

# The crystal structure of LiMgAlH<sub>6</sub> from combined neutron and synchrotron X-ray powder diffraction

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## Abstract

LiMgAlH<sub>6</sub> is the intermediate phase when LiMg(AlH<sub>4</sub>)<sub>3</sub> is heated. It contains 9.4 wt.% hydrogen, of which 4.8 wt.% is released during the decomposition step to MgH<sub>2</sub> and LiH. Deuterated LiMgAlD<sub>6</sub> was prepared by heat-treating LiMg(AlD<sub>4</sub>)<sub>3</sub> at 130 °C. Powder neutron and synchrotron X-ray diffraction patterns were measured and the structure was refined using the Rietveld technique on both patterns simultaneously. LiMgAlD<sub>6</sub> crystallizes in the trigonal space group *P*321 with *a* = 7.9856(4) Å and *c* = 4.3789(3) Å. The structure consists of isolated AlD<sub>6</sub> octahedra connected through octahedrally coordinated Mg- and Li-atoms.

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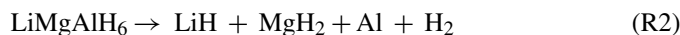
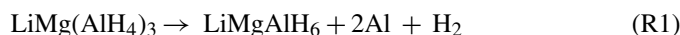
**Keywords:** Metal hydrides; Mixed alانات; Crystal structure; Synchrotron X-ray powder diffraction; Neutron powder diffraction

## 1. Introduction

Since the reversible hydrogenation of Ti-enhanced sodium alanate (NaAlH<sub>4</sub>) at reasonable conditions was discovered by Bogdanovic [1], much attention has been given to alانات. NaAlH<sub>4</sub> is still considered to be among the most promising materials for hydrogen storage. When alkali tetra-alanate MAlH<sub>4</sub> compounds are heated, the intermediate state hexa-alanate M<sub>3</sub>AlH<sub>6</sub> compounds are usually formed, followed by the monohydride MH upon further heating. The homoleptic alkali hexa-alانات include Na<sub>3</sub>AlH<sub>6</sub> [2], Li<sub>3</sub>AlH<sub>6</sub> [3], and K<sub>3</sub>AlH<sub>6</sub> [4–6]. The earth alkaline alanate Ca(AlH<sub>4</sub>)<sub>2</sub> also decomposes through an intermediate phase consisting of AlH<sub>6</sub> units in CaAlH<sub>5</sub> [7], but Mg(AlH<sub>4</sub>)<sub>2</sub> is directly dehydrogenated to magnesium hydride MgH<sub>2</sub> [8,9]. Alانات containing more than one alkali or alkaline earth atom, mixed alانات, have also been investigated, with LiNa<sub>2</sub>AlH<sub>6</sub> and K<sub>2</sub>NaAlH<sub>6</sub> being the

first examples [1,10–14]. The existence of the mixed hexa-alanate K<sub>2</sub>LiAlH<sub>6</sub> was predicted by a modelling study [4,15], which was later confirmed by experiments [12,16]. Recently the mixed lithium magnesium hexa-alanate LiMgAlH<sub>6</sub> was reported [17,18]. The crystal structure of this compound has also been predicted by first-principles calculations to be in the trigonal space group *P*321 [19].

LiMgAlH<sub>6</sub> is an intermediate product when LiMg(AlH<sub>4</sub>)<sub>3</sub> decomposes:



The first step (R1) appears at 100–130 °C and is exothermic; thus the tetra-alanate is not useful for reversible hydrogen storage. The second step (R2) appears at 150–180 °C and is endothermic, which demonstrates the stability of the intermediate LiMgAlH<sub>6</sub> [17,18]. LiMgAlH<sub>6</sub> contains 9.4 wt.% hydrogen of which 4.8 wt.% hydrogen is released through the reaction (R2). Another 3.6 wt.% can be released when heating to about 250 °C (reaction (R3)), at significant higher temperatures also

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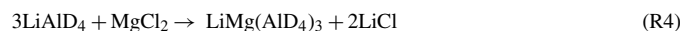
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LiH decomposes. This paper describes the crystal structure of LiMgAlD<sub>6</sub> determined by a combined synchrotron and neutron diffraction study.

## 2. Experimental

All chemicals were purchased from commercial sources. LiAlD<sub>4</sub> (>98 atom% D and chemical assay >95%; Sigma–Aldrich) and MgCl<sub>2</sub> (≥98%, Merck) were obtained as powders. Diethyl ether, Et<sub>2</sub>O, was dried over and distilled from Na/benzophenone. Toluene was also anhydrous grade from Sigma–Aldrich and was dried over and distilled from Na/anthracene. All handling of samples were carried out under Ar atmosphere. Deuterated samples were used in this investigation because of the higher purity of commercial LiAlD<sub>4</sub> and the significantly reduced incoherent scattering with deuterium compared to hydrogen.

LiMg(AlD<sub>4</sub>)<sub>3</sub> was synthesized as described in detail in a previous publication [20]; LiAlD<sub>4</sub> and MgCl<sub>2</sub> were ball milled for 3 h, giving LiMg(AlD<sub>4</sub>)<sub>3</sub> and LiCl:



Recrystallization in diethyl ether/toluene gave a highly crystalline sample containing about 15% LiCl. Thermal desorption spectroscopy (TDS) studies were performed with the in-house-built TDS-instrument. The sample (0.3 g) was heated in dynamic vacuum at a constant rate of 2 °C/min from 22 °C to 130 °C and then kept at this temperature for 6 h. Mamatha et al. reported somewhat lower desorption temperatures for (R1) in their study (100 °C) [18], we believe that this may be explained by a higher degree of impurities in their sample and the use of hydride instead of deuteride. This gave a sample containing LiCl in addition to LiMgAlD<sub>6</sub> and Al, according to (R1). No other impurity phases than Al and LiCl were included in the following structural refinements (see below).

Synchrotron radiation powder X-ray diffraction (SR-PXD) data at 22 °C were collected at the Swiss-Norwegian beam line (station BM01B) at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France. The sample was placed in a rotating 0.8 mm boron-silica glass capillary. Intensities were measured in steps of  $\Delta(2\theta) = 0.005^\circ$ . The wavelength 0.50059 Å was obtained from a channel-cut Si(1 1 1) monochromator.

Powder neutron diffraction (PND) data at 22 °C were collected with the PUS instrument at the JEEP II reactor at Kjeller, Norway [21]. Neutrons with the wavelength 1.5556 Å were obtained from a Ge(5 1 1) focusing monochromator. The detector unit consists of two banks of seven position-sensitive <sup>3</sup>He detectors, each covering 20° in  $2\theta$  (binned in steps of 0.05°). Data were collected from 10° to 130° in  $2\theta$ . The sample was placed in a rotating cylindrical vanadium sample holder with 6 mm diameter.

The atomic positions were determined with the program Free Objects for Xtal structures (FOX Version 1.6.0.2.) [22]. FOX uses global-optimization algorithms (simulated annealing, parallel tempering) to solve the crystal structure by performing trials in direct space. Rietveld refinements were carried out using Fullprof Version 3.20 [23]. X-ray form factors and neutron scattering lengths were taken from the Fullprof library. Pseudo-Voigt profile functions were used. The SR-XRD refinement backgrounds were modelled by interpolation between manually chosen points, while a 12-coefficient Fourier-cosine polynomial was used to fit the background in the PND refinements. Three different phases were refined: LiMgAlD<sub>6</sub>, Al, and LiCl. In addition, one impurity peak was found in the PND pattern. This impurity peak at  $2\theta = 42.5^\circ$  is probably due to oxidation and is consistent with Al<sub>2</sub>O<sub>3</sub>. The region  $2\theta = 42.10\text{--}43.10^\circ$  was excluded from the neutron pattern. In total, 37 parameters were refined in the final refinement: 17 structural parameters and 2 profile parameters for LiMgAlD<sub>6</sub>; 2 structural parameters and 3 profile parameters for LiCl; 2 structural parameters and 3 profile parameters for Al; and 2 zero points and 6 scale factors. The instrumental resolution of SR-PXD was found by a LaB<sub>6</sub> standard.  $B_{\text{iso}}$  for Li is set to 1.5 Å<sup>2</sup>; since the weak scattering for Li both in the X-ray and neutron radiation makes refinement of the displacement parameter difficult.

## 3. Results and discussion

A recent density-functional modeling study [19] has predicted that LiMgAlH<sub>6</sub> crystallizes in the trigonal space group

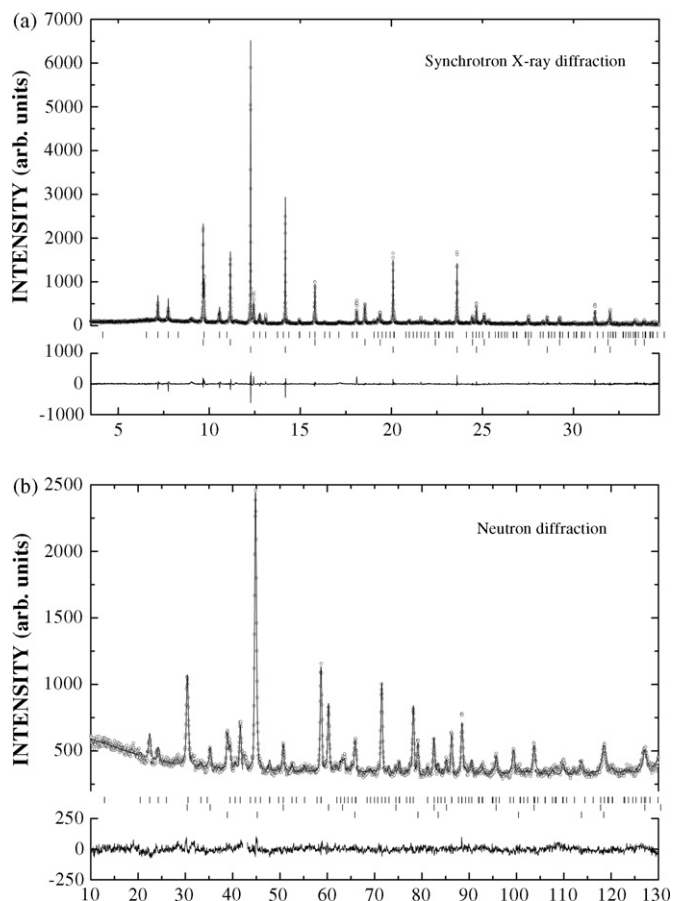


Fig. 1. (a) SR-PXD and (b) PND patterns for LiMgAlD<sub>6</sub> at 295 K showing observed (circles), calculated (upper line) and difference (bottom line) plots. The positions of the Bragg reflections are shown for LiMgAlD<sub>6</sub> (upper), LiCl (middle) and Al (lower).

*P*321. The cell parameters from that study were relaxed to  $a = b = 8.0006 \text{ \AA}$ ,  $c = 4.3837 \text{ \AA}$ . The volume of the unit cell is  $V = 243.00 \text{ \AA}^3$ , and there are three formula units and six asymmetric units in the unit cell ( $Z = 3$ ). It is worth noting that non-hydrogen atoms can be described in the space group  $P\bar{6}2m$ .

Structural determination using FOX within the *P*321 space group gave the same result as the theoretical calculation; with cell parameters:  $a = 7.9856(4) \text{ \AA}$  and  $c = 4.3789(3) \text{ \AA}$ . The *a*-axis is somewhat shorter than in the calculated structure, but overall a very good correspondence. (Since the calculated structure at 0 K has not been corrected for temperature or zero point contributions, a larger difference had been expected.) Rietveld refinement of the structure concluded that the sample contained about 35 wt.% LiMgAlD<sub>6</sub>, 42 wt.% Al, and 23 wt.% LiCl. According to (R1) the weight percent of aluminium should be 3/4 of LiMgAlD<sub>6</sub>. The refined wt.% aluminium is somewhat higher, indicating that some of the LiMgAlD<sub>6</sub> may have decomposed according to (R2). Another possibility is that a part of the LiMgAlD<sub>6</sub> phase is amorphous, and thus not contributing to the Bragg peaks. The fit of the combined Rietveld refinements, with the SR-PXD and PND data refined simultaneously, is shown in Fig. 1. The structure was refined with a relative contribution of 0.25 for the SR-PXD data and the 0.75 for PND data (Table 1).

Table 1  
The refined crystal structure of LiMgAlD<sub>6</sub> at 295 K

Atom	x	y	z	B (Å <sup>2</sup> )
Mg	1	0.3570(13)	0	0.005(3)
Li	0	0.686(6)	0.5	0.01900
Al1	0	0	0	0.003(3)
Al2	1/3	2/3	0.492(10)	0.003(3)
D1	0.540(3)	0.763(2)	0.278(3)	0.0223(11)
D2	0.119(3)	0.576(2)	0.734(3)	0.0223(11)
D3	0.904(2)	0.117(2)	0.228(3)	0.0223(11)

The space group is *P*321 and unit cell dimensions are: *a* = 7.985550(2) Å, *c* = 4.378942(7) Å, *V* = 241.83(2) Å<sup>3</sup>, and *Z* = 3. Atomic coordinates and isotropic displacement factors are given below. Standard deviations are given in parentheses. Reliability factors are *R*<sub>w</sub> = 17.6% and  $\chi^2$  = 3.03 for the PXD data and *R*<sub>w</sub> = 5.1% and  $\chi^2$  = 1.11 for the PND data.

Table 2  
Selected atomic distances (Å) in the first coordination sphere in the crystal structure of LiMgAlD<sub>6</sub>

Atom 1	Atom 2	Minimum (Å)	Maximum (Å)
Li	D	1.89(4)	1.92(4)
Mg	D	1.91(2)	1.97(2)
Al1	D	1.78(1)	1.78(2)
Al2	D	1.71(3)	1.83(3)
Li	Mg	3.42(4)	
Li	Al2	2.74(3)	
Mg	Al1	2.85(1)	

The structure of LiMgAlD<sub>6</sub> is related to the Na<sub>2</sub>SiF<sub>6</sub> structure type [24] with hexagonal close packing (hcp) of the deuterium atoms with Al, Mg, and Li occupying half of the octahedral sites, selected distances are given in Table 2. The crystal structure consists of isolated AlD<sub>6</sub>-octahedra tied together by octahedral LiD<sub>6</sub> and MgD<sub>6</sub> atoms in a three dimensional network. There are two symmetrically independent Al sites connected to three symmetrically unique D atoms. The structure can be described as alternating AlMg<sub>3</sub> and Al<sub>2</sub>Li<sub>3</sub> layers, see Fig. 2. In the Al<sub>2</sub>Li<sub>3</sub> layer the AlD<sub>6</sub> octahedra are sharing edges with three LiD<sub>6</sub>; each LiD<sub>6</sub>-octahedron is sharing edges with two Al-octahedra, and forming a two dimensional network (Fig. 2a). The AlMg<sub>3</sub> layer consists of isolated AlD<sub>6</sub>-octahedra sharing edges with three MgD<sub>6</sub>, and each Mg-octahedron shares edges with only

one Al-octahedron; isolated AlMg<sub>3</sub>-units are formed (Fig. 2b). The layers are interconnected by corner sharing of the AlD<sub>6</sub>-octahedron and six Mg/LiD<sub>6</sub> octahedra. All corners connect to Mg-, Li- and Al-octahedra, i.e. all D are bound to one Mg, one Li and one Al atom.

LiMg(AlD<sub>4</sub>)<sub>3</sub> was previously described as a very distorted hexagonal close packing of the AlD<sub>4</sub>-units showing some resemblance to Mg(AlD<sub>4</sub>)<sub>2</sub>, but without similarities to LiAlD<sub>4</sub> [20]. LiMgAlD<sub>6</sub> on the other hand can be compared to Li<sub>3</sub>AlD<sub>6</sub>. During the structure analysis of LiMgAlD<sub>6</sub>, it was noted that the PXD diagram of Li<sub>3</sub>AlD<sub>6</sub> [3] showed some similarities to LiMgAlD<sub>6</sub>. Excluding Li and D atoms and only including Al atoms in the calculated X-ray PXD diagram for Li<sub>3</sub>AlD<sub>6</sub> gave a diagram with same main features as the experimental data for LiMgAlD<sub>6</sub>. This indicates that the unit cells of the structures of LiMgAlD<sub>6</sub> and Li<sub>3</sub>AlD<sub>6</sub> are similar.

Both LiMgAlD<sub>6</sub> and Li<sub>3</sub>AlD<sub>6</sub> crystallize in trigonal space groups, LiMgAlD<sub>6</sub> in *P*321 and Li<sub>3</sub>AlD<sub>6</sub> in *R* $\bar{3}$ . The size and the shape of the unit cells are similar; the only significant difference is that *c* is twice as long for Li<sub>3</sub>AlD<sub>6</sub>. For a better comparison we divide the cell of Li<sub>3</sub>AlD<sub>6</sub> into two with *c'* = (1/2)*c*. The Al atoms in Li<sub>3</sub>AlD<sub>6</sub> are now placed at each corner of the hexagon. In addition Al atoms are at (1/3, 2/3, 1/3) and (2/3, 1/3, 2/3), respectively. In LiMgAlD<sub>6</sub> the Al atoms are in the same positions, in the corners, but the Al atoms in the middle of the unit cell are translated along *c* towards each other to the positions (1/3, 2/3, 0.5) and (2/3, 1/3, 0.5). The Al atoms in the Li<sub>3</sub>AlD<sub>6</sub> structure have eight nearest neighbours and six more distant neighbours, while the two different Al atoms in LiMgAlD<sub>6</sub> have two nearest neighbours and twelve more distant neighbours, and three nearest neighbours and six more distant neighbours, respectively. Li<sub>3</sub>AlD<sub>6</sub> can be described as a distorted bcc structure of the AlD<sub>6</sub>-units. The cubic structure has rhombohedral distortion. The structure of the AlD<sub>6</sub>-units in LiMgAlD<sub>6</sub> is best described as trigonal prism.

Table 3 shows a comparison with structures of related alanates. The table consists of all known metal alanates with metal atom number ≤ 20 that consist of AlD<sub>6</sub>-units. In CaAlH<sub>5</sub> the AlH<sub>6</sub> units are connected as helical chains [7]; they are connected by Ca<sup>2+</sup>-ions with coordination numbers exceeding six (C.N. > 6). MgAlH<sub>5</sub> has not been observed. In LiMgAlD<sub>6</sub>,

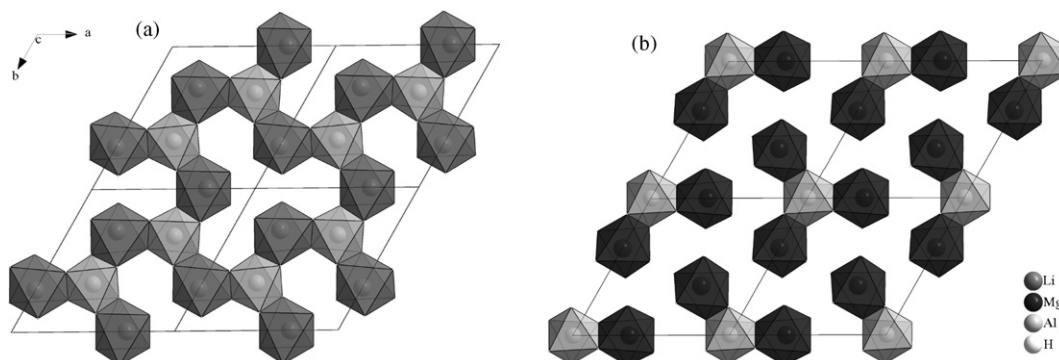


Fig. 2. The different sheets in LiMgAlD<sub>6</sub>: (a) Li<sub>3</sub>Al<sub>2</sub> and (b) Mg<sub>3</sub>Al. The sheets are connected in the *c*-direction, giving a three-dimensional structure.

Table 3  
Comparison of crystal structures for all known light alanates that contain  $\text{AlH}_6/\text{AlD}_6$ -units

	$A_mB_n$							
	$\text{K}_2\text{LiAlH}_6$ $R\bar{3}m^a$	$\text{K}_2\text{NaAlH}_6$ $Fm\bar{3}m^a$	$\text{LiNa}_2\text{AlD}_6$ $Fm\bar{3}m^a$	$\text{LiMgAlD}_6$ $P321^a$	$\text{Li}_3\text{AlD}_6$ $R\bar{3}^a$	$\text{Na}_3\text{AlD}_6$ $P2_1/n^a$	$\text{K}_3\text{AlH}_6$ $P2_1/n^a$	$\text{CaAlH}_5$ $P2_1/n^a$
$a$ (Å)	5.621	8.118	7.385	7.986	8.071	5.390	6.0250	8.325
$b$ (Å)	5.621	8.118	7.385	7.986	8.071	5.514	6.1020	6.967
$c$ (Å)	27.399	8.118	7.385	4.384	9.513	7.725	8.5820	9.780
$\alpha$ (°)	90	90	90	90	90	90	90	90
$\beta$ (°)	90	90	90	90	90	89.86	89.60	94.22
$\gamma$ (°)	120	90	90	120	120	120	90	90
$V$ (Å <sup>3</sup> )/ $Z$	124.9	133.7	100.7	80.6	89.4	114.8	157.8	70.7
Al–H/D	1.78	1.76	1.76	1.77	1.75	1.76	1.78	1.74
Al–A	3.42	3.52	3.69	2.74	2.91	3.20	3.58	3.05
Al–B	2.53	4.06	3.20	2.85	N.A.	N.A.	N.A.	N.A.
A–H/D	2.82	2.88	1.93	1.91	2.00	2.38	2.73	2.35
B–H/D	2.02	2.30	2.61	1.94	N.A.	N.A.	N.A.	N.A.

The compounds are on the form  $A_mB_n$ :  $\text{AlH}_6$  or  $\text{CaAlH}_5$ . All data are taken from experimental studies [2,3,7,13,14,20], except for the data for  $\text{K}_3\text{AlH}_6$  and  $\text{K}_2\text{LiAlH}_6$  that are taken from theoretical calculations [4,16]. Metal to hydrogen distances are given as the average of the first coordination sphere. A/B–Al distances are given as minimum values.

<sup>a</sup> Space group.

$\text{Mg}^{2+}$ -ions have C.N. equal to 6. It is likely that C.N. > 6 for the relative small  $\text{Mg}^{2+}$  is not a very stable modification. The Al–D distances, in the range from 1.71(3) to 1.83(3) Å in  $\text{LiMgAlD}_6$ , are comparable with the Al–D distances found in all the other compounds. This is consistent with previous studies, which have concluded that the Al–H complex is very stable in these hydrides, and influenced to a very low degree by temperature or the particular cation. The similarity with  $\text{Li}_3\text{AlD}_6$ , noted above, reflects also in this table. A similar trigonal cell is only found for two of the compounds:  $\text{LiMgAlD}_6$  and  $\text{Li}_3\text{AlD}_6$ . The Al–Li distance in  $\text{LiMgAlD}_6$ , 2.74(3) Å, is fairly short compared to all the other compounds, except for  $\text{Li}_3\text{AlD}_6$  (2.91 Å). The volume per formula unit is also very similar in  $\text{LiMgAlD}_6$  and  $\text{Li}_3\text{AlD}_6$ . The two compounds have also very similar Li–D distances, varying from 1.89(4) to 1.92(3) Å in  $\text{LiMgAlD}_6$  and 1.89(1) to 1.95(1) Å for  $\text{Li}_3\text{AlD}_6$  [3]. The Mg–D distances in  $\text{LiMgAlD}_6$  vary from 1.91(2) to 1.97(2) Å, somewhat longer than what was found in  $\text{Mg}(\text{AlH}_4)_2$  (1.833(7) Å) [8,9]. The shortest intermetallic distance 2.74(3) Å is between Li and the Al in the center of the alanate which is bi-coordinated (sharing edge) to Li. The shortest Mg–Al distance 2.85(1) Å is between Mg and the alanate bi-coordinated to Mg. The shortest Al to Al distance is 4.38 Å.

#### 4. Conclusions

The structure of  $\text{LiMgAlD}_6$  has been solved and described. The PND and PXD data are in good agreement with the previously predicted theoretical structure [19]. The refined structure is in the trigonal space group  $P321$ , exhibiting hexagonal close packing of hydrogen atoms with cations occupying half of the octahedral sites. The structure consists of isolated  $\text{AlD}_6$ -octahedra linked together by Li- and Mg-atoms.

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