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

IV *. THE PREPARATION AND STRUCTURE OF THE NOVEL SELENIDE $K[CH_3Se\{Al(CH_3)_3\}_3] \cdot 2C_6H_6$

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Summary

The crystal structure of $K[CH_3Se\{Al(CH_3)_3\}_3] \cdot 2C_6H_6$ has been determined from single-crystal X-ray diffraction data collected by counter methods. The compound crystallizes in the triclinic space group $P\bar{1}$ with cell dimensions $a = 17.165(7)$, $b = 10.144(7)$, $c = 10.156(7)$ Å, $\alpha = 119.26(5)$, $\beta = 104.07(5)$, $\gamma = 80.51(5)°$, and $Dc = 1.12$ g cm^{-3} for $Z = 2$. Least-squares refinement gave a final R value of 0.083 for 1967 independent observed reflections. One of the two benzene molecules in the asymmetric unit has been located by difference Fourier techniques. Because of either extreme disorder or high thermal motion, the aromatics make practically no contribution to the X-ray scattering. The selenium atom in the anion exhibits tetrahedral coordination. The Al—Se bond lengths average 2.578(5) Å, and the Se—C distance is 1.93(2) Å.

Introduction

Anionic organoaluminum compounds have come under scrutiny [1—8] for the past several years for a variety of reasons. First, the thermal decomposition of these substances has provided a variety of interesting results [1,4,6—8]. In the most thoroughly studied system [1,8], $K[Al_2(CH_3)_6SCN]$, the sulfur-bridged anion has been shown to decompose at temperatures above 120° C to yield the (isopropylidenamino)dimethylaluminum dimer:

$$K[Al_2(CH_3)_6SCN] \xrightarrow{120^\circ C} \{(CH_3)_3Al\text{—}NC\text{—}CH_3\} \rightarrow [(CH_3)_2AlNC(CH_3)_2]_2$$

For the production of the dimer a mechanism based on the existence of a head-to-tail bridged anion in the melt has been proposed as in eq. 1—3.

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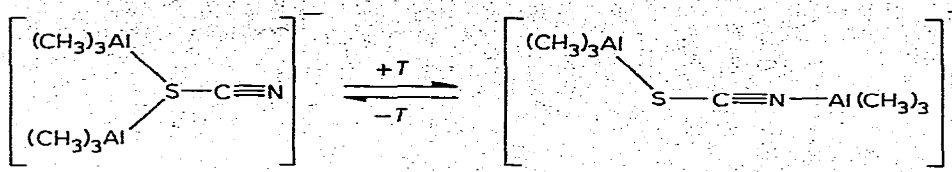
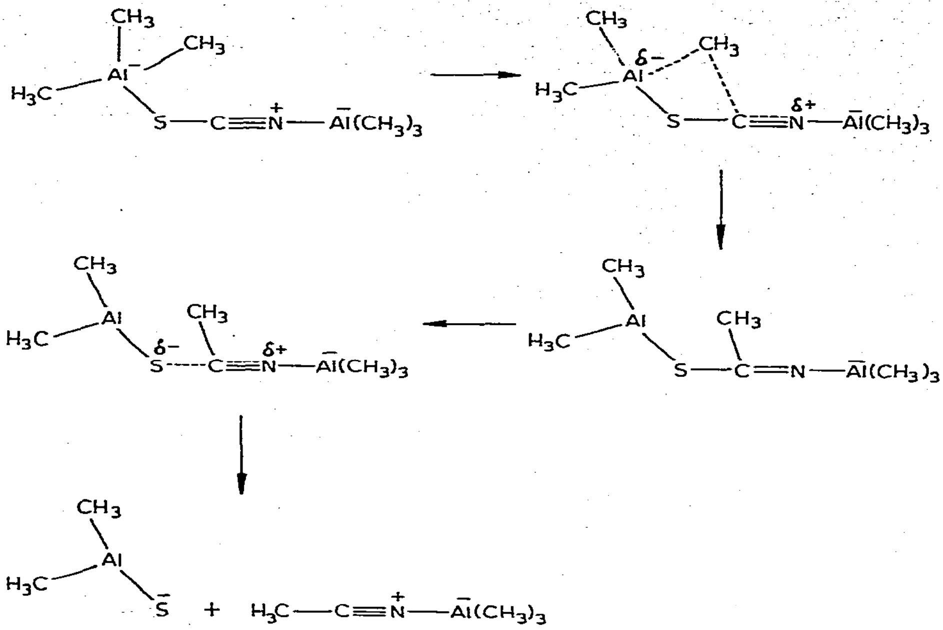
* For part III see ref. 12.



(1)



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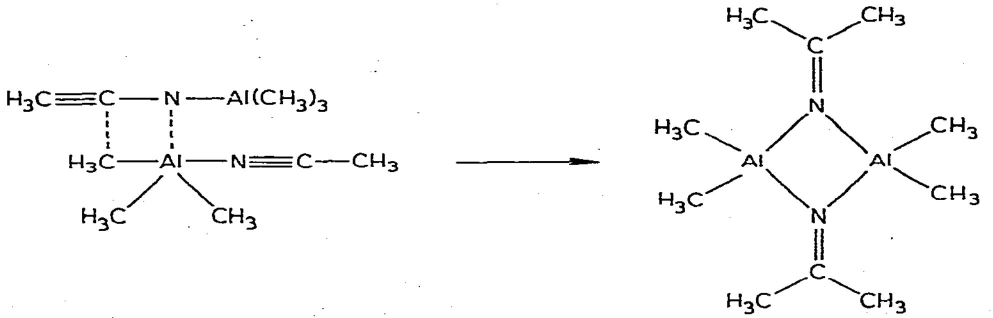

(3)

The sequence shown in eq. 2 involves well-known ability of the trimethylaluminum group to function as an alkylating agent, while eq. 3 is the thermal nitrile rearrangement [9]. Thus, under relatively mild conditions, the sulfur—carbon bond in the SCN^- group may be ruptured; no definitive experimental evidence as to the nature of the sulfur-containing fragment has been produced.

A second area of investigation has been the study of the solution behavior [10—14] of certain of the compounds. Many of the parent 2 : 1 complexes, $M[Al_2R_6X]$, have been found to trap a variety of small aromatic molecules in a nonstoichiometric fashion [14].

We have now prepared and investigated (via single-crystal X-ray diffraction techniques) a compound which illustrates a new type of thermal decomposition for anionic organoaluminum compounds. In addition, the X-ray structure presents an unusual form of solid state entrapment of solvent molecules by an organometallic complex.

Experimental

The preparation of a very small sample of $K[CH_3Se\{Al(CH_3)_3\}_3] \cdot 2C_6H_6$ was accomplished via the sealed tube thermal decomposition of $K[Al_2(CH_3)_6SeCN] \cdot nC_6H_6$ at 80° C. The major product of the thermolysis was a white crystalline substance which elemental analysis showed to be $K[Al_2(CH_3)_6CN]$ *.

$K[Al_2(CH_3)_6SeCN] \cdot nC_6H_6$ was prepared by the sealed tube reaction of a 1 : 2 mole ratio of KSeCN and $Al(CH_3)_3$ in benzene. The liquid-layering effect [14] was noted even at room temperature, but the more dense solution was too unstable to permit an NMR analysis of benzene/anion ratio. We were likewise unable to synthesize and analyze a pure sample of the potassium salt of $[Al_2(CH_3)_6SeCN]^-$ **.

Single crystals of the colorless, air sensitive compound were sealed in thin-walled glass capillaries. Preliminary unit cell parameters were determined from Weissenberg and precession (Cu-K_α) photographs. Final lattice parameters as determined from a least-squares refinement of the angular settings of 12 reflections accurately centered on a diffractometer are given in Table 1.

Data were taken on an ENRAF—NONIUS CAD-4 diffractometer with Ni-filtered copper radiation. The diffracted intensities were collected by the $\omega - 2\theta$ scan technique with a take-off angle of 1.5°. The scan rate was variable and was determined by a fast 20° min^{-1} prescan. If more than 20 net counts were found, a slow scan was carried out; if not, the reflection was considered to be unobserved. Calculated speeds for the slow scan based on the net intensity gathered in the prescan ranged from 7° min^{-1} to 0.7° 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:

$$\text{scan range} = A + B \operatorname{tg} \theta$$

where A = 0.7° and B = 0.45°. Aperture settings were determined in a like manner with A = 4 mm and B = 4 mm. The crystal-to-source and crystal-to-detector distances were 21.6 and 20.8 cm, respectively. The lower level and upper level discriminators of the pulse height analyzer were set to obtain a 95% window centered on the Cu-K_α) peak. Two standard peaks were measured periodically during data collection; no significant variation in the intensities was noticed.

The standard deviations of the intensities, σ_I, were estimated from the formula:

$$\sigma = \{[C_N + (T_C/2T_B)^2(B_1 + B_2)] + k^2[C_N + (T_C/2T_B)^2(B_1 + B_2)]^2\}^{1/2}$$

where C_N is the counts collected during scan time T_C, B_1 and B_2 are background intensities, each collected during the background time T_B, and k = 0.03.

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* A satisfactory elemental analysis was obtained for $K[Al_2(CH_3)_6CN]$, but not for $K[CH_3Se\{Al(CH_3)_3\}_3] \cdot 2C_6H_6$ or $K[Al_2(CH_3)_6SeCN]$.

** The existence of $[N(CH_3)_4][Al_2(CH_3)_6SeCN]$ has been demonstrated (see [5]).

TABLE 1

CRYSTAL DATA

Mol. formula:	$K[CH_3Se\{Al(CH_3)_3\}_3] \cdot 2C_6H_6$
Mol. wt.:	505.58
Linear abs. coeff.:	39.82 cm^{-1}
Calc. density:	1.12 g cm^{-3}
Max crystal dimensions:	0.70 × 0.25 × 0.05 mm
Space group:	triclinic, $P\bar{1}$
Molecules/unit cell:	2
Cell constants [a]:	$a = 17.165(7)$, $b = 10.144(7)$, $c = 10.156(7)$ Å
	$\alpha = 119.26(5)$, $\beta = 104.07(5)$, $\gamma = 80.51(5)°$
Cell volume:	1494.3 Å

[a] Cu-K_α radiation, $\lambda = 1.54051$ Å. Ambient temperature of 23 ± 1°C.

One independent hemisphere of data was measured out to $2\theta = 130°$; a total of 1977 reflections had intensities greater than three times their estimated standard deviations. These reflections were deemed to be "observed," and were used in the subsequent structure determination and refinement. The intensities were corrected for Lorentz and polarization effects, but not for absorption ($\mu = 39.82$ cm^{-1}).

Fourier calculations were made with the ALFF [15] program. The full-matrix, least-squares refinement was carried out using the Busing and Levy program ORFLS [16]. The function $w(|F_o| - |F_c|)^2$ was minimized. No corrections were made for extinction or anomalous dispersion. Neutral atom scattering factors were taken from the compilations of Cromer and Waber [17] for Se, K, Al, and C. The scattering by selenium and potassium was corrected for the real and imaginary components of anomalous dispersion using Cromer's table [18]. Final bond distances, angles, and errors were computed with the aid of the Busing, Martin, and Levy ORFFE program [19]. Crystal structure illustrations were obtained with the program ORTEP [20].

Solution and refinement of structure

Statistical tests based on normalized structure factors indicated the space group to be the acentric $P1$. The Patterson map was interpreted to give the coordinates of the two selenium atoms in the two independent molecules in the asymmetric unit, and subsequent Fourier and difference Fourier maps afforded the positions of the remaining nonhydrogen atoms in the $K[CH_3Se\{Al(CH_3)_3\}_3]$ unit. Isotropic refinement converged initially at 0.29, but unusually high parameter correlations led to the calculation of a center of inversion relating the two molecules. Isotropic refinement in $P\bar{1}$ led to $R = 0.13$. The space group was therefore subsequently taken to be $P\bar{1}$.

Up to this point, only the nonhydrogen atoms of the $K[CH_3Se\{Al(CH_3)_3\}_3]$ unit were included; since there was not enough of the compound to permit an elemental analysis, it was not known that the substance was a disolvate. Inspection of the structure showed two features which indicated something was missing: (*1*) the calculated density was only 0.78 g cm^{-3}, and (*2*) there appeared to be a large, empty region in the unit cell. It must be emphasized that the R value

TABLE 2

FINAL FRACTIONAL COORDINATES AND THERMAL PARAMETERS [a] FOR $K[CH_3Se\{Al(CH_3)_3\}_3]\cdot 2C_6H_6$

Atom	x/a	y/b	z/c	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Se	0,6430(1)	0,8200(2)	0,7148(2)	0.0056(1)	0,0134(3)	0,0144(2)	—0,0005(1)	0.0023(1)	0,0006(2)
K(1)	0,5000	0.5000	0,0000	0,0062(2)	0,0144(6)	0,0141(6)	—0,0013(2)	0.0019(2)	0,0064(4)
K(2)	0,0000	0.5000	0.5000	0,0076(2)	0,0222(7)	0,0213(7)	—0,0002(3)	0.0012(3)	0.0077(5)
Al(1)	0,6772(4)	1,0401(6)	0,6807(6)	0,0066(3)	0,0143(9)	0,0191(9)	—0,0022(4)	0.0038(4)	0,0078(8)
Al(2)	0,6773(4)	0,8577(6)	0,9921(6)	0,0064(3)	0,0156(9)	0,0133(8)	—0,0021(4)	0,0022(4)	0,0066(7)
Al(3)	0,6843(3)	0,5554(6)	0,5140(6)	0,0058(3)	0,0137(8)	0,0144(8)	0,0002(4)	0.0018(4)	0,0058(7)
C(1)	0,6592(18)	0,9511(28)	0.4552(25)	0,0186(22)	0,0271(52)	0,0223(43)	0,0031(26)	0.0067(25)	0,0153(40)
C(2)	0,7920(14)	1,0578(31)	0,7962(35)	0.0065(13)	0,0393(43)	0,0534(47)	—0,0041(22)	0.0049(25)	0,0209(49)
C(3)	0,6004(13)	1,2053(21)	0,7882(23)	0.0104(14)	0,0158(32)	0,0261(39)	0,0036(16)	0,0080(19)	0,0108(30)
C(4)	0,6035(13)	0.7213(22)	0,9862(21)	0.0095(17)	0,0236(37)	0,0214(34)	—0,0056(17)	0,0021(16)	0,0149(30)
C(5)	0,7889(13)	0.7822(31)	0,9935(29)	0,0068(13)	0,0463(45)	0,0382(36)	—0,0050(22)	—0,0044(20)	0,0311(43)
C(6)	0,6524(14)	1,0825(22)	1,1070(23)	0.0114(15)	0,0154(34)	0,0228(37)	—0,0023(17)	0.0062(19)	—0,0010(28)
C(7)	0,6635(14)	0,4173(23)	0,5877(25)	0.0113(15)	0,0185(36)	0,0268(41)	—0,0014(17)	0.0041(19)	0,0111(32)
C(8)	0,7974(14)	0,5869(22)	0,5318(23)	0.0052(15)	0,0348(35)	0,0224(34)	0,0017(17)	0.0039(18)	0,0091(29)
C(9)	0,6094(14)	0,5339(22)	0,3199(24)	0.0089(15)	0,0156(37)	0,0179(35)	0,0001(17)	0.0002(19)	0,0072(30)
C(10)	0,5270(12)	0,8270(24)	0.6624(26)	0,0060(12)	0,0234(41)	0,0325(46)	—0,0010(16)	0.0018(18)	0,0127(36)
RC(1)	0,127 [b]	0,752	0,808						
RC(2)	0,126	0,638	0,858						
RC(3)	0,061	0,584	0,879						
RC(4)	—0,002	0,684	0,881						
RC(5)	—0,008	0,808	0,836						
RC(6)	0,055	0,845	0,810						

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[a] Anisotropic thermal parameters defined by $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$. [b] RC symbolizes the ring carbon atoms of one of the two independent benzene molecules. These coordinates were taken from a difference Fourier map and were not refined, nor included in the refinement.

was quite reasonable for a complete structure at the isotropic stage of refinement.

Analysis of a difference Fourier map at this point revealed many peaks of $\sim 1.5\ e^- \text{Å}^{-3}$ in the empty regions of the unit cell. By a process of trial and error it was finally possible to resolve the carbon atom coordinates of one of the two benzene rings in the asymmetric unit. The peaks associated with these positions ranged from $0.8 \rightarrow 1.5\ e^- \text{Å}^{-3}$. Although there were many other possibilities, the derived parameters were chemically reasonable, and there is little doubt that the ring is correctly located. The individual carbon atoms of the second independent benzene molecule could not be positioned, but a smear of electron density appeared in the region in which the ring must reside.

The positional parameters of the benzene carbon atoms were refined with a damping factor of 0.5 for three cycles. The bond lengths and angles remained reasonable, but it was apparent that many cycles would be required in order to obtain the potential minimum. Variation of the isotropic B's (originally fixed at 15.0 Å^2) resulted in values of $25 \rightarrow 30\ \text{Å}^2$ after two cycles. The R factor was approximately 2% higher with the scattering of the carbon atoms of the ring accounted for. In view of these difficulties, it was believed that the best values of the bonding parameters of the anion would be obtained without the inclusion of contributions from the aromatic groups. The coordinates are, however, included in Table 2, and the locations of the rings are depicted in Fig. 4.

Inspection of the structure factor list (calculated without the benzene molecule contributions) revealed generally excellent agreement between observed and calculated values. The exceptions were a group of ten low-angle ($\sin\theta/\lambda < 0.15$) reflections for which the observed and calculated structure factors disagreed by almost an order of magnitude. Removal of this group *, the (010), $(\bar{1}00)$, $(\bar{1}10)$, $(20\bar{2})$, (220), $(\bar{3}1\bar{2})$, $(33\bar{1})$, (410), $(\bar{4}11)$, and $(50\bar{1})$, followed by isotropic least-squares refinement led to $R = 0.107$. Conversion to anisotropic temperature factors and additional cycles of refinement led to final values :

$$R_1 = \sum(|F_\text{o}| - |F_\text{c}|)/\sum|F_\text{o}| = 0.083$$

$$R_2 = [\sum w(|F_\text{o}| - |F_\text{c}|)^2/\sum wF_\text{o}^{\,2}]^{1/2} = 0.107$$

Neither the benzene molecules of solvation, nor the hydrogen atoms associated with the anion were included. The weighting scheme was based on unit weights; unobserved reflections and the group of ten mentioned previously were not included. For the remainder of the X-ray data no systematic variation of $w(|F_\text{o}| - |F_\text{c}|)^2$ vs. $|F_\text{o}|$ or $\sin\theta/\lambda$ was noted. The largest parameter shifts in the final cycle of refinement were less than 0.20 of their estimated standard deviations. The final value for the standard deviation of an observation of unit weight was 2.95. The final values of the positional and thermal parameters are given in Table 2 **.

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* Although it is not in general a good practice to remove a group of reflections from a data set, in this case it is believed that by so doing a refinement was obtained which produced the most accurate possible parameters for the structure. Further justification for this action is given in the discussion.

** The table of structure factors can be obtained from the authors.

Discussion

A. Crystallographic problems associated with the crystal structure

It is at first surprising to note that the *R* factor is 0.083 for 1967 reflections when neither molecule of solvation has been included in the refinement. In fact the carbon atoms of the benzene molecules comprise 32% of the nonhydrogen electron density in the unit cell, and if one includes the hydrogen atoms, 43% of the total electron density is unaccounted. The small contribution to the X-ray scattering made by hydrogen atoms is well-known. Even for a structure which exhibits low thermal motion, hydrogen atoms have an appreciable effect on only those reflections with low $\sin\theta/\lambda$ value.

In order to explain the lack of importance of the benzene molecules to the total X-ray scattering, two possibilities may be envisioned. First, extremely high thermal motion of the aromatic rings would have the effect of reducing the scattering ability of the carbon atoms. More specifically, such atoms would be expected to contribute strongly to only a few low angle reflections. The same net effect could also be achieved by a highly disordered arrangment of benzene rings. Either of these two possibilities fits the experimental data: excellent agreement between observed and calculated structure factors for 1967 reflections, but very poor agreement for 10 low angle reflections.

For the situation exemplified by $K[CH_3Se\{Al(CH_3)_3\}_3] \cdot 2C_6H_6$, the limitations of the X-ray method must be emphasized: the benzene molecules are essentially "invisible". In the absence of an elemental analysis, however, the number of benzene molecules may be readily deduced from the size of the "vacancies" in the unit cell. It should also be noted that there must be exactly $2C_6H_6$ units. In normal clathrate structures the holes or channels may be empty, filled, or partially filled by guest molecules. For $K[CH_3Se\{Al(CH_3)_3\}_3] \cdot 2C_6H_6$, the benzene molecules are not simply guests: they are an integral part of the unit cell. Amplification of these arguments is given in part *C*.

B. Description of the anion

The preparation of $K[CH_3Se\{Al(CH_3)_3\}_3] \cdot 2C_6H_6$ could be viewed in the simplest terms as the conversion of K[SeCN] to $K[SeCH_3]$:

$$Se{-}C{\equiv}N \rightarrow Se{-}CH_3$$

The three molecules of $Al(CH_3)_3$ would then add to the three lone pairs of electrons on the Se atom.

Since crystallographic difficulties are known to be associated with this crystal structure, it is necessary to fully justify that the carbon moiety bonded to the selenium atom is a methyl, not a cyano group. To accomplish this purpose, two arguments may be presented. A close examination of Fig. 1 shows that the methyl carbon atom exhibits normal thermal behavior. If this group were actually two atoms (as in C≡N), one would expect the thermal ellipsoid to be greatly elongated in the direction of the C—Se bond.

The Se—C bond length of 1.93(2) Å is also characteristic of an alkyl group linkage. For this case Sutton's tabulations [21] show a range of values from 1.95 to 1.98 Å, whereas the Se—C-(cyano) lengths average 1.83 Å for two recent determinations of the triselenocyanate anion [22,23].

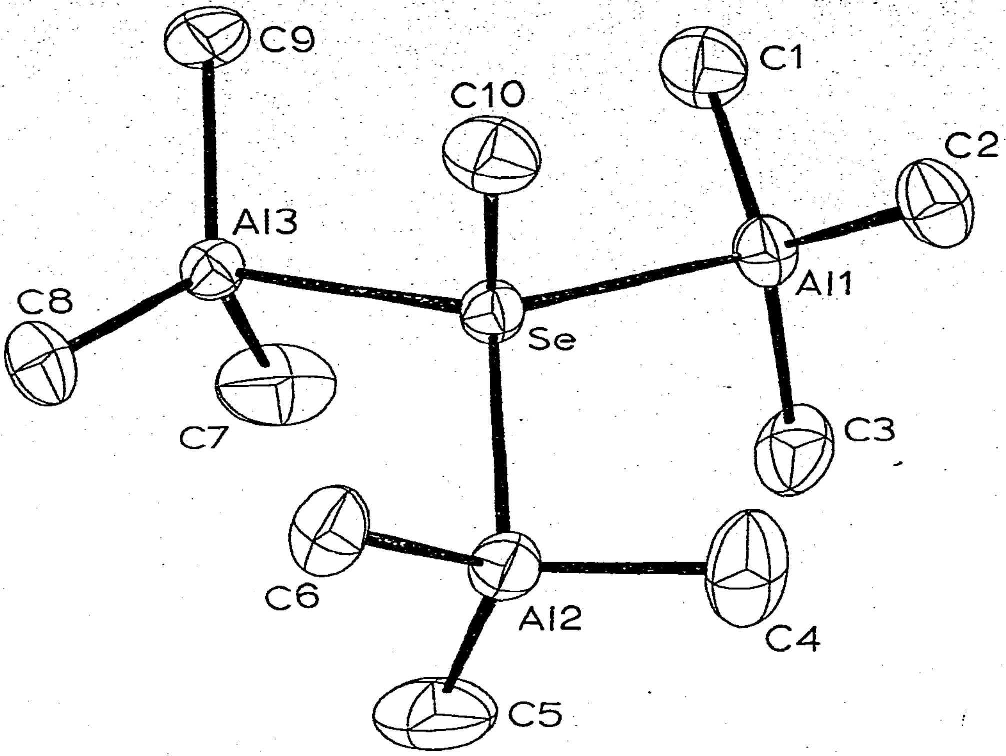

Fig. 1. Atom labeling scheme for the [$CH_3Se\{Al(CH_3)_3\}_3$]$^-$ anion with the atoms displayed as their 40% probability ellipsoids for thermal motion.

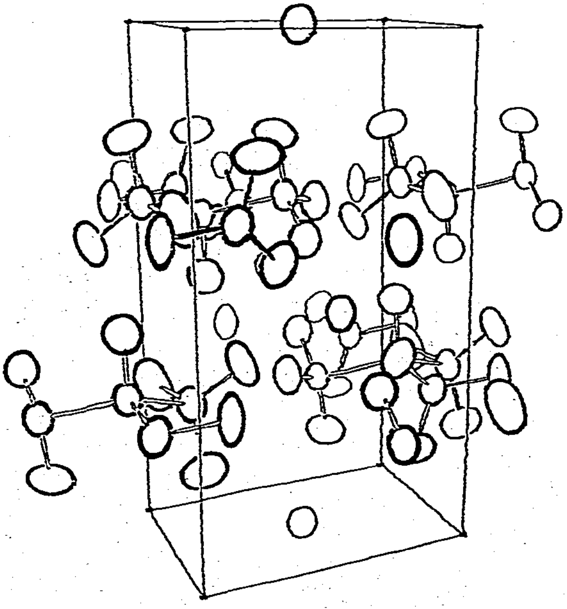

Fig. 2. View of the unit cell of K[$CH_3Se\{Al(CH_3)_3\}_3$] · $2C_6H_6$ normal to the *ab* plane.

TABLE 3

BOND LENGTHS (Å) AND ANGLES (°) FOR $K[CH_3Se\{Al(CH_3)_3\}_3] \cdot 2C_6H_6$

Bond			
Se—Al(1)	2.594(6)	Se—Al(3)	2.566(5)
Se—Al(2)	2.575(5)	Se—C(10)	1.93(2)
Al(1)—C(1)	1.97(2)	Al(2)—C(4)	1.99(2)
Al(1)—C(2)	2.01(3)	Al(2)—C(5)	1.94(2)
Al(1)—C(3)	1.98(2)	Al(2)—C(6)	2.02(2)
Al(3)—C(7)	1.99(2)	Al(3)—C(8)	1.97(2)
Al(3)—C(9)	2.00(2)		
K(1)—C(4) [a]	3.15(2)	K(2)—C(8) [f]	3.49(2)
K(1)—C(4) [b]	3.15(2)	K(2)—C(8) [b]	3.49(2)
K(1)—C(9)	3.21(2)	K(2)—Al(3) [f]	5.375(6)
K(1)—C(9) [c]	3.21(2)	K(2)—Al(3) [b]	5.375(6)
K(1)—C(3) [d]	3.22(2)	K(2)—C(2) [f]	5.98(3)
K(1)—C(3) [e]	3.22(2)	K(2)—C(2) [b]	5.98(3)
Angle			
Al(1)—Se—C(10)	102.0(7)	Al(1)—Se—Al(2)	115.6(2)
Al(2)—Se—C(10)	105.4(7)	Al(1)—Se—Al(3)	114.4(2)
Al(3)—Se—C(10)	103.5(7)	Al(2)—Se—Al(3)	113.9(2)
Se—Al(1)—C(1)	101.8(8)	C(1)—Al(1)—C(2)	117.8(12)
Se—Al(1)—C(2)	96.9(8)	C(1)—Al(1)—C(3)	117.3(10)
Se—Al(1)—C(3)	102.3(6)	C(2)—Al(1)—C(3)	115.4(11)
Se—Al(2)—C(4)	102.8(6)	C(4)—Al(2)—C(5)	114.7(10)
Se—Al(2)—C(5)	97.5(7)	C(4)—Al(2)—C(6)	117.3(9)
Se—Al(2)—C(6)	99.8(7)	C(5)—Al(2)—C(6)	119.1(10)
Se—Al(3)—C(7)	104.2(7)	C(7)—Al(3)—C(8)	117.3(10)
Se—Al(3)—C(8)	99.3(7)	C(7)—Al(3)—C(9)	113.8(10)
Se—Al(3)—C(9)	102.7(7)	C(8)—Al(3)—C(9)	116.1(10)

[a] Related to the atom given in Table 2 by $(x, y, z-1)$. [b] $(1-x, 1-y, 1-z)$. [c] $(1-x, 1-y, -z)$. [d] $(x, y-1, z-1)$. [e] $(1-x, 2-y, 1-z)$. [f] $(x-1, y, z)$.

The coordination about the selenium atom is essentially tetrahedral (Fig. 1): the Al—Se—Al angles average 114.6°, and the Al—Se—C angles, 103.8°. These small distortions may be explained either in terms of greater steric interference of the more bulky $Al(CH_3)_3$ groups, or in terms of secondary hybridization considerations. The more electronegative carbon atom demands more *p*-character in the Se—C bond, and as a result the selenium atom places more *s*-character in the bonds to the aluminum atoms: hence the larger Al—Se—Al bond angles.

The Al—Se bond lengths given in Table 3 are important in that they are the first such distances to be obtained crystallographically. The average value, 2.578(5) Å, is significantly longer than expected on the basis of covalent radii. Brauer and Stucky [24] found an Al—S distance of 2.348(2) Å in $[(CH_3)_2AlSCH_3]_n$, and Pauling's tables [25] give a difference of 0.13 Å between the radii of Se and S. Thus, one might expect the Al—Se length to be ~2.48 Å. The observed value is in fact similar to the 2.58 Å standard given for the Al—I bond in $(CH_3)_3N \cdot Al(CH_3)_2I$ [26]. The explanation of the lengthening of the Al—Se bond is probably found in a combination of steric and electronic effects which arise from the fact that the selenium atom is bonded to four groups.

None of the individual Al—C bond lengths differ significantly from the average value of 1.99 Å, which is similar to those found in related compounds [24,27]. The C—Al—C bond angles are also normal.

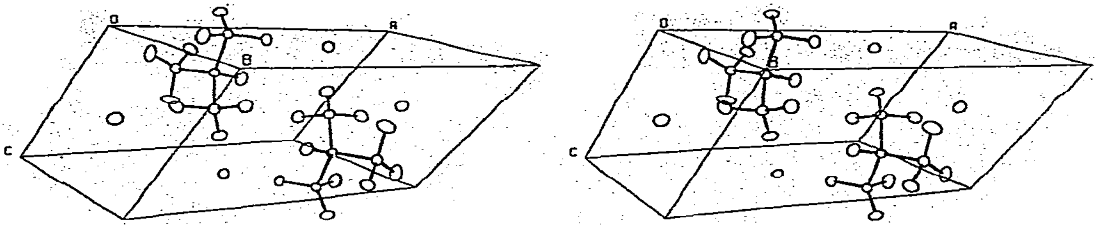

Fig. 3. Stereo view of the unit cell packing in $K[CH_3Se\{Al(CH_3)_3\}_3] \cdot 2C_6H_6$.

C. Entrapment of the benzene molecules

In the asymmetric unit there are two crystallographically independent potassium ions. Although both reside on centers of symmetry, the fact that the environments are quite different can be seen from the data given in Table 3, and in Fig. 2, 3, and 4. One potassium ion, K(1), is packed within the methyl groups of the anions: The K—C distances of 3.15 → 3.22 Å are normal [11,12]. The other potassium ion, K(2), is associated with two carbon atoms at 3.49 Å, and the remainder of its contacts must be with the benzene molecules. For one of the two independent C_6H_6 molecules the important bond lengths and angles are given in Table 4. K(2) is symmetrically disposed over the benzene molecule at K—C distances of 3.33 → 3.47 Å. In fact, since K(2) is situated on a center of inversion, the symmetry related ring completes a sandwich-like arrangement.

Careful inspection of Fig. 4 affords the observation that the centers of the symmetry related benzene rings lie close to one of the *bc* face diagonals. The remaining aromatic molecules must reside with their centers close to the other *bc*

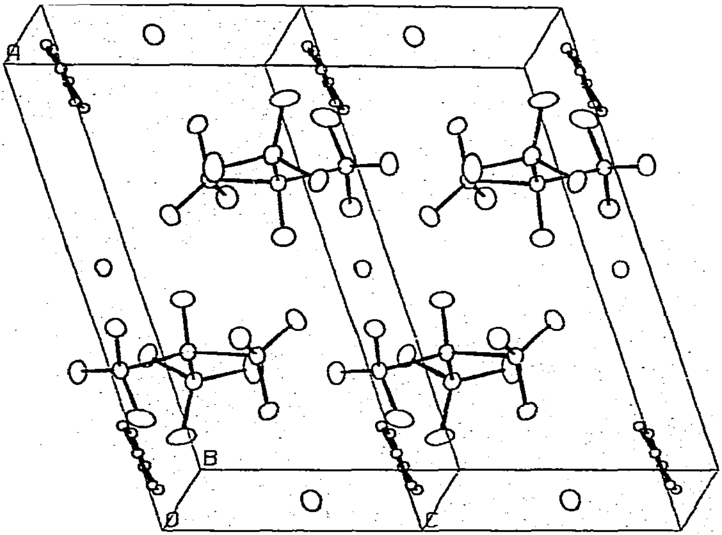

Fig. 4. Two unit cells with one of the two independent benzene molecules shown.

TABLE 4

BOND LENGTHS (Å) AND ANGLES (°) ASSOCIATED WITH ONE OF THE BENZENE MOLECULES OF $K[CH_3Se\{Al(CH_3)_3\}_3] \cdot 2C_6H_6$ [a]

Bond			
RC(1)—RC(2)	1.48	RC(4)—RC(5)	1.52
RC(2)—RC(3)	1.42	RC(5)—RC(6)	1.32
RC(3)—RC(4)	1.35	RC(6)—RC(1)	1.42
K(2)—RC(1)	3.42	K(2)—RC(4)	3.43
K(2)—RC(2)	3.47	K(2)—RC(5)	3.33
K(2)—RC(3)	3.45	K(2)—RC(6)	3.38
Angle			
RC(1)—RC(2)—RC(3)	131	RC(4)—RC(5)—RC(6)	122
RC(2)—RC(3)—RC(4)	106	RC(5)—RC(6)—RC(1)	117
RC(3)—RC(4)—RC(5)	127	RC(6)—RC(1)—RC(2)	115

[a] The ring carbon atoms were not included in the least-squares refinement.

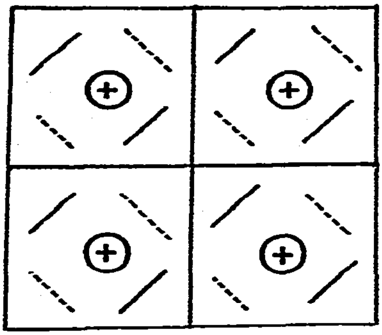

Fig. 5. Approximate orientation of the benzene molecules with respect to the potassium ions (viewed down the *a* axis). Solid line indicates an aromatic found on the difference Fourier; dashed line indicates predicted location of an aromatic.

face diagonal. An idealized view of the unit cell along the *a* direction is given in Fig. 5. K(2) is therefore in all probability found to be at the center of a distorted octahedron composed of C(8), C(8′), and the centers of four aromatic rings.

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