Journal of Organometallic Chemistry, 65 (1974) 145—154

THE INTERACTION OF AROMATIC HYDROCARBONS WITH ORGANOMETALLIC COMPOUNDS OF THE MAIN GROUP ELEMENTS

II. SOLUTION BEHAVIOR AND CRYSTAL STRUCTURE OF $K[Al_2(CH_3)_6N_3]$

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(Received May 30th, 1973)

Summary

$K[Al_2(CH_3)_6N_3]$ interacts with benzene or toluene to form liquid complexes with from 2.5 to 5 aromatic molecules per anionic unit. The relation of this behavior to the structure of the anion is discussed. The crystal structure of $K[Al_2(CH_3)_6N_3]$ has been determined from three-dimensional X-ray data measured by counter methods. The substance crystallizes in the triclinic space group $P\bar{1}$ with cell dimensions a 11.960(5), b 11.327(5), c 10.705(5) Å, α 93.68(4), β 97.33(4), γ 99.20(4)°, and $\rho_{calc.}$ 1.06 $g{\cdot}cm^{-3}$ for $Z = 4$. Least-squares refinement gave a conventional R value of 0.095 (weighted $R = 0.079$) for 1631 observed reflections. In the asymmetric unit there are two geometrically different single-nitrogen bridged anions. One has approximate C_{2v} point symmetry and exhibits an eclipsed configuration of the methyl groups viewed down the aluminum-aluminum vector, while the other is best described by the C_s point group and shows a staggered methyl group conformation.

Introduction

Over the past several years, a wide range of organoaluminum compounds have been studied in a variety of aromatic solvents [1 - 3]. It was only, however, with the preliminary report [4,5] of the formation of "complexes" between benzene, toluene, or *p*-xylene and $M[Al_2(CH_3)_6X]$ (M = K, Cs, $N(CH_3)_4$; $X = N_3$, SCN, SeCN, Cl, I) that an interaction of appreciable strength was defined. In this paper we wish to present the details of the completed crystal structure of $K[Al_2(CH_3)_6N_3]$, and to relate the geometrical parameters of the anion to the nature of the complex of this and related compounds with aromatic molecules.

TABLE 1

FINAL FRACTIONAL COORDINATES AND THERMAL PARAMETERS[a,b] FOR $K[Al_2(CH_3)_6N_3]$

Atom	x/a	y/b	z/c	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
K(1)	0.2986(3)	0.1683(3)	−0.0732(3)	0.0125(4)	0.0115(4)	0.0122(3)	−0.0010(3)	0.0003(3)	0.0021(3)
K(2)	0.2041(3)	0.6343(3)	0.3928(2)	0.0149(4)	0.0138(4)	0.0063(3)	−0.0018(3)	−0.0002(3)	0.0010(3)
Al(1)	0.3340(4)	0.5921(4)	−0.1871(3)	0.0105(5)	0.0106(5)	0.0067(4)	0.0003(4)	0.0002(3)	0.0019(3)
Al(2)	0.1454(4)	0.7568(4)	0.0045(3)	0.0150(6)	0.0121(5)	0.0068(4)	0.0004(4)	0.0032(4)	0.0022(3)
Al(3)	0.2098(4)	0.2490(4)	0.3971(4)	0.0118(5)	0.0139(6)	0.0112(5)	0.0006(5)	0.0004(4)	0.0041(4)
Al(4)	0.2874(4)	0.0652(4)	0.6132(4)	0.0117(5)	0.0108(5)	0.0099(4)	−0.0007(4)	0.0017(4)	0.0021(4)
N(1)	0.1720(8)	0.6327(9)	−0.1306(7)	0.0072(11)	0.0135(14)	0.0067(9)	−0.0004(10)	−0.0003(8)	0.0005(9)
N(2)	0.0776(10)	0.5708(11)	−0.1840(9)	0.0069(12)	0.0195(14)	0.0080(10)	−0.0030(9)	0.0007(9)	0.0046(10)
N(3)	−0.0065(10)	0.5149(11)	−0.2314(9)	0.0110(14)	0.0215(15)	0.0120(10)	−0.0055(11)	−0.0014(10)	0.0016(10)
N(4)	0.3241(9)	0.1965(9)	0.5258(8)	0.0058(11)	0.0142(15)	0.0094(9)	−0.0002(10)	0.0007(9)	0.0018(9)
N(5)	0.4255(10)	0.2550(10)	0.5573(8)	0.0083(13)	0.0181(14)	0.0100(11)	0.0036(12)	0.0024(10)	0.0066(10)
N(6)	0.5226(11)	0.3112(11)	0.5855(10)	0.0095(13)	0.0217(15)	0.0149(10)	−0.0027(11)	0.0009(11)	0.0061(9)
C(1)	0.3430(14)	0.4259(12)	−0.1530(12)	0.0195(15)	0.0105(16)	0.0153(14)	0.0026(13)	0.0003(13)	0.0062(12)
C(2)	0.2937(13)	0.6046(14)	−0.3528(9)	0.0179(17)	0.0255(16)	0.0039(14)	0.0025(14)	0.0007(12)	0.0036(12)
C(3)	0.4655(11)	0.7101(12)	−0.0949(10)	0.0104(14)	0.0158(13)	0.0084(13)	−0.0026(11)	−0.0008(12)	−0.0004(12)
C(4)	0.2357(14)	0.6993(12)	0.1396(10)	0.0213(15)	0.0133(14)	0.0076(15)	0.0003(11)	−0.0020(12)	0.0051(12)
C(5)	0.2147(15)	0.9157(11)	−0.0302(12)	0.0262(15)	0.0079(15)	0.0141(14)	0.0005(16)	0.0077(15)	0.0032(16)
C(6)	−0.0359(15)	0.7513(15)	0.0064(12)	0.0214(15)	0.0237(17)	0.0110(15)	0.0071(15)	0.0060(14)	0.0051(15)
C(7)	0.3229(13)	0.3708(12)	0.3708(12)	0.0172(15)	0.0172(15)	0.0140(14)	0.0121(11)	0.0121(11)	0.0074(11)
C(8)	0.1597(20)	0.0997(15)	0.2776(13)	0.0415(18)	0.0154(19)	0.0122(16)	−0.0042(18)	−0.0108(18)	0.0003(16)
C(9)	0.0645(13)	0.3135(14)	0.4727(13)	0.0095(16)	0.0219(15)	0.0201(15)	0.0036(14)	0.0027(14)	0.0099(13)
C(10)	0.1346(12)	0.1119(13)	0.6811(11)	0.0108(14)	0.0198(15)	0.0118(13)	0.0004(14)	0.0024(13)	0.0034(13)
C(11)	0.4463(12)	0.0698(12)	0.7234(11)	0.0122(13)	0.0149(15)	0.0108(14)	0.0020(12)	0.0023(14)	0.0053(12)
C(12)	0.2567(16)	−0.0895(13)	0.5015(11)	0.0286(15)	0.0114(16)	0.0102(15)	−0.0014(15)	0.0025(16)	0.0006(14)

[a] Standard deviations in parentheses refer to *last digit quoted.* [b] Anisotropic thermal parameters defined by exp $[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$

Experimental

The preparation of $K[Al_2(CH_3)_6N_3]$ may be accomplished in two ways. The sealed tube reaction of 0.01 mole of KN_3 with 0.02 mole of $Al(CH_3)_3$ goes to completion in one hour at 90°, but it is difficult to achieve purity on the microscopic level. In fact, to obtain a satisfactory elemental analysis it was often necessary to open the original tube, crush the white crystalline substance, and add excess $Al(CH_3)_3$ as many as three times. (At no time did we note any evidence for the formation of a compound of stoichiometry higher than 2:1.) A second and more desirable synthesis is based on the initial formation of the liquid complex with toluene. If 0.01 mole of KN_3, 0.02 mole of $Al(CH_3)_3$, and 0.10 mole of toluene are sealed together, complete reaction is obtained in only a few minutes at 50°. Large colorless crystals of $K[Al_2(CH_3)_6N_3]$ may then be formed by cooling the liquid complex. The process is a reversible one:

$$K[Al_2(CH_3)_6N_3]\cdot n\ C_6H_5CH_3 \underset{50^\circ}{\overset{-196^\circ}{\rightleftharpoons}} K[Al_2(CH_3)_6N_3] + n\ C_6H_5CH_3$$

($n = 2.5 \rightarrow 5$). It should also be noted that the initial reaction may be visually monitored, since the more dense liquid complex is immiscible with the excess toluene.

Single crystals of $K[Al_2(CH_3)_6N_3]$ were grown by the slow cooling of the toluene complex, but since they were invariably too large for an X-ray study, they were cut before being sealed in thin-walled glass capillaries. The crystal system is triclinic, and statistical tests based on normalized structure factors indicate the space group to be the centric $P\bar{1}$. The lattice parameters as determined from a least-squares refinement of the angular settings of 12 reflections ($2\theta > 80°$; CuK_α, λ = 1.54051 Å; T ≈ 24°) accurately centered on an Enraf-Nonius CAD-4 diffractometer are:

a 11.960(5), b 11.327(5), c 10.705(5)Å; α 93.68(4),

β 97.33(4), γ 99.20(4)°; $\rho_{calc.}$ 1.06 g cm^{-1} for $Z = 4$.

Data were collected on the CAD-4 diffractometer with Ni-filtered copper radiation. The crystal, an irregular polyhedron of approximate dimensions 0.30 mm × 0.30 mm × 0.50 mm, was aligned on the diffractometer such that no major crystallographic axis was coincident with the ϕ axis of the diffractometer.

The diffracted intensities were collected by the ω - 2θ scan technique with a take-off angle of 1.5°. The scan rate was variable and was determined by a fast (20°$\cdot$min^{-1}) pre-scan. Calculated speeds based on the net intensity gathered in the pre-scan ranged from 7° to 0.4°$\cdot$min^{-1}. Background counts were collected for 25% of the total scan time at each end of the scan range. For each intensity the scan width was determined by the equation: scan range = $A + B \tan\theta$, where A is 1.0° and B is 0.5°. Aperture settings were determined in a like manner with A 4 mm and B 3 mm. Other diffractometer parameters and the method of calculating standard deviations have been described previously [6]. As a check on the stability of the instrument and the crystal, one standard peak was measured periodically during data collection. A decay of about 20% was noted; the reflection data were broken into groups and scaled appropriately.

One unique hemisphere of data was measured out to 2θ 140°; 1631 reflections with intensities greater than background were recorded. The intensities were corrected in the usual manner for Lorentz and polarization effects but not for absorption because of the moderate size of the linear absorption coefficient (μ 39.5 cm^{-1}) and the highly irregular shape of the crystal.

Fourier calculations were made with the ALFF [7] program. The full-matrix, least squares refinement was carried out using the Busing and Levy program ORFLS [8]. The function Σ W $(|F_o| - |F_c|)^2$ was minimized. No corrections were made for extinction or anomalous dispersion. Neutral atom scattering factors were taken from the compilations of Cromer and Waber [9]. Final bond distances, angles, and errors were computed with the aid of the Busing, Martin, and Levy ORFFE [10] program. The crystal structure illustrations were obtained with the program ORTEP [11].

Structure determination and refinement

Partial structure solution was accomplished by direct methods [12]. An electron density map phased on the initial potassium and aluminum positions revealed the coordinates of all nonhydrogen atoms in the asymmetric unit. Subsequent isotropic refinement led to a reliability index of $R_1 = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|$ of 0.17. Conversion to anisotropic refinement yielded R_1 0.109; introduction of a weighting scheme ($W = 1/\sigma^2$) and several cycles of

TABLE 2

INTERATOMIC DISTANCES (Å) AND ANGLES (DEG) FOR $K[Al_2(CH_3)_6N_3]$

Bond Distance			
Al(1) - N(1)	2.00(1)	Al(3) - N(4)	2.03(1)
Al(2) - N(1)	2.03(1)	Al(4) - N(4)	2.00(1)
Al(1) - C(1)	1.99(1)	Al(3) - C(7)	2.00(1)
Al(1) - C(2)	2.00(1)	Al(3) - C(8)	2.02(1)
Al(1) - C(3)	1.97(1)	Al(3) - C(9)	2.02(1)
Al(2) - C(4)	2.00(1)	Al(4) - C(10)	1.98(1)
Al(2) - C(5)	2.03(1)	Al(4) - C(11)	2.01(1)
Al(2) - C(6)	1.94(1)	Al(4) - C(12)	2.01(1)
N(1) - N(2)	1.24(1)	N(4) - N(5)	1.22(1)
N(2) - N(3)	1.10(1)	N(5) - N(6)	1.17(1)
Bond angles			
Al(1) - N(1) - Al(2)	128.2(5)	Al(3) - N(4) - Al(4)	127.8(5)
C(1) - Al(1) - C(2)	115.6(6)	C(7) - Al(3) - C(8)	111.6(6)
C(1) - Al(1) - C(3)	114.3(6)	C(7) - Al(3) - C(9)	115.6(6)
C(2) - Al(1) - C(3)	116.6(6)	C(8) - Al(3) - C(9)	116.7(6)
C(4) - Al(2) - C(5)	116.5(6)	C(10) - Al(4) - C(11)	116.2(6)
C(4) - Al(2) - C(6)	112.2(5)	C(10) - Al(4) - C(12)	112.8(6)
C(5) - Al(2) - C(6)	111.8(6)	C(11) - Al(4) - C(12)	111.9(6)
C(1) - Al(1) - N(1)	100.2(5)	C(7) - Al(3) - N(4)	102.4(5)
C(2) - Al(1) - N(1)	101.1(5)	C(8) - Al(3) - N(4)	104.5(5)
C(3) - Al(1) - N(1)	106.0(5)	C(9) - Al(3) - N(4)	104.0(5)
C(4) - Al(2) - N(1)	104.2(5)	C(10) - Al(4) - N(4)	103.6(5)
C(5) - Al(2) - N(1)	105.2(5)	C(11) - Al(4) - N(4)	103.4(5)
C(6) - Al(2) - N(1)	106.0(5)	C(12) - Al(4) - N(4)	107.7(6)
Al(1) - N(1) - N(2)	114.5(8)	Al(3) - N(4) - N(5)	116.6(7)
Al(2) - N(1) - N(2)	117.2(6)	Al(4) - N(4) - N(5)	115.5(6)
N(1) - N(2) - N(3)	179.6(1.1)	N(4) - N(5) - N(6)	178.8(1.0)

TABLE 3

ROOT-MEAN-SQUARE AMPLITUDES OF VIBRATION (Å)

Atom	Min	Intermed.	Max
K(1)	0.25(1)	0.28(1)	0.30(1)
K(2)	0.21(1)	0.27(1)	0.32(1)
Al(1)	0.21(1)	0.25(1)	0.26(1)
Al(2)	0.20(1)	0.27(1)	0.30(1)
Al(3)	0.25(1)	0.27(1)	0.31(1)
Al(4)	0.24(1)	0.26(1)	0.28(1)
N(1)	0.19(2)	0.23(2)	0.30(2)
N(2)	0.18(2)	0.22(2)	0.37(2)
N(3)	0.21(2)	0.30(2)	0.40(2)
N(4)	0.18(2)	0.26(2)	0.30(2)
N(5)	0.21(2)	0.22(2)	0.35(2)
N(6)	0.22(2)	0.30(2)	0.39(2)
C(1)	0.21(2)	0.33(2)	0.35(2)
C(2)	0.16(2)	0.32(2)	0.40(2)
C(3)	0.20(2)	0.27(2)	0.34(2)
C(4)	0.17(2)	0.27(2)	0.32(2)
C(5)	0.21(2)	0.28(2)	0.40(2)
C(6)	0.25(2)	0.32(2)	0.41(2)
C(7)	0.19(2)	0.32(2)	0.34(2)
C(8)	0.22(2)	0.33(2)	0.54(2)
C(9)	0.22(2)	0.30(2)	0.42(2)
C(10)	0.24(2)	0.28(2)	0.35(2)
C(11)	0.24(2)	0.26(2)	0.32(2)
C(12)	0.25(2)	0.28(2)	0.41(2)

least-squares refinement gave final values of $R_1 = 0.095$ and

$R_2 = [\Sigma W(|F_o| - |F_c|)^2/\Sigma(F_o)^2]^{1/2} = 0.079$.

Hydrogen atoms were not located, and unobserved reflections were not included. The largest parameter shifts in the final cycle of refinement were less than 0.05 of their estimated standard deviations. A final difference Fourier map showed no feature greater than 0.8 $e^-/\text{Å}^3$. No systematic variation of $W(|F_o| - |F_c|)^2$ vs. $|F_o|$ or $(\sin\theta)/\lambda$ was observed. The final values of the positional and thermal parameters are given in Table 1*.

Discussion

In the asymmetric unit there are two $Al_2(CH_3)_6N_3^-$ ions which are geometrically as well as crystallographically nonequivalent. One has approximate C_{2v} point symmetry and exhibits an eclipsed configuration of the methyl groups viewed down the aluminum-aluminum vector (Fig. 1). The other belongs to the C_s point groups and shows a staggered methyl group conformation (Fig. 2). As is indicated in Table 2, the bond distances and angles in both forms are quite similar. The root-mean-square amplitudes of vibration along the principal axes of vibration are given in Table 3. The direction of vibration may be seen in Fig. 2 and 3.

The conformation of methyl groups or of methyl hydrogen atoms in

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[*] The Table of structure factors has been deposited as NAPS Document No. 02236, with ASIS/NAPS, c/o Microfiche Publications, 305 E. 46th Street, New York, New York 10017. A copy may be secured by citing the document and remitting $1.50 for microfiche or $5.00 for photocopies. Advance payment is required. Make checks payable to Microfiche Publications.

TABLE 4

WEIGHTED[a] LEAST-SQUARES PLANES

Plane
$A(C_s$ anion$)\ -0.0950X + 0.7730Y - 0.6273Z - 6.5453 = 0$
$B(C_{2v}$ anion$)\ 0.5343X - 0.5479Y - 0.6437Z + 3.2260 = 0$

DEVIATIONS OF ATOMS FROM PLANES (Å)

Atom	Plane A	Atom	Plane B
Al(1)	−0.04[b]	Al(3)	−0.08
Al(2)	−0.07	Al(4)	0.00
N(1)	−0.01	N(4)	−0.02
N(2)	0.01	N(5)	−0.05
N(3)	0.01	N(6)	−0.07
C(3)	0.06	C(7)	0.13
C(6)	0.05	C(11)	0.08

[a] The weight of each atom was inversely proportional to the mean variance of the positional parameters of that atom. [b] The standard deviation for the distance of each atom from the plane is less than 0.02 Å.

compounds of this general type has been the subject of some speculation [13]. In fact, Weller and Dehnicke [14,15] have determined from spectroscopic studies that the anions in $[N(CH_3)_4][Al_2(CH_3)_6N_3]$ and $[N(CH_3)_4]$-$[Al_2(CH_3)_6SCN]$ are of C_{2v} symmetry. In the present situation, the existence of both C_{2v} and C_s forms may be taken as an indication of the closeness in overall energy of the two configurations in solution. The lattice and coordination requirements of the counter-ion might therefore in most cases be expected to subtly influence the geometry of the anion [16]. It is also worth noting that the barrier to rotation of the methyl groups appears to be quite high: the thermal motion of the carbon atoms is not excessive, and the best plane calculations (Table 4) show that the plane of symmetry is rather closely maintained for each anion.

As the two independent anions differ geometrically, so also do the two potassium ions differ environmentally (Fig. 3,4). One is most closely associated with the terminal nitrogen atoms of two azide ions (2.92(1), 2.93(1)Å), with

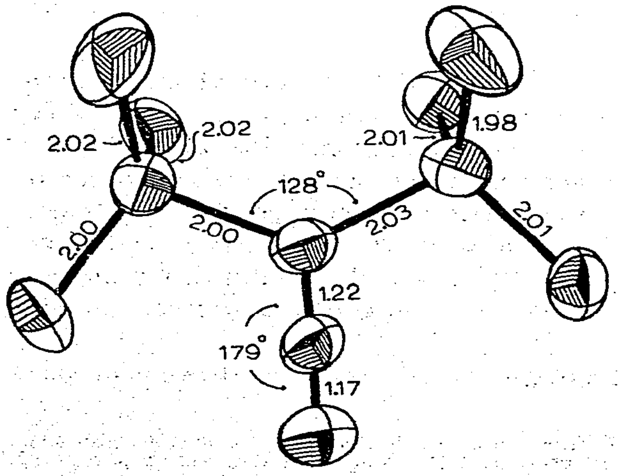


Fig. 1. Structure of the C_{2v} anion of $K[Al_2(CH_3)_6N_3]$ with the atoms displayed as 50% probability ellipsoids for thermal motion.

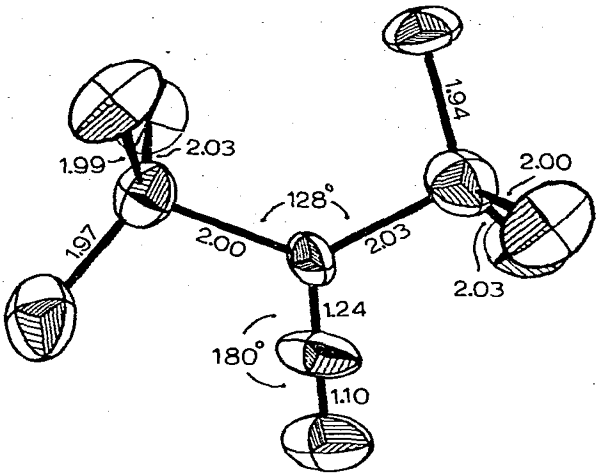


Fig. 2.Structure of the C_s anion of $K[Al_2(CH_3)_6N_3]$ with the atoms displayed as 50% probability ellipsoids for thermal motion.

four methyl carbon atoms filling out the coordination sphere. The other potassium ion finds itself in the less favorable electrostatic position of having no portion of an azide ion within 4.90Å. Its neighborhood consists of six methyl carbon atoms at distances from 3.08(1) to 3.24(1)Å.

Within each azide ion, there are two distinctly different N—N bond lengths. For the two independent anions, the longer distances occuring between the middle nitrogen atom and the nitrogen atom bonded to the aluminum atoms are essentially equivalent: 1.22(1) and 1.24(1)Å. On the other hand, the two terminal N—N bond lengths differ significantly: 1.17(1) and 1.10(1)Å. There appears, however, to be no electronic reason for the difference, and the best values for the bond lengths in the azide ions probably lie close to the average of the two determinations. The distances thus arrived at, 1.23 and 1.14Å, agree very well with those found by Fehlhammer and Dahl [17] for the

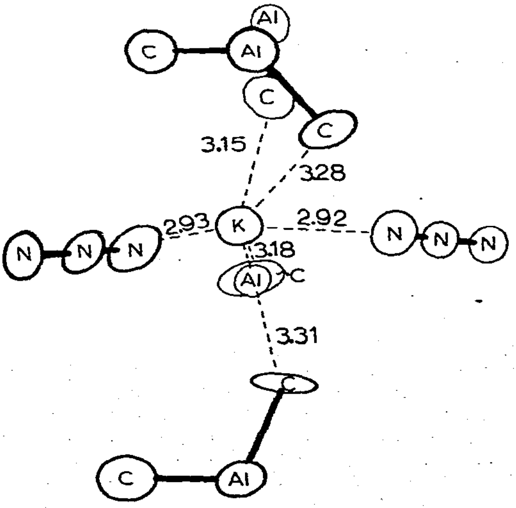


Fig. 3. Environment of the potassium ion associated with the azide groups. All contacts less than 3.50Å are shown.

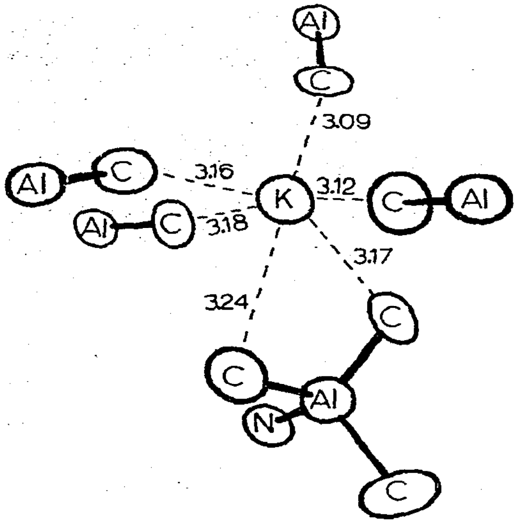


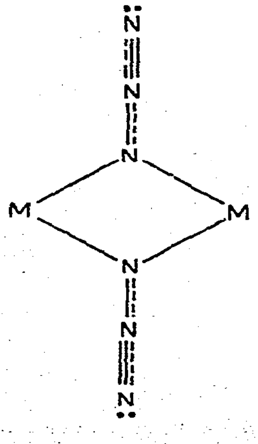
Fig. 4. Environment of the potassium ion packed near the methyl groups. All contacts less than 3.70Å are shown.

bridging azide groups in the $Pd_2(N_3)_6^{2-}$ anion (I), 1.239(14) and 1.142(13)Å and are in contrast to the close equivalence noted in both ionic [18] and head-to-tail bridged species (II) [19].

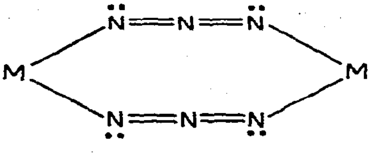
The aluminum—nitrogen—aluminum bond angles of 128° are appreciably larger than the normal value for sp^2 hybridization. This is due primarily to the repulsion between the nonbonded methyl groups, and is an indication that in $M[Al_2R_6N_3]$ where R is more bulky than the methyl group, the single-nitrogen bridged structure may well represent a higher energy configuration than that of the head-to-tail bridged moiety.

Although the aluminum—carbon bond lengths which range from 1.94(1) to 2.03(1)Å are statistically quite different, no physical significance should be placed on the spread of values. The average, 2.00Å, is normal for a compound of this nature where hydrogen atoms have not been located [6].

It has been previously noted [4,5] that $M[Al_2(CH_3)_6X]$ (M = K, Rb, Cs, $N(CH_3)_4$; X = N_3, SCN, SeCN, Cl, I) interacts in a dramatic fashion with



(I)



(II)

aromatic molecules. Liquid complexes which may contain as many as five or as few as two aromatic molecules per anionic unit may be formed by mild heating (~70°) of either the components MX and $(CH_3)_3Al$ or the compound $M[Al_2(CH_3)_6X]$ in the presence of an aromatic "solvent". The complexes are immiscible with excess aromatic material, and in some cases exhibit remarkable thermal stability [20]. It is always possible to recover the pure $M[Al_2(CH_3)_6X]$ and "solvent" by chilling the complex in liquid nitrogen.

A possible explanation of the formation and bonding in complexes of the type $M[Al_2(CH_3)_6X]\cdot n$(aromatic) would involve the formation of cages or layers of $Al_2(CH_3)_6X^-$ units with the counter-ions and aromatic molecules trapped inside. The principal force of stabilization would be the interaction of the positive ions with the negative charge which is localized to a large extent on one very accessible portion of $Al_2(CH_3)_6X^-$ (as the structure of $K[Al_2(CH_3)_6N_3]$ illustrates). The role of the aromatic molecules is then to shield the positive ions in the cage from each other by utilization of the polarizable π-cloud in the classic ion-induced dipole fashion. It would be difficult for the aromatic molecules to extract themselves from such a situation without causing a collapse back to the solid lattice, hence the failure of the complexed solvent to exchange with bulk solvent.

In the formation of the liquid complex, four factors stand out as being of major importance: (1) the lattice energy of $M[Al_2(CH_3)_6X]$, (2) the location or availability of the anionic charge, (3) the size of the positive ion, and (4) the size of the aromatic molecule. Thus, complex formation and stability should be favored by a low lattice energy of the compound $M[Al_2(CH_3)_6X]$, a localized and available negative charge on the anion, a small cation, and a small aromatic molecule.

Acknowledgements

We are grateful to the National Science Foundation for support of this work under Grant GP-24852, and for departmental assistance in the purchase of the X-ray diffractometer.

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