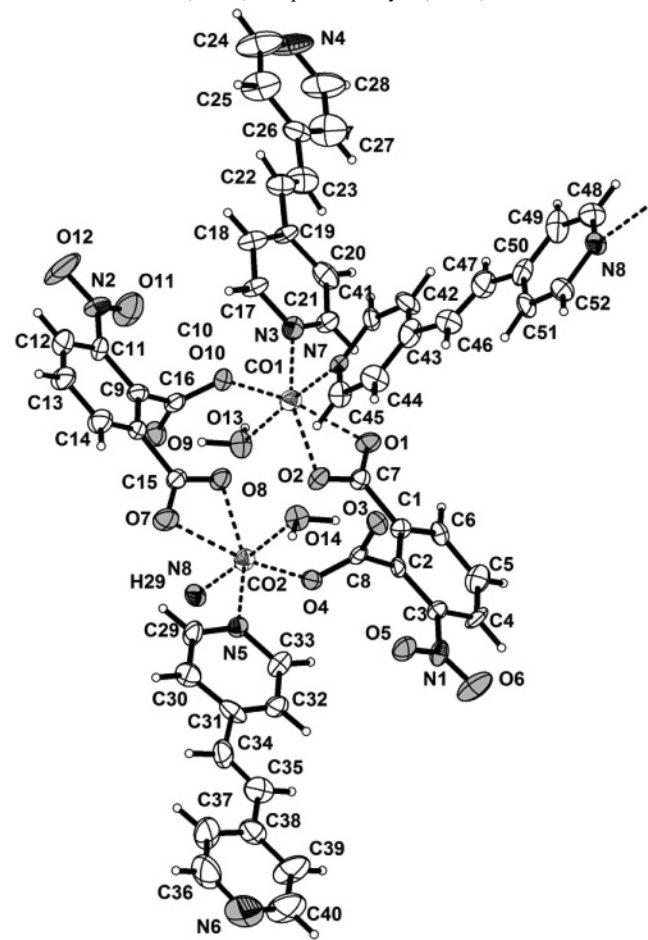


Crystal structure of poly[*diaqua*-(1,2-di(4-pyridyl)ethylene- $\kappa^2N:N'$)-bis(1,2-di(4-pyridyl)ethylene- κN)-bis(3-nitro-1,2-benzenedicarboxylato- $\kappa^3O,O':O''$)]cobalt(II)], $C_{52}H_{40}Co_2N_8O_{14}$

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Abstract

$C_{52}H_{40}Co_2N_8O_{14}$, triclinic, $P\bar{1}$ (no. 2), $a = 11.423(5)$ Å, $b = 13.051(6)$ Å, $c = 17.776(8)$ Å, $\alpha = 74.185(5)^\circ$, $\beta = 86.110(6)^\circ$, $\gamma = 70.758(6)^\circ$, $V = 2407(2)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0933$, $wR_{\text{ref}}(F^2) = 0.1817$, $T = 296$ K.

Source of material

A mixture of 3-nitro-1,2-benzenedicarboxylic acid (0.1 mmol, 18.7 mg), 1,2-di(4-pyridyl)ethylene (0.2 mmol, 36.8 mg), $\text{Co}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ (0.1 mmol, 24.8 mg), KOH (0.1 mmol), and H_2O (7 ml) were sealed in a 23 ml Teflon-lined autoclave, heated to 393 K for 4 days, and then slowly cooled down to room temperature for crystallization. Carmine red, block-shaped crystals of the title compound were obtained.

Table 1. Data collection and handling.

Crystal:	pink blocks, size 0.180×0.250×0.330 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	7.69 cm ^{−1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{\text{max}}$:	50.7°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	17330, 8650
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 5484
$N(\text{param})_{\text{refined}}$:	686
Programs:	SHELX [12], DIAMOND [13]

Discussion

Recently interest in metal-organic frameworks (MOFs) is rapidly increasing not only for their appealing structures, but also for their potential applications in magnetism, ion exchange, catalysis, photoluminescence, gas storage, electric conductivity and so on [1–4]. It is well-known that organic ligands play crucial roles in the design and construction of desirable frameworks. With regard to the aromatic benzenedicarboxylic acid and its derivatives (such as 1,*n*-benzenedicarboxylic acid, $n = 2, 3, 4$) are widely used as building blocks to link metal ions to produce metal-organic frameworks with interesting structures and properties [5–9]. However, the coordination chemistry based on 3-nitro-1,2-benzenedicarboxylic acid (H_2nbdc), a derivative of 1,2-benzenedicarboxylic acid, has rarely been studied [10]. Though the nitro group is not tangled in coordination, H_2nbdc may provide the potential to construct unpredictable and interesting network structures due to the existence of a non-coordinating electron-withdrawing nitro-group on the aromatic backbone, which will have a profound impact on the electron density of such a ligand and therefore different physical and chemical properties [11]. With the aim of the understanding of the coordination chemistry of rigid aromatic multi-carboxylate complexes with the nitro group, we have chosen 3-nitro-1,2-benzenedicarboxylic acid as a bridging ligand to construct new coordination polymers. Single-crystal X-ray analysis shows that the title compound is a 1D chain structure. The asymmetric unit contains two Co(II) cations, two completely deprotonated nbdc^{2-} anions, three *bpe* molecules (*bpe* = 1,2-di(4-pyridyl)ethylene) and two coordinating waters as shown in the Figure. In this complex, each cobalt atom is six-coordinated in a distorted octahedral manner [CoN_2O_4] by two N atoms from two *bpe* ligands, four oxygen atoms from two nbdc^{2-} anions and one coordinating water molecules. All the Co–O bond lengths are between 2.028(4) and 2.314(5) Å, and the Co–N bond lengths are in the range of 2.077(5) to 2.153(5) Å, respectively. Two Co(II) neighbours are connected by two nbdc^{2-} ligands adopting single and chelating-coordination modes to form one dinuclear unit with the Co–Co distance of 5.205(2) Å. The carboxyl-bridged dinuclears are extended along the [110] direction by *bpe* coligands to produce one 1D coordination polymer with

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the Co–Co separation of 13.680(4) Å. In the structure, there are two kinds of H-bonds. Firstly, there exist intra-chain H-bonding interactions between the coordinated water and the carboxylate oxygen of *nbc*²⁻. Secondly, inter-chain H-bond interactions in coordinated water and the carboxylate oxygen connect to favor formation of 2D layer structure. No H-bonds are observed between the layers, which stack in a slightly off-set parallel fashion and are cohered only by the weak van der Waals force to accomplish its entire 3D supramolecular structure.

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1W)	2i	0.4487	0.7811	0.0827	0.061
H(2W)	2i	0.4736	0.8726	0.0116	0.061
H(4W)	2i	0.3082	0.9754	0.4081	0.057
H(3W)	2i	0.2857	0.8824	0.4790	0.057
H(4)	2i	0.7536	1.2016	0.2674	0.051
H(5A)	2i	0.7185	1.2505	0.1317	0.059
H(6)	2i	0.5799	1.1869	0.0804	0.038
H(12A)	2i	−0.0067	0.5607	0.2261	0.062
H(13A)	2i	0.0230	0.5194	0.3583	0.062
H(14A)	2i	0.1573	0.5860	0.4041	0.057
H(17)	2i	0.1226	0.9377	0.0041	0.048
H(18)	2i	0.0348	0.9908	−0.1153	0.056
H(20)	2i	0.2472	1.1832	−0.1917	0.059
H(21)	2i	0.3238	1.1336	−0.0646	0.050

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
Co(1)	2i	0.3079(1)	0.97686(9)	0.09688(6)	0.0286(7)	0.0296(7)	0.0251(7)	−0.0075(6)	−0.0018(5)	−0.0112(5)
Co(2)	2i	0.4478(1)	0.77660(9)	0.38772(6)	0.0323(7)	0.0267(7)	0.0223(6)	−0.0108(6)	0.0002(5)	−0.0080(5)
O(1)	2i	0.4287(5)	1.0943(5)	0.0745(3)	0.043(4)	0.049(4)	0.023(3)	−0.020(3)	−0.005(3)	−0.007(3)
O(2)	2i	0.4210(5)	0.9752(5)	0.1863(3)	0.041(4)	0.042(4)	0.032(3)	−0.022(3)	−0.001(3)	−0.012(3)
O(3)	2i	0.3811(5)	1.0535(4)	0.3339(3)	0.035(4)	0.030(3)	0.033(3)	−0.004(3)	0.007(3)	−0.013(3)
O(4)	2i	0.5356(5)	0.8899(4)	0.3410(3)	0.029(3)	0.031(3)	0.031(3)	−0.007(3)	−0.002(3)	−0.009(3)
O(5)	2i	0.6193(6)	1.0161(5)	0.4228(3)	0.071(5)	0.049(4)	0.039(4)	−0.025(4)	−0.017(3)	−0.012(3)
O(6)	2i	0.7300(6)	1.1225(6)	0.3994(3)	0.081(5)	0.115(6)	0.042(4)	−0.065(5)	0.000(4)	−0.025(4)
O(7)	2i	0.3369(5)	0.6562(5)	0.4069(3)	0.055(4)	0.052(4)	0.029(3)	−0.023(3)	−0.011(3)	−0.004(3)
O(8)	2i	0.3284(5)	0.7880(5)	0.2987(3)	0.044(4)	0.038(4)	0.030(3)	−0.022(3)	−0.004(3)	−0.004(3)
O(9)	2i	0.3699(5)	0.6983(5)	0.1480(3)	0.037(4)	0.035(4)	0.049(4)	−0.011(3)	0.007(3)	−0.013(3)
O(10)	2i	0.2196(5)	0.8633(4)	0.1428(3)	0.035(4)	0.028(3)	0.040(3)	−0.015(3)	0.000(3)	−0.008(3)
O(11)	2i	0.1306(8)	0.7345(7)	0.0632(4)	0.121(7)	0.086(6)	0.048(5)	−0.061(5)	−0.011(5)	−0.009(4)
O(12)	2i	0.0072(8)	0.6382(7)	0.0915(4)	0.114(7)	0.134(8)	0.093(6)	−0.085(6)	−0.027(5)	−0.037(5)
O(13)	2i	0.4629(5)	0.8524(4)	0.0672(3)	0.044(4)	0.037(4)	0.042(3)	−0.014(3)	0.016(3)	−0.016(3)
O(14)	2i	0.2930(5)	0.9046(4)	0.4232(3)	0.043(4)	0.041(4)	0.030(3)	−0.014(3)	0.008(3)	−0.011(3)
N(1)	2i	0.6683(7)	1.0793(6)	0.3785(4)	0.049(5)	0.034(5)	0.031(4)	−0.021(4)	0.010(4)	−0.017(4)
N(2)	2i	0.0816(8)	0.6778(7)	0.1110(5)	0.055(6)	0.057(6)	0.065(7)	−0.016(5)	−0.031(5)	−0.019(5)
N(3)	2i	0.2304(6)	1.0323(6)	−0.0170(4)	0.033(4)	0.037(5)	0.044(5)	−0.013(4)	0.007(4)	−0.016(4)
N(4)	2i	0.0112(9)	1.228(1)	−0.5475(5)	0.065(7)	0.17(1)	0.022(5)	−0.051(7)	−0.018(5)	0.003(6)
N(5)	2i	0.5120(6)	0.7164(5)	0.5029(4)	0.037(4)	0.032(4)	0.024(4)	−0.012(4)	−0.002(3)	−0.009(3)
N(6)	2i	0.729(1)	0.5266(9)	1.0371(5)	0.115(9)	0.104(9)	0.036(6)	−0.028(7)	−0.021(6)	−0.006(6)
N(7)	2i	0.1592(6)	1.0938(5)	0.1414(4)	0.029(4)	0.027(4)	0.034(4)	−0.011(3)	−0.006(3)	−0.009(3)
N(8)	2i	−0.4050(6)	1.6597(5)	0.3430(4)	0.034(4)	0.029(4)	0.039(4)	0.000(3)	0.001(3)	−0.018(4)
C(1)	2i	0.5423(7)	1.0948(7)	0.1823(4)	0.029(5)	0.032(5)	0.025(5)	−0.009(4)	0.002(4)	−0.011(4)
C(2)	2i	0.5623(7)	1.0624(6)	0.2653(4)	0.026(5)	0.016(5)	0.034(5)	−0.004(4)	0.002(4)	−0.011(4)
C(3)	2i	0.6413(7)	1.1057(7)	0.2932(4)	0.030(5)	0.032(5)	0.023(4)	−0.012(4)	−0.001(4)	−0.010(4)
C(4)	2i	0.7008(8)	1.1751(7)	0.2462(5)	0.039(6)	0.051(6)	0.053(6)	−0.031(5)	−0.008(5)	−0.015(5)
C(5)	2i	0.6788(8)	1.2046(8)	0.1651(5)	0.047(6)	0.054(7)	0.055(7)	−0.031(5)	0.016(5)	−0.016(5)
C(6)	2i	0.5966(8)	1.1649(7)	0.1340(5)	0.041(6)	0.022(5)	0.028(5)	−0.009(4)	0.006(4)	−0.003(4)
C(7)	2i	0.4563(7)	1.0527(7)	0.1455(5)	0.035(5)	0.032(5)	0.024(5)	−0.014(4)	0.001(4)	−0.009(4)
C(8)	2i	0.4848(8)	0.9958(7)	0.3179(4)	0.031(5)	0.030(5)	0.022(4)	−0.016(4)	−0.002(4)	−0.010(4)
C(9)	2i	0.2107(7)	0.6660(7)	0.3000(5)	0.026(5)	0.025(5)	0.042(5)	−0.009(4)	−0.003(4)	−0.009(4)
C(10)	2i	0.1933(7)	0.6932(7)	0.2204(5)	0.028(5)	0.034(5)	0.030(5)	−0.005(4)	−0.005(4)	−0.012(4)
C(11)	2i	0.1083(8)	0.6523(7)	0.1948(5)	0.043(6)	0.028(5)	0.058(6)	−0.014(5)	−0.006(5)	−0.018(5)
C(12)	2i	0.0472(9)	0.5874(8)	0.2459(6)	0.048(7)	0.041(6)	0.073(8)	−0.023(5)	−0.003(6)	−0.013(6)
C(13)	2i	0.0647(9)	0.5623(8)	0.3239(6)	0.053(7)	0.050(7)	0.055(7)	−0.036(5)	−0.014(5)	0.010(5)
C(14)	2i	0.1450(9)	0.6019(8)	0.3504(5)	0.048(6)	0.049(7)	0.048(6)	−0.025(5)	−0.004(5)	−0.004(5)
C(15)	2i	0.2965(7)	0.7053(7)	0.3383(5)	0.021(5)	0.032(5)	0.052(6)	−0.004(4)	0.000(4)	−0.023(5)

Table 2. continued.

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(22)	2i	0.0140	1.0942	−0.2537	0.056
H(23)	2i	0.2054	1.1711	−0.3079	0.074
H(24)	2i	−0.1066	1.1495	−0.5020	0.111
H(25)	2i	−0.0467	1.1014	−0.3742	0.085
H(27)	2i	0.2007	1.2640	−0.4440	0.096
H(28)	2i	0.1295	1.3058	−0.5695	0.109
H(29)	2i	0.4487	0.5884	0.5308	0.050
H(30)	2i	0.5004	0.5263	0.6627	0.055
H(32)	2i	0.6445	0.7714	0.6343	0.050
H(33)	2i	0.5922	0.8298	0.5020	0.053
H(34)	2i	0.5844	0.5348	0.7738	0.068
H(35)	2i	0.6797	0.7013	0.7608	0.075
H(36)	2i	0.6394	0.4266	1.0316	0.128
H(37)	2i	0.6014	0.4737	0.9035	0.095
H(39)	2i	0.7643	0.7039	0.8798	0.097
H(40)	2i	0.8060	0.6432	1.0083	0.109
H(41)	2i	0.0728	1.1999	0.0457	0.042
H(42)	2i	−0.0625	1.3301	0.1034	0.055
H(44)	2i	0.0771	1.1340	0.3108	0.057
H(45)	2i	0.2155	1.0084	0.2517	0.045
H(46)	2i	−0.0830	1.3072	0.3135	0.061
H(47)	2i	−0.1679	1.4415	0.1721	0.071
H(48)	2i	−0.4575	1.7388	0.2349	0.052
H(49)	2i	−0.3322	1.6159	0.1719	0.064
H(51)	2i	−0.1806	1.4227	0.3824	0.050
H(52)	2i	−0.3163	1.5510	0.4395	0.048

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(16)	2i	0.2673(8)	0.7565(7)	0.1648(5)	0.032(5)	0.037(6)	0.036(5)	−0.013(5)	−0.006(4)	−0.015(4)
C(17)	2i	0.1471(8)	0.9895(8)	−0.0352(5)	0.032(5)	0.058(6)	0.038(5)	−0.023(5)	−0.004(4)	−0.013(5)
C(18)	2i	0.0975(8)	1.0181(8)	−0.1077(5)	0.033(5)	0.066(7)	0.043(6)	−0.018(5)	−0.007(5)	−0.016(5)
C(19)	2i	0.1362(7)	1.0858(7)	−0.1703(4)	0.034(5)	0.050(6)	0.011(4)	−0.024(5)	0.000(4)	−0.006(4)
C(20)	2i	0.2208(8)	1.1325(7)	−0.1526(5)	0.052(6)	0.039(6)	0.039(6)	−0.009(5)	0.006(5)	0.007(5)
C(21)	2i	0.2662(8)	1.1026(7)	−0.0753(5)	0.036(6)	0.044(6)	0.050(6)	−0.016(5)	−0.006(5)	−0.016(5)
C(22)	2i	0.0860(8)	1.1108(8)	−0.2490(5)	0.033(6)	0.059(7)	0.044(6)	−0.009(5)	−0.001(5)	−0.015(5)
C(23)	2i	0.1338(9)	1.1543(9)	−0.3131(6)	0.042(7)	0.081(9)	0.058(7)	−0.011(6)	0.009(6)	−0.025(6)
C(24)	2i	−0.042(1)	1.171(1)	−0.4893(6)	0.069(9)	0.17(1)	0.041(7)	−0.057(9)	−0.007(6)	−0.011(8)
C(25)	2i	−0.009(1)	1.143(1)	−0.4126(5)	0.069(8)	0.12(1)	0.027(6)	−0.047(8)	0.010(5)	−0.003(6)
C(26)	2i	0.0854(9)	1.1796(8)	−0.3931(5)	0.041(6)	0.049(6)	0.038(6)	0.005(5)	−0.011(5)	−0.013(5)
C(27)	2i	0.137(1)	1.239(1)	−0.4538(6)	0.072(9)	0.11(1)	0.050(7)	−0.039(8)	0.015(6)	−0.004(7)
C(28)	2i	0.095(1)	1.263(1)	−0.5296(6)	0.076(9)	0.14(1)	0.038(7)	−0.050(9)	−0.017(6)	0.021(7)
C(29)	2i	0.4880(8)	0.6270(7)	0.5516(5)	0.061(7)	0.036(6)	0.034(5)	−0.030(5)	−0.007(5)	0.000(4)
C(30)	2i	0.5185(8)	0.5887(8)	0.6313(5)	0.061(7)	0.045(6)	0.026(5)	−0.018(5)	−0.008(5)	0.004(4)
C(31)	2i	0.5771(9)	0.6468(8)	0.6623(5)	0.054(7)	0.037(6)	0.019(5)	0.013(5)	−0.008(4)	−0.011(4)
C(32)	2i	0.6036(8)	0.7332(8)	0.6143(5)	0.047(6)	0.044(6)	0.040(6)	−0.008(5)	−0.011(5)	−0.024(5)
C(33)	2i	0.5717(8)	0.7691(8)	0.5339(5)	0.046(6)	0.051(6)	0.048(6)	−0.025(5)	0.003(5)	−0.021(5)
C(34)	2i	0.6050(9)	0.5987(8)	0.7483(6)	0.051(7)	0.031(6)	0.089(8)	−0.007(5)	0.001(6)	−0.025(6)
C(35)	2i	0.6547(9)	0.6404(9)	0.7877(6)	0.055(7)	0.062(8)	0.064(8)	−0.014(6)	−0.009(6)	−0.011(6)
C(36)	2i	0.668(1)	0.483(1)	1.0005(7)	0.17(2)	0.055(9)	0.08(1)	−0.024(9)	−0.07(1)	0.008(7)
C(37)	2i	0.642(1)	0.5115(9)	0.9242(6)	0.14(1)	0.055(8)	0.053(7)	−0.030(8)	−0.036(7)	−0.016(6)
C(38)	2i	0.6776(9)	0.5990(9)	0.8765(5)	0.066(7)	0.047(7)	0.032(6)	−0.004(6)	−0.012(5)	−0.013(5)
C(39)	2i	0.738(1)	0.645(1)	0.9098(6)	0.11(1)	0.12(1)	0.031(6)	−0.068(9)	−0.025(6)	0.003(6)
C(40)	2i	0.762(1)	0.608(1)	0.9874(7)	0.11(1)	0.13(1)	0.046(8)	−0.053(9)	−0.027(7)	−0.013(8)
C(41)	2i	0.0760(7)	1.1867(6)	0.0998(5)	0.033(5)	0.021(5)	0.051(6)	−0.007(4)	0.002(4)	−0.011(4)
C(42)	2i	−0.0068(8)	1.2653(7)	0.1346(5)	0.034(6)	0.032(6)	0.057(7)	0.004(4)	−0.008(5)	−0.005(5)
C(43)	2i	−0.0068(8)	1.2480(8)	0.2137(6)	0.042(6)	0.049(6)	0.062(7)	−0.016(5)	0.020(5)	−0.039(6)
C(44)	2i	0.0764(8)	1.1498(8)	0.2566(5)	0.051(7)	0.056(7)	0.033(5)	−0.008(6)	0.008(5)	−0.020(5)
C(45)	2i	0.1594(8)	1.0736(7)	0.2212(5)	0.047(6)	0.040(6)	0.030(5)	−0.015(5)	0.010(4)	−0.017(4)
C(46)	2i	−0.0889(9)	1.3288(9)	0.2591(6)	0.050(7)	0.048(7)	0.051(6)	−0.011(5)	−0.008(5)	−0.009(5)
C(47)	2i	−0.1621(9)	1.4209(9)	0.2264(6)	0.051(7)	0.049(7)	0.081(8)	−0.023(6)	−0.001(6)	−0.015(6)
C(48)	2i	−0.4022(8)	1.6729(8)	0.2653(5)	0.034(6)	0.051(6)	0.045(6)	−0.008(5)	0.007(5)	−0.018(5)
C(49)	2i	−0.3269(9)	1.6000(8)	0.2261(6)	0.054(7)	0.044(7)	0.070(7)	−0.023(6)	0.028(6)	−0.026(6)
C(50)	2i	−0.2430(8)	1.5023(8)	0.2693(6)	0.042(6)	0.043(6)	0.057(6)	−0.022(5)	0.011(5)	−0.028(5)
C(51)	2i	−0.2385(7)	1.4861(7)	0.3515(5)	0.030(5)	0.023(5)	0.067(7)	0.002(4)	0.004(5)	−0.019(5)
C(52)	2i	−0.3197(8)	1.5641(7)	0.3854(5)	0.044(6)	0.036(6)	0.035(5)	−0.011(5)	−0.001(4)	−0.002(4)

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