SHORT COMMUNICATION

(*tert*-Butoxy)aluminium and -gallium Hydrides☆

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The crystal structures obtained by X-ray methods of (*tert*-butoxy)alane and -gallane (tBuOMH$_2$) are isotypic and are composed of discrete centrosymmetric dimers. The dimers arise from almost symmetrical Al–O–Al [1.810(3), 1.815(3) Å] and Ga–O–Ga [1.902(9), 1.908(9) Å] bridges forming a central rhombohedral four membered M_2O_2 cycle [O–Al–O 81.0(2)°, O–Ga–O 78.6(5)°] with the metallic atoms in a distorted tetrahedral environment. When one of the hydride ligands on the metals is further substituted by *tert*-butoxy, the formation of (tBuO)$_2$AlH and (tBuO)$_2$GaH is observed. These two compounds crystallize in different lattices and space groups, but they are nevertheless very similar in their structure: the centrosymmetric M_2O_2 cycle is maintained [Al–O 1.817(3), 1.817(3) Å; Ga–O 1.904(4), 1.907(4) Å]. As expected, the terminal Al–O [1.675(3) Å] and Ga–O [1.783(4) Å] distances are considerably shorter than the bridging ones.

Very recently the synthesis and crystal structure determination of bis(*tert*-butyl)methoxyalane (tBu$_2$HC–O–AlH$_2$) and the corresponding gallane were described[1]. We have independently studied quite similar compounds. Our intention has been to "bridge" the structural gap between AlH$_3$[2] and Al(OtBu)$_3$[3] with the missing mixed compounds, e.g. tBuOAlH$_2$ and (tBuO)$_2$AlH, and to examine the homologous gallium compounds. We were interested in the structures of these compounds because we had used them in CVD processes[4]. A profound synthetic work on the aluminium compounds has already been published in 1968 by Nöth and Suchy[5], and some predictions of structures were made on the basis of IR spectroscopy and molecular weight determinations.

The synthesis of the compounds tBuOMH$_2$ and (tBuO)$_2$MH (M = Al, Ga) was achieved by alcoholysis of diethyl ether solutions of alane and gallane[5] [equation (1)].

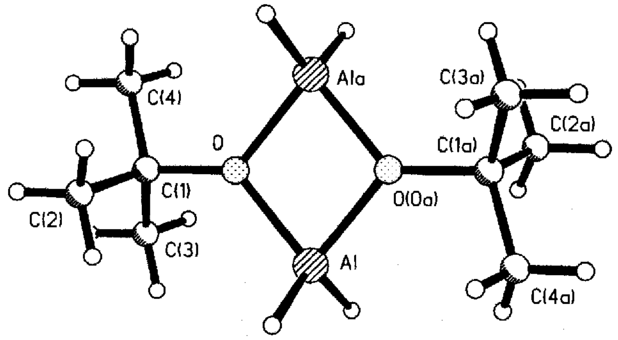
$$\mathrm{Et_2O\cdot MH_3} + n\,t\mathrm{BuOH} \rightarrow \frac{n}{2}\mathrm{H_2} + \mathrm{Et_2O} + (t\mathrm{BuO})_n\mathrm{MH}_{3-n} \qquad (1)$$

$$\mathrm{M = Al, Ga};\ n = 1 \text{ or } 2$$

The correct stoichiometry of the reaction is important for obtaining the desired products and the yields have been very satisfactory. The products were isolated by sublimation and crystallization. tBuOAlH$_2$ and tBuOGaH$_2$ show very simple NMR spectra in solution: in the ^{1}H-NMR spectrum, the *tert*-butyl group exhibits a singlet, and in the ^{13}C-NMR spectrum two lines for the primary and quaternary carbon atoms are detected. The ^{1}H and ^{13}C-NMR spectra of (tBuO)$_2$AlH and (tBuO)$_2$GaH are much more complex. In each case two sets of two signals of equal intensity were found that can be attributed to four chemically different *tert*-butyl groups. In the aluminium derivative, the ratio of the sets of signals is approximately 45:55, whereas it is 33:66 in the case of gallium derivative. We tentatively ascribed the four signals in the ^{1}H-NMR spectra to a mixture of *cis* and *trans* isomers [*cis* and *trans* refer to the position of the hydrides with respect to the M_2O_2 central ring (see structure below)]. The *trans* isomer presumably leads to higher intensities. The chemical shifts of the signals change with the temperature of the NMR probe, but no changes of the intensities or a coalescence phenomenon have been observed[6]. All four compounds are dimeric in benzene, as found by cryoscopy.

The data for the X-ray structure determination on single crystals of tBuOMH$_2$ and (tBuO)$_2$MH (M = Al, Ga) have been assembled in Table 1; the most important bond lengths and angles are summarized in Table 2[7]. As a result of the structure analyses, the molecule tBuOAlH$_2$ (tBuOGaH$_2$ is isotypic) has been depicted in Figure 1, and the molecules (tBuO)$_2$MH (M = Al, Ga) in Figures 2 and 3, respectively. All compounds form cyclic dimers with a central, centrosymmetric, four-membered M_2O_2 cycle. As can be deduced from the interatomic distances, these dimers show only van-der-Waals type interactions within the lattices.

Figure 1. A projection of the X-ray crystal structure of the dimeric *tert*-butylalane [tBuOAlH$_2$]$_2$[11] Atoms not numberd are hydrogen atoms; the "a" added to the atom numbering designate atoms related by the inversion symmetry



In each compound, aluminium or gallium is tetracoordinated and approximately tetrahedral-, all bridging oxygen atoms are in a trigonal planar environment (see angle sum for the oxygen atom in Table 2). The hydride atoms could be located from difference fourier maps, but their positions should be observed with caution, especially in the gallium compounds, because of the big difference in the X-ray scattering of the atoms to which they are linked.

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Figure 2. The molecular dimer in the crystal structure of $[(t\mathrm{BuO})_2\mathrm{AlH}]_2$[11]. Atoms without numbering are hydrogen atoms; for "a" notation see Figure 1

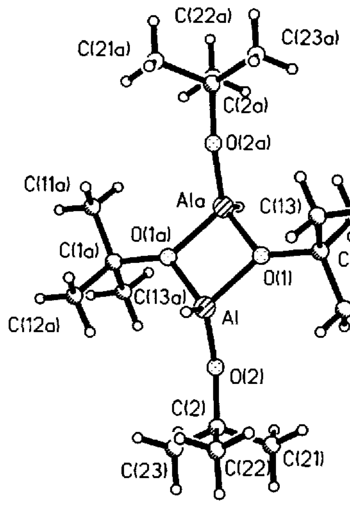
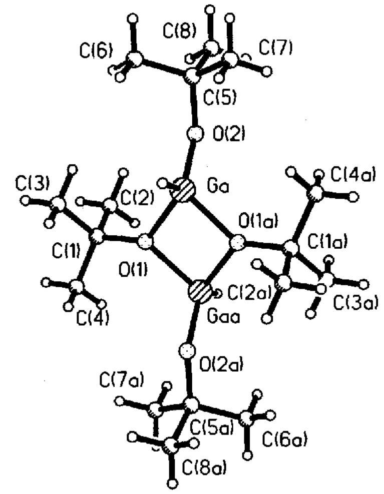


Figure 3. The molecular dimer in the crystal structure of $[(t\mathrm{BuO})_2\mathrm{GaH}]_2$[11]. Atoms without numbering are hydrogen atoms; for "a" notation see Figure 1



The most striking difference in our results compared to those obtained by C. L. Raston et al.[1] on $t\mathrm{Bu_2CHOAlH_2}$ is that this molecule is a tetramer in the crystal while $t\mathrm{BuOAlH_2}$ is a dimer. Consequently, the bond lengths are affected by the higher coordination numbers at aluminium in the tetramer and by the different electronic factors of the ligands. The mean Al–O distance in $[(t\mathrm{Bu})_2\mathrm{CHOAlH_2}]_4$ is 1.863(2) Å for pentacoordinate Al atoms and 1.834(2) Å for tetracoordinate, whereas it is 1.813(2) Å in $[t\mathrm{BuOAlH_2}]_2$.

The two gallanes $[t\mathrm{Bu_2CHOGaH_2}]_2$[1] and $[t\mathrm{BuOGaH_2}]_2$ are similar in that they form comparable dimers by oxygen bridging. The mean Ga–O–Ga angle in the first compound is 99.3(3)°, which is 2.1° less than in $[t\mathrm{BuOGaH_2}]_2$. This might be attributed to the bulkier $\mathrm{CH}(t\mathrm{Bu})_2$ group compared to *tert*-butyl group that elongates the ring along the O–O diagonal. The Ga–O distances in $[t\mathrm{Bu_2CHOGaH_2}]_2$ (as in the aluminium case) are distinctly longer (mean 1.93(1) Å) than in $[t\mathrm{BuOGaH_2}]_2$ (mean 1.905(9) Å). This is because *tert*-butyl is in an α-position with respect to oxygen in $t\mathrm{BuOMH_2}$ while it has a β-position in the other case.

The substitution of one of the hydride atoms by *tert*-butoxy in $(t\mathrm{BuOAlH_2})_2$ or $(t\mathrm{BuOGaH_2})_2$ does not affect the geometry of the central rings much, as can be deduced from Table 2 (the angles remain almost unchanged). The substitution also has no effect on the Ga–O bond; the bond lengths in $[t\mathrm{BuOGaH_2}]_2$ and $[(t\mathrm{BuO})_2\mathrm{GaH}]_2$ compare well with one another. It nevertheless has a minor effect on the Al–O bond, which becomes slightly longer (0.008 Å, which is within the standard errors) in $[(t\mathrm{BuO})_2\mathrm{AlH}]_2$ than in $(t\mathrm{BuO})\mathrm{AlH_2}$. As expected the coordination number at the oxygen atoms has a direct influence on the Al–O and Ga–O distances, which are about 0.142(3) Å or 0.122(4) Å shorter in the terminal positions (see also Table 2). The Al–O–C angle of the terminal bond [144.3(4)°] is considerably greater than the corresponding Ga–O–C angle [126.0(4)°]. This might be due to steric repulsion that is larger in the case of aluminium than in gallium (see also different arguments in lit.[8]), because the dimensions within $[(t\mathrm{BuO})_2\mathrm{GaH}]_2$ are comparatively larger.

Table 1. Crystal data for $t\mathrm{BuOMH_2}$ and $(t\mathrm{BuO})_2\mathrm{MH}$ (M = Al, Ga)

formula	$[\mathrm{tBuO\text{-}AlH_2}]_2$ $\mathrm{C_8H_{22}Al_2O_2}$	$[\mathrm{tBuO\text{-}GaH_2}]_2$ $\mathrm{C_8H_{22}Ga_2O_2}$	$[(\mathrm{tBuO})_2\mathrm{AlH}]_2$ $\mathrm{C_{16}H_{38}Al_2O_4}$	$[(\mathrm{tBuO})_2\mathrm{GaH}]_2$ $\mathrm{C_{16}H_{38}Ga_2O_4}$
fw	204.22	289.70	348.42	433.90
a [Å]	6.911(9)	7.089(5)	8.137(4)	8.780(5)
b [Å]	12.18(2)	12.247(7)	16.547(8)	11.890(8)
c [Å]	8.325(11)	8.380(5)	8.507(5)	21.353(12)
α [°]	90	90	90	90
β [°]	99.96(11)	101.25(5)	97.92(4)	90
γ [°]	90	90	90	90
V [Å^3]	690(2)	713.5(8)	1135(1)	2229(2)
Z	2	2	2	4
Crystal System	monoclinic	monoclinic	monoclinic	orthorhombic
Space Group	$P2_1/n$	$P2_1/n$	$P2_1/c$	Pbca
δ [g/cm^3]	0.983	1.348	1.02	1.293
μ (MoK$_α$) [mm^{-1}]	0.182	3.748	0.140	2.429
number of reflections	900	627	1361	1965
parameters	74	48	106	104
R_1 [I>2σ(I)]	0.066	0.090	0.071	0.065
wR_2	0.197	0.297	0.194	0.139
largest diff. peak	0.422	0.806	0.451	0.470
and hole [eÅ^{-3}]	-0.227	-0.653	-0.272	-0.306

In the series $\mathrm{AlH_3}$, $\mathrm{AlH_2(O}t\mathrm{Bu)}$, $\mathrm{AlH(O}t\mathrm{Bu)_2}$ and $\mathrm{Al(O}t\mathrm{Bu)_3}$, the compounds become molecular with the second member compound and the association is reduced to dimers by introducing the tBuO ligand. The central $\mathrm{Al_2O_2}$ ring becomes larger with higher amount of alkoxy groups (Al–O: 1.812 < 1.817 < 1.824) whereas the terminal Al–O–C angles remain unchanged (approximately 144°). In the corresponding gallium series, all compounds are dimers, even the starting molecule (gas phase) which has hydride bridges[9]; no important changes in comparable bond lengths have been observed.

The IR-spectra of $[\mathrm{H_2MO}t\mathrm{Bu}]_2$ (gas cell) and $[\mathrm{HM(O}t\mathrm{Bu)_2}]_2$ (Nujol) showed characteristic[5] absorption bands for M–H stretching (degenerated) $[\mathrm{H_2MO}t\mathrm{Bu}]_2$: 1845 cm^{-1} (M = Al); 1906 cm^{-1} (M = Ga) and $[\mathrm{HM(O}t\mathrm{Bu)_2}]_2$: 1859 cm^{-1} (M = Al); 1949 cm^{-1} (M = Ga) and deformation $[\mathrm{H_2MO}t\mathrm{Bu}]_2$: 775, (731) cm^{-1} (M = Al); (739), 700 (M = Ga) and $[\mathrm{HM(O}t\mathrm{Bu)_2}]_2$: 776 cm^{-1} (M = Al); 764 cm^{-1} (M = Ga) modes.

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Table 2. Selected bond lengths [Å] and bond angles [°] for $[t\mathrm{BuOAlH_2}]_2$, $[t\mathrm{BuOGaH_2}]_2$, $[(t\mathrm{BuO})_2\mathrm{AlH}]_2$ and $[(t\mathrm{BuO})_2\mathrm{GaH}]_2$ (see also ref.[12])

$[\mathrm{tBuO\text{-}AlH_2}]_2$			
Al-O	1.810(3)	Al-H(1)	1.55(4)
Al-O'	1.815(3)	Al-H(2)	1.55(5)
		O-C(1)	1.444(5)
O-Al-O'	81.0(2)	C(1)-O-Al	131.4(3)
C(1)-O-Al'	129.6(3)	Al-O-Al'	99.0(2)
H(1)-Al-H(2)	122(2)		
$[\mathrm{tBuO\text{-}GaH_2}]_2$			
Ga-O	1.908(9)	O-C(1)	1.49(2)
Ga-O'	1.902(9)	Ga-H(1)	1.61(10)
		Ga-H(2)	1.60(12)
O-Ga-O'	78.6(5)	C(1)-O-Ga	129.2(8)
C(1)-O-Ga'	129.2(8)	Ga-O-Ga'	101.4(5)
$[(\mathrm{tBuO})_2\mathrm{AlH}]_2$			
Al-O(1)	1.817(3)	Al-O(1')	1.817(3)
Al-O(2)	1.675(3)	Al-H	1.51(5)
C(1)-O(1)	1.469(6)	C(2)-O(2)	1.411(6)
O(1)-Al-O(2)	113.7(2)	H-Al-O(1)	111(2)
H-Al-O(1')	117(2)	H-Al-O(2)	117(2)
O(2)-Al-O(1')	111.5(2)	O(1)-Al-O(1')	80.8(2)
Al-O(1)-Al'	99.2(2)	C(1)-O(1)-Al	130.6(3)
C(1)-O(1)-Al'	129.7(3)	C(2)-O(2)-Al	144.3(4)
$[(\mathrm{tBuO})_2\mathrm{GaH}]_2$			
Ga-O(1)	1.904(4)	Ga-O(2)	1.783(4)
Ga-O(1')	1.907(4)	Ga-H	1.53(7)
C(1)-O(1)	1.439(7)	C(5)-O(2)	1.416(7)
O(1)-Ga-O(2)	106.9(2)	O(1')-Ga-O(2)	108.9(2)
O(1)-Ga-O(1')	79.4(2)	O(1)-Ga-H	116(3)
O(1')-Ga-H	114(2)	O(2)-Ga-H	123(2)
C(1)-O(1)-Ga	129.2(3)	C(1)-O(1)-Ga'	128.4(3)
Ga-O(1)-Ga'	100.6(2)	C(5)-O(2)-Ga	126.0(4)

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Experimental

All experimental procedures were performed under dry N_2 by using standard Schlenk techniques. Solvents were dried by refluxing with the appropriate drying agent and were distilled before use. NMR spectra have been measured in benzene with 5% deuteriobenzene (also internal standard) on a Bruker 200 MHz AC 200P. Infrared spectra were recorded on the model 883 of the Perkin Elmer Company. The intensity measurements of the X-ray reflections were performed on a Siemens AED 2 diffractometer.

Preparation of $[\mathrm{HAl(OtBu)_2}]_2$ and $[\mathrm{H_2AlOtBu}]_2$: Following established routes[5], 4.554 g (120 mmol) $\mathrm{LiAlH_4}$ were dissolved in 80 ml of diethyl ether in a flask with a reflux condenser. With cooling 5.334 g (40 mmol) of aluminium trichloride were dissolved in 80 ml diethyl ether and added to the lithium aluminium hydride in a steady flow at room temperature. LiCl precipitates from the mixture. To this suspension 23.718 g (320 mmol) or 11.859 g (160 mmol) *tert*-butyl alcohol was added dropwise, and formation of hydrogen was observed. The reaction was completed by stirring the reaction mixture at ambient temperature for 4 h. After separation of the lithium chloride by filtration, the solvent was condensed in a cooling trap, and the residue was sublimed at 60 °C/0.16 mbar or 20 °C/1 mbar. 27.7 g (90%) colorless crystals of bis[(di-*tert*-butoxy)aluminium hydride) and 15.2 g (93%) colorless crystals of bis[(*tert*-butoxy)aluminium dihydride), respectively. *$[\mathrm{H_2AlOtC_4H_9}]_2$:* m.p. 71 °C, dec. >120 °C; ^{1}H NMR ($\mathrm{C_6D_6}$): δ = 1.22 [18 H, $\mathrm{C(CH_3)_3}$], 4.43 (broad, 4 H, $\mathrm{AlH_2}$); ^{13}C NMR ($\mathrm{C_6D_6}$): δ = 30.36 ($\mathrm{CH_3}$, br.), 76.43 [$\mathrm{C(CH_3)_3}$, br]. – IR: $\tilde{\nu}$ = 1845 cm^{-1} (Al-H), 775; (731) cm^{-1} (δ Al-H). – $\mathrm{C_8H_{22}Al_2O_2}$: calcd. C 47.00; H 10.86; found C 47.02, H 10.65. – $[\mathrm{HAl(O}t\mathrm{C_4H_9]_2)_2}$: m.p. 82 °C dec. >260 °C, ^{1}H NMR ($\mathrm{C_6D_6}$, toluene): δ = 1.376 [s, 18 H, 55%, $\mathrm{C(CH_3)_3}$], 1.378 (s, 18 H 45%, $\mathrm{C(CH_3)_3}$], 1.414 [s, 18 H 55%, $\mathrm{C(CH_3)_3}$], 1.420 [s, 18 H 45%, $\mathrm{C(CH_3)_3}$]. – ^{13}C NMR ($\mathrm{C_6D_6}$, toluene): δ = 31.51 ($\mathrm{CH_3}$, broad), 34.03 ($\mathrm{CH_3}$), 34.05 ($\mathrm{CH_3}$), 69.41 [$C\mathrm{(CH_3)_3}$], 69.53 [$C\mathrm{(CH_3)_3}$], 76.39 [$C\mathrm{(CH_3)_3}$], 76.43 [$C\mathrm{(CH_3)_3}$]. – IR: $\tilde{\nu}$ = 1859 cm^{-1} (Al-H), 776 cm^{-1} (δ Al-H). – $\mathrm{C_{16}H_{38}Al_2O_4}$: calcd. C 55.4; H 10.99; found C 55.15, H 10.77.

Preparation of $[\mathrm{HGa(OtC_4H_9)_2}]_2$ and $[\mathrm{H_2GaOtC_4H_9}]_2$: The synthesis of $[\mathrm{H}_{3\text{-}x}\mathrm{Ga(O}t\mathrm{C_4H_9)}_x]_2$ is similar to that of $[\mathrm{H}_{3\text{-}x}\mathrm{Al(O}t\mathrm{C_4H_9)}_x]_2$, and $\mathrm{GaH_3 \cdot OEt_2}$ was prepared according to the literature[10]. To a freshly prepared solution of 10 mmol $\mathrm{GaH_3}$ in diethyl ether, 0.741 g (10 mmol) *tert*-butanol was added dropwise at 0 °C. The formation of hydrogen was weaker than in the alcoholysis reaction of $\mathrm{AlH_3 \cdot OEt_2}$. The reaction was completed after stirring (3 h) at ambient temperature. After separation of the lithium chloride by filtration, the diethyl ether was evaporated under vacuum. In the case of $[\mathrm{H_2GaO}t\mathrm{C_4H}]_2$, the remaining oily solid was sublimed under dynamic vacuum from a bath at 0 °C on a cold finger at –78 °C, where it was collected as a colorless crystalline solid. The yield was 1.271 g (4.38 mmol, 87%) based on 10 mmol $\mathrm{GaH_3}$. In the case of $[\mathrm{HGa(O}t\mathrm{C_4H_9)_2}]_2$ the remaining oily liquid was separated by fractional sublimation or distillation under vacuum. The last fraction was collected at –78 °C. The yield was only 0.737 g (1.7 mmol, 34%) $[\mathrm{HGa(O}t\mathrm{C_4H_9)_2}]_2$ based on 10 mmol $\mathrm{GaH_3}$. The yield increased if the oily liquid was refluxed with $[\mathrm{Ga(O}t\mathrm{C_4H_9)_3}]_2$ in hexane before isolating the product by sublimation. After removing hexane under vacuum, the oily solid is sublimed at 70 °C and 0.18 mbar. Yield 1.713 g, 79%. *$[\mathrm{H_2GaOtC_4H_9}]_2$:* m.p. 47 °C, dec. >110 °C; ^{1}H NMR ($\mathrm{C_6D_6}$): δ = 1.15 [s, 18 H, $\mathrm{C(CH_3)_3}$], 5.69 (broad, 4 H, $\mathrm{GaH_2}$); ^{13}C NMR ($\mathrm{C_6D_6}$): δ = 30.50 ($\mathrm{CH_3}$), 74.74 [$\mathrm{C(CH_3)_3}$]. – IR: $\tilde{\nu}$ = 1906 cm^{-1} (Ga-H); (739), 700 cm^{-1} (δ Ga-H). $\mathrm{C_8H_{22}Ga_2O_2}$: calcd. C 33.16, H 7.65, Ga 48.13; found C 31.54, H 7.59, Ga 48.55. *$[\mathrm{HGa(OtC_4H_9)_2}]_2$:* m.p. 75 °C, dec. >180 °C; ^{1}H NMR ($\mathrm{C_6D_6}$, toluene): δ = 1.390 [s, 18 H 33%, $\mathrm{C(CH_3)_3}$], 1.395 [s, 18 H 66%, $\mathrm{C(CH_3)_3}$], 1.403 [s, 18 H 66%, $\mathrm{C(CH_3)_3}$], 1.416 [s, 18 H 33%, $\mathrm{C(CH_3)_3}$]. ^{13}C NMR ($\mathrm{C_6D_6}$, toluene): δ = 31.91 ($\mathrm{CH_3}$); 32.01 ($\mathrm{CH_3}$), 34.26 ($\mathrm{CH_3}$), 34.28 ($\mathrm{CH_3}$), 71.31 [$C\mathrm{(CH_3)_3}$], 71.36 [$C\mathrm{(CH_3)_3}$], 76.29 [$C\mathrm{(CH_3)_3}$, broad]. – IR: $\tilde{\nu}$ = 1949 cm^{-1} (Ga-H); 764 cm^{-1} (δ Ga-H). – $\mathrm{C_{16}H_{38}Ga_2O_4}$: calcd. C 44.29, H 8.83, Ga 32.13; found C 40.82, H 7.95, Ga 31.79 (carbide formation).

☆ Dedicated to Prof. *H. W. Roesky* on the occasion of his 60th birthday.

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