Crystal structure and physical properties of $^{243}$AmPd$_5$Al$_2$

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We report on the crystal structure, magnetic susceptibility, specific heat, electrical and thermophysical properties of AmPd$_5$Al$_2$, the americium counterpart of the unconventional superconductor NpPd$_5$Al$_2$. AmPd$_5$Al$_2$ crystallizes in the ZrNi$_2$Al$_5$-type of structure with lattice parameters: \(a = 4.1298\ \text{Å}\) and \(c = 14.7925\ \text{Å}\). Magnetic measurements of AmPd$_5$Al$_2$ indicate a paramagnetic behavior with no hint of magnetic ordering nor superconductivity down to 2 K. This aspect is directly related to its 5$f$ electronic configuration with \(J = 0\). The specific heat measurements confirm the non magnetic ground state of this compound. The low temperature electronic specific heat \(\gamma_c \sim 20\ \text{mJ\ mol}^{-1}\ \text{K}^{-2}\) is clearly enhanced as compared to americium metal. All transport measurements obtained point to a metallic behavior in AmPd$_5$Al$_2$.

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I. INTRODUCTION

The discovery of unconventional superconductivity in transuranium based intermetallics such as PuCo$_5$Ga$_5$[1] and PuRhGa$_5$[2] has led the scientific community to investigate the properties of numerous transuranium compounds, especially at low temperatures. NpPd$_5$Al$_2$[3] is the third transuranium and the first neptunium based superconductor discovered within the last few years. Despite the fact that superconducting properties of NpPd$_5$Al$_2$[3] are analogous to 4$f$ or 5$f$ well studied Ce- or U-based heavy fermion superconductors[4] there are still many open questions that have not been resolved such as the pairing mechanism and the symmetry of the order parameter. In the case of transuranium based superconductors several scenarios were proposed recently to account for their superconductivity but the situation is still unclear [see Ref. 1 8 9]. While NpPd$_5$Al$_2$ is a heavy-fermion superconductor, the isostructural ThPd$_5$Al$_2$[10] and UPd$_5$Al$_2$[11] exhibit a paramagnetic ground state and PuPd$_5$Al$_2$ orders antiferromagnetically below 5.6 K[10]. The rare earth (RE) based compounds of the same structure such as CePd$_5$Al$_2$[12] and LuPd$_5$Al$_2$[13] have also been examined and some of them present fascinating features such as pressure induced unconventional superconductivity[13].

To explore further the f-electron properties and the richness of behavior intrinsically related to this ZrNi$_2$Al$_5$ structure, we have considered to substitute another transuranium element, namely americium. Americium (Am$^{3+}$) presents a non magnetic ground state due to its $5f^6$ electronic configuration (\(J = 0\)). In the case of americium metal, this implies a multitude of interesting properties such as superconductivity[15] and an extreme sensitivity to external parameters like pressure[16] with a complex structural phase diagram[17]. It suggests then that an americium based compound in this structure should present interesting properties.

Here we report on the crystal structure and magnetic, transport and thermal properties of AmPd$_5$Al$_2$. It is worth to note the challenge and the difficulties when working with americium: it is a highly radioactive element and only one isotope allows low temperature studies ($^{243}$Am, $t_{1/2} = 7.38 \times 10^6$ years, $Q = 6.3\ \mu\text{W}\ \text{mg}^{-1}$). This isotope in metal form at quantity required for sample preparation (∼0.1 g scale) and physical property measurements is extremely rare. Such difficulties have generally prevented studies of americium based intermetals or compounds in the last decades especially considering electrical transport properties (resistivity, magnetoresistance, thermopower) and specific heat at low temperature. This study constitutes therefore a clear advance on trans-plutonium basic properties at low temperatures. This should be added to the few ones reported[18 19] and would help to understand the nature of the unconventional superconductivity in NpPd$_5$Al$_2$, especially the magnetic fluctuation scenario related to 5$f$-electrons.

II. EXPERIMENTAL

The polycrystalline samples were prepared by arc melting stoichiometric amounts of the pure metals components. Starting materials were used in the form of 4N Pd pellets, 5N Al wire as supplied by A. D. Mackay Inc. and 99.85 % Am metal[24]$^{243}$Am isotope[20] produced originally by thermo reduction process[21] with 3N purity. Other actinides (Np, Pu) are present at ppm level[22] in Am after long term storage (∼10 years). They do not contribute significantly to basic properties but decrease purity level with time. Due to the contamination risk generated by the radiotoxicity of americium, all operations of preparation and encapsulation have been performed in gloveboxes under inert N$_2$ atmosphere. Moreover the self radiation and the self damage induced by α disintegration constrain us to work very rapidly (within days). This aspect is even more drastic at low temperature (T $<$ 10 K). The arc melting was performed un-
der a high purity argon atmosphere on a water-cooled copper hearth, using a Zr alloy as an oxygen/nitrogen getter. The arc melted buttons were turned over and melted three times in order to ensure homogeneity. The weight losses after melting were smaller than 0.2 %. The magnetic properties were determined using a Quantum Design (QD) MPMS-7 device in the temperature range 2-300 K and in magnetic fields up to 7 T. The heat capacity, electrical resistivity and Hall effect were measured from 2.5 to 300 K with a QD PPMS-14 device up to 14 T on polycrystals. The thermoelectric power was measured from 3 to 300 K in a home-made setup using pure copper as a reference material. For transport properties, all measurements have been determined by a four probe DC technique voltage measurement on a polycrystal sample polished on 2 opposite faces to present a flat surface with typical size 1x0.4x0.2 mm³. Electrical resistivity measurement has been performed with the applied current $I = 5$ mA along the flat surface and the voltage $V$ was extracted parallel to the current $I$ while the applied magnetic field $B$ was perpendicular to the flat surface for magnetoresisitivity determination. When applying negative field at fixed temperature, no parasitic Hall effect was observed for magnetoresisitivity. The Hall resistance ($R_H$) was determined by voltage measurements $V_H$ under field alternatively at +14 and −14 T. Field response $V_H(B)$ at fixed temperatures has been measured to confirm results obtained when ramping in temperature. Excepted crystallographic structure determination, no orientation information related could be extracted from the basic properties observed.

III. CRYSTALLOGRAPHIC STRUCTURE

Small single crystals of typical size 50x50x20 µm³ suitable for crystal structure determination were mechanically extracted from the button and mounted inside a capillary for single crystal x-ray diffraction (Fig. 1 top). They were examined on an Enraf-Nonius CAD-4 diffractometer with the graphite monochromatized MoKα-radiation. The crystal structure was solved by the direct method using SHELX97[25] and the data processing using the WinGX package[26] are reported in Table I. The atomic coordinates obtained are presented in Table II. The crystal structure was refined from the single-crystal x-ray data and was corrected for Lorentz and polarization effects. The results confirm the tetragonal unit cell. Samples were then examined by powder x-ray diffraction using a Bruker D8 diffractometer with the monochromated CuKα₁ radiation ($\lambda = 1.54059$ Å) equipped with a Vantec detector. The powder diffraction pattern (Fig. 2) was analyzed by a Rietveld profile refinement method[23] using the WinplotR-Fullprof program[24]. Results are similar to those determined by x-ray single crystal diffraction confirming the structure, the crystallographic parameters and good homogeneity of the polycrystals. Finally, the phase composition was determined by energy dispersive

![FIG. 1: (Color online) X-ray single crystal diffraction pattern obtained for a single crystal for a random orientation and along the main directions a, b and c. Bottom, energy dispersive x-ray (EDX) analysis performed on a polycrystal showing the homogeneity of the material.](image)

![FIG. 2: (Color online) X-ray powder diffraction pattern ($\lambda = 1.54059$ Å) recorded for $^{243}$AmPd$_5$Al$_2$. The solid red line through experimental points is the Rietveld refinement profile calculated for tetragonal AmPd$_5$Al$_2$. The blue line corresponds to the difference between measured and calculated Rietveld refinement. The crystal structure with the ZrNi$_2$Al$_3$ unit cell and the crystallographic parameters $a = 4.1298(9)$ Å and $c = 14.792(4)$ Å are presented.](image)
x-ray (EDX) analysis performed on a Philips XL40 scanning electron microscope (SEM). The microprobe analysis indicates good homogeneity (Fig. 1 bottom) for the samples and a single phase with stoichiometry close to 1:5:1.70 (Am$_{0.13}$Pd$_{0.65}$Al$_{0.22}$). This slight deficiency in aluminium on the bulk could be explained by the difficulty to integrate the Al signal vs. Am. In conclusion, AmPd$_5$Al$_2$ adopts a tetragonal ZrNi$_2$Al$_5$-type of structure (s.g. $I4/mmm$) like NpPd$_5$Al$_2$ \cite{9} with lattice parameters $a = 4.1298(9)$ Å and $c = 14.792(4)$ Å.

### IV. MAGNETIC PROPERTIES

The temperature dependence of the magnetic susceptibility of polycrystalline AmPd$_5$Al$_2$ is presented in Fig. 3. The compound shows a paramagnetic behavior with almost no temperature dependence down to 50 K. Below, an upturn leads to a slightly enhanced magnetic susceptibility at low temperature with $\chi_{\Sigma}K \sim 3.5 \times 10^{-3}$ emu mol$^{-1}$. A similar but weaker behavior has been reported for the magnetic susceptibility of americium metal \cite{27} and is presented on the same figure for comparison. The field dependence of the magnetization ($\sigma$) taken at 5 K (see inset in Fig. 3) indicates a paramagnetic response. The non magnetic nature of AmPd$_5$Al$_2$ is expected from the ground state of americium (5f$^3$). In LS coupling the six 5f-electrons of Am$^{3+}$ will occupy the J = 0 state. In this case, due to the proximity of the first excited state J = 1 the susceptibility will mainly be governed by a Van Vleck term. On the other hand in jj model the six electrons will occupy $j$=5/2 sub-shell separated by spin-orbit coupling from $j$=7/2 sub-level. So, the susceptibility coming from a Pauli exchange reinforced term and an orbital contribution is rather anticipated. However, the exchange interactions are still present leading to intermediate coupling of the 5f-states. As has been shown in Am an intermediate coupling is very close to the jj limit so only a small 5f-electron occupation is observed in $j$=7/2 sub-band \cite{28, 29}. Therefore, the enhanced susceptibility values obtained for AmPd$_5$Al$_2$ compared to americium metal may come from a stronger Van Vleck contribution and/or Pauli paramagnetism. The presence of Pu and more specifically Np atoms created during the long term storage could be considered but can not explain quantitatively the behavior at low temperature. They can be present in the material, participating to a relative local disorder in the lattice without signatures on the XRD powder diffraction patterns or EDX measurements. No

| Atom | site | x   | y   | z   | U$_{eq}$ |
|------|------|-----|-----|-----|---------|
| Am   | 2a   | 0   | 0   | 0   | 0.0060  |
| Pd1  | 8g   | 0.5 | 0   | 0.1453(2) | 0.0069  |
| Pd2  | 2b   | 0.5 | 0.5 | 0   | 0.0056  |
| Al   | 4e   | 0   | 0   | 0.2545(20) | 0.0106  |

The structure was refined with anisotropic displacement parameters for all atoms. The last column contains the equivalent isotropic U values (Å$^2$).

**FIG. 3:** Temperature dependence of the magnetic susceptibility $\chi$ of $^{243}$AmPd$_5$Al$_2$ for $H = 70$ kOe. The magnetic susceptibility reported by Kanellakopoulos et al. \cite{27} for Am metal is also plotted on the graph. Inset: Magnetization $\sigma$ measured at 5 K for increasing and decreasing magnetic field $H$. The value is very small even at the highest applied magnetic field ($\sim 0.4$ emu g$^{-1}$).

**TABLE I: Crystallographic data for $^{243}$AmPd$_5$Al$_2$ obtained from a single crystal.**

| Composition          | $^{243}$AmPd$_5$Al$_2$ |
|----------------------|------------------------|
| Space Group          | $I4/mmm$ (No. 139)     |
| Lattice Parameters ( Å) | $a = 4.1298(9)$       |
|                      | $c = 14.792(4)$        |
| Cell Volume ( Å$^3$)  | 252.29                 |
| Formula units per cell | Z = 2                  |
| Formula Mass         | 829.07                |
| Calculated density (g/cm$^3$) | 10.91            |
| Crystal size (mm$^3$) | 0.03 x 0.20 x 0.05    |
| Radiation            | MoK$_\alpha$ ($\lambda = 0.71073$ Å) |
| Scans up to $\theta$ | 70 $^\circ$           |
| Linear absorption coefficient | 32.00 mm$^{-1}$     |
| Total number of reflections | 1008               |
| Reflections with $F_o > 4\sigma(F_o)$ | 154                   |
| Goodness of fit      | 1.134                 |
| Conventional residual R | 0.0772 (F > 4$\sigma$) |

**TABLE II: Crystallographic parameters for $^{243}$AmPd$_5$Al$_2$ obtained by single crystal x-ray diffraction.**

![Graph showing temperature dependence of magnetic susceptibility](image-url)
sign of superconductivity nor magnetic order has been observed down to 2 K in AmPd₅Al₂.

V. TRANSPORT PROPERTIES

A. Electrical resistivity

The electrical resistivity and magnetoresistance of polycrystalline samples of AmPd₅Al₂ are presented in Fig. 4. The overall behavior of the electrical resistivity of AmPd₅Al₂ is clearly metallic with a relatively high value of the residual resistivity ρ₀ and a low value of the residual resistivity ratio (RRR = 1.35). The electrical resistivity value is small and point to the absence of magnetic scattering phenomena such as Kondo or RKKY interactions. It is interesting to note the similarity of behavior for all 4f and 5f counterparts (RE,An)Pd₅Al₂ with electrical resistivity ρ₉₀₀Ｋ around 15–30 μΩ cm [3, 4, 12]. Only NpPd₅Al₂ presents a higher electrical resistivity at room temperature ~ 90 μΩ cm [3, 5].

Assuming the validity of Matthiessen rule the resistivity of a non-magnetic metallic compound should follow the Bloch-Gruneisen-Mott relation [31, 32]:

\[ \rho(T) = \rho_0 + 4R\Theta_R \left( \frac{T}{\Theta_R} \right)^5 \int_0^{\Theta_R} \frac{x^5 dx}{(e^x - 1)(1 - e^{-x})} - KT^3 \]  

(1)

where Θ_R is the Debye temperature obtained by resistivity and R is a constant, whereas the third term KT³ describes interband electron scattering processes on the s-d bands in the case of transition metal alloys and s-f bands in the case of f metals. The magnitude of the constant Κ and its sign depend on the density of states at the Fermi level. The adjustment of the electrical resistivity curve by a LSQ fit as shown in Fig. 4 according to Eq. (1) gives the following results: residual resistivity ρ₀ = 17.0 μΩ cm, phonon scattering term R = 2.372x10⁻² μΩ cm K⁻¹, Debye-temperature Θ_R = 209 K and scattering coefficient of the conduction electrons into a narrow d band near the Fermi level K = 2.978x10⁻⁸ μΩ cm K⁻³. This value of Debye temperature is roughly of the same order of magnitude as obtained by heat capacity (200–300 K) but slightly reduced. A second fit using the Debye temperature obtained by heat capacity measurements (Θ_R = 295 K, see IV) is presented on the same figure Fig. 4. The values are then ρ₀ = 17.1 μΩ cm, R = 2.50x10⁻² μΩ cm K⁻¹, and K = 5.18x10⁻⁸ μΩ cm K⁻³. This fit does not reproduce well the overall shape of electrical resistivity especially at low temperature. The discrepancy between the two fits can be explained by 2 origins.

On one side, the different phonons branches contributes differently to the 2 properties (heat capacity and electronic transport). The electrical conductivity (σ ~ 1/ρ) is predominantly limited by the interaction between conduction electrons and longitudinal phonons, whereas the low temperature specific heat is dominated by transverse phonons [37]. This is clearly reinforced in the case of anisotropic structures presenting highly selective modes.

On the other side, the RRR can be reduced by the disorder created by the ²⁴³Am decay. We suggest to consider an increased interaction at low temperature of some defects induced by self disintegration of ²⁴³Am atoms creating Frenkel pairs in the lattice. These defects appear quite fast despite the short delay between synthesis and measurement (within a week) and play a much stronger role than the presence of Np and Pu atoms created during long term storage that should not contribute significantly to the transport properties as for the magnetization. They could lead to an "impurity effect" with time, reducing the RRR as their effect is even more visible at low temperature when phonon modes are reduced. This phenomenon has been clearly observed in intermetallic systems especially in the case of superconductors [58].

We get the same value for the residual resistivity ρ₀ for both adjustments but slightly enhanced values for R and K constants for the Θ_R = 209 K case. The R constant is of the same order of magnitude than non magnetic ⁵f [33] or localized/mixed valence ⁴f intermetallics ~ 10⁻² μΩ cm K⁻¹ such as YbTi₅ (T=Rh, Ir) [42] and YbAl₅ [41] but K constant is clearly reduced by one or two orders of magnitude (10⁻⁸ μΩ cm K⁻³ for AmPd₅Al₂) in comparison to UTGa₅(T=Co, Rh) for instance (~ 10⁻⁶ μΩ cm K⁻³ for URhGa₅) [40]. As the value of K as well as its sign depend on the density of states at the Fermi level, this illustrates the localized aspect of the material and the absence of ⁵f-electrons at the Fermi level.

The inset in Fig. 4 presents the magnetoresistivity...
(MR) data $\Delta \rho/\rho \times 1000$ taken at 2.5 and 100 K, respectively. As may be seen from the figure we observe, in AmPd$_5$Al$_2$, an extremely small positive magnetoresistivity ($\sim 1 \%$). At 2.5 K the field dependence of the resistivity may be well described by a power law behavior $B^{3/2}$ (see the solid line in the inset of Fig. 3a). It is slightly different from $\rho(B)$ observed in pure simple metals where resistivity grows quadratically with field $[32]$. This can be related to the large anisotropy of the crystallographic structure as reported for other counterparts of the 1:5:2 family $[5, 13]$ and by the "impurity effect" $[32]$. This impurity effect is noticeable on magnetotransport because all other contributions are negligible: the 5f-electrons are localized and are not participating to the bonding. The small positive values of $MR$ points to closed 5f shell characteristics. Only remaining $s$,$p$,$d$ electrons can interact and lead to a scattering effect noticeable on the metallic-like shape of the material and on the weak magnetotransport properties. To estimate the electronic carriers qualitatively and quantitatively we have performed Hall and Seebeck effect measurements.

B. Hall and Seebeck effects

The temperature dependence of the Hall coefficient $R_H$ measured in a magnetic field of 14 T is shown in Fig. 5. $R_H$ is positive up to 70 K where it changes sign to negative. Unlike simple metals, $R_H$ of AmPd$_5$Al$_2$ is strongly temperature dependent. This could suggest, in AmPd$_5$Al$_2$ the presence of a complex electronic structure with multiple electron and hole bands with different temperature variations of carrier concentrations and mobilities. The higher value of Hall effect at low temperatures could come from the reduction of the carrier mobility when going down to 0 K. Above 100 K the $R_H$ is of the order of $-2 \times 10^{-10}$ m$^2$ C$^{-1}$, which corresponds in a one band model to the effective electron concentration $n_{Hall} \sim 3.2 \times 10^{22}$ cm$^{-3}$. It agrees well with the thermoelectric data (see below).

The inset of Fig. 5 shows the temperature dependence of the thermoelectric power of AmPd$_5$Al$_2$. It is worth noting that, up to our best knowledge, it is the first measurement of the Seebeck coefficient of a Am-based system. The thermopower is negative in the whole temperature range suggesting the dominant role of electrons in the electrical and heat conduction. The low-temperature dependence of the thermoelectric power $S(T)$ shows a clear extremum at $T_{ext} \approx 45$ K. Then, with decreasing temperature $S$ decreases down to zero when $T\rightarrow 0$ K, as expected. This extremum of the thermopower can be related to the phonon drag effect, where the position is a measure of the Debye temperature $[\Theta_D \approx T_{ext}/5$ (see Ref $[35]$), which we estimate for AmPd$_5$Al$_2$ to be of about 230 K. It is of the same order as $\Theta_D$ values estimated from the electrical resistivity and specific heat measurements (see below). Above 150 K the thermopower is roughly proportional to the temperature, hence indicating that the main contribution to $S(T)$ comes from diffusion of the carriers due to the applied temperature gradient. This mechanism is generally expressed as $[35]$.

$$S(T) = \frac{k_B^2 \pi^2 T}{3eE_F}. \quad (2)$$

Within a single-band model the value of the thermoelectric power measured at room temperature (-1.1 $\mu$V K$^{-1}$) implies the Fermi energy and effective carrier concentrations to be about $E_F = 6.5$ eV and $n_{Seebeck} \sim 7.5 \times 10^{22}$ cm$^{-3}$, respectively. The value of $n$ is very close to the one obtained from the Hall effect measurements and is also similar to $n$ observed in usual metals such as Al or Cu $[32, 33]$. However, taking into account the simplicity of the model applied, the estimated carrier concentration could account for the upper limit of carrier concentration in AmPd$_5$Al$_2$. Altogether, it strongly suggests the absence of 5f-electrons in the electronic properties: only $s$-$p$-$d$ carriers participate to the bonding while 5f-electrons are well localized in AmPd$_5$Al$_2$.

VI. HEAT CAPACITY

The specific heat measurements of AmPd$_5$Al$_2$ are presented on Fig. 6. The absence of magnetic order and/or superconductivity in this system down to 3 K is clearly confirmed. Near room temperature $C_p$ has a value of about 180 J mol$^{-1}$K$^{-1}$ which is slightly lower than the Dulong-Petit limit, i.e., $C_p = 3rR = 199$ J/mol K, where $r$ is the number of atoms per molecule and $R$ is the gas constant. The low temperature part of the specific heat allows to determine the electronic contribution $\gamma_e$ and
Debye temperature $\theta_D$ by a linear fit of $C_p/T \sim \gamma_e + \beta T^2$, with $\beta$ related to $\theta_D$ (see inset of Fig. 6). For AmPd$_3$Al$_2$ we get $\gamma_e = 19$ mJ mol$^{-1}$ K$^{-2}$ and $\theta_D = 295$ K. The value of $\theta_D$ is very close to the other non magnetic counterparts of the family, $\theta_D = 290$ K for ThPd$_3$Al$_2$ and 310 K for LuPd$_3$Al$_2$. The value of $\gamma_e$ obtained is relatively enhanced especially when compared to americium metal$^{[34]}$ or to other classical metals. However this value is much smaller than all other parent compound of the ZrNi$_2$Al$_3$ structure (325 mJ mol$^{-1}$ K$^{-2}$ for NpPd$_3$Al$_2^{[6]}$ and 61 mJ mol$^{-1}$ K$^{-2}$ for PuPd$_3$Al$_2^{[10]}$) and is rather close to the one for ThPd$_3$Al$_2$ (4.4 mJ mol$^{-1}$ K$^{-2}$) as this latter one does not present any $5f$ contribution. It suggests the presence of well localized $5f$-electrons in AmPd$_3$Al$_2$. The slight enhancement of $\gamma_e$ could come from the $5d$-$6d$ exchange correlations between Pd and Am atoms. However, further measurements using synchrotron techniques as magnetic x-ray circular dichroism are needed to conclude on this point.

VII. DISCUSSION AND CONCLUSION

In conclusion, the Am-based intermetallic compound AmPd$_3$Al$_2$ crystallizes, like other members of the AnPd$_3$Al$_2$ family in the tetragonal ZrNi$_2$Al$_3$-type of structure (s.g. $I4/mmm$ with lattice parameters $a = 4.1298(9)$ Å and $c = 14.793(4)$ Å, as determined by single crystal studies. The magnetic measurements revealed that AmPd$_3$Al$_2$ shows a temperature independent paramagnetic behavior, enhanced compared to Am metal. In agreement with the electronic configuration of americium, it does not show any hint of a magnetic nor superconducting signature down to 2 K. The non magnetic ground state, governed by the J=0 configuration, is also supported by specific heat measurements. The electrical resistivity, Hall effect and thermoelectric power are characteristic of a good metallic system with the carrier concentration of the order of $10^{22}$ cm$^{-3}$. This clearly point to the presence in AmPd$_3$Al$_2$ of well localized $5f$-electrons. Moreover, the absence of superconductivity in this system strongly emphasizes the importance of magnetic interactions as a possible medium of the unconventional superconductivity in NpPd$_3$Al$_2$. Electronic structure calculations would be of great interest to determine the position of $5f$ states versus Fermi energy in AmPd$_3$Al$_2$ and compare it to the other members of the (An,RE)Pd$_3$Al$_2$ family.

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