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Neopentyl-Based Aminoalanes. Syntheses and Molecular Structures of $[Neo_2Al–N(H)R]_2$ (Neo = Neopentyl (CH_2CMe_3); R = Adamantyl, *t*-Bu, $Ph(i\text{-}Pr)_2$, Ph′ (2-Biphenyl))

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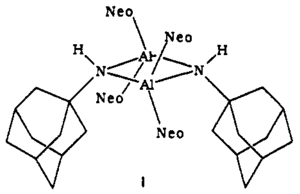
The first neopentyl-based aminoalanes, $[Neo_2Al–N(H)Ada]_2$ (I), $[Neo_2Al–N(H)Ph']_2$ (II), $[Neo_2Al–N(H)Ph(i\text{-}Pr)_2]_2$ (III), and $[Neo_2Al–N(H)\text{-}t\text{-}Bu]_2$ (IV), have been prepared by reaction of trineopentylaluminum, Neo_3Al (Neo = CH_2CMe_3), with 1-adamantanamine ($H_2N–Ada$), 2-aminobiphenyl ($H_2N–Ph'$, Ph′ = 2-biphenyl), 2,6-diisopropylaniline ($H_2N–Ph(i\text{-}Pr)_2$), and *tert*-butylamine ($H_2N\text{-}t\text{-}Bu$), respectively, in toluene. These dimers, each residing about Al_2N_2 four-membered cores, have been characterized by ^1H NMR, elemental analyses, and single-crystal X-ray diffraction. I crystallizes in the rhombohedral space group $R\bar{3}c$ (No. 167) with unit cell parameters $a = 18.665$ (7) Å, $c = 61.507$ (4) Å, $V = 18538$ Å^3, and $D_{calc} = 1.03$ g cm^{-3} for $Z = 18$. Least-squares refinement based on 878 observed reflections with intensities $I > 2\sigma(I)$ in the range $3.5 < 2\theta < 40.0°$ converged at $R = 0.095$ and $R_w = 0.099$. II crystallizes in the monoclinic space group $P2_1/n$ (No. 14) with unit cell parameters $a = 12.883$ (4) Å, $b = 20.825$ (7) Å, $c = 15.947$ (5) Å, $\beta = 94.41$ (2)°, $V = 4266$ Å^3, and $D_{calc} = 1.05$ g cm^{-3} for $Z = 4$. Least-squares refinement based on 3444 observed reflections with intensities $I > 3\sigma(I)$ in the range $3.5 < 2\theta < 45.0°$ converged at $R = 0.051$ and $R_w = 0.064$. III crystallizes in the monoclinic space group $P2_1/c$ (No. 14) with unit cell parameters $a = 10.537$ (5) Å, $b = 40.134$ (14) Å, $c = 11.570$ (4) Å, $\beta = 110.51$ (3)°, $V = 4583$ Å^3, and $D_{calc} = 1.00$ g cm^{-3} for $Z = 4$. Least-squares refinement based on 2539 observed reflections with intensities $I > 3\sigma(I)$ in the range $3.50 < 2\theta < 45.0°$ converged at $R = 0.054$ and $R_w = 0.065$. IV crystallizes in the orthorhombic space group $Aba2$ (No. 41) with unit cell parameters $a = 19.361$ (6) Å, $b = 9.745$ (3) Å, $c = 17.972$ (5) Å, $V = 3391$ Å^3, and $D_{calc} = 0.95$ g cm^{-3} for $Z = 4$. Least-squares refinement based on 825 observed reflections with intensities $I > 3\sigma(I)$ in the range $3.50 < 2\theta < 45.0°$ converged at $R = 0.075$ and $R_w = 0.091$. The Al_2N_2 cores in I and IV are decidedly nonplanar, having fold angles of 32.7° and 28.9°, respectively, between the Al_2N planes. The Al_2N_2 cores of II and III constitute nearly perfect planes. All four aminoalanes reside in cis conformations in the solid state. ^1H NMR suggests that I undergoes a trans–cis isomerization in solution (in approximately a 1:3 ratio).

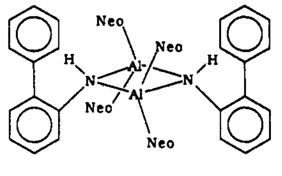
Introduction

The interaction of organoaluminum moieties with amines has been studied for decades.[1] As demonstrated by the seminal studies of Smith et al.,[2] aluminum–nitrogen condensation products possessing extensive Al–N association are often obtained from reaction of organoaluminum species with simple primary or secondary amines. Recently, the utilization of sterically demanding Lewis bases has been brought to the fore[3,4] as a means by which low coordination number aluminum complexes may be accessed. These investigations have endeavored to address, inter alia, fundamental issues regarding the extent of π-bonding in Al–E fragments (E = N, P, O, S). It is interesting that the vast majority of studies germane to the organometallic chemistry of aluminum have generally involved sterically unremarkable, and readily available, organoaluminum reagents such as trimethyl- or triethylaluminum. Indeed, the literature reveals a paucity of analogous investigations concerning sterically demanding, and less commonly available, aluminum alkyls.

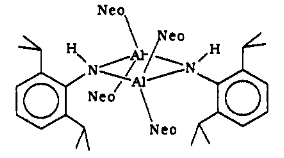
Chart I



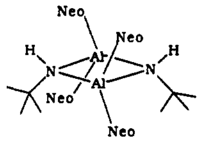
I



II



III



IV

Herein, we report the syntheses, characterization, and molecular structures of $[Neo_2Al–N(H)Ada]_2$ (I), $[Neo_2Al–N(H)Ph']_2$ (II), $[Neo_2Al–N(H)Ph(i\text{-}Pr)_2]_2$ (III), and $[Neo_2Al–N(H)\text{-}t\text{-}Bu]_2$ (IV) (Chart I) isolated from reaction of the sterically demanding aluminum alkyl tris(2,2-dimethylpropyl)aluminum, commonly known as trineopentylaluminum, Neo_3Al (Neo = CH_2CMe_3), with the primary amines 1-adamantanamine ($H_2N–Ada$), 2-aminobiphenyl ($H_2N–Ph'$, Ph′ = 2-biphenyl), 2,6-diisopropylaniline ($H_2N–Ph(i\text{-}Pr)_2$), and *tert*-butylamine ($H_2N\text{-}t\text{-}Bu$), respectively.

These organoaminoalanes, the first such compounds based on neopentylaluminum which have been structurally

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characterized, are dimeric assuming cis conformations. The preparation of these aminoalanes is given by eq 1.

$$\underset{\text{I–IV}}{\mathrm{Neo_3Al + H_2N\text{–}R \rightarrow \tfrac{1}{2}\ \mathit{cis}\text{-}[Neo_2Al\text{–}N(H)R]_2 + Neo\text{–}H}} \tag{1}$$

R = Ada (I), Ph′ (II), Ph(*i*-Pr)$_2$ (III), *t*-Bu (IV)

Experimental Section

General Comments. Owing to the extreme air and water sensitivity of the reactants and products, efforts were taken to exclude oxygen and moisture. Standard Schlenk techniques and an inert-atmosphere drybox (Vacuum Atmospheres HE-43 Dri-Lab) were employed. Aluminum bromide, neopentyl chloride, lithium powder, 1-adamantanamine, 2-aminobiphenyl, 2,6-diisopropylaniline, and *tert*-butylamine, purchased from Aldrich Chemical Co., were used without further purification. Toluene and hexane were distilled under an atmosphere of dried nitrogen over sodium/benzophenone or calcium hydride, respectively, prior to use. Neopentyllithium was prepared by the method described by Schrock and Fellmann,[5] while trineopentylaluminum was prepared as described by Beachley and Victoriano.[6] Elemental analyses were performed by E+R Microanalytical Laboratories, Corona, NY. ^{1}H NMR data were recorded on a Bruker AC300 FT-NMR spectrometer. Single-crystal X-ray diffraction studies were performed on a Nicolet R3mV diffractometer with graphite-monochromated Mo Kα radiation (λ = 0.710 73 Å). Calculations, structure solution, and refinement were performed using the SHELXTL[7] package of computer programs.

Synthesis of [Neo$_2$Al–N(H)Ada]$_2$ (I). In the drybox, H$_2$N–Ada (0.51 g, 3.4 mmol) was added to a reaction vessel containing 25 mL of toluene. Trineopentylaluminum (0.82 g, 3.4 mmol) was added dropwise into the solution. The reaction vessel was removed from the drybox and heated in an oil bath for 3 h to 60 °C. Upon cooling to room temperature, colorless, hexagonal crystals of I formed in quantitative yield (based on the amine), mp 300 °C. ^{1}H NMR (C$_6$D$_6$) δ 0.35 (s, 4 H, AlC*H*$_2$, cis isomer), 0.41 (s, 4 H, AlC*H*$_2$, cis isomer), 0.48 (s, 8 H, AlC*H*$_2$, trans isomer), 0.86 (s, 2 H, N*H*, cis isomer), 0.95 (s, 2 H, N*H*, trans isomer), 1.05 (s, 18 H, AlCH$_2$(C*H*$_3$)$_3$, cis isomer), 1.07 (s, 18 H, AlCH$_2$(C*H*$_3$)$_3$, cis isomer), 1.09 (s, 36 H, AlCH$_2$(C*H*$_3$)$_3$, trans isomer), 1.62 (s, 12 H, Ada C*H*$_2$), 1.93 (dd, 12 H, Ada C*H*$_2$), 2.09 (bs, 6 H, Ada C*H*). Anal. Found (Calcd) for C$_{40}$H$_{76}$Al$_2$N$_2$: C, 74.24 (75.18); H, 11.82 (11.99).

Synthesis of [Neo$_2$Al–N(H)Ph′]$_2$ (II). A reaction of vessel containing 20 mL of freshly distilled toluene was taken into the drybox where H$_2$N–Ph′ (0.70 g, 4.1 mmol) was added and dissolved, producing a slightly purple solution. Trineopentylaluminum (1.0 g, 4.2 mmol) was slowly added dropwise to the solution. The color of the solution gradually changed upon mixing to a light brown solution. The reaction vessel was removed from the drybox and was heated in an oil bath to 80 °C for 3 h. Cooling to room temperature resulted in the quantitative formation of colorless, air-sensitive, rectangular crystals, mp 235–236 °C. ^{1}H NMR (C$_6$D$_6$) δ 0.76 (s, 4 H, AlC*H*$_2$), 0.61 (s, 4 H, AlC*H*$_2$), 0.83 (s, 18 H, AlCH$_2$(C*H*$_3$)$_3$), 0.97 (s, 18 H, AlCH$_2$(C*H*$_3$)$_3$), 4.46 (s, 2 H, N*H*), 6.8–7.7 (m, 18 H, Ph′*H*). Anal. Found (Calcd) for C$_{44}$H$_{64}$Al$_2$N$_2$: C, 78.15 (78.30); H, 9.29 (9.56).

Synthesis of [Neo$_2$Al–N(H)Ph(*i*-Pr)$_2$]$_2$ (III). Inside the drybox a stoichiometric quantity of Neo$_3$Al (0.85 g, 3.5 mmol) was slowly added to a toluene solution (20 mL) of H$_2$N–Ph(*i*-Pr)$_2$ (0.66 mL, 3.5 mmol). A light-brown solution resulted. The reaction vessel was removed from the drybox, and the system was heated in an oil bath at 80 °C for 3 h. Colorless, rectangular, extremely air-sensitive, crystals were obtained in quantitative yield (based on the amine) upon removal of some solvent and subsequent cooling to −20 °C overnight, mp 140–141 °C. ^{1}H NMR (C$_6$D$_6$) δ 0.42 (s, 4 H, AlC*H*$_2$), 0.63 (s, 4 H, AlC*H*$_2$), 0.89 (s, 18 H, AlCH$_2$(C*H*$_3$)$_3$), 0.95 (s, 18 H, AlCH$_2$(C*H*$_3$)$_3$), 1.23 (d, 24 H, PhCH(C*H*$_3$)$_2$, 3.47 (sept, 4 H, PhC*H*(CH$_3$)$_2$), 4.39 (s, 2 H, N*H*), 6.9–7.1 (m, 6 H, C$_6$*H*$_3$). Anal. Found (Calcd) for C$_{44}$H$_{80}$Al$_2$N$_2$: C, 75.58 (76.41); H, 11.74 (11.67).

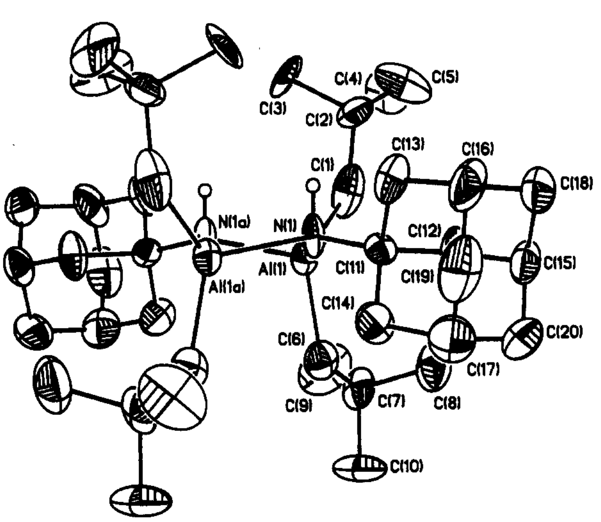

Figure 1. Thermal ellipsoid plot of [Neo$_2$Al–N(H)Ada]$_2$, I, showing the atom-labeling scheme. Thermal ellipsoids show 35% probability levels. Hydrogen atoms, except those bound to nitrogen, have been omitted.

Synthesis of [Neo$_2$Al–N(H)-*t*-Bu]$_2$ (IV). IV was prepared in a manner analogous to that employed for I–III. A quantity of Neo$_3$Al (0.85 g, 3.5 mmol) was added to a reaction vessel containing toluene (15 mL) inside the drybox. A stoichiometric quantity of *tert*-butylamine (0.40 mL, 3.8 mmol) was slowly added to this solution via syringe. The system was removed from the drybox and heated in an oil bath (90 °C) for 4 h. Cooling to −20 °C in the freezer overnight afforded colorless, extremely air-sensitive crystals of IV in high yield, mp 216–218 °C. ^{1}H NMR (C$_6$D$_6$) δ 0.59 (s, 4 H, AlC*H*$_2$), 0.66 (s, 4 H, AlC*H*$_2$), 1.21 (s, 18 H, AlCH$_2$(C*H*$_3$)$_3$), 1.27 (s, 18 H, AlCH$_2$(C*H*$_3$)$_3$), 1.11 (s, 2 H, N*H*), 1.33 (s, 18 H, NC(C*H*$_3$)$_3$). Anal. Found (Calcd) for C$_{28}$H$_{64}$Al$_2$N$_2$: C, 69.66 (69.58); H, 13.36 (13.54).

X-ray Structural Solution and Refinement for [Neo$_2$Al–N(H)Ada]$_2$ (I), [Neo$_2$Al–N(H)Ph′]$_2$ (II), [Neo$_2$Al–N(H)Ph(*i*-Pr)$_2$]$_2$ (III), and [Neo$_2$Al–N(H)-*t*-Bu]$_2$ (IV). Single crystals of I–IV, were mounted in thin-walled glass capillaries under an inert atmosphere of argon. Cell parameters and an orientation matrix for data collection at 21 °C were obtained from a least-squares fitting of several carefully centered high-order reflections. All structures were solved by direct methods and refined by full-matrix least-squares analysis. For I and IV, the asymmetric unit consists of one monomer unit situated about a crystallographic 2-fold axis which generates the remainder of the dimer; in II and III, the entire dimer unit is unique, occupying a general position in the unit cell. The methyl carbon atoms of one neopentyl group per monomer unit of I and IV were found to be disordered, with alternate sites related by rotation about the CH$_2$–CMe$_3$ bond. In I the major component atoms (C(3)–C(5)) were assigned 65% occupancy and minor component atoms (C(3a)–C(5a)), 35% occupancy, while in IV two sets of half-occupancy atoms (C(8)–C(10a)) were included. In both cases occupancies were determined by optimization of a multiplicity factor in the latter stages of refinement. For all of the compounds, full-occupancy non-hydrogen atoms were refined with anisotropic thermal parameters and partial-occupancy atoms were refined using isotropic thermal parameters. Hydrogen atoms, other than those of the disordered methyl groups, were located by standard difference Fourier techniques and were included in the structure factor calculation at idealized positions ($d_{\mathrm{C-H}}$ = 0.96 Å, $d_{\mathrm{N-H}}$ = 0.90 Å) and were allowed to ride on the atom to which they were bonded.

Isotropic group thermal parameters were refined for the hydrogen atoms (U_{iso} = 0.18 (2) Å^2 for I; 0.107 (2) Å^2 for II; 0.108 (4) Å^2 for III). The somewhat higher residuals of I and IV are due, in part, to the presence of disorder and, for I, a paucity of observed data. An attempt to determine the absolute structure of IV (the direction of the polar *z* axis), which crystallizes in a polar space group, was inconclusive. Relevant crystallographic

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Table I. Crystal Data for [Neo_2Al–N(H)Ada]$_2$ (I), [Neo_2Al–N(H)Ph′]$_2$ (II), [Neo_2Al–N(H)Ph(*i*-Pr)$_2$]$_2$ (III), and [Neo_2Al–N(H)-*t*-Bu]$_2$ (IV)

	I	II	III	IV
chem formula	$C_{44}H_{76}N_2Al_2$	$C_{44}H_{64}N_2Al_2$	$C_{44}H_{80}N_2Al_2$	$C_{28}H_{64}N_2Al_2$
mol wt	639.02	674.97	691.09	482.79
size, mm	0.10 × 0.32 × 0.32	0.23 × 0.30 × 0.36	0.24 × 0.31 × 0.52	0.16 × 0.14 × 0.37
color; habit	colorless; hexagonal plate	colorless; parallelepiped	colorless; parallelepiped	colorless; parallelepiped
space group	$R\bar{3}c$ (No. 167)	$P2_1/n$ (No. 14)	$P2_1/c$ (No. 14)	*Aba*2 (No. 41)
	rhombohedral (hexagonal setting)	monoclinic	monoclinic	orthorhombic
a, Å	18.655 (7)	12.883 (4)	10.537 (5)	19.361 (6)
b, Å		20.825 (7)	40.134 (14)	9.745 (3)
c, Å	61.507 (4)	15.947 (5)	11.570 (4)	17.972 (5)
β, deg		94.41 (2)	110.51 (3)	
vol, Å^3	18538 (18)	4266 (2)	4583 (3)	3391 (2)
Z	18	4	4	4
D_{calc}, g cm^{-3}	1.03	1.05	1.00	0.95
temp, °C		21		
reflns coll	5015	6039	6399	1156
reflns unique (R_{int})	2233 (0.024)	5564 (0.026)	5903 (0.026)	1156
reflns obsd	878 ($I > 2\sigma(I)$)	3444 ($I > 3\sigma(I)$)	2539 ($I > 3\sigma(I)$)	825 ($I > 3\sigma(I)$)
2θ range, deg	3.5–40.0	3.5–45.0	3.5–45.0	3.5–45.0
R	0.095	0.050	0.054	0.075
R_w	0.099	0.064	0.065	0.091

Table II. Atomic Coordinates (×10^4) and Equivalent Isotropic Thermal Parameters (Å^2 × 10^3) for [Neo_2Al–N(H)Ada]$_2$ (I)

	x	*y*	*z*	*U*(eq)^a
Al(1)	3267 (3)	389 (3)	632 (1)	58 (3)
N(1)	4036 (7)	24 (7)	725 (1)	67 (7)
C(1)	3710 (12)	1358 (11)	426 (2)	113 (15)
C(2)	4570 (11)	2176 (10)	406 (3)	83 (11)
C(3)	4805 (20)	2736 (17)	611 (5)	103 (20)
C(4)	4579 (19)	2740 (18)	223 (4)	121 (22)
C(5)	5264 (15)	1959 (18)	347 (6)	126 (21)
C(3a)	4871 (38)	2305 (35)	624 (10)	85 (21)
C(4a)	4179 (33)	2769 (34)	383 (9)	111 (21)
C(5a)	4812 (50)	2027 (51)	192 (13)	182 (32)
C(6)	2157 (8)	−617 (8)	567 (2)	82 (11)
C(7)	1556 (11)	−694 (12)	378 (2)	93 (13)
C(8)	1977 (11)	−644 (11)	156 (2)	133 (15)
C(9)	1268 (13)	−68 (12)	396 (3)	147 (16)
C(10)	775 (11)	−1607 (11)	391 (3)	129 (12)
C(11)	4278 (9)	−494 (10)	601 (2)	51 (9)
C(12)	4174 (10)	−453 (10)	352 (2)	87 (12)
C(13)	5206 (11)	−172 (10)	640 (2)	95 (12)
C(14)	3811 (11)	−1389 (10)	671 (3)	98 (11)
C(15)	4475 (12)	−963 (11)	230 (2)	82 (13)
C(16)	5547 (12)	−691 (14)	523 (3)	113 (15)
C(17)	4078 (14)	−1919 (11)	540 (3)	106 (15)
C(18)	5401 (12)	−600 (11)	273 (3)	102 (13)
C(19)	4997 (16)	−1605 (14)	595 (3)	120 (19)
C(20)	3984 (11)	−1861 (11)	301 (3)	110 (14)

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

data are given in Table I. Final fractional atomic coordinates are given in Tables II and IV–VI while selected bond distances and angles are given in Table III. Molecular structures of the title compounds are given in Figures 1–4.

Results and Discussion

As evidenced by the preparation of a series of interesting five-[8-10] and six-coordinate[11,12] organoaluminum complexes, the organometallic coordination chemistry of aluminum progressed greatly in the last decade. These studies have often employed sterically unremarkable organoaluminum moieties (i.e., Me_3Al, Et_3Al). Corresponding studies utilizing sterically demanding aluminum alkyls are noteworthy in their absence.

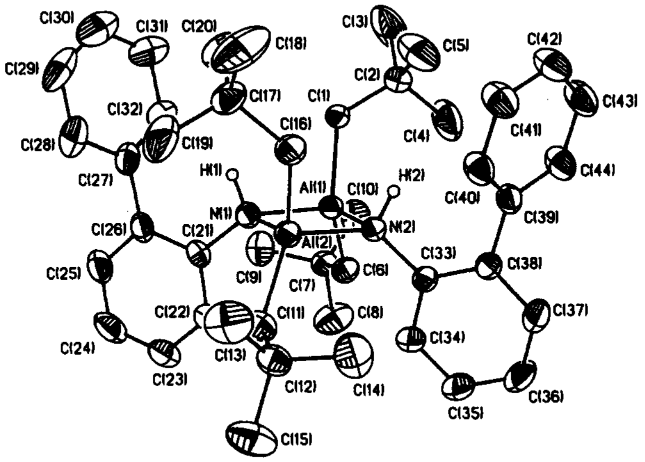

Figure 2. Thermal ellipsoid plot of [Neo_2Al–N(H)Ph′]$_2$, II, showing the atom-labeling scheme. Thermal ellipsoids show 35% probability levels. Hydrogen atoms, except those bound to nitrogen, have been omitted.

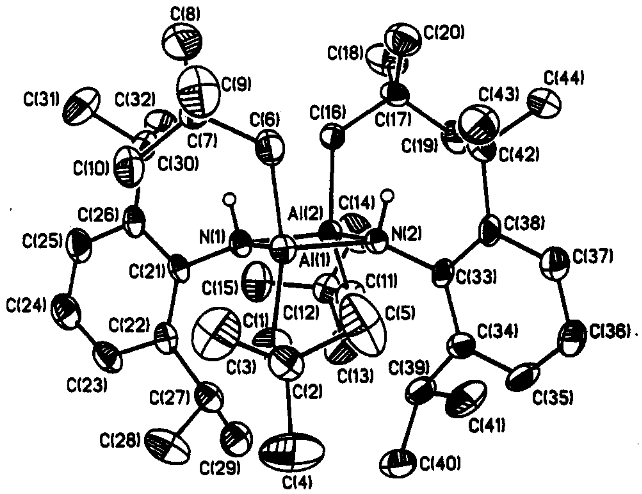

Figure 3. Thermal ellipsoid plot of [Neo_2Al–N(H)Ph(*i*-Pr)$_2$]$_2$, III, showing the atom-labeling scheme. Thermal ellipsoids show 35% probability levels. Hydrogen atoms, except those bound to nitrogen, have been omitted.

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Table III. Selected Bond Lengths (Å) and Angles (deg)

[Neo$_2$Al–N(H)Ada]$_2$ (I)			
Al(1)–N(1)	1.956 (16)	Al(1)–C(1)	2.016 (16)
Al(1)–C(6)	2.022 (12)	N(1)–C(11)	1.469 (24)
Al(1)–N(1A)	1.971 (10)	C(1)–C(2)	1.571 (21)
N(1)–Al(1A)	1.971 (10)	Al(1)···Al(1A)	2.877 (9)
C(6)–C(7)	1.570 (23)		
N(1)–Al(1)–C(1)	116.9 (8)	N(1)–Al(1)–C(6)	108.9 (6)
C(1)–Al(1)–C(6)	118.5 (7)	N(1)–Al(1)–N(1A)	81.6 (6)
C(1)–Al(1)–N(1A)	113.9 (6)	C(6)–Al(1)–N(1A)	111.3 (5)
Al(1)–N(1)–C(11)	126.6 (8)	Al(1)–N(1)–Al(1A)	94.2 (6)
C(11)–N(1)–Al(1A)	128.5 (10)	Al(1)–C(1)–C(2)	132.9 (13)
Al(1)–C(6)–C(7)	125.9 (10)	N(1)–C(11)–C(13)	108.5 (11)
N(1)–C(11)–C(12)	113.3 (16)	N(1)–C(11)–C(14)	112.4 (12)
[Neo$_2$Al–N(H)Ph′]$_2$ (II)			
Al(1)–N(2)	1.979 (3)	Al(1)–N(1)	1.999 (3)
Al(1)–C(6)	1.980 (4)	Al(1)–C(1)	1.986 (4)
Al(2)–N(2)	1.996 (3)	Al(2)–N(1)	1.980 (3)
Al(2)–C(16)	1.980 (4)	Al(2)–C(11)	1.975 (4)
N(2)–C(33)	1.456 (4)	N(1)–C(21)	1.448 (5)
C(6)–C(7)	1.532 (6)	C(1)–C(2)	1.530 (5)
C(16)–C(17)	1.537 (5)	C(11)–C(12)	1.532 (6)
		Al(1)···Al(2)	2.881 (2)
N(1)–Al(1)–N(2)	87.1 (1)	N(1)–Al(1)–C(1)	103.7 (1)
N(2)–Al(1)–C(1)	111.3 (2)	N(1)–Al(1)–C(6)	114.3 (1)
N(2)–Al(1)–C(6)	111.8 (1)	C(1)–Al(1)–C(6)	122.7 (2)
N(1)–Al(2)–N(2)	87.1 (1)	N(1)–Al(2)–C(11)	109.7 (2)
N(2)–Al(2)–C(11)	115.2 (2)	N(1)–Al(2)–C(16)	112.4 (2)
N(2)–Al(2)–C(16)	102.7 (1)	C(11)–Al(2)–C(16)	123.6 (2)
Al(1)–N(1)–Al(2)	92.8 (1)	Al(1)–N(1)–C(21)	121.9 (2)
Al(2)–N(1)–C(21)	122.6 (2)	Al(1)–N(2)–Al(2)	92.9 (1)
Al(1)–N(2)–C(33)	123.3 (2)	Al(2)–N(2)–C(33)	123.3 (2)
Al(1)–C(1)–C(2)	124.7 (3)	Al(1)–C(6)–C(7)	123.9 (3)
Al(2)–C(11)–C(12)	124.8 (3)	Al(2)–C(16)–C(17)	126.2 (3)
N(1)–C(21)–C(22)	119.3 (3)	N(1)–C(21)–C(26)	121.7 (3)
N(2)–C(33)–C(38)	121.9 (3)	N(2)–C(33)–C(34)	118.2 (3)
[Neo$_2$Al–N(H)Ph(*i*-Pr)$_2$]$_2$ (III)			
Al(1)–N(1)	2.013 (5)	Al(1)–N(2)	2.007 (5)
Al(1)–C(1)	1.964 (7)	Al(1)–C(6)	1.989 (8)
Al(2)–N(1)	2.011 (5)	Al(2)–N(2)	2.030 (5)
Al(2)–C(11)	1.969 (8)	Al(2)–C(16)	1.994 (7)
N(2)–C(33)	1.478 (9)	N(1)–C(21)	1.457 (8)
		Al(1)···Al(2)	2.954 (3)
N(1)–Al(1)–N(2)	86.0 (2)	N(1)–Al(1)–C(1)	117.0 (2)
N(2)–Al(1)–C(1)	112.3 (3)	N(1)–Al(1)–C(6)	104.2 (3)
N(2)–Al(1)–C(6)	102.7 (2)	C(1)–Al(1)–C(6)	126.5 (3)
N(1)–Al(2)–N(2)	85.4 (2)	N(1)–Al(2)–C(11)	118.0 (3)
N(2)–Al(2)–C(11)	120.3 (3)	N(1)–Al(2)–C(16)	102.7 (3)
N(2)–Al(2)–C(16)	105.1 (3)	C(11)–Al(2)–C(16)	119.4 (3)
Al(1)–N(1)–Al(2)	94.5 (2)	Al(1)–N(1)–C(21)	135.2 (3)
Al(2)–N(1)–C(21)	125.3 (4)	Al(1)–N(2)–Al(2)	94.1 (2)
Al(1)–N(2)–C(33)	118.9 (3)	Al(2)–N(2)–C(33)	138.9 (4)
Al(1)–C(1)–C(2)	126.7 (5)	Al(1)–C(6)–C(7)	126.3 (5)
Al(2)–C(11)–C(12)	124.8 (6)	Al(2)–C(16)–C(17)	126.0 (5)
N(1)–C(21)–C(22)	121.2 (6)	N(1)–C(21)–C(26)	119.4 (6)
N(2)–C(33)–C(34)	121.9 (6)	N(2)–C(33)–C(38)	118.7 (6)
[Neo$_2$Al–N(H)-*t*-Bu]$_2$ (IV)			
Al(1)–N(1)	1.970 (7)	Al(1)–N(1A)	1.983 (7)
N(1)–Al(1A)	1.983 (7)	Al(1)–C(6)	1.955 (12)
Al(1)–C(1)	2.018 (11)	C(1)–C(2)	1.535 (16)
N(1)–C(11)	1.502 (11)	Al(1)···Al(1A)	2.870 (5)
C(6)–C(7)	1.577 (19)		
Al(1)–N(1)–Al(1A)	93.1 (3)	N(1)–Al(1)–N(1A)	83.5 (3)
C(1)–Al(1)–C(6)	114.7 (5)	N(1)–Al(1)–C(1)	109.4 (4)
N(1A)–Al(1)–C(1)	109.5 (4)	N(1)–Al(1)–C(6)	118.7 (4)
N(1A)–Al(1)–C(6)	116.9 (4)	Al(1)–N(1)–C(11)	128.9 (6)
Al(1A)–N(1)–C(11)	129.0 (6)	Al(1)–C(1)–C(2)	126.6 (8)
Al(1)–C(6)–C(7)	131.1 (9)	N(1)–C(11)–C(12)	107.5 (11)
N(1)–C(11)–C(13)	110.3 (10)	N(1)–C(11)–C(14)	110.2 (11)

Table IV. Atomic Coordinates (×10^4) and Equivalent Isotropic Thermal Parameters (Å^2 × 10^3) for [Neo$_2$Al–N(H)Ph′]$_2$ (II)

	x	*y*	*z*	*U*(eq)
Al(1)	2211 (1)	4882 (1)	3166 (1)	40 (1)
Al(2)	3071 (1)	3888 (1)	2172 (1)	42 (1)
N(1)	3133 (2)	4837 (1)	2215 (2)	42 (1)
N(2)	2206 (2)	3932 (1)	3156 (2)	40 (1)
C(1)	885 (3)	5234 (2)	2637 (2)	47 (1)
C(2)	−197 (3)	5070 (2)	2911 (3)	56 (2)
C(3)	−945 (4)	5586 (3)	2661 (5)	161 (4)
C(4)	−174 (4)	4955 (4)	3832 (3)	122 (3)
C(5)	−581 (4)	4464 (3)	2473 (4)	115 (3)
C(6)	2895 (3)	5228 (2)	4225 (2)	51 (1)
C(7)	2962 (3)	5945 (2)	4438 (2)	51 (1)
C(8)	3684 (5)	6042 (3)	5233 (3)	95 (2)
C(9)	3390 (4)	6324 (2)	3725 (3)	72 (2)
C(10)	1910 (4)	6216 (2)	4595 (3)	76 (2)
C(11)	4482 (3)	3529 (2)	2412 (3)	55 (2)
C(12)	4770 (3)	2821 (2)	2309 (3)	55 (2)
C(13)	4666 (4)	2637 (3)	1379 (3)	94 (2)
C(14)	4064 (4)	2394 (2)	2778 (3)	92 (2)
C(15)	5889 (4)	2707 (3)	2633 (4)	94 (2)
C(16)	2070 (3)	3570 (2)	1262 (2)	54 (2)
C(17)	1990 (4)	3794 (2)	341 (2)	64 (2)
C(18)	1425 (6)	3291 (3)	−212 (3)	124 (3)
C(19)	3050 (5)	3920 (3)	52 (3)	122 (3)
C(20)	1370 (4)	4404 (3)	248 (3)	91 (2)
C(21)	4104 (3)	5189 (2)	2228 (2)	44 (1)
C(22)	4891 (3)	5055 (2)	2852 (3)	53 (2)
C(23)	5822 (3)	5378 (2)	2893 (3)	65 (2)
C(24)	5988 (3)	5845 (3)	2309 (4)	82 (2)
C(25)	5227 (3)	5983 (2)	1691 (3)	72 (2)
C(26)	4276 (3)	5656 (2)	1626 (2)	52 (1)
C(27)	3458 (3)	5826 (2)	932 (3)	58 (2)
C(28)	3713 (5)	5729 (3)	89 (3)	85 (2)
C(29)	2964 (7)	5896 (3)	−553 (3)	105 (3)
C(30)	2036 (6)	6140 (3)	−385 (4)	103 (3)
C(31)	1805 (4)	6244 (3)	430 (3)	91 (2)
C(32)	2541 (3)	6086 (2)	1077 (3)	64 (2)
C(33)	2406 (3)	3543 (2)	3910 (2)	40 (1)
C(34)	3316 (3)	3655 (2)	4414 (2)	52 (2)
C(35)	3519 (3)	3318 (2)	5157 (3)	67 (2)
C(36)	2837 (4)	2866 (3)	5388 (3)	83 (2)
C(37)	1939 (4)	2744 (2)	4883 (3)	69 (2)
C(38)	1711 (3)	3080 (2)	4139 (2)	45 (1)
C(39)	732 (3)	2921 (2)	3628 (2)	47 (1)
C(40)	733 (3)	2689 (2)	2810 (2)	55 (2)
C(41)	−193 (3)	2542 (2)	2344 (3)	68 (2)
C(42)	−1125 (3)	2619 (2)	2690 (3)	71 (2)
C(43)	−1134 (3)	2834 (3)	3502 (3)	73 (2)
C(44)	−221 (3)	2980 (2)	3967 (3)	64 (2)

Although preparations of triphenylaluminum[13,14] have been known for decades, synthetic procedures for other sterically demanding aluminum alkyls have generally not been forthcoming. The literature of late, however, reveals imaginative synthetic procedures for some sterically demanding aluminum alkyls. Indeed, the syntheses and molecular structures of Mes$_3$Al,[15] Mes$_3$Ga,[16] and Mes$_3$In[17] (Mes = mesityl) were recently reported. Each of these products were shown to be monomeric with the metal atoms residing in nearly idealized trigonal planar geometries. With regard to neopentyl derivatives of group 13 metals, the preparations of Neo$_3$Al,[6] Neo$_3$Ga,[18] and Neo$_3$In[19] have also been reported.

As an extension of earlier aminoalane studies performed by this laboratory,[20-23] and in an attempt to explore the

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Table V. Atomic Coordinates (×10^4) and Equivalent Isotropic Thermal Parameters (Å^2 × 10^3) for [$Neo_2Al-N(H)Ph(i-Pr)_2]_2$ (III)

	x	*y*	*z*	*U*(eq)
Al(1)	2354 (2)	1004 (1)	1922 (2)	40 (1)
Al(2)	4483 (2)	1481 (1)	3290 (2)	38 (1)
N(1)	2449 (5)	1458 (1)	2685 (5)	39 (2)
N(2)	4380 (5)	1031 (1)	2460 (4)	38 (2)
C(1)	1491 (8)	973 (2)	121 (6)	57 (3)
C(2)	1003 (8)	650 (2)	−654 (7)	56 (3)
C(3)	−312 (9)	531 (2)	−551 (8)	98 (5)
C(4)	737 (11)	719 (2)	−2004 (7)	123 (6)
C(5)	2049 (9)	378 (2)	−229 (9)	122 (5)
C(6)	2076 (7)	696 (2)	3159 (7)	63 (4)
C(7)	744 (7)	642 (2)	3404 (7)	49 (3)
C(8)	995 (9)	681 (2)	4764 (7)	93 (5)
C(9)	231 (9)	286 (2)	3023 (9)	98 (5)
C(10)	−354 (7)	888 (2)	2663 (7)	70 (4)
C(11)	5323 (7)	1864 (2)	2771 (6)	54 (3)
C(12)	5591 (7)	2212 (2)	3428 (7)	52 (3)
C(13)	6101 (9)	2452 (2)	2659 (8)	96 (5)
C(14)	6667 (8)	2184 (2)	4706 (7)	93 (5)
C(15)	4315 (8)	2357 (2)	3537 (8)	89 (5)
C(16)	4995 (7)	1380 (2)	5082 (5)	51 (3)
C(17)	6328 (7)	1217 (2)	5907 (6)	52 (3)
C(18)	6938 (8)	1418 (2)	7095 (7)	95 (4)
C(19)	7316 (7)	1197 (2)	5227 (7)	81 (4)
C(20)	6034 (8)	869 (2)	6282 (7)	80 (4)
C(21)	1541 (6)	1743 (2)	2473 (6)	37 (3)
C(22)	1141 (6)	1922 (2)	1341 (7)	43 (3)
C(23)	267 (8)	2195 (2)	1193 (7)	64 (4)
C(24)	−184 (8)	2301 (2)	2111 (9)	76 (4)
C(25)	230 (8)	2130 (2)	3214 (8)	67 (4)
C(26)	1083 (6)	1852 (2)	3432 (7)	46 (3)
C(27)	1514 (7)	1827 (2)	222 (7)	52 (3)
C(28)	255 (8)	1750 (2)	−914 (7)	87 (4)
C(29)	2308 (8)	2104 (2)	−129 (7)	75 (4)
C(30)	1492 (7)	1697 (2)	4717 (7)	53 (3)
C(31)	266 (9)	1597 (2)	5056 (8)	90 (5)
C(32)	2424 (8)	1928 (2)	5713 (7)	71 (4)
C(33)	5080 (6)	847 (2)	1754 (6)	37 (3)
C(34)	5292 (7)	987 (2)	711 (6)	46 (3)
C(35)	5841 (8)	792 (2)	22 (7)	60 (4)
C(36)	6234 (8)	464 (2)	333 (8)	66 (4)
C(37)	6067 (7)	334 (2)	1373 (7)	58 (4)
C(38)	5493 (7)	517 (2)	2099 (6)	46 (3)
C(39)	4993 (8)	1347 (2)	305 (7)	60 (4)
C(40)	4112 (9)	1391 (2)	−1055 (7)	87 (5)
C(41)	6358 (9)	1530 (2)	561 (8)	93 (5)
C(42)	5422 (7)	333 (2)	3242 (6)	52 (3)
C(43)	4614 (9)	8 (2)	2912 (8)	80 (4)
C(44)	6846 (8)	259 (2)	4163 (7)	77 (4)

organoaluminum chemistry of sterically demanding aluminum alkyls, we endeavored to examine the reaction of Neo_3Al with sterically demanding primary amines. Herein we report the syntheses, characterization, and molecular structures of the neopentyl-based aminoalanes [$Neo_2Al-N(H)Ada]_2$ (I), [$Neo_2Al-N(H)Ph']_2$ (II), [$Neo_2Al-N(H)-Ph(i-Pr)_2]_2$ (III), and [$Neo_2Al-N(H)$-*t*-$Bu]_2$ (IV).

A number of points are worthy of note regarding structure and bonding in these aminoalanes. All four compounds have cis confirmations, with both aza hydrogen atoms oriented toward the same side of the Al_2N_2 ring. Within experimental error, the bonding to aluminum is equivalent in all four compounds, with Al–N bonds ranging from 1.96 (2) to 2.013 (5) Å and Al–C bonds ranging from 1.964 (7) to 2.02 (2) Å. Significant distortion from an ideal tetrahedral geometry is observed for all of the atoms in the constrained four-membered rings; ring angles at aluminum range from 81.6 (6) to 87.1 (1)° and at nitrogen from 92.8 (1) to 94.5 (2)°.

Table VI. Atomic Coordinates (×10^4) and Equivalent Isotropic Thermal Parameters (Å^2 × 10^3) for [$Neo_2Al-N(H)$-*t*-$Bu]_2$ (IV)

	x	*y*	*z*	*U*(eq)
Al(1)	5573 (1)	933 (3)	5000	44 (1)
N(1)	5434 (3)	−1039 (7)	4811 (4)	45 (3)
C(1)	5759 (5)	1231 (11)	6093 (6)	59 (4)
C(2)	6347 (7)	2080 (13)	6432 (7)	62 (5)
C(3)	6287 (7)	1992 (15)	7314 (7)	92 (6)
C(4)	7042 (6)	1351 (21)	6234 (11)	130 (8)
C(5)	6337 (12)	3516 (16)	6214 (10)	149 (10)
C(6)	6173 (6)	1973 (12)	4331 (7)	69 (5)
C(7)	6225 (7)	1995 (17)	3455 (8)	69 (5)
C(8)	6833 (22)	2821 (43)	3174 (25)	120 (16)
C(9)	5549 (14)	2767 (27)	3150 (15)	81 (7)
C(10)	6235 (22)	623 (42)	3067 (23)	136 (13)
C(8A)	6614 (20)	3289 (39)	3222 (22)	99 (11)
C(9A)	5551 (22)	1505 (45)	3076 (25)	146 (13)
C(10A)	6777 (21)	714 (37)	3271 (22)	122 (12)
C(11)	5922 (5)	−2211 (9)	4949 (9)	59 (3)
C(12)	5985 (9)	−2400 (20)	5811 (9)	122 (7)
C(13)	6636 (7)	−1830 (18)	4714 (11)	137 (8)
C(14)	5629 (10)	−3492 (18)	4660 (14)	189 (13)

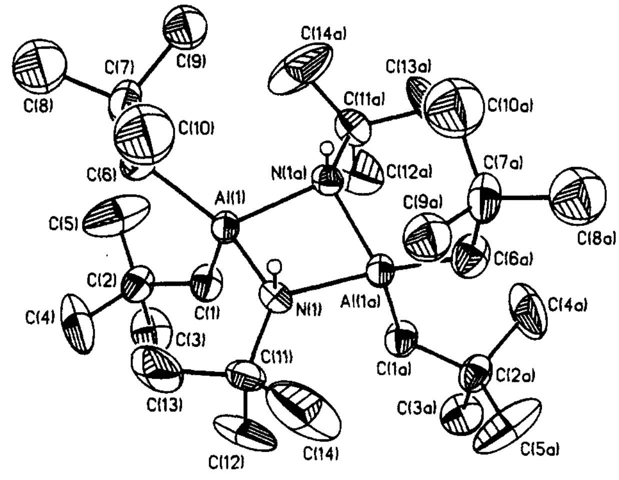

Figure 4. Thermal ellipsoid plot of [$Neo_2Al-N(H)$-*t*-$Bu]_2$, IV, showing the atom-labeling scheme. Thermal ellipsoids show 35% probability levels. Hydrogen atoms, except those bound to nitrogen, have been omitted.

A significant structural feature of these neopentyl-based aminoalanes, the planarity of the Al_2N_2 four-membered ring, appears to be related to the steric requirements of the group bound to nitrogen. For I and IV, the steric bulk of the amine substituent is limited to one side of the ring plane. The neopentyl groups bound to aluminum in both compounds assume a similar conformation, in which the CMe_3 portion of the neopentyl groups on the same side (as N–R) of the Al_2N_2 ring are oriented away from the center of the ring while those on the opposite side of the ring are oriented in toward the center of the ring. This conformation brings the latter neopentyl groups into close proximity. So as to minimize this interaction, the ring becomes puckered, affording an Al_2N plane fold angle of 32.7° for I and 28.9° for IV. For II and III, the steric bulk of the amine substituents extends over both sides of the Al_2N_2 plane. In both of these compounds, the neopentyl groups on either side of the Al_2N_2 plane assume an intermediate conformation relative to the extremes found for I and IV. This more symmetrical orientation of neopentyl groups results in a planar Al_2N_2 ring (mean devia-

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tion is 0.022 Å for II and 0.019 Å for III).

The relative positioning of the α-carbon atoms and associated *tert*-butyl fragments of the neopentyl groups is also interesting and may best be illustrated by considering the Al$\cdots$Al approach as a "bond" and then examining the resulting Al$\cdots$Al–C$_\alpha$ "bond angles". In I and IV, the α-carbon atoms cis to the N–H groups are directed away from the central cavity at angles of 139.1 (5) and 142.0 (4)°, respectively, while the *tert*-butyl fragments are oriented back toward the central cavity. Alternatively, the α-carbon atoms trans to the N–H groups are directed in toward the central cavity (Al$\cdots$Al–C$_\alpha$ angles of 102.4 (4) and 103.2 (3)°, respectively) and the *tert*-butyl fragments are turned away from the center. In II and III, the opposite positioning is seen. The α-carbon atoms cis to N–H are oriented into the cavity (Al$\cdots$Al–C$_\alpha$ angles of 112.8 (1) and 112.7 (1)° for II and 107.1 (2) and 107.6 (2)° for III), while those in the trans position are oriented away (Al$\cdots$Al–C$_\alpha$ angles of 124.5 (1) and 123.5 (1)° for II and 126.2 (2) and 132.9 (2)° for III). The *tert*-butyl fragments in II and III are rotated to positions intermediate to the two extremes seen in I and IV; a pseudo-C_2 axis normal to the Al_2N_2 ring relates the two Neo_2Al groups.

An appreciation of some of the ramifications of the sterically demanding neopentyl substituent as to cis or trans conformations may be realized by examining other organoaminoalanes. The dimethylaluminum analog of I, $[Me_2Al{-}N(H)Ada]_2$ (V),[24,25] provides an informative comparison as it resides in a trans conformation about a planar Al_2N_2 four-membered ring. It is important to note that the ^1H NMR spectrum of I clearly indicates the presences of both cis and trans isomers in approximately a 3:1 ratio. The trans isomers of II–IV were not observed. The fact that V assumed a trans conformation while the *seemingly* more sterically demanding I resides in a cis conformation is intriguing. A similar phenomenon is observed with the methylaluminum derivative of II, $[Me_2Al{-}N(H)Ph']_2$ (VI).[26] However, the isobutyl derivative of II, $[(i\text{-}Bu)_2Al{-}N(H)Ph']_2$ (VII),[26] resides in a cis conformation. The dimethylaluminum derivative of III, $[Me_2Al{-}N(H)Ph(i\text{-}Pr)_2]_2$ (VIII),[25] resides in a trans conformation. It is also interesting that the Al_2N_2 ring of VIII was found to be decidely nonplanar, having a fold angle of 22.7° between the Al_2N planes.

The fact that each of the four aminoalanes reported herein reside in cis conformations is noteworthy. The available data suggest that cis conformations are preferred in cases in which sterically demanding aluminum alkyls have been employed, whereas trans conformations are often observed when sterically less-demanding aluminum alkyls are involved. Isobutyl appears to possess the minimum steric requirements to afford cis conformations. Relative to core planarity, the steric bulk of the amine appears to be a more substantive factor than the steric bulk of the aluminum alkyl in determining the planarity of the Al_2N_2 ring of the given aminoalane. Additional studies concerning the ramifications of sterically demanding aluminum alkyls are forthcoming from this laboratory.

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Supplementary Material Available: A summary of data collection and refinement, plots of molecules, and tables of crystal data, bond distances and angles, final fractional coordinates, and thermal parameters (38 pages). Ordering information is given on any current masthead page.

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