

Syntheses and Characterization of Mixed-Ligands Lead(II) Complexes, $[\text{Pb}(\text{bpy})(\text{CH}_3\text{COO})\text{X}]$ ($\text{X} = \text{I}^-$, NO_3^- , and ClO_4^-), Crystal Structure of $[\text{Pb}(\text{bpy})(\text{NO}_3)(\text{CH}_3\text{COO})]_n$ (A New 1-D Polymeric Compound)

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Lead(II) complexes with 2,2'-bipyridine (bpy) containing two different anions, $[\text{Pb}(\text{bpy})(\text{CH}_3\text{COO})\text{X}]$ ($\text{X} = \text{I}^-$, NO_3^- , and ClO_4^-), have been synthesized and characterized by CHN elemental analysis, IR-, ^1H NMR- and ^{13}C NMR spectroscopy. The structure of $[\text{Pb}(\text{bpy})(\text{ClO}_4)(\text{CH}_3\text{COO})]_n$ was confirmed by X-ray crystallography. The complex is a one-dimensional polymer as a result of perchlorate ligand bridging. The Pb atom has an unsymmetrical eight-coordinate geometry. The arrangement of the bpy, acetate and nitrate ligands leaves a coordination gap at the Pb(II) ion, occupied probably by a stereo-active lone pair of electrons. There is a π - π stacking interaction between the parallel aromatic rings that may be formed by influence lone pair activity.

Key words: Lead(II) Complexes, Crystal Structure, Mixed-Anion Complexes,
2,2'-Bipyridine Ligand

Introduction

The coordination chemistry of lead(II) with N and O-donor ligands has been investigated in the past decade and frequently discussed in regard to the coordination and stereoactivity of the valence shell lone electron pairs [1–5]. Extensive recent structural studies of lead(II) compounds [6–7] in particular have provided a basis for rather detailed analysis of the evidence for coordination sphere distortions which may be a consequence of the presence of such electron pairs. It appears that in complexes of lead(II) {and probably in those of related species such as Tl(I) [8–10] and Bi(III) [11–15]}, the nature and form of the coordination sphere is generally determined by a number of factors, including lone pair-bond pair repulsions, so that seemingly minor differences in ligands can have quite marked effects upon the coordination stereochemistry. Since the presence of a lone pair is not directly detected but inferred on the basis of the spatial distribution of donor atoms surrounding the metal, the identification of these donor atoms is fundamental to the analysis of any particular system. Interestingly, this alone is not a straightforward process. An interesting

point is that crystal packing may affect the size and extent of the lone-pair in the coordination sphere. Building new molecular species and modifying their architectures in order to control their physical properties, has been a topic for many research groups. Mixed ligand complexes such as $\text{Pb}(\text{bpy})(\text{CH}_3\text{COO})\text{X}$ ($\text{X} = \text{I}^-$, NO_3^- , and ClO_4^-), with differences only in one part of the coordination sphere are particularly interesting.

Recent reports of the crystal structure of the 1:1 adducts $[\text{Pb}(\text{phen})(\text{O}_2\text{CCH}_3)(\text{O}_2\text{ClO}_2)]$ [16], $[\text{Pb}(\text{phen})(\text{O}_2\text{CCH}_3)(\text{O}_2\text{NO})]$ [17], $[\text{Pb}(\text{phen})(\text{O}_2\text{CCH}_3)(\text{NCS})]$ [18] and the 1:2 adducts of $[\text{Pb}(\text{phen})_2(\text{CH}_3\text{COO})\text{X}]$ ($\text{X} = \text{NCS}^-$, NO_3^- and ClO_4^-) [19] described the presence of bridging acetate and its influence upon the coordination stereochemistry of the lead(II) ion and also on their structures. In the present paper, we report the synthesis and characterization of some new 1:1 adducts of $\text{Pb}(\text{bpy})(\text{CH}_3\text{COO})\text{X}$ ($\text{X} = \text{I}^-$, NO_3^- , and ClO_4^-).

Experimental Section

Physical measurements

IR spectra were recorded as nujol mulls using Perkin-Elmer 597 and Nicolet 510P spectrophotometers. Micro-

analyses were carried out using a Heraeus CHN-O- Rapid analyzer. Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. The ^{207}Pb solution NMR spectra were recorded on a Bruker DRX-500 AVANCE spectrometer at 104.6 MHz using a 5-mm broad-band probe. Lead chemical shifts are reported (ppm) downfield from tetramethyllead using PbPh_4 ($\delta_{\text{Pb}} = -178.0$ ppm, saturated in CDCl_3) as an external standard for 0.01 M solution and of 25 °C. Each lead spectrum was acquired in 3–10 hours.

Preparation of $[\text{Pb}(\text{bpy})(\text{NO}_3)(\text{CH}_3\text{COO})]$

2,2'-Bipyridine (0.156 g, 1 mmol) was placed in one arm of a branched tube and a mixture of lead(II) acetate (0.36 g, 1 mmol) and sodium nitrate (0.085 g, 1 mmol) in the other. Methanol was carefully added to fill both arms, then the tube was sealed and the ligand-containing arm immersed in a bath at 60 °C, while the other was at ambient temperature. After 8 d, white crystals, (m.p. 225 °C) had deposited in the cooler arm. Yield: 0.291 g, 60%. $\text{C}_{12}\text{H}_{11}\text{N}_3\text{O}_5\text{Pb}$: calcd. C 29.72, H 2.27, N 8.66; found C 29.40, H 2.44, N 8.90. – IR (film) selected bands: $\nu = 720(\text{s})$, $850(\text{s})$, $1380(\text{vs})$, $1590(\text{s})$, $1648(\text{s})$, $2980(\text{w})$, $3140(\text{w}) \text{ cm}^{-1}$. – ^1H NMR (DMSO): $\delta = 1.60$ (s, 3H), 7.80 (t, 2H), 8.20 (q, 2H), 8.70 (d, 2H), and 7.78 (d, 2H). – $^{13}\text{C}\{^1\text{H}\}$ NMR (DMSO): $\delta = 27.20$ ($^{13}\text{CH}_3\text{-COO}$), 178.58 ($\text{CH}_3\text{-}^{13}\text{COO}$), 117.50, 124.20, 127.62, 1141.65, 149.96, and 150.62. ^{207}Pb NMR (DMSO): $\delta = -1337.06$ ppm.

Preparation of $[\text{Pb}(\text{bpy})(\text{ClO}_4)(\text{CH}_3\text{COO})]$

2,2'-Bipyridine (0.156 g, 1 mmol) as described above, with lead(II) acetate (0.36 g, 1 mmol) and sodium perchlorate (0.123 g, 1 mmol). White crystals (m.p. 235 °C) deposited. Yield: 0.260 g, 50%. $\text{C}_{12}\text{H}_{11}\text{ClN}_2\text{O}_6\text{Pb}$: calcd. C 27.61, H 2.20, N 5.37; found C 27.30, H 2.50, N 5.70. – IR (film) selected bands: $\nu = 622(\text{s})$, $850(\text{s})$, $1110(\text{vs})$, $1430(\text{s})$, $1585(\text{s})$, $1640(\text{s})$, $2980(\text{w})$, $3140(\text{w}) \text{ cm}^{-1}$. – ^1H NMR (DMSO): $\delta = 1.65$ (s, 3H), 7.80 (t, 2H), 8.25 (q, 2H), 8.70 (d, 2H), and 7.78 (d, 2H). – $^{13}\text{C}\{^1\text{H}\}$ NMR (DMSO): $\delta = 27.30$ ($^{13}\text{CH}_3\text{-COO}$), 178.60 ($\text{CH}_3\text{-}^{13}\text{COO}$), 117.50, 124.20, 127.60, 1141.60, 149.95, and 150.65. – ^{207}Pb NMR (DMSO): $\delta = -1350.50$ ppm.

Preparation of $[\text{Pb}(\text{bpy})(\text{CH}_3\text{COO})\text{I}]$

2,2'-Bipyridine (0.156 g, 1 mmol), as described above, with lead(II) acetate (0.36 g, 1 mmol) and sodium iodide (0.150 g, 1 mmol). After 15 d yellow crystals (m.p. 280 °C) deposited. Yield: 0.275 g, 50%. $\text{C}_{12}\text{H}_{11}\text{N}_2\text{O}_2\text{IPb}$: calcd. C 25.4, H 1.81, N 5.60; found C 25.60, H 1.90, N 5.80. – IR (film) selected bands: $\nu = 720(\text{s})$, $850(\text{s})$, $1340(\text{s})$, $1592(\text{s})$, $1650(\text{s})$, $2985(\text{w})$ and $3135(\text{w}) \text{ cm}^{-1}$. – ^1H NMR (DMSO): $\delta = 1.60$ (s, 3H), 7.80 (t, 2H), 8.25

Table 1. Crystal data and structure refinement for $[\text{Pb}(\text{bpy})(\text{NO}_3)(\text{CH}_3\text{COO})]$.

Empirical formula	$\text{C}_{24}\text{H}_{22}\text{N}_6\text{O}_{10}\text{Pb}_2$
Formula weight	968.86
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P2_1/c$
Unit cell dimensions	$a = 8.240(2)$ Å $b = 19.828(5)$ Å, $\beta = 92.216(5)^\circ$ $c = 8.844(2)$ Å
Volume	$1443.8(6)$ Å ³
Z	2
Density (calculated)	2.229 g/cm^3
Absorption coefficient	11.710 mm^{-1}
$F(000)$	904
Crystal size	$0.8 \times 0.7 \times 0.5 \text{ mm}^3$
Theta range for data collection	2.68 to 27.10.
Index ranges	$-10 \leq h \leq 10$, $-25 \leq k \leq 16$, $-10 \leq l \leq 11$
Reflections collected	8228
Independent reflections	3095 [$R(\text{int}) = 0.0514$]
Completeness to $\theta = 27.10$	97.0%
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.564 and 0.007
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3095 / 0 / 190
Goodness-of-fit on F^2	1.116
Final R indices for 2307 refl. [$I > 2\sigma(I)$]	$R1 = 0.0430$, $wR2 = 0.0923$
R Indices (all data)	$R1 = 0.0603$, $wR2 = 0.0974$
Largest diff. Peak, hole	$1.335, 2.457 \text{ e}^{-\text{Å}^{-3}}$

(q, 2H), 8.75 (d, 2H), and 7.80 (d, 2H). – $^{13}\text{C}\{^1\text{H}\}$ NMR (DMSO): $\delta = 27.25$ ($^{13}\text{CH}_3\text{-COO}$), 178.75 ($\text{CH}_3\text{-}^{13}\text{COO}$), 117.70, 124.30, 127.60, 1141.60, 149.80, and 150.40. – ^{207}Pb NMR (DMSO): $\delta = -1280.50$ ppm.

Crystallography

Determination of the structure

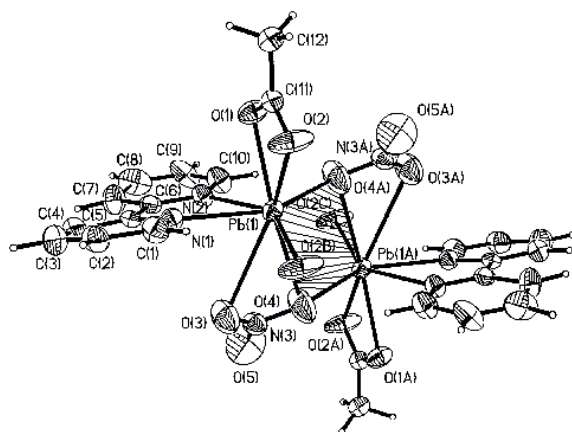
Intensity data were collected at 298(2) K within the range $2.68 \leq \theta \leq 27.10^\circ$ using a Siemens R3m/V diffractometer and graphite monochromated $\text{Mo-K}\alpha$ radiation ($\lambda = 0.71073$ Å). Accurate unit cell parameters and an orientation matrix for data collection were obtained from a least-squares refinement. Intensities of 8228 reflections were measured, from which 2307 with $I > 2\sigma(I)$ were used in the refinement. The structure was solved by direct methods and refined by full-matrix least-squares on F^2 .

The hydrogen atoms were placed idealized positions and included in the calculations of the structure factors as fixed contributions. Each hydrogen atom was assigned an isotropic displacement parameter. R , R_w with goodness of

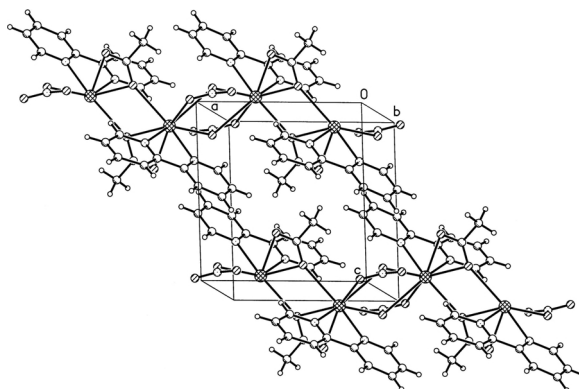
Table 2. Selected bond lengths (Å) and angles (°) for the $[\text{Pb}(\text{bpy})(\text{NO}_3)(\text{CH}_3\text{COO})]_n$.

Pb(1)-O(1)	2.429(5)	Pb(1)-N(1)	2.485(6)
Pb(1)-N(2)	2.526(6)	Pb(1)-O(2)	2.589(6)
Pb(1)-O(2) ^{#1}	2.691(5)	Pb(1)-O(4) ^{#2}	2.714(8)
Pb(1)-O(4)	2.973(8)	Pb(1)-O(3)	2.982(9)
Pb(1)-C(11)	2.900(7)	Pb(1)-C(1)	3.369(9)
Pb(1)-C(6)	3.385(8)	Pb(1)-C(5)	3.394(8)
Pb(1)-C(10)	3.410(8)	Pb(1)-C(11) ^{#1}	3.823(8)
Pb(1)-Pb(1) ^{#1}	4.419(11)	Pb(1)-Pb(1) ^{#2}	4.581(11)
O(4)-O(3)	2.053(11)	O(5)-O(3)	2.129(11)
O(4)-O(5)	2.095(14)	N(3)-O(5)	1.139(9)
N(3)-O(3)	1.218(9)	N(3)-O(4)	1.278(12)
O(1)-Pb(1)-N(1)	82.2(2)	O(1)-Pb(1)-N(2)	77.46(19)
N(1)-Pb(1)-N(2)	64.39(19)	O(2)-Pb(1)-O(1)	50.12(18)
N(1)-Pb(1)-O(2)	88.1(3)	N(2)-Pb(1)-O(2)	124.2(2)
O(1)-Pb(1)-O(2) ^{#1}	115.41(18)	N(1)-Pb(1)-O(2) ^{#1}	85.7(2)
N(2)-Pb(1)-O(2) ^{#1}	146.3(2)	O(2)-Pb(1)-O(2) ^{#1}	66.3(2)
O(1)-Pb(1)-O(4) ^{#2}	83.2(2)	N(1)-Pb(1)-O(4) ^{#2}	144.1(2)
N(2)-Pb(1)-O(4) ^{#2}	80.5(3)	O(2)-Pb(1)-O(4) ^{#2}	106.9(3)
O(2) ^{#1} -Pb(1)-O(4) ^{#2}	130.1(3)	O(1)-Pb(1)-O(4)	152.0(2)
N(1)-Pb(1)-O(4)	109.1(2)	N(2)-Pb(1)-O(4)	84.5(2)
O(2)-Pb(1)-O(4)	151.2(2)	O(1) ^{#1} -Pb(1)-O(4)	91.4(2)
O(4) ^{#2} -Pb(1)-O(4)	72.8(3)	O(1)-Pb(1)-O(3)	143.6(2)
N(1)-Pb(1)-O(3)	68.9(2)	N(2)-Pb(1)-O(3)	70.3(2)
O(2)-Pb(1)-O(3)	144.8(2)	O(2) ^{#1} -Pb(1)-O(3)	85.0(2)
O(4) ^{#2} -Pb(1)-O(3)	107.2(2)	O(4)-Pb(1)-O(3)	40.3(2)
O(5)-N(3)-O(3)	129.2(11)	O(5)-N(3)-O(4)	120.1(10)
O(4)-N(3)-O(3)	110.7(8)	N(3)-O(4)-O(3)	33.7(4)
N(3)-O(4)-O(5)	28.1(5)	O(3)-O(4)-O(5)	61.8(5)
Pb(1) ^{#2} -O(4)-Pb(1)	107.2(3)	N(3)-O(5)-O(4)	31.8(6)
N(3)-O(5)-O(3)	26.3(6)	O(4)-O(5)-O(3)	58.1(4)

^{#1} $-x+1, -y+1, -z$; ^{#2} $-x, -y+1, -z$.

Fig. 1. View of the Pb_2O_2 rhomb forming the basic link of the repeating units of the $[\text{Pb}(\text{bpy})(\text{NO}_3)(\text{CH}_3\text{COO})]_n$ polymer (ORTEP diagram).

fit on F^2 $1.116R = 0.0430$, $R_w = 0.0923$. The final difference density map showed maximum peak and hole of 1.335 and $-2.457 \text{ e} \cdot \text{\AA}^{-3}$, respectively. Corrections for the Lorentz and polarization effects as well as the empirical correction

Fig. 2. The unit cell of $[\text{Pb}(\text{bpy})(\text{NO}_3)(\text{CH}_3\text{COO})]_n$, showing the π - π stacking between bipyridine ligands.

for absorption using the Sadabs programs were applied. All structural calculations were carried out with a PDP-11/23+ computer using the SDP-PLUS program package [20–21].

Crystal data and details of structure refinement are given in Table 1. Selected bond lengths and angles are given in Table 2. Anisotropy thermal parameters, observed and calculated structure factors, full lists of bond distances, bond angles and torsion angles have been deposited at Cambridge Crystallography Data Centre. Supplementary data are available from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK on request, quoting the deposition number 226181 for $[\text{Pb}(\text{bpy})(\text{NO}_3)(\text{CH}_3\text{COO})]_n$. ORTEP diagrams and a perspective view of the packing in the unit cell are shown in Figures 1 and 2.

Results and Discussion

Synthesis

Upon reaction between 2,2'-bipyridine (bpy) and an equimolar mixture of lead(II) acetate with sodium iodide, sodium nitrate or sodium perchlorate, the $[\text{Pb}(\text{bpy})(\text{CH}_3\text{COO})\text{X}]$ ($\text{X} = \text{I}^-, \text{NO}_3^-, \text{ClO}_4^-$) complexes were isolated. The IR spectra of all three complexes show absorption bands resulting from the skeletal vibrations of aromatic rings in the 1400 – 1600 cm^{-1} range. The characteristic bands of the acetate anions appear at about $1640 \nu_{\text{as}}(\text{C}-\text{O})$ and $1430 \nu_{\text{sym}}(\text{C}-\text{O}) \text{ cm}^{-1}$. The Δ value ($\nu_{\text{as}} - \nu_{\text{sym}}$) indicates that the acetate anions coordinate to the Pb^{II} center in a bridging mode. Attempts for preparation of $[\text{Pb}(\text{bpy})_2(\text{CH}_3\text{COO})\text{X}]$ complexes were not successful and each time the 1:1 adducts were isolated.

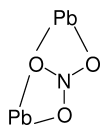
Crystal structure of $[\text{Pb}(\text{bpy})(\text{NO}_3)(\text{CH}_3\text{COO})]_n$

The crystal structure of this compound consists of polymeric units of $[\text{Pb}(\text{bpy})(\text{NO}_3)(\text{CH}_3\text{COO})]_n$. Each

lead atom is chelated by two nitrogen atoms of the bpy ligand with Pb–N distances of 2.485 and 2.526 Å, by the acetate anions with Pb–O distances of 2.429, 2.589 and 2.691 Å and also by oxygen atoms of nitrate anions with Pb–O distances of 2.714, 2.973 and 2.982 Å (Table 2). The coordination number in this complex is eight. The interaction of the lead(II) atom with oxygen atoms of a nitrate group of adjacent molecules produces polymeric units in the solid state in a new, rarely observed 1D framework type and a new mixed-ligand complex containing three ligands. The Pb–Pb distances in the polymeric units are Pb(1)–Pb(1)#1 = 4.419 and Pb(1)–Pb(1)#2 = 4.581 Å (Table 2).

The arrangement of the bpy ligand and the acetate and nitrate anions suggests a gap or hole in the coordination geometry around the metal ion [the O(2)–Pb–O(3) angle is 144.8°], occupied probably by a stereo-active lone pair of electrons on lead(II). The observed shortening of the Pb–N bonds opposite to the putative lone pair (2.485 Å) compared with 2.526 Å for the adjacent bond supports the presence of this feature [22].

There is some evident similarity in the two mixed-anion lead(II) complexes, Pb(L)(CH₃COO)(NO₃) (L=bpy and phen). Both complexes are polymeric and the coordination number is eight. Despite these similarities, it comes as a surprise to find significant differences between them, showing the influence of the different ligands (phen and and bpy). In the [(phen)Pb(O₂CCH₃)(O₂NO)] complex, both acetate oxygen atoms are bridging to form the polymer and the nitrate anion is asymmetrically bidentate towards the lead atom but does not appear to be involved in further bridging interactions. In the [Pb(bpy)(NO₃)(CH₃COO)]_n, however, only one of the acetate oxygen atoms is bridging and lead(II) interacts the with oxygen atoms of nitrate groups of adjacent molecules to produce a polymer. Indeed, two of the oxygen atoms of a nitrate anion are chelating and another pair is linked to a second lead atom.



It is very interesting that three bond lengths N–O and also three bond angles ONO of the nitrate anion in [Pb(bpy)(NO₃)(CH₃COO)]_n are different, [N(3)–O(5) = 1.139, N(3)–O(3) = 1.218, N(3)–O(4) =

1.278 Å, O(5)–N(3)–O(3) = 129.2°, O(5)–N(3)–O(4) = 120.1° and O(4)–N(3)–O(3) = 110.7°] and consequently the O–O distances are different [O(4)–O(3) = 2.053, O(5)–O(3) = 2.129 and O(4)–O(5) = 2.095 Å]. This point shows that the nitrate anion is highly distorted by the bridging.

The possible stereo-chemical activity of the lone pair in divalent lead compounds has recently been discussed by Shimoni–Livny *et al.* based on a thorough review of crystal data available in the Cambridge Structural Database (CSD) [7]. They classify lead coordination as holodirected, which refers to complexes in which the bonds to ligand atoms are directed throughout the surface of an encompassing sphere, while hemidirected refers to those cases in which the bonds to ligand atoms are directed in only part of the coordination sphere, leaving a gap in the distribution of bonds to the ligand. The latter, present in all Pb(II) compounds with coordination number 2 to 5, is quite common also for compounds with coordination numbers 6, 7 and 8, but does not exist in lead complexes with higher coordination numbers, where a holodirected geometry is the rule. For the structure described here, the coordination around the lead atoms is hemidirected with a significant gap trans to the chelating 2,2′-bipyridine.

A striking difference between [Pb(phen)(NO₃)(CH₃COO)]_n and [Pb(bpy)(NO₃)(CH₃COO)]_n is the π – π stacking [23–24] mode between the parallel aromatic rings belonging to adjacent chains, as shown in Fig. 2. The pyridyl rings are almost parallel and separated by a distance of about 3.5 Å, close to that of the layers in graphite. Parallel arrays of the planes of the aromatic moieties indicate that these interactions are of the “ π -stacking” type, rather than “edge-to-face” or “vertex-to-face” types [25–28]. A projection of the structure perpendicular to the ring plane shows the overall form of “slipped” stacking [28–29], which can be rationalized qualitatively in terms of optimizing the attraction between atoms of opposite charges [30].

A simple model to describe the nature of π – π interactions has been developed by Hunter *et al.* [30]. This model predicts that face-to-face π -stacked interactions will be disfavored due to the dominance of π – π repulsion. However, in offset π -stacked, and edge-on or T-shaped geometries, favorable π – σ attractions dominate. The polarization of aromatic systems through the introduction of heteroatoms, electron-withdrawing groups or electron-donating groups, alters the nature

of any $\pi - \pi$ interactions. It has been shown [30–33] that electron-deficient aromatic groups interact most strongly with electron-rich aromatic groups. Hence, since the molecules of this packing are equally or almost equally electron-deficient, or electron-rich, it can be expected that face-to-face π -stacking interactions

should be disfavored due to the dominance of π - π repulsion.

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