Crystal Structure of Di-μ-phenyl-bis(dimethylaluminium)

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Crystals of the title compound are triclinic, space group $P\bar{1}$, with three dimeric molecules in the reduced cell of dimensions $a = 12{\cdot}313$, $b = 12{\cdot}755$, $c = 9{\cdot}352$ Å, $\alpha = 102{\cdot}8$, $\beta = 91{\cdot}6$, $\gamma = 61{\cdot}3°$. The structure was determined by Patterson and Fourier methods from 3126 visually estimated photographic data, and refined by least-squares to R 10·6%. One third of the molecules lie on centres of symmetry with the $\overline{\text{Al–C–Al–C}}$ ring necessarily planar, and the remainder are in general positions, with the central ring ' folded ' by 14° about the Al ··· Al line. Al ··· Al is 2·687, Al–C(bridge) 2·144, and Al–C(terminal) 1·977 Å; the Al–C–Al bridge angle is 77·6°.

We have shown[1] that triphenylaluminium is dimeric with Al–C–Al bridges which may be formed by two-electron three-centre bonds similar to those in trimethylaluminium dimer. Examination[2] of the ^{1}H n.m.r. spectra of mixtures of composition $Me_{6-x}Ph_xAl_2$ showed that the phenyl groups preferentially occupy the bridging positions. Similar experiments[3] on mixtures of trimethylaluminium and tri(cyclopropyl)aluminium show that the cyclopropyl group displaces methyl from the bridge position. The present analysis was undertaken to confirm the preference of phenyl in the bridging position; a preliminary account has appeared.[4]

EXPERIMENTAL

Crystal Data.—$C_{16}H_{22}Al_2$, $M = 268{\cdot}3$, Triclinic, $a = 12{\cdot}313 \pm 0{\cdot}02$, $b = 12{\cdot}755 \pm 0{\cdot}02$, $c = 9{\cdot}352 \pm 0{\cdot}02$ Å, $\alpha = 102{\cdot}8 \pm 0{\cdot}3$, $\beta = 91{\cdot}6 \pm 0{\cdot}03$, $\gamma = 61{\cdot}3 \pm 0{\cdot}2°$, $U = 1250{\cdot}9$ Å^3, $Z = 3$, $D_c = 1{\cdot}07$, $F(000) = 432$. Space group $P\bar{1}$. Cu-K_α radiation, $\lambda = 1{\cdot}5418$ Å; μ(Cu-K_α) = 14 cm^{-1}.

Structure Determination.—Cell dimensions were determined from zero-level precession and Weissenberg photographs (Mo-K_α, $\lambda = 0{\cdot}7107$ Å), and the dimensions given define the reduced cell according to the convention adopted in Crystal Data.[5] The density of the crystals was not determined because of their very high reactivity with air, but with $Z = 3$ the volume per molecule is 417 Å^3, while interpolation between the values for Me_6Al_2 (270 Å^3) and Ph_6Al_2 (718 Å^3) indicates a molecular volume of 419 Å^3.

The compound was prepared by reaction of aluminium with a mixture of dimethyl- and diphenyl-mercury in toluene solution. Crystals were protected by a coating of grease, transferred under nitrogen and sealed in soft glass capillaries. The crystal used for the recording of the intensity data was rotated about the [10$\bar{1}$] axis, and equi-inclination Weissenberg photographs of layers $h - l = 0$—13 were taken.

The structure was solved from the Patterson synthesis and successive Fourier syntheses, yielding a structure conforming to space group $P\bar{1}$. Least-squares refinement of co-ordinates, isotropic temperature factors, and 14 layer scale factors gave R 15·9%, and allowance for anisotropic vibrations reduced R to 11·9%. In the block-diagonal approximations used here satisfactory convergence was obtained although the full matrix would be singular. A difference-Fourier synthesis computed at this stage showed peaks close to expected positions for all 33 hydrogen atoms. The 15 phenyl hydrogens were included in calculated positions and the 18 methyl hydrogens were included in their observed positions; after further least-squares refinement in which only the hydrogen positions and isotropic temperature factors were allowed to vary, R was reduced to 10·6%. The least-squares program of Smith and Cruickshank was used; scattering factors for aluminium and carbon were taken from ref. 6 and those for bonded hydrogen from ref. 7. The weighting scheme in the final cycles was $w = 1/(5 + F_o + 0{\cdot}04F_o^2 + 0{\cdot}002F_o^3)$. The final co-ordinate and vibration parameters, with their estimated standard deviations, are given in Table 1. Observed and calculated structure factors are listed in Supplementary Publication No. SUP 20530 (19 pp., 1 microfiche †).

DISCUSSION

The structure analysis shows that the dimeric molecules have the phenyl groups in the bridging positions.

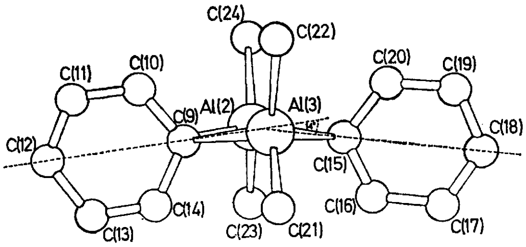

Figure 1 Molecule (2) showing atom numbering

One third of the molecules lie on centres of symmetry and the remainder occupy general positions. The asymmetric unit is therefore one and one half molecules. Figure 1 shows the molecule which occupies a general position [molecule (2)], and the atom numbering is given. The centrosymmetric molecule [molecule (1)] is similar except that its central ring is necessarily planar. Figure 2 gives an indication of the molecular packing

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† For details see Notice to Authors No. 7 in *J. Chem. Soc.* (*A*), 1970, Issue No. 20.

[1] J. F. Malone and W. S. McDonald, preceding paper.

[2] E. A. Jeffery, T. Mole, and J. K. Saunders, *Chem. Comm.*, 1967, 696.

[3] J. W. Moore, D. A. Sanders, P. A. Scherr, M. D. Glick, and J. P. Oliver, *J. Amer. Chem. Soc.*, 1971, **93**, 1035.

[4] J. F. Malone and W. S. McDonald, *Chem. Comm.*, 1970, 280.

[5] ' Crystal Data Determinative Tables,' 2nd edn., eds. J. D. H. Donnay, G. Donnay, E. G. Cox, O. Kennard, and M. V. King, Amer. Cryst. Assoc., Washington, 1963.

[6] ' International Tables for *X*-Ray Crystallography,' vol. III, Kynoch Press, Birmingham, 1962.

[7] R. F. Stewart, E. R. Davidson, and W. T. Simpson, *J. Chem. Phys.*, 1965, **42**, 3175.

and the bond lengths and angles with their estimated standard deviations are given in Table 2.

Corresponding bond lengths in the two molecules do not differ significantly, and are similar to those in triphenylaluminium. The Al–C(bridging) distances range from 2·128(6) to 2·163(6) Å. Although these extreme values differ by *ca.* 3σ from the mean of 2·144 Å it is unlikely that the differences are real. The mean terminal Al–C bond length is 1·977 Å, with the Al–C–Al bridge angle 77·6°. As in triphenylaluminium the bridging phenyl groups are perpendicular to the central ring (or normal to the Al · · · Al vector). The equations of mean planes are given in Table 3, together with some dihedral angles.

TABLE 1

Final atomic parameters with estimated standard deviations in parentheses

(*a*) Fractional co-ordinates of the heavy atoms

Atom	x	y	z
Al(1)	0·1047(2)	−0·0230(2)	−0·0728(2)
Al(2)	0·2386(2)	0·4038(2)	0·3987(2)
Al(3)	0·4502(2)	0·2006(2)	0·4157(2)
C(1)	0·0767(6)	−0·1371(5)	0·0400(7)
C(2)	0·0819(7)	−0·2446(5)	−0·0542(8)
C(3)	0·1427(8)	−0·3585(6)	−0·0197(10)
C(4)	0·1993(8)	−0·3703(7)	0·1099(12)
C(5)	0·1967(8)	−0·2706(9)	0·2041(11)
C(6)	0·1340(7)	−0·1531(7)	0·1737(8)
C(7)	0·1392(10)	−0·1012(7)	−0·2846(9)
C(8)	0·2179(8)	0·0299(7)	0·0332(11)
C(9)	0·4085(6)	0·3164(5)	0·2621(7)
C(10)	0·4768(7)	0·3818(6)	0·2753(8)
C(11)	0·5324(8)	0·3885(7)	0·1535(9)
C(12)	0·5238(8)	0·3300(8)	0·0152(8)
C(13)	0·4592(7)	0·2650(6)	−0·0047(7)
C(14)	0·4028(6)	0·2577(5)	0·1180(7)
C(15)	0·2696(6)	0·2689(5)	0·5214(7)
C(16)	0·2106(7)	0·1995(7)	0·4682(9)
C(17)	0·1404(8)	0·1772(8)	0·5633(11)
C(18)	0·1295(8)	0·2256(7)	0·7141(10)
C(19)	0·1866(7)	0·2932(7)	0·7702(9)
C(20)	0·2545(6)	0·3160(6)	0·6757(7)
C(21)	0·5101(8)	0·0319(6)	0·2978(9)
C(22)	0·5517(8)	0·2421(9)	0·5633(9)
C(23)	0·1039(7)	0·4153(7)	0·2722(10)
C(24)	0·2276(9)	0·5528(6)	0·5357(9)

(*b*) Anisotropic temperature factors (Å² × 10³) of the heavy atoms, in the form $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{23}klb^*c^* + 2U_{31}lhc^*a^* + 2U_{12}hka^*b^*)]$

Atom	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
Al(1)	44(1)	33(1)	41(1)	6(1)	8(2)	−33(1)
Al(2)	41(1)	34(1)	32(1)	12(1)	1(2)	−29(1)
Al(3)	40(1)	45(1)	25(1)	19(1)	−2(2)	−29(1)
C(1)	43(3)	39(3)	41(4)	18(5)	−12(6)	−27(5)
C(2)	65(4)	33(3)	50(4)	10(5)	22(7)	−32(6)
C(3)	80(6)	42(3)	78(6)	28(7)	30(9)	−32(7)
C(4)	53(5)	68(5)	123(8)	79(10)	−6(10)	−47(8)
C(5)	52(5)	108(6)	97(7)	120(11)	−52(9)	−79(9)
C(6)	57(4)	83(5)	44(4)	52(7)	−40(7)	−70(7)
C(7)	138(8)	57(4)	47(5)	−10(7)	71(10)	−66(10)
C(8)	63(5)	72(5)	117(8)	46(10)	−46(10)	−82(8)
C(9)	47(4)	50(3)	28(3)	12(5)	−8(5)	−52(5)
C(10)	57(4)	62(4)	42(4)	25(6)	−9(6)	−73(7)
C(11)	67(5)	71(4)	61(5)	46(8)	−7(8)	−74(8)
C(12)	64(5)	85(5)	42(5)	43(8)	17(8)	−53(8)
C(13)	60(4)	62(4)	29(4)	21(6)	6(6)	−28(7)
C(14)	53(4)	45(3)	26(3)	8(5)	−4(6)	−41(5)
C(15)	46(4)	52(3)	32(3)	23(5)	4(6)	−53(6)
C(16)	71(5)	64(4)	61(5)	37(7)	−19(8)	−86(8)
C(17)	73(6)	93(6)	99(7)	97(10)	−40(10)	−110(10)
C(18)	54(5)	78(5)	80(6)	72(9)	7(9)	−52(8)
C(19)	58(5)	65(4)	50(5)	45(7)	24(8)	−25(7)
C(20)	45(4)	52(3)	36(4)	21(6)	13(6)	−26(6)
C(21)	86(6)	43(3)	59(5)	6(7)	14(9)	−8(7)
C(22)	60(5)	121(7)	48(5)	53(9)	−40(8)	−91(10)
C(23)	51(4)	74(5)	76(6)	57(8)	−40(8)	−48(7)
C(24)	114(7)	47(4)	58(5)	−7(7)	18(9)	−75(8)

TABLE 1 (*Continued*)

(*c*) Fractional co-ordinates and isotopic temperature factors (Å²) in the form $\exp[-8\pi^2 U_{\rm iso}\sin^2\theta/\lambda^2]$ for the hydrogen atoms. Each hydrogen has the number of its attached carbon, with methyl hydrogens distinguished by A, B, or C

Atom	x	y	z	$U_{\rm iso}$
H(2)	0·037(9)	−0·235(8)	−0·159(10)	0·16(3)
H(3)	0·131(8)	−0·437(7)	−0·100(9)	0·12(3)
H(4)	0·249(8)	−0·458(7)	0·144(9)	0·12(3)
H(5)	0·212(10)	−0·267(8)	0·289(10)	0·12(4)
H(6)	0·124(6)	−0·084(6)	0·245(8)	0·09(2)
H(7A)	0·055(12)	−0·101(10)	−0·301(13)	0·17(5)
H(7B)	0·225(5)	−0·213(4)	−0·304(6)	0·08(1)
H(7C)	0·179(13)	−0·042(11)	−0·327(13)	0·19(5)
H(8A)	0·163(10)	0·092(8)	0·132(11)	0·21(4)
H(8B)	0·309(10)	−0·055(9)	0·072(12)	0·21(4)
H(8C)	0·226(7)	0·103(7)	−0·016(8)	0·11(3)
H(10)	0·472(10)	0·428(9)	0·355(11)	0·10(4)
H(11)	0·577(9)	0·442(8)	0·171(10)	0·12(3)
H(12)	0·558(8)	0·349(7)	−0·066(9)	0·13(3)
H(13)	0·451(10)	0·212(8)	−0·114(10)	0·12(4)
H(14)	0·340(7)	0·219(6)	0·094(8)	0·07(2)
H(16)	0·246(9)	0·139(8)	0·371(10)	0·13(4)
H(17)	0·097(6)	0·125(6)	0·513(7)	0·09(2)
H(18)	0·067(10)	0·209(9)	0·795(11)	0·13(4)
H(19)	0·174(9)	0·318(8)	0·882(10)	0·16(3)
H(20)	0·294(5)	0·371(5)	0·730(6)	0·05(2)
H(21A)	0·448(13)	0·021(11)	0·270(15)	0·14(5)
H(21B)	0·583(11)	0·006(9)	0·227(12)	0·14(4)
H(21C)	0·545(5)	−0·027(4)	0·360(6)	0·07(1)
H(22A)	0·478(10)	0·345(9)	0·604(10)	0·20(4)
H(22B)	0·633(10)	0·224(9)	0·530(11)	0·08(4)
H(22C)	0·610(9)	0·152(8)	0·652(10)	0·27(4)
H(23A)	0·138(7)	0·321(6)	0·205(8)	0·11(2)
H(23B)	0·104(6)	0·477(6)	0·218(8)	0·09(2)
H(23C)	0·010(13)	0·442(12)	0·319(15)	0·28(6)
H(24A)	0·298(7)	0·549(7)	0·589(8)	0·12(3)
H(24B)	0·128(8)	0·595(8)	0·612(10)	0·08(3)
H(24C)	0·203(12)	0·616(10)	0·467(12)	0·22(5)

The most remarkable feature of the structure is the presence of two kinds of molecule differing by a folding of the central ring. Molecule (1) of crystallographic symmetry $\bar{1}$ (C_i) closely approximates to *mmm* (D_{2h}) symmetry. Molecule (2) has no crystallographically imposed symmetry but closely approximates to *mm*2 (C_{2v}). The hydrogen atoms also conform to these symmetries to within the accuracy with which we could locate them. This may be seen for molecule (1) in Figure 3 which shows the hydrogen atoms; the hydrogens were found in corresponding positions in molecule (2). The lowering of the *mmm* symmetry of molecule (1) to *mm*2 in molecule (2) results from a folding by 14° about the Al · · · Al line, accompanied by a rotation of the $AlMe_2$ groups so as to preserve approximate equality of the C(terminal)–Al–C(bridge) angles.

We have attempted to understand the comparable stabilities of both planar and folded molecules by calculating intramolecular H · · · H distances as a function of the fold angle, for an idealised molecule of *mm*2 symmetry. It was assumed that the ratio of the angle by which the $AlMe_2$ groups are rotated to the angle of fold is constant and equal to that found in the folded molecule, and C–H distances were given their standard values.

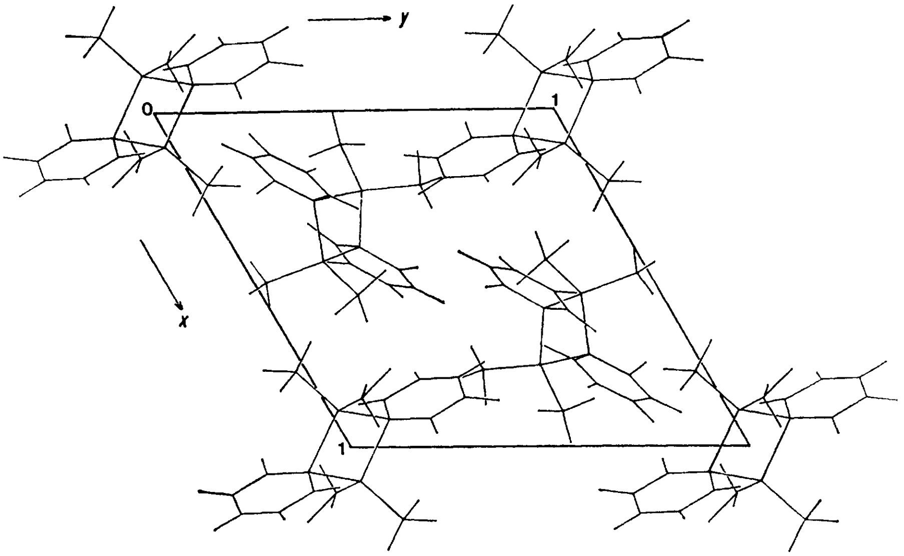

FIGURE 2 A projection of the unit cell down the c axis

TABLE 2

Bond lengths (Å) and bond angles (°) with estimated standard deviations in parentheses

(a) Distances

Molecule (1)		Molecule (2)			
Al(1)–Al(1′)	2·690(4)	Al(2)–Al(3)	2·684(3)		
C(1)–C(1′)	3·331(12)	C(9)–C(15)	3·325(9)		
Al(1)–C(1)	2·129(6)	Al(2)–C(9)	2·218(6)	Al(3)–C(9)	2·163(6)
Al(1)–C(1′)	2·152(6)	Al(2)–C(15)	2·155(6)	Al(3)–C(15)	2·135(7)
Al(1)–C(7)	1·965(9)	Al(2)–C(23)	1·977(8)	Al(3)–C(21)	1·965(8)
Al(1)–C(8)	1·978(9)	Al(2)–C(24)	1·988(8)	Al(3)–C(22)	1·991(9)
C(1)–C(2)	1·429(9)	C(9)–C(10)	1·429(9)	C(15)–C(16)	1·401(9)
C(2)–C(3)	1·388(10)	C(10)–C(11)	1·384(11)	C(16)–C(17)	1·418(12)
C(3)–C(4)	1·382(13)	C(11)–C(12)	1·371(11)	C(17)–C(18)	1·391(13)
C(4)–C(5)	1·364(13)	C(12)–C(13)	1·383(11)	C(18)–C(19)	1·370(12)
C(5)–C(6)	1·411(12)	C(13)–C(14)	1·400(9)	C(19)–C(20)	1·398(10)
C(6)–C(1)	1·423(10)	C(14)–C(9)	1·407(9)	C(20)–C(15)	1·415(9)
C(2)–H(2)	1·12(9)	C(10)–H(10)	0·83(10)	C(16)–H(16)	1·00(9)
C(3)–H(3)	1·18(8)	C(11)–H(11)	1·05(9)	C(17)–H(17)	1·07(7)
C(4)–H(4)	1·10(8)	C(12)–H(12)	1·00(9)	C(18)–H(18)	1·21(10)
C(5)–H(5)	0·81(9)	C(13)–H(13)	1·13(9)	C(19)–H(19)	1·02(9)
C(6)–H(6)	0·93(7)	C(14)–H(14)	1·09(7)	C(20)–H(20)	1·06(5)
C(7)–H(7A)	1·04(12)	C(23)–H(23A)	1·10(7)	C(21)–H(21A)	0·86(13)
C(7)–H(7B)	1·27(5)	C(23)–H(23B)	1·03(7)	C(21)–H(21B)	1·00(11)
C(7)–H(7C)	1·21(12)	C(23)–H(23C)	1·10(14)	C(21)–H(21C)	0·97(5)
C(8)–H(8A)	1·06(10)	C(24)–H(24A)	0·98(8)	C(22)–H(22A)	1·16(10)
C(8)–H(8B)	1·24(11)	C(24)–H(24B)	1·24(9)	C(22)–H(22B)	0·96(10)
C(8)–H(8C)	1·18(7)	C(24)–H(24C)	1·07(11)	C(22)–H(22C)	1·45(9)

(b) Angles

C(1)–Al(1)–C(1′)	102·2(0·2)	C(9)–Al(2)–C(15)	101·8(0·2)	C(9)–Al(3)–C(15)	101·3(0·2)
C(1)–Al(1)–C(7)	108·9(0·3)	C(9)–Al(2)–C(23)	108·4(0·3)	C(9)–Al(3)–C(21)	106·8(0·3)
C(1)–Al(1)–C(8)	109·0(0·3)	C(9)–Al(2)–C(24)	107·7(0·3)	C(9)–Al(3)–C(22)	107·6(0·3)
C(1′)–Al(1)–C(7)	107·5(0·3)	C(15)–Al(2)–C(23)	107·3(0·3)	C(15)–Al(3)–C(21)	109·5(0·3)
C(1′)–Al(1)–C(8)	106·8(0·3)	C(15)–Al(2)–C(24)	109·0(0·3)	C(15)–Al(3)–C(22)	108·2(0·3)
C(7)–Al(1)–C(8)	120·9(0·4)	C(23)–Al(2)–C(24)	121·0(0·4)	C(21)–Al(3)–C(22)	121·5(0·4)
Al(1)–C(1)–Al(1′)	77·8(0·2)	Al(2)–C(9)–Al(3)	77·4(0·2)	Al(2)–C(15)–Al(3)	77·5(0·2)
C(6)–C(1)–C(2)	116·0(0·6)	C(14)–C(9)–C(10)	115·9(0·6)	C(20)–C(15)–C(16)	116·0(0·6)
C(1)–C(2)–C(3)	121·9(0·7)	C(9)–C(10)–C(11)	122·0(0·6)	C(15)–C(16)–C(17)	122·0(0·7)
C(2)–C(3)–C(4)	120·1(0·7)	C(10)–C(11)–C(12)	119·9(0·7)	C(16)–C(17)–C(18)	119·2(0·8)
C(3)–C(4)–C(5)	120·4(0·8)	C(11)–C(12)–C(13)	120·9(0·7)	C(17)–C(18)–C(19)	120·4(0·8)
C(4)–C(5)–C(6)	121·0(0·8)	C(12)–C(13)–C(14)	119·6(0·7)	C(18)–C(19)–C(20)	120·1(0·7)
C(5)–C(6)–C(1)	120·5(0·7)	C(13)–C(14)–C(9)	121·7(0·6)	C(19)–C(20)–C(15)	122·2(0·6)

The shortest H · · · H distances which vary as a result of the folding are those between one of the methyl hydrogens and the *ortho*-hydrogen of a phenyl group [such as H(7′A) · · · H(6) in Figure 3]. There are eight equivalent contacts of this kind in the planar molecule and all are increased very slightly in the folded molecule. The distances, initially 2·51 Å increase to 2·55 Å on one side of the Al_2C_2 plane and 2·56 Å on the other, when the fold angle is 14°. For the same fold angle the distances between pairs of methyl hydrogens [such as

H(7′A) ··· H(8A)] go from 3·20 to 2·43 Å on one side and to 3·96 Å on the other. This assumes that no rotation of the methyl groups about the Al–C bonds occurs; it seems likely that such rotation is prevented by the H(Ph) ··· H(Me) repulsion. The distances between pairs of phenyl hydrogens [such as H(6) ··· H(2′)] always remain much greater than the sum of the van der Waals radii. These values suggest that folding the molecule causes no great change in the van der Waals energy of the molecule until a fold angle of *ca.* 14° (the observed value) is reached, at which point the H(Me) ··· H(Me) distance is reduced to a value close to twice the van der Waals radius (Pauling [8] gives *ca.* 1·2 Å). Further folding would produce a considerable additional H ··· H repulsion. We conclude, therefore, that for fold angles in the range ±14° the H ··· H repulsion energy may vary little, and that it is plausible to ascribe the existence of two types of molecule to the effects of packing forces.

TABLE 3

(*a*) Equations of best least-squares planes referred to orthogonal axes where X is parallel to a^*, and Z is parallel to c. Distances (Å) of atoms from the planes are given in square brackets

Plane (1):

Al(1), Al(1′), C(1), C(1′) $0{\cdot}2509X + 0{\cdot}6097Y + 0{\cdot}7519Z = 0$

[Al(1) 0, Al(1′) 0, C(1) 0, C(1′) 0]

Plane (2):

C(1)—(6) $0{\cdot}8505X + 0{\cdot}2533Y - 0{\cdot}4609Z = 0{\cdot}0347$

[C(1) 0·012, C(2) −0·004, C(3) −0·002, C(4) −0·001, C(5) 0·010, C(6) −0·015]

Plane (3):

Al(2), Al(3), C(9) $0{\cdot}3163X + 0{\cdot}8200Y + 0{\cdot}4770Z = 7{\cdot}2970$

Plane (4):

Al(2), Al(3), C(15) $0{\cdot}2068X + 0{\cdot}7198Y + 0{\cdot}6627Z = 6{\cdot}8347$

Plane (5):

C(9)—(14) $0{\cdot}8380X - 0{\cdot}4796Y + 0{\cdot}2604Z = 0{\cdot}9944$

[C(9) 0·004, C(10) −0·003, C(11) 0·002, C(12) −0·002, C(13) 0·003, C(14) −0·004]

Plane (6):

C(15)—(20) $0{\cdot}8229X - 0{\cdot}4936Y + 0{\cdot}2813Z = 1{\cdot}0680$

[C(15) −0·002, C(16) −0·001, C(17) 0·001, C(18) 0·003, C(19) −0·007, C(20) 0·006]

(*b*) Angles (°) between planes

(1)—(2)	88·8	(3)—(5)	89·8
(3)—(4)	13·7	(4)—(6)	89·9

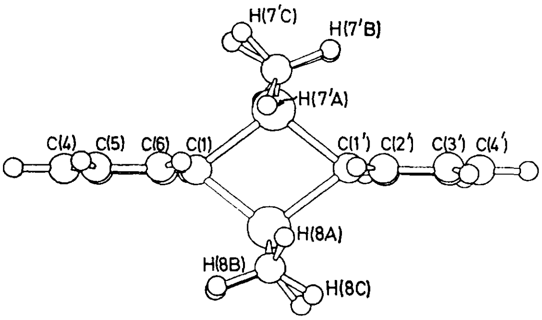

FIGURE 3 Molecule (1) showing hydrogen atoms

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[8] L. Pauling, 'The Nature of the Chemical Bond,' Cornell University Press, Ithaca, New York, 1960, p. 260.