

Synthesis and characterization of a novel 3-amino-1,2,4-triazole lead(II) coordination polymer

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Abstract: A novel $[\text{Pb}_2(\mu\text{-atrz})_2(\mu\text{-CH}_3\text{COO})(\text{CH}_3\text{COO})]_n$ (atrz = 3-amino-1,2,4-triazole) complex was synthesized and characterized by IR spectroscopy, thermogravimetric analysis, and single crystal X-ray diffraction. The molecular structure shows that the complex is a coordination polymer in which there are 2 different lead(II) ions with coordination number of 5, Pb(2), and, with a coordination number of 4, Pb(1). Each atrz acts as a bridging ligand between 2 Pb(II) ions through the 2 adjacent nitrogen atoms of the triazole ring. The acetate anions show different types of coordination mode: one acts as a bridge by coordination of the same oxygen atom with Pb(II) ions and the other as a monodentate coordination by one of the oxygen atoms with Pb(2).

Key words: 3-Amino-1,2,4-triazole, 1,2,4-triazole, acetate, lead(II) complexes, coordination polymer

1. Introduction

The synthesis of lead(II)-organic coordination polymers has attracted great interest due to their unusual structures^{1–10} and some interesting physical properties such as luminescence,^{11–14} nonlinear optics,^{15,16} and ion exchange.¹⁷ It is well known that several factors, including the nature of ligands, coordination number and geometry of the metal ions, temperatures, pH, solvents, etc. might be very important to form the final structures of coordination polymers.¹⁸ Among these factors mentioned above, coordination modes and geometries of ligands and metal ions are the most important ones to build polymeric structures with different dimensions.¹⁹ Various ligands have been employed to design and synthesize Pb(II) complexes. The carboxylate ligands with their diverse coordination modes have been extensively used to form many lead(II) supramolecular compounds.²⁰ N-donor ligands such as bipyridines^{11,21–26} and 1,10-phenanthroline derivatives^{27–32} are widely used in the construction of lead(II) coordination polymers. 1,2,4-Triazole (trz) and its derivatives have attracted great interest in coordination chemistry because of the position of the donor atoms in the 5-membered ring. They can coordinate as bridging ligands between metal ions. This bridging capacity (N1,N2 or N2,N4) enables them to play a very important role in creating coordination polymers and metal complexes in various geometries in the solid state.^{33,34} Lead(II) ion is capable of forming flexible variable coordination numbers from 2 to 10 due to the presence of its $6s^2$ outer electron configuration with large ionic radius. This flexibility in the coordination numbers influences the coordination geometry of lead(II) to be holodirected (the bonds to the ligand atoms are

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directed throughout the surrounding sphere) and hemidirected (the bonds to the ligand atoms are directed to one part of the coordination sphere) coordination.³⁵

In this paper, we report the preparation, characterization, and crystal structure of a new lead(II) coordination polymer with 3-amino-1,2,4-triazole (atrz) ligand and acetate anions.

2. Results and discussion

The synthesis of a mixed-ligand lead(II) complex with atrz and 2 different anions, acetate and nitrite, was intended. However, the reaction between atrz, mixtures of lead(II) acetate, and sodium nitrite in 3:1:1 molar ratio, respectively, gave a mixed-anion lead(II) complex with atrz and acetate anions, $[\text{Pb}_2(\mu\text{-atrz})_2(\mu\text{-CH}_3\text{COO})(\text{CH}_3\text{COO})]_n$. The complex is air stable and does not melt, but decomposes at 106 °C. It is moderately soluble in DMSO but highly soluble in water.

The IR spectrum of $[\text{Pb}_2(\mu\text{-atrz})_2(\mu\text{-CH}_3\text{COO})(\text{CH}_3\text{COO})]_n$ shows characteristic absorption bands for atrz and acetate anions. The weak band at around 3137 cm^{-1} is assigned to the $\nu(\text{C-H})$ mode of the aromatic atrz rings in the complex. The stretching vibrations of $\text{C}=\text{N}$ of atrz as a very strong band are observed at 1627 cm^{-1} and are shifted to a higher frequency compared with the free ligand (1594 cm^{-1}). The strong absorption band observed at 3356 cm^{-1} can be attributed to the $\nu_{\text{asym}}(\text{N-H})$ of the NH_2 groups in the complex. The weak bands at 3294 and 3186 cm^{-1} are assigned to the symmetric stretching of the NH_2 group of atrz. A series of bands in the range of $1200\text{--}970\text{ cm}^{-1}$ are proposed for the rocking or twisting vibrational modes of the NH_2 groups in the complex.³⁶ The relatively weak band at 2955 cm^{-1} corresponds to C-H vibrational mode of the acetate anion. Four strong bands at 1549 and 1526 cm^{-1} for the $\nu_{\text{asym}}(\text{COO})$ and 1497 and 1434 cm^{-1} for the $\nu_{\text{sym}}(\text{COO})$ are assigned to the stretching vibrations of the acetate anions. Two new bands that appeared at 443 and 470 cm^{-1} in the complex were attributed to the Pb-N vibrations and eventually to be evidence of coordination of the atrz with the lead atom.

The thermal decomposition behavior of the complex was investigated between 30 and 700 °C in flowing nitrogen by thermogravimetric analysis (TGA). The TGA curve of $[\text{Pb}_2(\mu\text{-atrz})_2(\mu\text{-CH}_3\text{COO})(\text{CH}_3\text{COO})]_n$ shows 3 exothermic processes at temperatures higher than 110 °C. Heating from 110 to 160 °C led to the loss of an atrz molecule and the observed mass loss was 10.81% (calcd. value: 12.04%). Decomposition of the other atrz and one acetate anion took place between 160 and 343 °C and the experimental weight loss of 20.92% is consistent with the calculated value of 20.49%. The mass loss from 343 to 500 °C was 11.72% (calcd. value: 8.45) and attributed to the elimination of the other acetate anion. Finally, the initial lead(II) complex oxidized to PbO at around 500 °C.

The molecular structure of the complex was determined by X-ray crystallographic study. The ORTEP view of the complex is shown in Figure 1 and the crystallographic data are summarized in the Table.³⁷ The lead(II) complex crystallizes in a monoclinic system with $Z = 4$ in space group $\text{P}2_1/\text{n}$. The structure determination of the $[\text{Pb}_2(\mu\text{-atrz})_2(\mu\text{-CH}_3\text{COO})(\text{CH}_3\text{COO})]_n$ showed the complex in the solid state to be a one-dimensional coordination polymer (Figure 2). The triazole anion acts as a 3-donor ligand and connects to 3 Pb(II) ions in $[\text{Pb}_2(\mu\text{-atrz})_2(\mu\text{-CH}_3\text{COO})(\text{CH}_3\text{COO})]_n$ (Scheme 1a). The triazole ligands are nearly planar; the torsion angle is -2.38° for $\text{N}6\text{N}7\text{C}4\text{N}8$ and 0.26° for $\text{C}1\text{N}1\text{C}2\text{N}2$. The acetate anions in this compound are coordinated and one acts as bridging manner where one of its oxygen atoms coordinate to Pb(2) and Pb(1) atoms while the other acts in terminal coordination with Pb(2) (Scheme 1b).

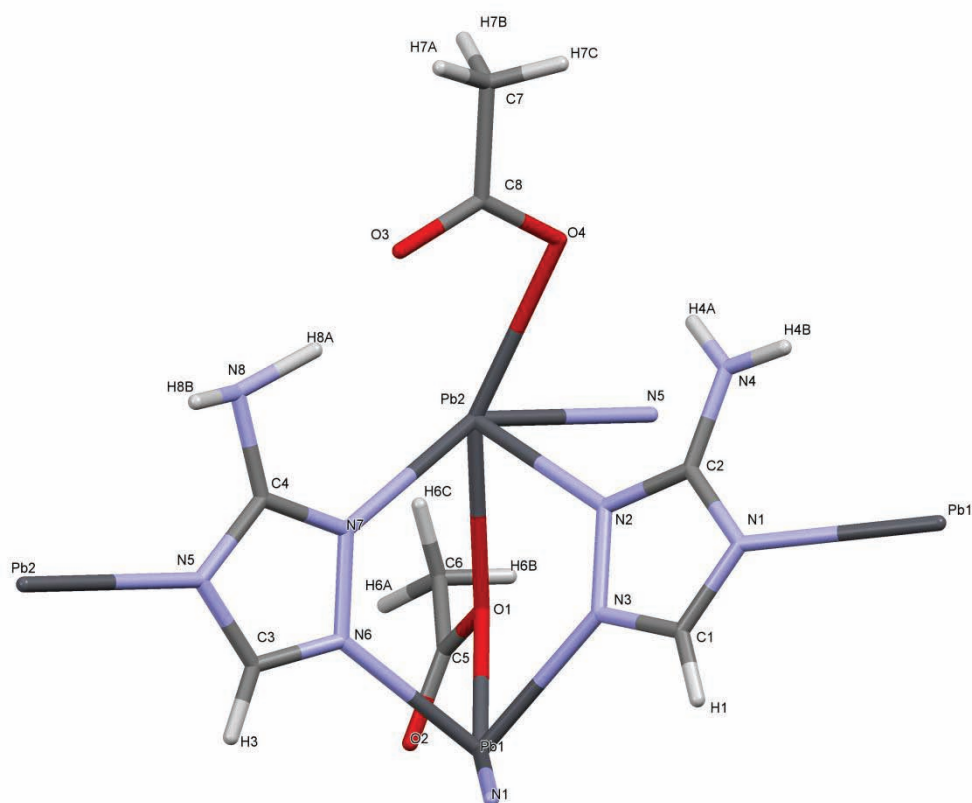


Figure 1. Drawing of $[\text{Pb}_2(\mu\text{-atrz})_2(\mu\text{-CH}_3\text{COO})(\text{CH}_3\text{COO})]_n$, with the atom numbering scheme; selected bond lengths (Å) and angles ($^\circ$) for complex: Pb1–O1 2.473(9), Pb1–N3 2.426(8), Pb1–N6 2.3512(9), Pb1–N1 2.603(8), Pb2–O1 2.5272(7), Pb2–O4 2.597(7), Pb2–N7 2.711(9), Pb2–N2 2.433(9), Pb2–N5 2.608(9), N6–N7 1.391(1), N6–C3 1.3161(1), O4–C8 1.261(1), C2–N4 1.363(1), C8–C7 1.493(1), N6–Pb1–O1 80.0(3), N6–Pb1–N3 85.3(3), O1–Pb1–N3 75.1(3), O4–Pb2–O1 153.7(3), O4–Pb2–N7 119.6(3), O4–Pb2–N2 86.5(3), O1–Pb2–N7 75.2(3), O1–Pb2–N2 75.1(3), N7–Pb2–N2 78.2(3), Pb1–N6–N7 123.2(7), Pb1–N6–C3 130.2(7), Pb2–O4–C8 99.9(7), Pb1–O1–Pb2 116.3(3), O3–C8–O4 124.0(1), O1–C5–O2 121.1(1).

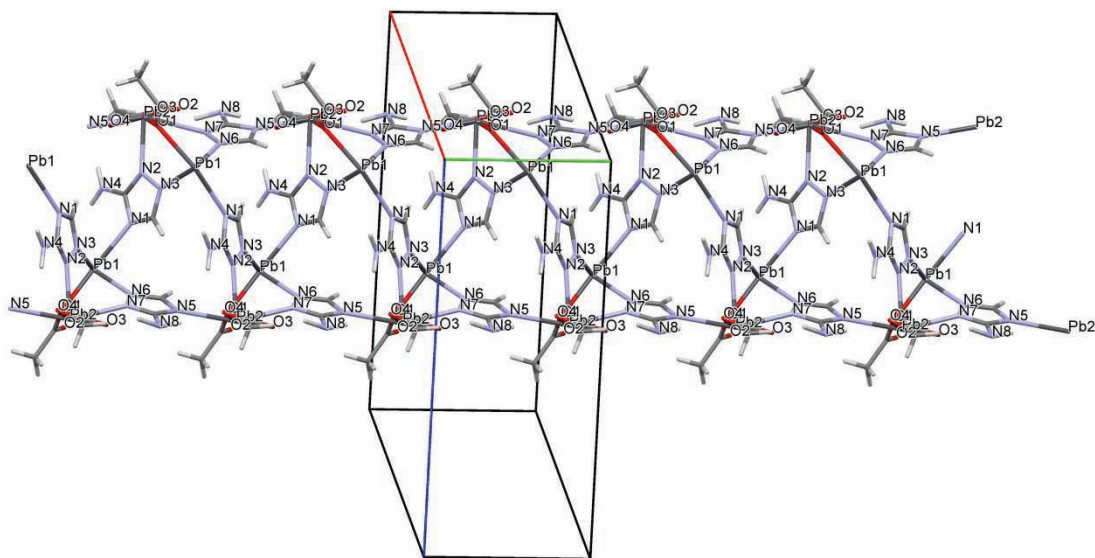
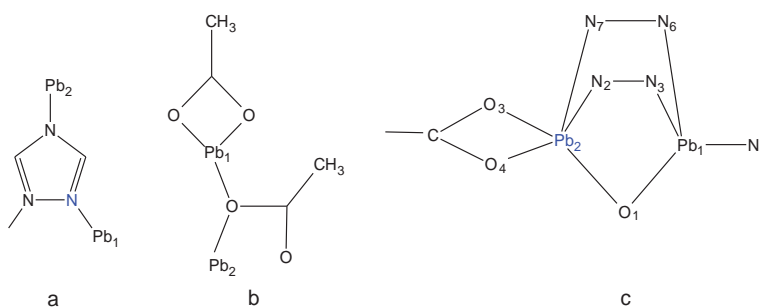


Figure 2. The 1-D polymeric network of $[\text{Pb}_2(\mu\text{-atrz})_2(\mu\text{-CH}_3\text{COO})(\text{CH}_3\text{COO})]_n$.

Table. Crystal data and structure refinement parameters for $[\text{Pb}_2(\mu\text{-atrz})_2(\mu\text{-CH}_3\text{COO})(\text{CH}_3\text{COO})]_n$.

Empirical formula	$\text{C}_8\text{H}_{12}\text{N}_8\text{O}_4\text{Pb}_2$
Formula weight	698.6
Temperature	100 (2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, P 21/n
Unit cell dimensions	$a = 14.1364 (6) \text{ \AA}$ $\alpha = 90^\circ$
	$b = 7.3307 (3) \text{ \AA}$ $\beta = 104.812 (2)^\circ$
	$c = 17.5364 (7) \text{ \AA}$ $\gamma = 90^\circ$
Volume	$1756.90 (13) \text{ \AA}^3$
Z	4
Density (calculated)	2.702 Mg/cm^3
Absorption coefficient	19.17 mm^{-1}
F(000)	1280
Theta range for data collection	1.7 to 28.6°
Index ranges	$-18 \leq h \leq 18, -9 \leq k \leq 8, -22 \leq l \leq 23$
Reflections collected	4374
Independent reflections	3902 [R(int) = 0.072]
Refinement method	Full-matrix least-squares on F^2
Function minimized	$\sum w(F_o^2 - F_c^2)^2$
Data/restraints/parameters	4374/0/185
Goodness-of-fit on F^2	1.06
Final R indices [$I > 2\sigma(I)$]	R1 = 0.0476, wR2 = 0.1326

**Scheme 1.** Schematic representation of coordination type of triazole, acetate, and 2 different Pb(II) in the $[\text{Pb}_2(\mu\text{-atrz})_2(\mu\text{-CH}_3\text{COO})(\text{CH}_3\text{COO})]_n$.

There are 2 types of Pb atoms with a coordination number of 5, $\text{Pb}(2)\text{O}_2\text{N}_3$, and with a coordination number of 4, $\text{Pb}(1)\text{ON}_3$. $\text{Pb}(2)$ is coordinated to 2 oxygen atoms of the acetate anions and 3 nitrogen atoms of triazole ligands. $\text{Pb}(1)$ is tetrahedrally coordinated by 3 nitrogen atoms from 3 different triazole ligands, and 1 oxygen atom from bridging acetate anion (Scheme 1c). The $\text{Pb}(1)\text{-O}$ distance is $2.473(9) \text{ \AA}$, while the $\text{Pb}(2)\text{-O}$ distances range from $2.527(7)$ to $2.597(7) \text{ \AA}$. In the core, the $\text{Pb}(1)\text{-N}$ distances are $2.3512(9)$ and $2.603(8) \text{ \AA}$, while the $\text{Pb}(2)\text{-N}$ distances are $2.433(9)$ and $2.711(9) \text{ \AA}$. The OCO bond angles of the carboxylate groups are $121(1)^\circ$ and $124(1)^\circ$, respectively, which are very similar to previously reported values.³⁸ The distance of $\text{Pb}(1)\text{-Pb}(2)$ is $4.247(9) \text{ \AA}$. The $\text{Pb(II)-N}_{\text{triazole}}$ bond length values are very close to those of a previously reported similar type coordination of $\text{Pb(II)-N}_{\text{triazole}}$ complex.⁵ From analysis of the bond angles

and the atom positions in the coordination core, the geometries around lead(II) atoms in $[\text{Pb}_2(\mu\text{-atrz})_2(\mu\text{-CH}_3\text{COO})(\text{CH}_3\text{COO})]_n$ can be described as hemidirected.³⁵

The molecular structure features 3 intramolecular hydrogen bonds: $\text{N8-H8B}\cdots\text{N5} = 2.357(12)$ Å with an $\text{N8}\cdots\text{N5}$ distance of $2.395(3)$ Å, $\text{N8-H8A}\cdots\text{O3} = 1.757(9)$ Å with an $\text{N8}\cdots\text{O3}$ distance of $2.837(9)$ Å, and $\text{N4-H4A}\cdots\text{O4} = 2.057(9)$ Å with an $\text{N4}\cdots\text{O4}$ distance of $2.871(15)$ Å. As shown in Figure 3, the one-dimensional layered structure of molecules is formed through 2 types of intermolecular hydrogen bonds: $\text{N8-H8B}\cdots\text{O4} = 2.874(12)$ Å, $\text{N8}\cdots\text{O4} = 2.013(17)$ Å, $\text{N8-H8B}\cdots\text{O4} = 174.6(3)^\circ$, $x, +y+1, +z$; $\text{N4-H4B}\cdots\text{O2} = 2.104(7)$ Å, $\text{N4}\cdots\text{O2} = 2.894(12)$ Å, $\text{N4-H4B}\cdots\text{O2} = 149.1(7)^\circ$, $x+1/2, -y+1/2, +z+1/2$. The $\text{Pb}\cdots\text{Pb}$ distances between the layers are 12.968 Å and 7.331 Å.

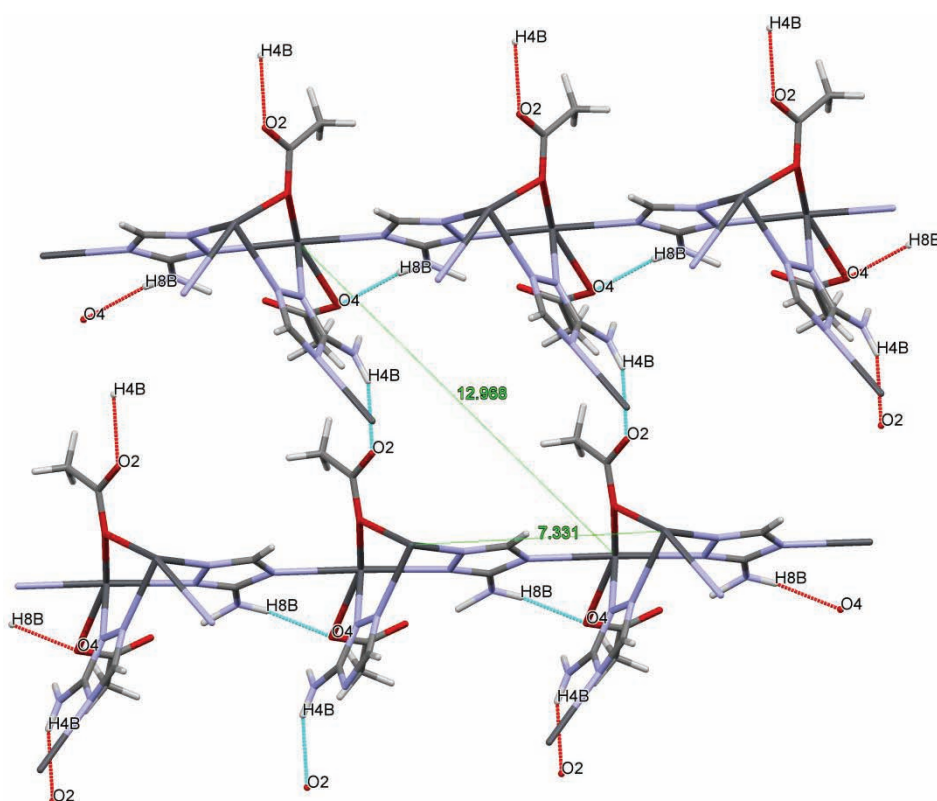


Figure 3. 1-D framework of $[\text{Pb}_2(\mu\text{-atrz})_2(\mu\text{-CH}_3\text{COO})(\text{CH}_3\text{COO})]_n$ through intermolecular hydrogen bonding along a direction.

3. Conclusions

We report the synthesis and characterization of a new lead(II) carboxylate complex with 3-amino-1,2,4-triazole. Lead(II) ion is capable of forming various structures due to its radius and extensive coordination environment. The binding ability of triazoles with 3 nitrogen donor atoms is noteworthy. Moreover, the ability of the carboxylate groups to form chelating and bridging coordination modes make them remarkable candidates to create variable coordination networks. In this work, the lead(II) compound shows a one-dimensional polymeric structure, which indicates that the 3-amino-1,2,4-triazole plays an important role in the formation of coordination polymer.

4. Experimental

4.1. General procedures

3-Amino-1,2,4-triazole and lead(II) acetate trihydrate were purchased from Sigma-Aldrich Co.; sodium nitrite was purchased from Carlo Erba. All chemicals and reagents were used as received without any purification.

IR spectra were recorded as KBr pellets using a PerkinElmer LR 64912C in the frequency range 4000 – 400 cm^{-1} . Thermogravimetric analysis was performed on a PerkinElmer Diamond TG/DTA by heating the sample at a rate of 20 °C/min under flowing nitrogen. Melting point was measured on an Electrothermal 9100 apparatus and was uncorrected.

4.2. X-ray crystallography

Diffraction data for the complex were collected with a Bruker AXS APEX CCD diffractometer equipped with a rotation anode at 296(2) K using graphite monochromated Mo $K\alpha$ radiation at $\lambda = 0.71073$ Å. Diffraction data were collected over the full sphere and were corrected for absorption. The data reduction was performed with the Bruker SMART³⁹ software package. For further crystal and data collection details, see the Table. Structure solution was carried out with the SHELXS-97⁴⁰ package using the direct methods and was refined SHELXL-97⁴¹ against F^2 using first isotropic and later anisotropic thermal parameters for all nonhydrogen atoms. Hydrogen atoms were added to the structure model at calculated positions. Geometric calculations were performed with PLATON.⁴²

4.3. Preparation of $[\text{Pb}_2(\mu\text{-atrz})_2(\mu\text{-CH}_3\text{COO})(\text{CH}_3\text{COO})]_n$

$\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$ (379 mg, 1 mmol) and sodium nitrite (69 mg, 1 mmol) were mixed as solids, and methanol (3 mL) was added. After dissolving both solids leaving a yellow solution over an ultrasonic bath in a few minutes, a solution of 3-amino-1,2,4-triazole (252 mg, 3 mmol) in methanol (7 mL) was added dropwise to the above-mentioned solution. The resulting mixture allowed to stand for 3–4 days for crystallization at room temperature. The yellow crystals were filtered off, washed with cold methanol and then diethyl ether, and dried in a vacuum desiccator. Yield: 260 mg, 37%. Decomp. at 106 °C. IR (cm^{-1}) selected bands: 443 *vs*, 470 *m*, 665 *vs*, 837 *s*, 1064 *s*, 1177 *m*, 1268 *s*, 1434 *vs*, 1497 *ms*, 1526 *s*, 1549 *ms*, 1627 *vs*, 2955 *w*, 3137 *w*, 3186 *w*, 3294 *w*, 3356 *s*.

Supplementary data: Crystallographic data can be obtained from the Cambridge Crystallographic Data Center, by quoting the reference number CCDC-928602. The data can be obtained free of charge at www.ccdc.cam.ac.uk/data_request/cif.

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