yield 1.95 g (65%); ¹H NMR (C₆D₆) δ 1.39 (s, 9H, *p*-*t*-Bu), 1.67 (s, 18H, *o*-*t*-Bu), 7.38 (s, 2H, *m*-H).

Ga(Mes*)Cl₂ (11). *n*-BuLi (9.4 mL of a 1.6 M hexane solution), cooled to ca. -10 °C in an ice/salt mixture, was added dropwise to an *n*-hexane/Et₂O (60:40) solution (75 mL) of Mes*Br (4.88 g, 15 mmol) with rapid stirring. The solution, which was allowed to come to room temperature and stirred for 10 h, was added dropwise to GaCl₃ (1.76 g, 10 mmol) in *n*-hexane (30 mL) with cooling in an ice bath. The mixture was refluxed for 10 h and filtered. The solvent was removed under reduced pressure, and the residue was redissolved in *n*-hexane (60 mL). The volume of the filtrate was reduced to ca. 20 mL. Cooling in a -40 °C bath for 20 h afforded the product 8 as colorless crystals: yield 1.35 g, 35%; mp 134-135 °C; ¹H NMR (C₆D₆) δ 1.43 (s, 9H, *p*-*t*-Bu), 1.59 (s, 18H, *o*-*t*-Bu), 7.42 (s, 2H, *m*-H).

In(Mes*)Cl₂ (12). The procedure used to generate LiMes* was identical with that for 10. This solution was added to InCl₃ (2.21 g, 10 mmol) in Et₂O (50 mL), and the mixture was refluxed overnight. The solvent was removed by distillation, and the residue was taken up in an *n*-hexane/Et₂O mixture (90:10, 60 mL). Filtration, followed by volume reduction to ca. 25 mL and storage in a -20 °C freezer overnight, gave the product 11 as a colorless crystals: yield 1.68 g, 39%; mp 158 °C; ¹H NMR (C₆D₆) δ 1.35 (s, 9H, *p*-*t*-Bu), 1.71 (s, 18H, *o*-*t*-Bu), 7.39 (s, 2H, *m*-H).

X-ray Crystallographic Studies. Crystals of the compounds were mounted on the diffractometer by transferring them from the Schlenk tubes under N₂ to Petri dishes and immediately covering them with a layer of hydrocarbon oil. A suitable crystal was selected, attached to a glass fiber with silicone grease, and immediately placed in the low-temperature nitrogen stream.¹⁷

The X-ray data for the compounds were collected by using Syntex P2₁ (1, 5, 6, 8, 9, 11), Siemens R3m/V (2, 3, 11, 12), and Siemens P3RA (4, 7) diffractometers equipped with locally modified Syntex LT-1 or a locally modified Enraf-Nonius LT (R3m/V) device for low-temperature work. All data were collected by using graphite-monochromated Mo K α radiation ($\lambda = 0.71069$ Å). Calculations for compound 1 were carried out on a Data General Eclipse computer, and those for the remaining compounds were performed on a Micro VAX 3200 computer using the SHELXTL PLUS program system. Important crystallographic parameters and refinement data are provided in Table I. An ω -scan method was used for all data collections as described in ref 17. No decay in the intensities of two standard reflections was observed during the course of the data collections. Scattering factors were from common sources.¹⁸ The structures of 1 and 3-9 were solved by direct methods; those of 2 and 10-12 were solved by Patterson synthesis. Hydrogen atoms were included at calculated positions by using a riding model with C-H = 0.96 Å and $U_{\text{iso}}(\text{H}) = 1.2[U_{\text{iso}}(\text{C})]$ or $1.2[U^*_{\text{iso}}(\text{C})]$ for 1-8, where U^*_{iso} is the equivalent isotropic thermal parameter and fixed $U(\text{H}) = 0.08$ Å (9), 0.03 Å (10), and 0.04 Å (11 and 12). Disorder was modeled for the C(25) isopropyl groups in compounds 7 and 8. An absorption correction was applied by using the method described in ref 19 for all compounds. Only the Ga atom was refined anisotropically in compound 9. The metal and halogen atoms were refined anisotropically in 7, 8, and 10-12. All non-hydrogen atoms were refined anisotropically in the remaining compounds. The coordinates of important atoms are given in Table II. Important bond distances and angles are listed in Table III.

Results and Discussion

General Considerations. The major objective of the work described in this paper is the synthesis of unassociated aryl/halide derivatives of aluminum, gallium, and indium. These compounds are of importance because they are key precursors for the synthesis of further low-coordinate derivatives of these elements. Of particular

(17) Hope, H. In *Experimental Organometallic Chemistry: A Practicum in Synthesis and Characterization*; Wayda, A L., Darenbourg, M. Y., Eds.; ACS Symposium Series 357; American Chemical Society: Washington, D.C., 1987; Chapter 10.

(18) *International Tables for X-ray Crystallography*; Kynoch Press: Birmingham, England, 1974; Vol. IV.

(19) The absorption correction was made by using the program XABS by H. Hope and B. Moezzi. The program obtains an absorption tensor from $F_o - F_c$ differences. Moezzi, B. Ph.D. Dissertation, University of California, Davis, CA, 1987.

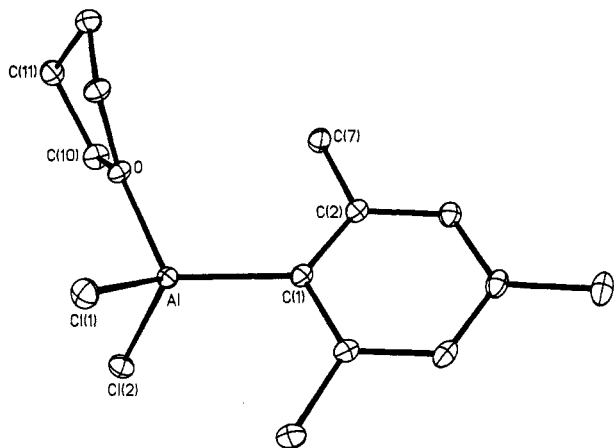


Figure 1. Thermal ellipsoid plot of $\text{Al}(\text{Mes})_2\text{Cl}_2\cdot\text{THF}$ (sharing 30% occupancy) (1). H atoms are omitted for clarity.

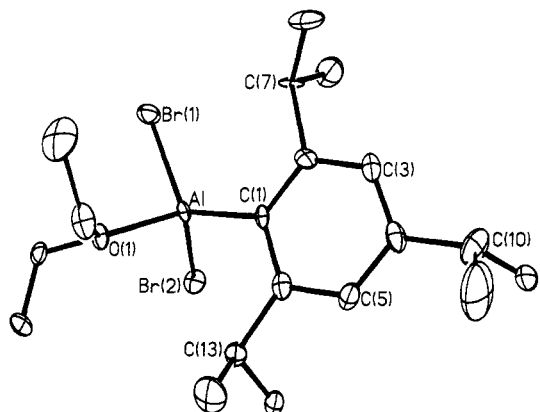


Figure 2. Thermal ellipsoid plot of $\text{Al}(\text{Trip})\text{Br}_2\cdot\text{Et}_2\text{O}$ (sharing 30% occupancy) (2). H atoms are omitted for clarity.

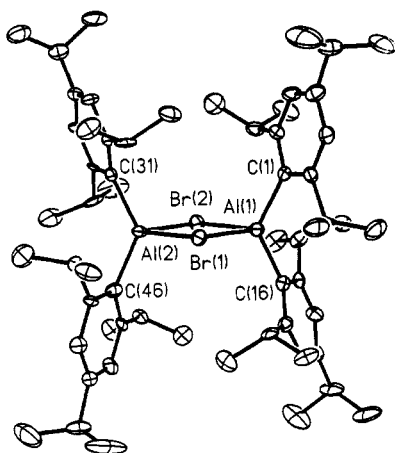
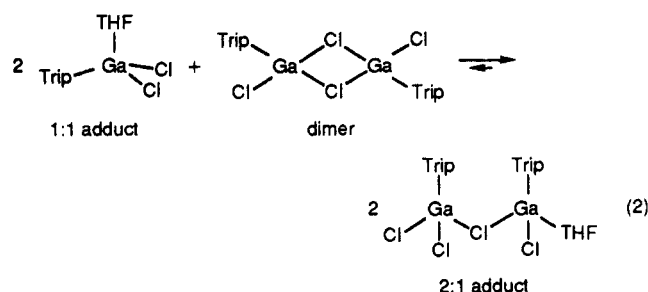


Figure 3. Thermal ellipsoid plot of $[\text{Al}(\text{Trip})_2\text{Br}]_2$ (sharing 30% occupancy) (3). H atoms are omitted for clarity.

relevance as possible objectives are species that could have homonuclear multiple bonds between the elements aluminum, gallium, and indium, or compounds that have π bonding between these metals and elements such as C, N, O, S, P, or As. Since multiple bonding of this kind has yet to be established with certainty,²⁰ it is important to have the widest possible range of precursors that have sufficient steric crowding to make association, via the formation of bridges, unlikely in their subsequent chemistry. It is also

desirable to have structural data on such compounds in order to derive the maximum possible information from comparisons with subsequent structural determinations.

A further objective of this work was to find the simplest possible synthetic routes to the title compounds. Frequently, conventional routes to the organic derivatives of metals (involving the reaction of the metal halide with an organolithium or Grignard reagent in ethers) are not readily applicable to derivatives of aluminum or gallium owing to the inclusion of strongly bound donor molecules (i.e. Et_2O or THF) in the products.²¹ The structures of 1 (Figure 1), 2 (Figure 2), 4 (Figure 4), and 5 (Figure 5) bear out this tendency. The presence of the donor molecule is also detectable in solution, as in the case of 1, 2, and 4. In some instances, however, an equilibrium with other species occurs. This may be what happens in the case of $\text{Ga}(\text{Trip})\text{Cl}_2\cdot\text{THF}$ in C_6D_6 , where $[\text{Ga}(\text{Trip})\text{Cl}_2]_2$ is also probably present in solution owing to the equilibrium defined by eq 2. The formation of such chlorine bridges



has precedent in Ga^{22} and Al^{23} chemistry. Even the hindered species $\text{Al}(\text{Mes})_3$ binds THF fairly strongly, and heating to 180–200 °C at 3 mmHg is required to remove the donor molecule.¹⁵ It is notable, therefore that the synthesis of compounds 3 and 7–12 proceed by the normal lithium or magnesium halide elimination route in the presence of ethers without the inclusion of donor molecules in the product.

At present, there is very little information available for mononuclear organometal halide derivatives of aluminum, gallium, or indium. The structures of a number of unassociated purely organic derivatives of these elements are, of course, well-known, e.g. MPh_3 ($\text{M} = \text{Ga}, \text{In}$),^{24,25} $\text{M}(\eta^1\text{-C}_5\text{H}_5)_3$ (Ga ;²⁶ the In analog²⁷ is associated with the metal coordinated by two terminal and two bridging ligands), MMes_3 ($\text{M} = \text{Al}$,²⁸ Ga ,²⁹ In ;³⁰), or $\text{In}[\text{CH}(\text{SiMe}_3)_2]_3$,³¹ which have essentially trigonal-planar metal coordination. However, if one or more of the organo groups are replaced by a halogen, the compounds generally become

(21) Mole, T.; Jeffery, E. A. *Organoaluminum Compounds*; Elsevier: Amsterdam, 1972.

(22) Wilson, I. L.; Dehnicke, K. *J. Organomet. Chem.* 1974, 67, 229. Dehnicke, K.; Wilson, I. L. *J. Chem. Soc., Dalton Trans.* 1973, 1428.

(23) Atwood, J. L. In *Inclusion Compounds-Structural Aspects of Inclusion Compounds Formed by Inorganic and Organometallic Host Lattices*; Atwood, J. L., Davies, J. E. D., MacNicol, D. D., Eds.; Academic: London, 1988; Vol. 1, Chapter 9, p 375.

(24) Malone, J. F.; McDonald, W. S. *J. Chem. Soc. A* 1970, 3362.

(25) Weak intermolecular association is apparent in these derivatives.

(26) Beachley, O. T.; Getman, T. D.; Kirss, R. U.; Hallock, R. B.; Hunter, W. E.; Atwood, J. L. *Organometallics* 1985, 4, 751.

(27) Einstein, F. W. B.; Gilbert, M. M.; Tuck, D. G. *Inorg. Chem.* 1972, 11, 2832.

(28) Jerius, J. J.; Hahn, J. M.; Rahman, A. F. M. M.; Mols, O.; Ilsley, W. H.; Oliver, J. P. *Organometallics* 1986, 5, 1812.

(29) Beachley, O. T.; Churchill, M. R.; Pazik, J. C.; Ziller, J. W. *Organometallic* 1986, 5, 1814.

(30) Leman, J. T.; Barron, A. R. *Organometallics* 1989, 8, 2214.

(31) Carty, A. J.; Gynane, M. J. S.; Lappert, M. F.; Miles, S. J.; Singh, A.; Taylor, N. J. *Inorg. Chem.* 1980, 19, 3637.

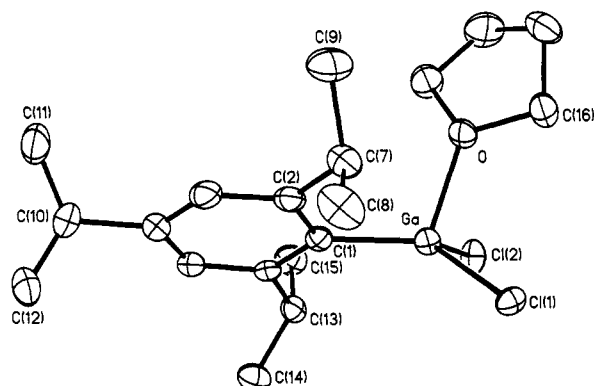


Figure 4. Thermal ellipsoid plot of $\text{Ga}(\text{Trip})\text{Cl}_2\cdot\text{THF}$ (sharing 30% occupancy) (4). H atoms are omitted for clarity.

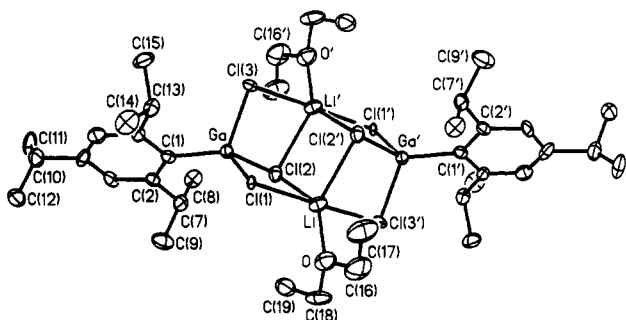


Figure 5. Thermal ellipsoid plot of $[(\text{Et}_2\text{O})\text{LiCl}_3\text{GaTrip}]_2$ (sharing 30% occupancy) (5). H atoms are omitted for clarity.

associated through halide bridging, as exemplified by the tetrameric character of $\text{Al}(t\text{-Bu})_2\text{F}^{32}$ and the dimeric structures of $[\text{Ga}(t\text{-Bu})_2\text{Cl}]_2^{33}$ and $[\text{Ga}(\text{CH}_2\text{SiMe}_3)_2\text{Cl}]_2^{34}$ in C_6H_6 solution. In addition, X-ray structural data for the dimers $[\text{Ga}(\eta^1\text{-C}_5\text{Me}_5)_2\text{Cl}]_2^{35}$, $[\text{Ga}(\eta^1\text{-C}_5\text{Me}_5)_2\text{Cl}]_2^{35}$, $[\text{Ga}(\text{Mes})_2\text{Cl}]_2$ (6), and $[\text{In}(\text{Mes})_2\text{Cl}]_2^{30}$ bear out this view. A notable feature of the structure of 6 is the asymmetry in the bridging Ga–Cl distances (2.407(1) vs 2.384(1) Å). Asymmetry is also present in the bridge bonds in the structure of $[\text{Al}(\text{Trip})_2\text{Br}]_2$ (vide infra), which may be indicative of incipient dissociation into monomers. This asymmetry in the bridging metal halogen distances has already been reported in the case of $[\text{Ga}(\eta^1\text{-C}_5\text{Me}_5)_2\text{Cl}]_2$, where Ga–Cl distances of 2.432(7) and 2.499(8) Å were observed.³⁵ It is significant that these distances are considerably longer than those observed in 6 and in $[\text{Ga}(\eta^1\text{-C}_5\text{Me}_5)_2\text{Cl}]_2$ (Ga–Cl = 2.352(3) and 2.372(3) Å).³⁵ This is, very probably, a consequence of the large size of the C_5Me_5 group. The weakness of the bridging in this compound is underscored by its monomeric nature in benzene. The species $\text{Al}\{\text{CH}(\text{SiMe}_3)_2\}_2\text{Cl}$ was also seen to be monomeric in this solvent.³⁶ Clearly, if large enough groups are employed, association can be prevented (at least in solution) in the case of the diorgano halide derivatives, although no structures of such species had been published prior to this work. For the less crowded organo dihalide derivatives no mononuclear species have been characterized either in solution or in the crystal phase.

(32) Lemhkuhl, J.; Olbrysch, O.; Nehl, H. *Justus Liebigs Ann. Chem.* 1973, 708.

(33) Schwering, H. U.; Jungk, E.; Weidlein, J. *J. Organomet. Chem.* 1975, 91, C4. Cleaver, W. M.; Barron, A. R. *Chemtronics* 1989, 4, 146.

(34) Beachley, O. T.; Simmons, R. G. *Inorg. Chem.* 1980, 19, 1021.

(35) Beachley, O. T.; Hallock, R. B.; Zhang, H. M.; Atwood, J. L. *Organometallics* 1985, 4, 1675.

(36) Al-Hashimi, S.; Smith, J. D. *J. Organomet. Chem.* 1978, 153, 253.

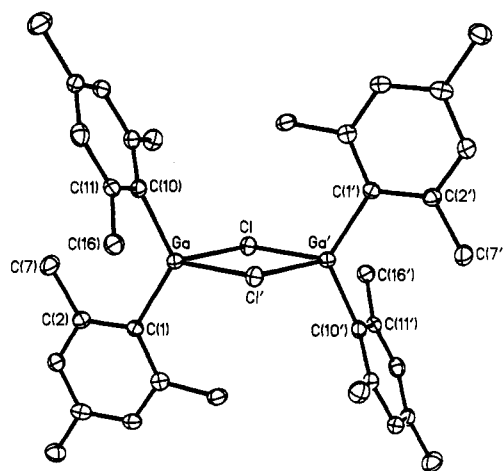


Figure 6. Thermal ellipsoid plot of $[\text{Ga}(\text{Mes})_2\text{Cl}]_2$ (sharing 30% occupancy) (6). H atoms are omitted for clarity.

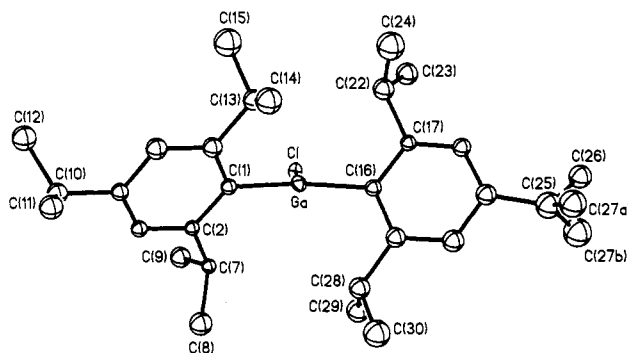


Figure 7. Thermal ellipsoid plot of $\text{Ga}(\text{Trip})_2\text{Cl}$ (sharing 30% occupancy) (7). H atoms are omitted for clarity.

Derivatives of 2,4,6-*i*-Pr₃C₆H₃– (Trip). Recent work has shown that the presence of two mesityl groups on boron is sufficient to prevent association in a variety of inorganic compounds, whereas smaller groups such as phenyl are unable to do so.³⁷ Owing to the greater sizes of aluminum (radius 1.3 Å) and gallium (radius 1.2 Å),³⁸ two mesityl groups are insufficient to prevent association, as is evident from the dimeric structures of $[\text{M}(\text{Mes})_2\text{Cl}]_2$ (M = Ga (6), In³⁰) (Figure 6). In the case of gallium, replacement of the mesityl substituents by larger Trip groups results in the monomeric structure from $\text{Ga}(\text{Trip})_2\text{X}$ (X = Cl (7), Br (8)) (illustrated by Figure 7). Both compounds have distorted-trigonal-planar coordination at the metal. The C–Ga–C angles, however, are a good deal wider (>13°) than the idealized value of 120°. This is no doubt due to the large size of the Trip substituents. Both structures are also characterized by rather high (60.5–69.2°) angles between the aromatic ring planes and the plane at gallium. The closest approaches of H's from the ortho *i*-Pr group to the Ga center are in the range 2.5–2.7 Å (cf. the much shorter approaches observed in $\text{Ga}(\text{Mes}^*)\text{Cl}_2$ below). It is notable that neither 7 nor 8 forms adducts with ether or THF, whereas $[\text{Ga}(\eta^1\text{-C}_5\text{H}_5)_2\text{Cl}]_2$ can do so, although in this case the THF can be removed at room temperature.

In spite of the congestion in 7 and 8, it is possible to fit one further Trip group around gallium by using slightly more forcing reaction conditions and excess TripMgBr to obtain $\text{Ga}(\text{Trip})_3$ (9) (Figure 8). This crowded molecule, which does not form complexes with THF or pyridine, has

(37) Power, P. P. *Angew. Chem., Int. Ed. Engl.* 1990, 29, 449.

(38) Huheey, J. E. *Inorganic Chemistry*, 3rd ed.; Harper and Row: New York, 1983; p 258. The radius of Ga is probably closer to 1.25 Å.

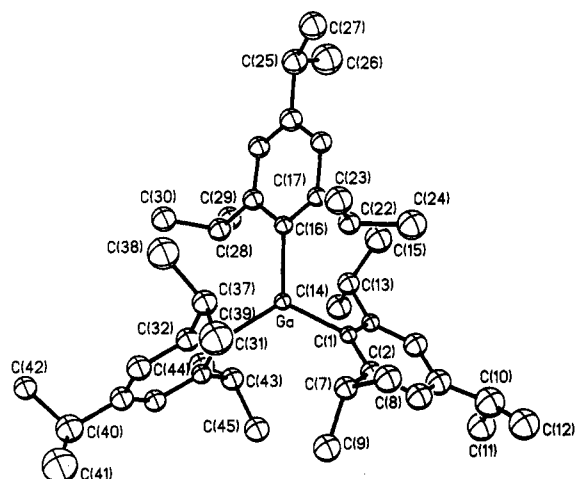


Figure 8. Thermal ellipsoid plot of $\text{Ga}(\text{Trip})_3$ (sharing 30% occupancy) (9). H atoms are omitted for clarity.

a structure that is very similar to that of GaMes_3 .²⁹ The average Ga–C distance in 9, 1.972(11) Å, is essentially identical with that found in GaMes_3 , 1.968(4) Å. As in the case of 7 and 8, the structure of 9 is characterized by high angles (ca. 60°) between the plane at gallium and those at the aromatic rings.

Whereas two or three Trip groups on gallium prevent association in the case of 7–9, the same is not true for the aluminum analogue $[\text{Al}(\text{Trip})_2\text{Br}]_2$ (3) (Figure 3), which is associated through Br bridges. The contrast between the structure of 3 and those of 7 and 8 underlines the greater Lewis acidity and larger size of aluminum vis à vis gallium. There is, however, evidence in the structural details that the association observed in 3 is weak. The Al–Br distances, which average a little less than 2.5 Å in length, are significantly longer than the bridging Al–Br distance in Al_2Br_6 (2.41 Å).³⁹ There is also the previously mentioned asymmetry in the bridging distances (two Al–Br bonds average 2.500(3) Å whereas the other two average 2.475(4) Å) that suggests dissociation into two monomers may be incipient. Indeed, cryoscopic measurements suggests that 3 is ca. 50% monomer in C_6H_6 . In addition, the C–Al–C angles (ca. 130°) are almost as wide as those observed in the gallium monomers 7 and 8 and the Al–C distances, ca. 1.97 Å, are normal for three-coordinate Al.

The structure of the compound $[(\text{Et}_2\text{O})\text{LiCl}_3\text{GaTrip}]_2$ (5) (Figure 5) is unique among the compounds discussed in this paper. This species, which is derived from the reaction of LiTrip with GaCl_3 in Et_2O in a 1:1 ratio, features two $[\text{Ga}(\text{Trip})\text{Cl}_3]^-$ moieties bridged by two Et_2O -solvated Li^+ ions. The structure is centrosymmetric, and two chlorines attached to gallium bridge μ_2 -wise to the Li^+ ion whereas the third Cl is bridge-bonded to the two lithiums and a gallium. The bond distances and angles at Ga, Li, and Cl are well within known ranges. The Ga–Cl distances are rather long, owing to the bridging nature of the chlorines. The Ga–C distance, however, is very similar to those observed in 4 or 11.

Derivatives of 2,4,6-*t*-Bu₃C₆H₂[−] (Mes*). A further increase in the size of the organo group to Mes* permits the isolation of the first unassociated organo dihalide derivatives of the elements aluminum, gallium, and indium.

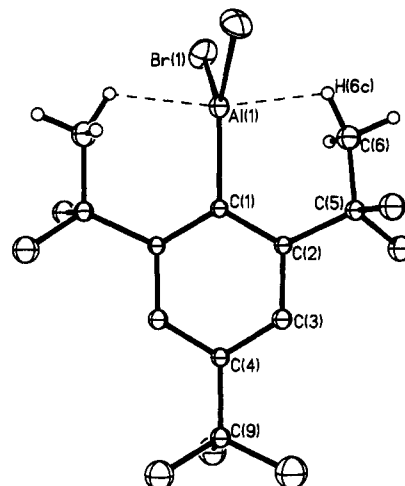


Figure 9. Thermal ellipsoid plot of $\text{Al}(\text{Mes}^*)\text{Br}_2$ (sharing 30% occupancy) (10). H atoms are omitted for clarity except hydrogens on methyl groups closest to the metal.

The steric protection afforded at the metal by the ortho *tert*-butyl groups can be appreciated if it is borne in mind that these three compounds (10–12) were synthesized in the presence of Et_2O . In the isomorphous aluminum and gallium derivatives 10 and 11 (illustrated by Figure 9), the molecules possess a 2-fold axis of symmetry (the para *t*-Bu group is disordered between two equivalent positions) along the M–C bond such that the plane of the Mes* ring is perpendicular to the plane at the metal. It is possible to argue that there is a weak interaction between one of the hydrogens of each of the ortho *t*-Bu groups and the metal. The closest H approach for aluminum is 2.11 Å, whereas for gallium it is only 1.86 Å. These distances are well within the sum of the van der Waals radii for these elements. Oddly, in the ortho *t*-Bu groups the angle at the central carbon atom subtended by the methyl group closest to the metal is wider (by ca. 4°) than those involving the other two methyls. This probably means that the interaction with the C(6) hydrogen is real, since minor strain is apparently induced in order to facilitate this interaction. Such differences are not observed in the indium species 12, although close approaches of ortho *t*-Bu hydrogens (2.08 and 2.24 Å) are apparent. The bond distances and angles at the metals in 10–12 are within known ranges. In 10–12, the angle between the halogens is significantly less than 120° owing to the different electronic properties of the halide and aryl groups and possibly to the large size of the Mes* group. The Al–C and Ga–C bond lengths are identical, which is in accordance with the smaller size of Ga relative to Al and the greater polarity of the Al–C bond. The Ga–Cl distance (2.242(4) Å) in 11 is quite close to that observed in 7 (2.220(5) Å). These are marginally longer than those in the four-coordinate species 4 (2.211(3) Å). They are also longer than the reported terminal Ga–Cl distance (2.10 Å) in Ga_2Cl_6 .

A remarkable feature of the data for compounds 1–11 in Table III is that there is very little variation (range 1.944–1.976 Å) in the M–C distances for the Al and Ga compounds in spite of the differences in the substituent sizes and coordination number of the metal. Thus, the formation of adducts with Et_2O or THF as exemplified by compounds 1, 2, and 4 does not result in a dramatic increase of the Al–C or Ga–C bond lengths in comparison to those of 10 or 11. Also, dimerization does not apparently result

(39) Shen, Q. Ph.D. Dissertation, Oregon State University, 1973. Some structural data for Al_2Br_6 and related species are quoted in: Wells, A. F. *Structural Inorganic Chemistry*, 4th ed.; Clarendon Press: Oxford, England, 1984; p 444.

in dramatic increases in M-C bond distances. Thus, the Ga-Cl bond in **6** is identical with that in **9** and marginally (0.02 Å) longer than those in **5**, **7**, and **8**. It may be noted that in the process of adduct formation or dimerization the hybridization change in the M-C bond is not as large as might be expected, since the angles at the metal involving the aryl and halide ligands remain relatively wide.

Conclusion

It has been shown that monomeric crystalline aryl-halogeno derivatives of aluminum, gallium, and indium can be obtained by using the bulky substituents Trip and Mes*. Moreover, many of these compounds may be

synthesized by straightforward synthetic procedures in the presence of ethers without giving ether-complexed products.

Acknowledgment. We thank the National Science Foundation and the donors of the Petroleum Research Fund, administered by the American Chemical Society, for financial support.

Supplementary Material Available: Tables of data collection parameters and refinement summaries and full tables of atom coordinates, bond distances and angles, anisotropic thermal parameters, and hydrogen coordinates (126 pages). Ordering information is given on any current masthead page.

OM920442M