The Use of Crown Ethers to Access New $M[Al_2R_6X]$ Species. Synthesis and Crystal Structure of $[K \cdot dibenzo\text{-}18\text{-}crown\text{-}6]$ $[Al_2Me_6Cl] \cdot 2\, C_6H_6$

JERRY L. ATWOOD,★ DUANE C. HRNCIR, and ROBIN D. ROGERS
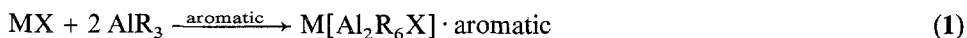*Department of Chemistry, University of Alabama, University, AL 35486, U.S.A.*

Abstract. Potassium chloride does not react with trimethylaluminium in benzene in the absence of a crown ether. With the addition of dibenzo-18-crown-6, a normal liquid clathrate ensues. The composition of the parent compound has been established by a single crystal X-ray diffraction study. The space group is monoclinic $P2_1/c$ with unit cell dimensions $a = 21.319(8)$, $b = 9.512(4)$, $c = 21.081(8)$ Å, $\beta = 93.79(3)°$, and $\rho_{calc} = 1.15$ g cm^{-3} for $Z = 4$. Refinement led to a final conventional R value of 0.049 for 2224 observed reflections. The anion has the expected angular geometry (Al – Cl – Al = 120.0(1)°). There are two different types of benzene in the crystalline solid. One is associated with the K^+ ion, while the other exists in layers normal to the a axis.

1. Introduction

Since the initial discovery of the novel solution behavior (liquid clathrate effect) of $K[Al_2Me_6N_3]$ [1], considerable effort has been devoted to (1) understanding the behavior [2], (2) developing uses for these systems [2,3], and (3) expanding the range of $M[Al_2R_6X]$ complexes [4,5]. The preferred method of synthesis is shown as reaction (**1**):

$$MX + 2\, AlR_3 \xrightarrow{\text{aromatic}} M[Al_2R_6X] \cdot \text{aromatic} \tag{1}$$

The span of MX species is wide indeed. Alkali metal halides gave way to alkali metal pseudohalides, and now M may include such cations as (η^6-tetralin) (η^5-cyclopentadienyl)iron$^+$ [6], while X may be virtually any simple anion. The principal limitation is that the lattice energy of MX must be overcome before the decomposition point of the AlR_3 (*ca.* 180 °C). Thus substances such as KCl, NaN_3, or $Ba(NO_3)_2$ were inaccessible according to (**1**). A major breakthrough was the use of crown ethers to 'solubilize' these problem MX compounds. In this contribution we discuss both the anionic structure of the title compound and the general utility of crown ethers in (**1**). In addition, we allude to the role of the molecules of crystallization in this and related substances.

2. Results and Discussion

Reaction of KCl with $AlMe_3$ in benzene according to (**1**) does not take place even at reflux temperature (60 °C) for several days. Addition of dibenzo-18-crown-6 in 1 : 1 ratio with the

---

★ Corresponding author.

KCl, however, immediately affords a liquid clathrate of composition [K · dibenzo-18-crown-6]-[Al_2Me_6Cl] · n C_6H_6 at 60 °C. Slow cooling to room temperature gives air-sensitive crystals of [K · dibenzo-18-crown-6] [Al_2Me_6Cl] · 2 C_6H_6. Similar reactivity has been observed for KNO_2, [7] KO_2 [8], KOH [9], and K_2SO_4 [10]. Two other observations should be noted. First, the rate of reaction can be accelerated by the use of crown ether even for reactions which occur as in (1). Second, the fact that the aluminum alkyls themselves form discrete complexes with crown ethers ([$AlMe_3$]$_2$[dibenzo-18-crown-6] [11], [$AlMe_3$]$_3$[dibenzo-18-crown-6] [12]) has no obvious effect on the outcome of the reaction with MX.
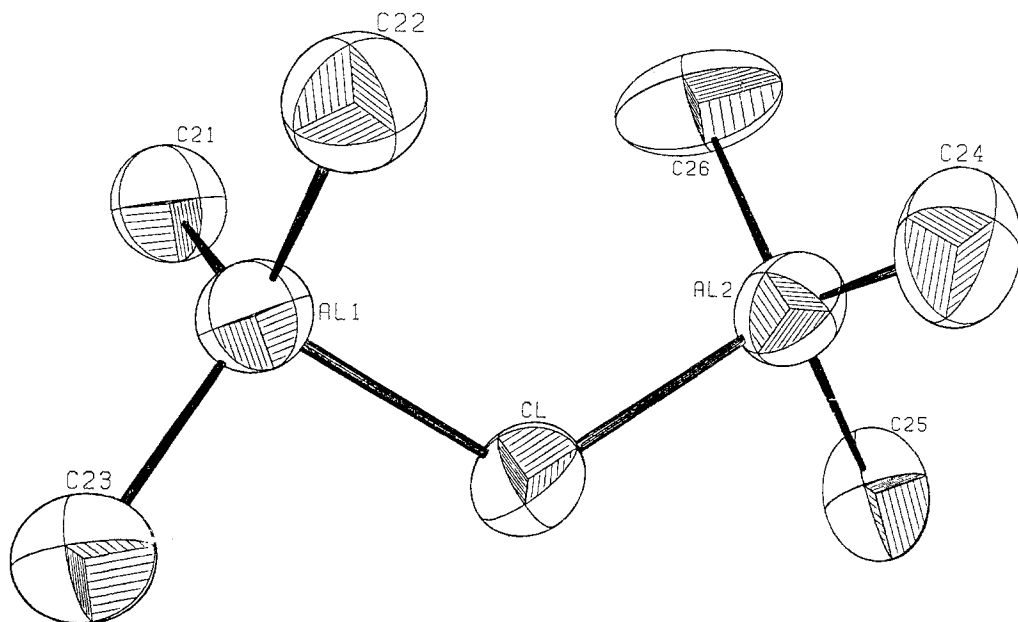

Fig. 1.   Structure of the [Al_2Me_6Cl]$^-$ anion.

The structure of the [Al_2Me_6Cl]$^-$ anion is shown in Figure 1. The angle at the chlorine atom, 120.0(1)°, gives the overall angular geometry which is prevalent in compounds which form liquid clathrates [2]. It is presumed that the chloride structure is typical of the numerous M[Al_2Me_6Br] and M[Al_2Me_6I] parent compounds. There are three X-ray determinations of complexes with fluoride in the bridging position, K[Al_2Et_6F] [13], K[Al_2Me_6F] [14], and Na[Al_2Me_6F] [15], and all possess linear Al – F – Al configurations. However, this is a peculiarity associated with F^-, H^- [16], and possibly CN^- [17]. Structures analogous to that in Figure 1 are known or assumed for the rest of the liquid clathrate parent anions [2].

The Al – Cl bond distances of 2.336(3) and 2.387(3)Å (Table I) as expected are longer than those found for terminal chlorines in K[$AlMeCl_3$] [18], 2.16 Å, and [$MeAlCl_2$]$_2$ [19], 2.05 Å. They are quite close to those found in other bridging situations (although the title compound is the first example of a single chlorine bridge): 2.34 Å in (η^5-C_5H_5)Fe(η^5-C_5H_4)Al_2Me_4Cl [20] and 2.38 Å in [(η^3-C_5Me_5)AlMeCl]$_2$ [21].

As Figure 1 illustrates, the methyl groups appear in a staggered configuration when viewed down the Al…Al bond vector. In K[$Al_2Me_6N_3$] there are two crystallographically different anions, one with staggered methyl groups and one in which they are eclipsed [22].

Table I. Bond lengths (Å) and angles (deg) for [K·dibenzo-18-crown-6]-[Al₂Me₆Cl]·2 C₆H₆

Atoms		Distance	Atoms		Distance
Cl	– Al(1)	2.336(3)	Cl	– Al(2)	2.387(3)
Al(1)	– C(21)	1.975(8)	Al(1)	– C(22)	1.968(8)
Al(1)	– C(23)	1.973(8)	Al(2)	– C(24)	1.952(8)
Al(2)	– C(25)	2.012(8)	Al(2)	– C(26)	1.988(9)
O(1)	– C(1)	1.388(9)	O(1)	– C(12)	1.429(8)
O(2)	– C(2)	1.374(9)	O(2)	– C(3)	1.435(9)
O(3)	– C(4)	1.426(9)	O(3)	– C(5)	1.404(9)
O(4)	– C(6)	1.426(9)	O(4)	– C(7)	1.381(9)
O(5)	– C(8)	1.370(9)	O(5)	– C(9)	1.422(9)
O(6)	– C(10)	1.414(9)	O(6)	– C(11)	1.416(9)
C(1)	– C(2)	1.40(1)	C(1)	– C(13)	1.35(1)
C(2)	– C(16)	1.37(1)	C(3)	– C(4)	1.48(1)
C(5)	– C(6)	1.49(1)	C(7)	– C(8)	1.40(1)
C(7)	– C(17)	1.36(1)	C(8)	– C(20)	1.38(1)
C(9)	– C(10)	1.49(1)	C(11)	– C(12)	1.48(1)
C(13)	– C(14)	1.38(1)	C(14)	– C(15)	1.39(1)
C(15)	– C(16)	1.37(1)	C(17)	– C(18)	1.40(1)
C(18)	– C(19)	1.36(1)	C(19)	– C(20)	1.38(1)
C(27)	– C(28)	1.32(1)	C(27)	– C(32)	1.34(1)
C(28)	– C(29)	1.31(1)	C(29)	– C(30)	1.36(2)
C(30)	– C(31)	1.38(2)	C(31)	– C(32)	1.35(2)
C(33)	– C(34)	1.32(4)	C(33)	– C(38)	1.34(2)
C(34)	– C(35)	1.37(3)	C(35)	– C(36)	1.31(2)
C(36)	– C(37)	1.29(2)	C(37)	– C(38)	1.32(2)
K	– O(1)	2.753(5)	K	– O(2)	2.747(5)
K	– O(3)	2.758(5)	K	– O(4)	2.762(5)
K	– O(5)	2.744(5)	K	– O(6)	2.705(5)
K	– C(22)	3.355(8)	K	– C(33)	3.50(3)
K	– C(34)	3.43(3)	K	– C(35)	3.32(1)
K	– C(36)	3.29(2)	K	– C(37)	3.34(2)
K	– C(38)	3.45(2)			

Atoms			Angle	Atoms			Angle
Al(1)	– Cl	– Al(2)	120.0(1)	Cl	– Al(1)	– C(21)	104.3(2)
Cl	– Al(1)	– C(22)	103.9(2)	C(21)	– Al(1)	– C(22)	115.9(3)
Cl	– Al(1)	– C(23)	99.7(3)	C(21)	– Al(1)	– C(23)	115.4(3)
C(22)	– Al(1)	– C(23)	114.7(3)	Cl	– Al(2)	– C(24)	103.5(3)
Cl	– Al(2)	– C(25)	97.5(2)	C(24)	– Al(2)	– C(25)	115.1(4)
Cl	– Al(2)	– C(26)	106.7(3)	C(24)	– Al(2)	– C(26)	114.9(4)
C(25)	– Al(2)	– C(26)	116.1(4)	C(1)	– O(1)	– C(12)	116.3(7)
C(2)	– O(2)	– C(3)	116.8(7)	C(4)	– O(3)	– C(5)	113.2(6)
C(6)	– O(4)	– C(7)	116.4(8)	C(8)	– O(5)	– C(9)	116.9(7)
C(10)	– O(6)	– C(11)	113.2(7)	O(1)	– C(1)	– C(2)	114.2(9)
O(1)	– C(1)	– C(13)	125(1)	C(2)	– C(1)	– C(13)	120.5(9)
O(2)	– C(2)	– C(1)	115.6(9)	O(2)	– C(2)	– C(16)	124(1)
C(1)	– C(2)	– C(16)	120.0(9)	O(2)	– C(3)	– C(4)	108.4(6)
O(3)	– C(4)	– C(3)	110.3(6)	O(3)	– C(5)	– C(6)	110.0(7)
O(4)	– C(6)	– C(5)	108.0(7)	O(4)	– C(7)	– C(8)	114(1)
O(4)	– C(7)	– C(17)	126(1)	C(8)	– C(7)	– C(17)	120(1)
O(5)	– C(8)	– C(7)	116.4(9)	O(5)	– C(8)	– C(20)	124(1)
C(7)	– C(8)	– C(20)	120(1)	O(5)	– C(9)	– C(10)	107.9(7)

Table I. Continued

Atoms	Angle	Atoms	Angle
O(6) – C(10) – C(9)	111.1(7)	O(6) – C(11) – C(12)	111.0(7)
O(1) – C(12) – C(11)	108.0(6)	C(1) – C(13) – C(14)	119.6(9)
C(13) – C(14) – C(15)	120.1(9)	C(14) – C(15) – C(16)	120.1(9)
C(2) – C(16) – C(15)	119.6(9)	C(7) – C(17) – C(18)	120(1)
C(17) – C(18) – C(19)	120(1)	C(18) – C(19) – C(20)	121(1)
C(8) – C(20) – C(19)	119(1)	C(28) – C(27) – C(32)	120(1)
C(27) – C(28) – C(29)	122(1)	C(28) – C(29) – C(30)	121(1)
C(29) – C(30) – C(31)	118(1)	C(30) – C(31) – C(32)	120(1)
C(27) – C(32) – C(31)	120(1)	C(34) – C(33) – C(38)	126(3)
C(33) – C(34) – C(35)	114(3)	C(34) – C(35) – C(36)	122(2)
C(35) – C(36) – C(37)	121(2)	C(36) – C(37) – C(38)	122(2)
C(33) – C(38) – C(37)	116(2)		

The role of the aromatic molecules in the solid state structure is of interest because of their crucial role in the liquid clathrates. One finds essentially two aromatic environments: those involved in an interaction with the cations and those which occupy space in the lattice (perhaps as in a typical solid inclusion situation). This is quite general for crystals derived from liquid clathrates [23]. The title compound possesses one benzene of each type, as will be demonstrated below.

The potassium ion resides at the center of the crown of six oxygen atoms, as shown in Figure 2. The average K...O contact of 2.74(2) Å is typical [8]. The six oxygen atoms are planar to within 0.09 Å, and the potassium ion lies 0.30 Å out of this plane. In [K · dibenzo-18-crown-6] [$Al_2Me_6O_2$] · 1.5 C_6H_6 [8] the mean K...O separation is 2.74(1)Å, and the K$^+$ resides 0.28 Å out of the plane of the crown.
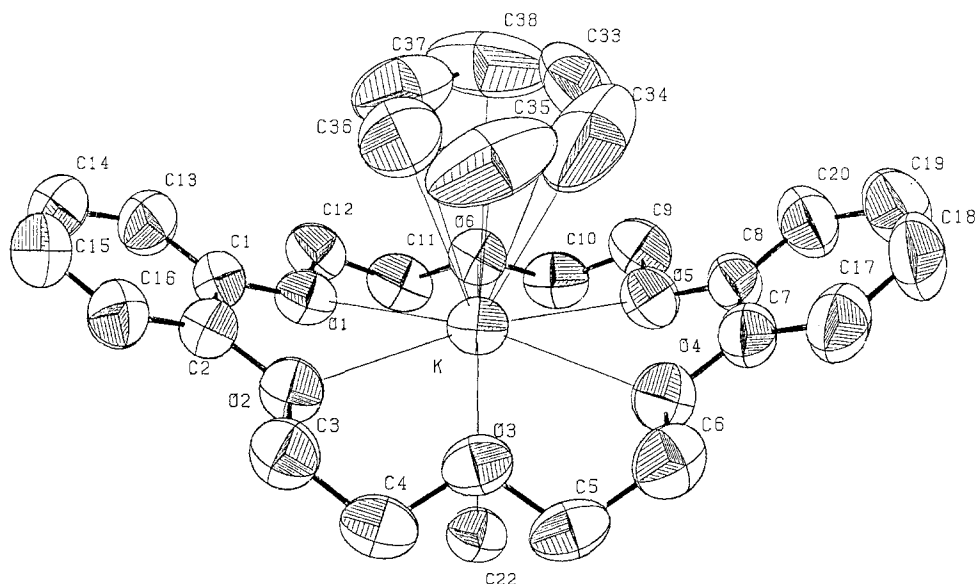

Fig. 2. Environment of the potassium ion: the six oxygen atoms of the crown ether occupy the equatorial positions, while one benzene molecule and a methyl group from the anion complete the coordination sphere.

One of the benzene rings is rather symmetrically positioned above the potassium ion. The K...C distances range from 3.29(2) to 3.50(3) Å and average 3.39(8)Å. A similar interaction has been found in [K · dibenzo-18-crown-6] [Al$_2$Me$_6$O$_2$] · 1.5 C$_6$H$_6$ with K...C(average) = 3.43(7)Å and in K[Al$_7$O$_6$C$_{16}$H$_{48}$] · C$_6$H$_6$ with K...C(average) = 3.33(5)Å [24]. This type of association of M$^+$ with aromatic pi-clouds is thought to be important in the stabilization of liquid clathrates.

The second independent benzene molecule has no intermolecular contacts out to 3.5 Å, and plays no role in the stabilization of the potassium ion. As may be seen from the stereoview in Figure 3, these aromatic moieties are arranged in layers normal to the crystallographic a axis.
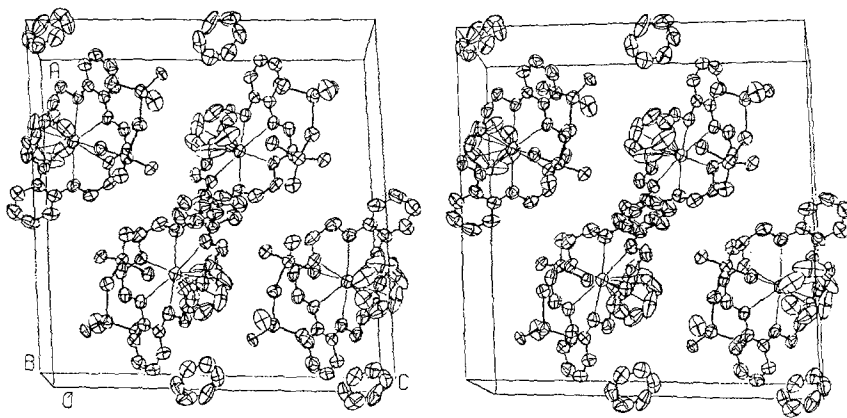

Fig. 3. Stereoscopic view of the unit cell packing in [K · dibenzo-18-crown-6] [Al$_2$Me$_6$Cl] · 2 C$_6$H$_6$. One half of the benzene molecules are arranged in layers normal to the crystallographic a axis.

3. Experimental

All manipulations were performed in an inert atmosphere glove box. Benzene was predried over sodium and freshly distilled from LiAlH$_4$ before use. Potassium chloride and trimethyl-aluminum were obtained from Alfa Ventron Corporation and used without further purification.

3.1. SYNTHESIS OF [K · Dibenzo-18-crown-6] [Al$_2$Me$_6$Cl] · 2 C$_6$H$_6$

Potassium chloride (0.75 g, 10 mmol) was mixed with 30 ml of benzene in a Fisher–Porter tube. Trimethylaluminum (2.0 ml, 20 mmol) was slowly syringed into the mixture. No visible sign of reaction was noted even after heating for several days at 80 °C. However, addition of 3.6 g (10 mmol) of dibenzo-18-crown-6 followed by heating to 80 °C afforded a liquid clathrate of composition {K · dibenzo-18-crown-6] [Al$_2$Me$_6$Cl] · n C$_6$H$_6$. Slow cooling to room temperature allowed isolation of air-sensitive crystals of the title compound.

3.2. X-RAY DATA COLLECTION, STRUCTURE DETERMINATION, AND REFINEMENT FOR [K · Dibenzo-18-crown-6] [Al$_2$Me$_6$Cl] · 2 C$_6$H$_6$

Single crystals of the air-sensitive compound were sealed under nitrogen in thin-walled glass capillaries. Final lattice parameters as determined from a least-squares refinement of

$((\sin \theta)/\lambda)^2$ values for 15 reflections ($\theta > 20°$) accurately centered on the diffractometer are given in Table II. The space group was uniquely determined as $P2_1/c$ from the systematic absences in $0k0$ for $k = 2n + 1$ and in $h0l$ for $l = 2n + 1$.

Data were collected on an Enraf–Nonius CAD–4 diffractometer by the θ–2θ scan technique. The method has been previously described [25]. A summary of data collection parameters is given in Table II. The intensities were corrected for Lorentz and polarization effects, but not for absorption.

Table II.   Crystal data and summary of intensity data collection and structure refinement

Compound	[K · dibenzo-18-crown-6] [Al_2Me_6Cl] · 2 C_6H_6
Mol wt.	735.4
Space group	$P2_1/c$
Cell constants	
a, Å	21.319(8)
b, Å	9.512(4)
c, Å	21.081(8)
β, deg	93.79(3)
Cell vol, Å^3	4265.6
Molecules/unit cell	4
ρ(calc), g cm^{-3}	1.15
μ(calc), cm^{-1}	2.71
Radiation	Mo$K\alpha$
Max crystal dimensions, mm	$0.20 \times 1.00 \times 1.38$
Scan width	$0.80 + 0.20 \tan\theta$
Standard reflections	400, 040, 004
Decay of standards	—
Reflections measured	3501
2θ range	2–36°
Reflections collected	2224
No. of parameters varied	433
GOF	2.21
R	0.049
R_w	0.049

Calculations were carried out with the SHELX system of computer programs [26]. Neutral atom scattering factors for K, Cl, Al, O, and C were taken from Cromer and Waber [27], and the scattering for potassium was correted for the real and imaginary components of anomalous dispersion using the table of Cromer and Libermann [28]. Scattering factors for H were from [29].

The structure was solved via the straightforward application of the direct methods program MULTAN [30]. Least-squares refinement with isotropic thermal parameters led to $R = (\Sigma||F_o| - |F_c||)/\Sigma|F_o| = 0.080$. The hydrogen atoms that could be placed at calculated positions were positioned 1.08 Å from the bonded carbon atom and were not refined. The methyl hydrogen atoms were located with the aid of a difference Fourier map. Refinement of the nonhydrogen atoms with anisotropic temperature factors led to final values of $R = 0.049$ and $R_w = 0.049$. A final difference Fourier showed no feature greater than $0.3\ e^-/$Å^3. The weighting scheme was based on unit weights; no systematic variation of $w(|F_o| - |F_c|)$ vs. $|F_o|$ or $(\sin \theta)/\lambda$ was noted. The final values of the positional parameters are given in Table III [31].

Table III. Final   fractional   coordinates   for   [K·dibenzo-18-crown-6]-[Al$_2$Me$_6$Cl]·2 C$_6$H$_6$

Atom	x/a	y/b	z/c
K	0.28932(8)	0.8839(2)	0.39629(7)
Cl	0.28886(9)	0.3779(2)	0.1883(1)
Al(1)	0.3701(1)	0.2368(2)	0.2326(1)
Al(2)	0.1838(1)	0.2897(3)	0.1780(1)
O(1)	0.1697(3)	0.9887(6)	0.3726(2)
O(2)	0.2114(3)	0.8177(5)	0.2916(2)
O(3)	0.3390(3)	0.7393(5)	0.2979(2)
O(4)	0.4162(2)	0.8222(5)	0.4044(3)
O(5)	0.3743(3)	0.9867(6)	0.4883(2)
O(6)	0.2500(3)	1.0888(5)	0.4733(2)
C(1)	0.1243(5)	0.904(1)	0.3419(4)
C(2)	0.1479(5)	0.809(1)	0.2986(5)
C(3)	0.2365(4)	0.7261(9)	0.2454(4)
C(4)	0.3032(5)	0.7638(8)	0.2394(4)
C(5)	0.4032(5)	0.7696(9)	0.2948(4)
C(6)	0.4381(4)	0.7327(9)	0.3563(5)
C(7)	0.4461(5)	0.810(1)	0.4642(6)
C(8)	0.4223(5)	0.900(1)	0.5095(6)
C(9)	0.3488(4)	1.076(1)	0.5341(4)
C(10)	0.3010(5)	1.1687(9)	0.5002(4)
C(11)	0.2023(5)	1.1724(9)	0.4423(4)
C(12)	0.1481(4)	1.084(1)	0.4186(4)
C(13)	0.0624(6)	0.906(1)	0.3518(4)
C(14)	0.0221(4)	0.813(2)	0.3188(6)
C(15)	0.0455(6)	0.716(1)	0.2773(5)
C(16)	0.1081(6)	0.716(1)	0.2665(4)
C(17)	0.4933(6)	0.718(1)	0.4816(6)
C(18)	0.5185(5)	0.716(1)	0.5443(9)
C(19)	0.4952(7)	0.803(2)	0.5880(6)
C(20)	0.4475(6)	0.896(1)	0.5714(6)
C(21)	0.3874(3)	0.1036(8)	0.1641(3)
C(22)	0.3350(3)	0.1544(8)	0.3082(3)
C(23)	0.4350(4)	0.3817(9)	0.2510(4)
C(24)	0.1559(4)	0.303(1)	0.2642(4)
C(25)	0.1479(3)	0.4356(9)	0.1168(3)
C(26)	0.1889(5)	0.0952(9)	0.1443(4)
C(27)	1.0047(9)	0.867(2)	0.1172(5)
C(28)	0.9581(8)	0.928(1)	0.084(1)
C(29)	0.9406(5)	0.889(2)	0.025(1)
C(30)	0.971(1)	0.782(3)	− 0.0028(6)
C(31)	1.019(1)	0.717(1)	0.032(1)
C(32)	1.0361(5)	0.761(2)	0.091(1)
C(33)	0.290(2)	0.842(3)	1.028(1)
C(34)	0.318(1)	0.915(3)	0.984(2)
C(35)	0.278(2)	0.954(1)	0.933(1)
C(36)	0.219(1)	0.913(2)	0.9282(9)
C(37)	0.1966(6)	0.836(2)	0.971(1)
C(38)	0.231(2)	0.794(2)	1.022(1)
H(1)[C(3)]	0.234	0.620	0.257
H(2)[C(3)]	0.211	0.730	0.201
H(3)[C(4)]	0.305	0.868	0.226
H(4)[C(4)]	0.322	0.710	0.202

Table III.   Continued

Atom	x/a	y/b	z/c
H(5)[C(5)]	0.421	0.718	0.255
H(6)[C(5)]	0.410	0.874	0.284
H(7)[C(6)]	0.486	0.741	0.351
H(8)[C(6)]	0.430	0.628	0.364
H(9)[C(9)]	0.326	1.021	0.568
H(10)[C(9)]	0.382	1.135	0.559
H(11)[C(10)]	0.323	1.221	0.466
H(12)[C(10)]	0.285	1.244	0.530
H(13)[C(11)]	0.187	1.250	0.470
H(14)[C(11)]	0.218	1.222	0.402
H(15)[C(12)]	0.111	1.143	0.399
H(16)[C(12)]	0.131	1.032	0.456
H(17)[C(13)]	0.043	0.976	0.384
H(18)[C(14)]	− 0.025	0.812	0.324
H(19)[C(15)]	0.014	0.645	0.253
H(20)[C(16)]	0.124	0.641	0.235
H(21)[C(17)]	0.510	0.648	0.446
H(22)[C(18)]	0.556	0.645	0.555
H(23)[C(19)]	0.516	0.796	0.634
H(24)[C(20)]	0.430	0.962	0.606
H(25)[C(21)]	0.433	0.045	0.180
H(26)[C(21)]	0.404	0.145	0.128
H(27)[C(21)]	0.344	0.052	0.142
H(28)[C(22)]	0.315	0.235	0.334
H(29)[C(22)]	0.371	0.103	0.336
H(30)[C(22)]	0.297	0.097	0.297
H(31)[C(23)]	0.449	0.464	0.217
H(32)[C(23)]	0.474	0.327	0.270
H(33)[C(23)]	0.419	0.444	0.280
H(34)[C(24)]	0.183	0.228	0.292
H(35)[C(24)]	0.160	0.408	0.298
H(36)[C(24)]	0.104	0.290	0.277
H(37)[C(25)]	0.185	0.460	0.084
H(38)[C(25)]	0.095	0.416	0.098
H(39)[C(25)]	0.168	0.435	0.162
H(40)[C(26)]	0.217	0.033	0.173
H(41)[C(26)]	0.176	0.039	0.103
H(42)[C(26)]	0.216	0.177	0.128
H(43)[C(27)]	1.017	0.902	0.162
H(44)[C(28)]	0.932	1.010	0.102
H(45)[C(29)]	0.902	0.938	− 0.002
H(46)[C(30)]	0.957	0.749	− 0.048
H(47)[C(31)]	1.043	0.633	0.011
H(48)[C(32)]	1.073	0.713	0.117
H(49)[C(33)]	0.322	0.815	1.068
H(50)[C(34)]	0.363	0.951	0.988
H(51)[C(35)]	0.297	1.017	0.896
H(52)[C(36)]	0.190	0.943	0.887
H(53)[C(37)]	0.148	0.803	0.967
H(54)[C(38)]	0.215	0.727	1.058

Acknowledgement

References

1. J. L. Atwood and W. R. Newberry: *J. Organomet. Chem.* **42**, C77 (1972).
2. J. L. Atwood: *Recent Dev. Sep. Sci.* **3**, 195 (1977).
3. J. J. Harrison and J. C. Montagna: *Sep. Sci. Tech.* **17**, 1151 (1982).
4. J. L. Atwood, J. E. D. Davies, and D. D. MacNicol, (eds): *Inclusion Compounds*, vol I. Academic Press, London (1983).
5. G. H. Robinson and J. L. Atwood: *J. Inclusion Phenom.*, to be submitted.
6. A. W. Coleman and J. L. Atwood, unpublished results.
7. R. D. Priester and J. L. Atwood, unpublished results.
8. D. C. Hrncir, R. D. Rogers, and J. L. Atwood: *J. Am. Chem. Soc.* **103**, 4277 (1981).
9. J. L. Atwood and J. Weeks, unpublished results.
10. J. L. Atwood and R. D. Rogers: *Organometallics*, in press.
11. J. L. Atwood, D. C. Hrncir, R. Shakir, M. S. Dalton, R. D. Priester, and R. D. Rogers: *Organometallics* **1**, 1021 (1982).
12. J. L. Atwood, R. D. Priester, R. D. Rogers, and L. G. Canada: *J. Inclusion Phenom.* **1**, 61 (1983).
13. G. Allegra and G. Perego: *Acta Crystallogr.* **16**, 185 (1963).
14. J. L. Atwood and W. R. Newberry: *J. Organomet. Chem.* **66**, 15 (1974).
15. J. L. Atwood, A. J. Baskar, and M. J. Zaworotko, unpublished results.
16. J. L. Atwood, D. C. Hrncir, R. D. Rogers, and J. A. K. Howard: *J. Amer. Chem. Soc.* **103**, 6787 (1981).
17. J. L. Atwood and R. E. Cannon: *J. Organomet. Chem.* **47**, 321 (1973).
18. J. L. Atwood, D. C. Hrncir, and W. R. Newberry: *Cryst. Struct. Commun.* **3**, 615 (1974).
19. G. Allegra, G. Perego, and A. Immirizi: *Macromol. Chem.* **61**, 69 (1963).
20. R. D. Rogers, W. J. Cook, and J. L. Atwood: *Inorg. Chem.* **18**, 279 (1979).
21. P. R. Schonberg, R. T. Paine, and C. F. Campana: *J. Amer. Chem. Soc.* **101**, 7726 (1979).
22. J. L. Atwood and W. R. Newberry: *J. Organomet. Chem.* **65**, 145 (1974).
23. J. L. Atwood and S. K. Seale: *J. Organomet. Chem.* **114**, 107 (1976); M. J. Zaworotko and J. L. Atwood, unpublished results.
24. J. L. Atwood, D. C. Hrncir, R. D. Priester, and R. D. Rogers: *Organometallics* **2**, 985 (1983).
25. J. Holton, M. F. Lappert, D. G. H. Ballard, R. Pearce, J. L. Atwood, W. E. Hunter: *J. Chem. Soc. Dalton Trans.* 46 (1979).
26. SHELX, a system of computer programs for X-ray structure determination by G. M. Sheldrick (1976).
27. D. T. Cromer, J. T. Waber: *Acta Crystallogr.* **18**, 104 (1965).
28. D. T. Cromer, D. Liberman: *J. Chem. Phys.* **53**, 1891 (1970).
29. *'International Tables for X-ray Crystallography'*, Vol. IV, Kynoch Press, Birmingham, England, 1974, p. 72.
30. G. Germain, P. Main, M. M. Woolfson: *Acta Crystallogr.* **3**, A27 (1971).
31. Tables of thermal parameters and structure factors are available from the BLLD by quoting SUP 90079.