

P=X bond (see angle β , Table 4). The anomeric stabilization from O(9) increases with increasing electronegativity of X. This explains the shortening of the P(2)–O(9) bond on going from SELOPOS to TIOPOS and OXOPOS.

The effect of increasing delocalization around O(9) can be seen from the angle P(2)–O(9)–C(10) which is significantly larger in OXOPOS compared to TIOPOS and SELOPOS.

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The Structure of Trimesic Acid, Its Hydrates and Complexes.

VI.* Glycine–Trimesic Acid Monohydrate, $\text{H}_3\text{N}^+\text{CH}_2\text{COO}^- \cdot \text{C}_9\text{H}_6\text{O}_6 \cdot \text{H}_2\text{O}$

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Abstract

The ternary adduct glycine–trimesic acid monohydrate is orthorhombic, $a = 7.971$ (5), $b = 24.615$ (10), $c = 6.476$ (5) Å, space group $Pna2_1$, $Z = 4$. The structure was solved by *MULTAN* and refined to $R = 4.42\%$, using 1570 reflections measured on a four-circle diffractometer with Mo $K\alpha$ radiation (graphite monochromator). The glycine is present in the zwitterionic form and the three moieties are joined together by a rather complicated three-dimensional arrangement of hydrogen bonds. These are all of standard dimensions except for the hydrogen bond between a carboxyl oxygen of glycine and a hydroxyl oxygen of trimesic acid; this is very short [$d(\text{O} \cdots \text{O}) = 2.464$ (3) Å] and

nearly symmetrical [$d(\text{O}_{\text{TMA}}-\text{H}) = 1.16$ (6) Å, $d(\text{O}_{\text{Gly}}^-\cdots\text{H}) = 1.31$ (6) Å]. This substance is classified as an acid salt in which the NH_3^+ group acts as cation and the two anions are the partially ionized carboxyl groups of trimesic acid and glycine.

1. Introduction

Aqueous solutions containing equimolar amounts of trimesic acid (1,3,5-benzenetricarboxylic acid; TMA) and glycine give crystals of the ternary complex glycine–trimesic acid monohydrate. Determination of the crystal structure shows that the glycine and TMA moieties are joined by a very short, nearly symmetrical hydrogen bond. Such bonds were first found in acid and basic salts of monobasic acids and monoacidic

* Part V: Herbstein & Kapon (1979).

Table 1. *Crystal data*

$\text{H}_3\text{N}^+\text{CH}_2\text{COO}^- \cdot \text{C}_9\text{H}_6\text{O}_6 \cdot \text{H}_2\text{O}$, $M_r = 303.23$	
Orthorhombic, $Pna2_1^*$ (No. 33)	
$Z = 4$, $F(000) = 632$	$V = 1270.6 \text{ \AA}^3$
$a = 7.971 (5) \text{ \AA}$	$D_x = 1.58 \text{ Mg m}^{-3}$
$b = 24.615 (10)$	$D_m = 1.56 \text{ Mg m}^{-3}$ (floatation in toluene- 1,1,2,2-tetrabromoethane)
$c = 6.476 (5)$	$\mu(\text{Mo } K\alpha) = 0.093 \text{ mm}^{-1}$

* By structure analysis.

bases (Speakman, 1972) but have also been encountered between O atoms in many other situations (e.g. Harlow & Simonsen, 1978; Catti, Ferraris & Ivaldi, 1979).

2. Experimental

The crystals (air-stable transparent plates) do not have a definite melting point but darken above 673 K and disintegrate above 873 K. After preliminary X-ray photography a crystal of size $0.6 \times 0.3 \times 0.1 \text{ mm}$ was mounted on the Philips PW 1100/20 four-circle diffractometer with z slightly displaced from the ϕ axis. Cell dimensions were determined from 25 centred reflections. Crystal data are given in Table 1.

Intensities of about 2100 reflections were measured using graphite-monochromated Mo $K\alpha$ radiation; the 2θ range was $4\text{--}55^\circ$; the scan speed and width in ω were $2.4^\circ \text{ min}^{-1}$ and 1.2° , respectively; background was counted for half the total scanning time on each side of a reflection; the crystal was stable during the measurements; no absorption corrections were applied as $\mu r < 0.06$.

3. Structure and refinement

The structure was solved using *MULTAN* (Main, Woolfson, Lessinger, Germain & Declercq, 1977). The E map based on the best set of phases revealed all the non-hydrogen atoms but two (a carbon of one of the carboxyl groups and the water oxygen); the initial R value was 39%. The refinement (for technical details see part V) converged after inclusion of all the atoms (anisotropic temperature factors for non-hydrogen and isotropic temperature factors for H atoms) at $R = 4.42\%$ ($R_w = 4.74\%$). 1570 reflections were used and 241 parameters were refined. The weighting factor obtained by refinement was $w(F) = 0.375/[\sigma^2(F) + 0.0023F^2]$. The z coordinate of C(1) was kept fixed during refinement. Final atomic coordinates are in Table 2.*

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35599 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Final fractional atomic coordinates*

Numbers in parentheses here and in other tables indicate e.s.d.'s in units of the least significant digit. The U_{iso} values for H atoms are $\times 10^3$.

	x	y	z	d^\dagger	$U_{\text{eq}}^\ddagger/U_{\text{iso}} (\text{\AA}^2)$
C(1)	0.3408 (3)	0.1656 (1)	0.2693*	0.003	0.0256 (13)
C(2)	0.3287 (3)	0.2215 (1)	0.3022 (7)	0.027	0.0265 (12)
C(3)	0.3963 (3)	0.2572 (1)	0.1571 (7)	0.006	0.0254 (12)
C(4)	0.4789 (4)	0.2369 (1)	0.0155 (7)	0.006	0.0286 (13)
C(5)	0.4940 (3)	0.1810 (1)	0.0455 (7)	0.005	0.0266 (13)
C(6)	0.4221 (3)	0.1456 (1)	0.0955 (7)	-0.028	0.0273 (13)
C(7)	0.2696 (3)	0.1252 (1)	0.4174 (7)	0.003	0.0277 (12)
C(8)	0.3807 (4)	0.3176 (1)	0.1826 (7)	-0.013	0.0284 (13)
C(9)	0.5869 (4)	0.1593 (1)	-0.2281 (6)	0.020	0.0290 (13)
C(10)	0.6269 (4)	0.5211 (1)	0.0244 (7)		0.0297 (13)
C(11)	0.5871 (3)	0.4629 (1)	-0.0313 (7)		0.0263 (12)
O(1)	0.2161 (3)	0.1463 (1)	0.5925 (6)	0.222	0.0368 (11)
O(2)	0.2633 (3)	0.0770 (1)	0.3780 (6)	0.174	0.0418 (12)
O(3)	0.4408 (3)	0.3445 (1)	0.0296 (6)	-0.115	0.0443 (11)
O(4)	0.3161 (3)	0.3389 (1)	0.3336 (6)	0.051	0.0392 (12)
O(5)	0.6501 (4)	0.1974 (1)	-0.3443 (6)	0.066	0.0494 (14)
O(6)	0.6020 (3)	0.1112 (1)	-0.2643 (6)	0.000	0.0395 (11)
O(7)	0.4562 (3)	0.4442 (1)	0.0566 (6)		0.0359 (11)
O(8)	0.6783 (3)	0.4389 (1)	0.1548 (6)		0.0350 (10)
O(9)	0.4338 (3)	-0.0166 (1)	0.4989 (7)		0.0427 (13)
N(1)	0.7682 (4)	0.5417 (1)	-0.0993 (7)		0.0320 (13)
H(1)	0.275 (4)	0.235 (1)	0.423 (7)		32 (9)
H(2)	0.531 (4)	0.260 (1)	-0.123 (6)		32 (8)
H(3)	0.435 (4)	0.107 (1)	0.076 (7)		44 (10)
H(4)	0.194 (5)	0.121 (2)	0.664 (8)		47 (11)
H(5)	0.441 (7)	0.391 (3)	0.040 (16)		121 (24)
H(6)	0.699 (6)	0.189 (2)	-0.433 (10)		59 (14)
H(7)	0.533 (4)	0.544 (1)	0.015 (6)		26 (8)
H(8)	0.648 (4)	0.523 (1)	0.167 (7)		32 (8)
H(9)	0.800 (5)	0.577 (2)	0.061 (8)		60 (12)
H(10)	0.852 (5)	0.523 (2)	0.078 (7)		38 (10)
H(11)	0.739 (6)	0.539 (2)	-0.252 (10)		80 (16)
H(12)	0.370 (6)	0.007 (2)	0.472 (9)		71 (15)
H(13)	0.425 (5)	0.042 (2)	0.404 (10)		71 (15)

* Arbitrarily assigned and held constant during the refinement.

† Deviations from planarity (\AA) (best plane calculated through the nine carbons of the TMA molecule). The equation of the plane (in terms of orthogonal axes X, Y, Z along the crystal axes) is $0.8572X + 0.0018Y + 0.5150Z - 3.2368 \text{ \AA}$. E.s.d.'s in the atom deviations are $\sim 0.005 \text{ \AA}$.

‡ U_{eq} † trace \bar{U} .

4. Results and discussion

4.1. The crystal structure

The structure is shown in z projection in Fig. 1, which corresponds to the *ORTEP* stereodiagram of Fig. 2. The structure can be described in convenient but formal terms as consisting of parallel slices of TMA molecules and of glycine and water molecules bound together by a complicated arrangement of hydrogen bonds. The slices are parallel to (010); there do not seem to be appreciable differences between the degree of hydrogen bonding within the slices as compared to that between them.

In the TMA slice adjacent molecules are related by the a glide perpendicular to b and are oriented so as to form a planar layer roughly in the (101) planes (Fig. 1). Adjacent TMA molecules are hydrogen bonded together [O(5) of 55501 to O(4) of 55404 (the *ORTEP* notation used to identify the molecules is described in the caption to Fig. 1)] and also *via* the carboxyl group of the glycine, forming a ring. Translationally equiva-

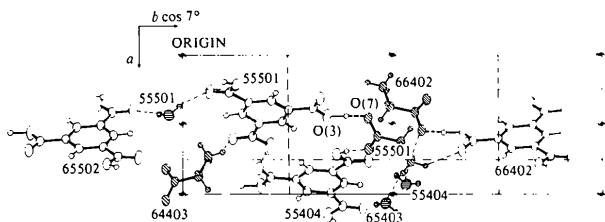


Fig. 1. Projection of part of the crystal structure down an axis rotated 7° anticlockwise from c about a . The codes of the various moieties are those used in *ORTEP* (Johnson, 1965). The reference molecules (those whose coordinates are given in Table 2) are denoted as 555, translations along the axes are specified by adding or subtracting integers from the reference code. The sequence of the symmetry elements is that given in *International Tables for X-ray Crystallography* (1952). Enough atoms are labelled in the reference molecules to allow identification of the others. The very short hydrogen bond is shown by a bolder broken line.

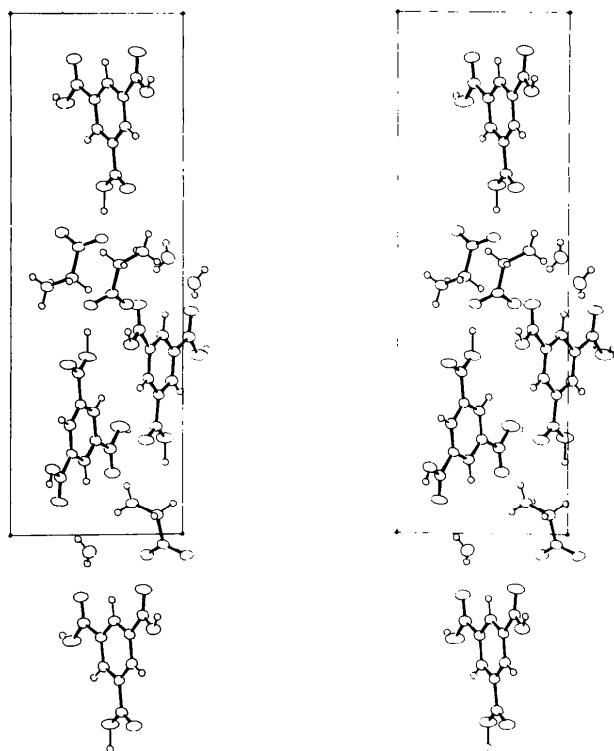


Fig. 2. *ORTEP* stereogram of part of the contents of the unit cell, orientation 90° from that of Fig. 1.

lent TMA molecules within a slice are arranged in stacks whose axes run parallel to a . The TMA slices are 'glued' together by the glycine + water slices, where adjacent molecules are related by the 2_1 axes parallel to c . Alternate slices of molecules of a particular type are related by the n glide normal to a .

Table 3. *Details of hydrogen bonding between the moieties*

Carbonyl oxygens of TMA are underlined (and have even numbers).

Hydrogen-bond donor (reference atom)	Hydrogen-bond acceptor	$d(N-H \cdots O)^*$ or $d(O-H \cdots O)$ (Å)	$\angle N-H \cdots O^*$ or $\angle O-H \cdots O$ ($^\circ$)
N(1)—H(10)	O(9) 65403	2.846	173
N(1)—H(9) [†]	O(6) 65503	2.951	132
	<u>O(4)</u> 66402	3.046	129
N(1)—H(11)	<u>O(7)</u> 66402	2.879	139
	O(9) 55404	2.982	123
O(9)—H(12)	<u>O(2)</u> 55501	2.786	165
O(9)—H(13)	<u>O(6)</u> 65502	2.901	168
O(1)—H(4)	<u>O(8)</u> 45604	2.678	171
O(3)—H(5)	O(7) 55501	2.464	174
O(5)—H(6)	<u>O(4)</u> 55404	2.627	175

* E.s.d.'s of bond lengths and bond angles are about 0.003 Å and 4° respectively.

[†] To avoid overlap in Fig. 1, this bond is shown between O(6) 55501 and N(1) 64403.

4.2. The hydrogen bonding

The relevant geometrical information is summarized in Table 3. The NH_3^+ group makes an almost linear hydrogen bond through H(10) to (water)O(9)(65403), but the other two H atoms both point between a pair of O atoms, *i.e.* these are bifurcated hydrogen bonds. Both O—H bonds of the water molecule form approximately linear hydrogen bonds to carbonyl oxygen acceptors. The three C—OH groups of the carboxylic acid groups of the TMA each form an almost linear hydrogen bond to carbonyl oxygen acceptors, one of another TMA molecule and the other two belonging to glycine carboxyl groups. The water molecule acts as donor in two hydrogen bonds and acceptor in one (from NH_3^+); the second acceptor position is blocked by an oblique N—H approach. All four carbonyl O atoms act as single acceptors of hydrogen bonds from other atoms: two of them are also blocked by oblique N—H approaches.

Thus the hydrogen-bonding capabilities of the three components are utilized almost to the full. The dimensions of the hydrogen bonds fall into the standard limits for the various types except for that involving the C(8) carboxyl group of TMA and the carboxyl group of the glycine molecule. The O(3)···O(7) distance here is 2.464 (3) Å, corresponding to a strong hydrogen bond (Speakman, 1972). H(5) has been located but with considerable uncertainty $\langle \sigma(r) \rangle = 0.08$ Å, $\sigma(z) = 0.10$ Å and $U_{iso} = 0.12$ Å²; it is closer to O(3)(TMA) [O(3)—H(5) = 1.16 (5) Å] than to O(7)(glycine) [O(7)···H(5) = 1.30 (6) Å] but the difference in these O···H lengths is significant only at the 8% level ($\Delta l/\sqrt{2}\sigma = 1.8$). We note further that the Debye-Waller factor for H(5) is 2–4 times larger than those of the other H atoms.

4.3. Glycine

The three H atoms attached to N show that glycine is present as a zwitterion; comparison (Table 4) of the torsion angles with those of α -glycine [the neutron diffraction results of Jönsson & Kvick (1972) are taken as standard] shows that the conformation is the same here as in α -glycine. The C–N distance here [1.472 (4) Å] agrees with that in α -glycine [1.476 (1) Å] but the dimensions in the rest of the molecule (Fig. 3) differ significantly from those in α -glycine. We ascribe these differences to the influence of the hydrogen bonding.

McGregor, Speakman & Lehmann (1977) have surveyed the dimensions of carboxyl groups joined by very short hydrogen bonds [$d(\text{O}\cdots\text{O}) < 2.5$ Å]. For eleven type A (symmetry-restricted) structures they find a mean difference between the two C–O lengths of $\Delta r = 0.066$ (2) Å, and between the C–C–O angles of $\Delta\theta = -8.0$ (5)°. In potassium hydrogen dicrotonate,

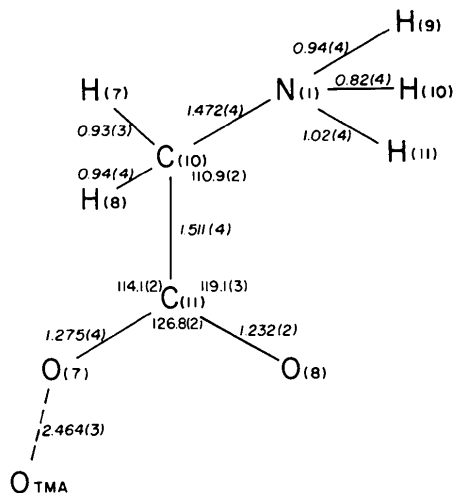


Fig. 3. Dimensions and numbering system of the glycine moiety.

Table 4. Torsion angles ($^{\circ}$) in glycine moiety

E.s.d.'s have been calculated using the formula of Stanford & Waser (1972). The torsion angles in α -glycine (Jönsson & Kvick, 1972) are used for comparison.

	τ	τ (α -glycine)*
H(8)–C(10)–C(11)–O(7)	62.1 (2.1)	78.3
H(8)–C(10)–C(11)–O(8)	–119.0 (2.1)	–101.0
H(7)–C(10)–C(11)–O(7)	–48.0 (2.1)	–40.1
N–C(10)–C(11)–O(8)	5.0 (0.3)	18.9
H(9)–N–C(10)–H(8)	–53.9 (3.8)	–62.6
H(9)–N–C(10)–H(7)	57.7 (3.8)	54.9
H(10)–N–C(10)–H(8)	62.3 (3.8)	59.7
H(10)–N–C(10)–C(11)	–59.8 (2.1)	–60.4
H(11)–N–C(10)–H(7)	–67.9 (3.8)	–64.4
H(11)–N–C(10)–C(11)	58.4 (2.1)	58.1

* The e.s.d.'s of the torsion angles in α -glycine are ~ 0.1 – 0.2° .

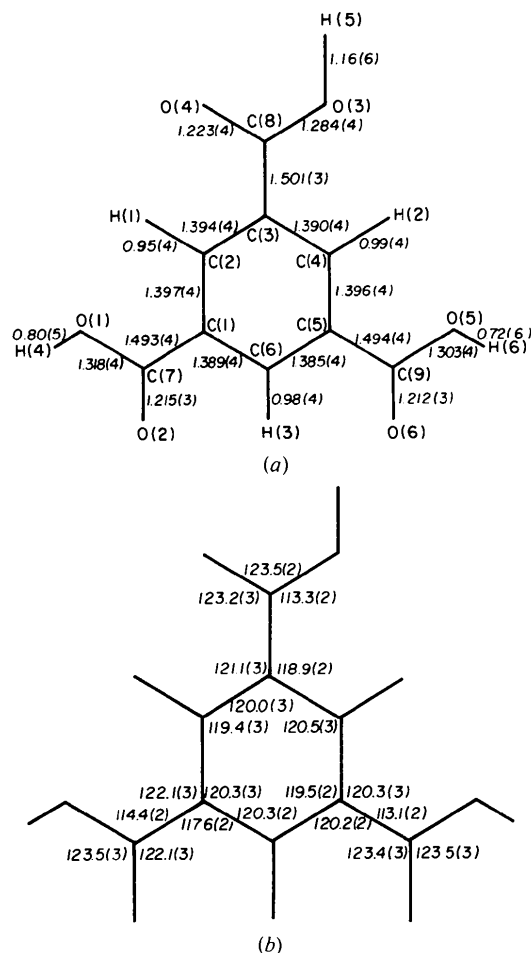


Fig. 4. (a) Bond lengths (Å) and numbering of TMA moiety, (b) bond angles ($^{\circ}$). Note that hydroxyl oxygens have been given odd numbers.

which is symmetry free and has an unsymmetrically located H atom [$d(\text{O}–\text{H}) = 1.141$ (3) Å, $d(\text{O}\cdots\text{H}) = 1.348$ (2) Å, by neutron diffraction], the corresponding values of Δr and $\Delta\theta$ are 0.073 Å and -3.3° for one carboxyl group and 0.047 Å and -0.2° for the other. We find 0.061 (6) Å and -9.9 (4)° in TMA and 0.043 Å and -5.0 (4)° in glycine. In neutral carboxyl groups $\Delta r \sim 0.09$ – 0.10 Å and $\Delta\theta \sim -9^{\circ}$ while in ionized groups both Δr and $\Delta\theta$ are essentially zero. Thus the carboxyl group of glycine is only partially ionized here and this influence carries over to the value of $d(\text{C}–\text{C})$ which is 1.511 (4) Å here compared to 1.526 (1) Å in the (completely ionized) α -glycine crystal.

4.4. Trimesic acid

The dimensions (Fig. 4; deviations from planarity in Table 2) do not differ appreciably from previous results except in the C(8) carboxyl group; these

dimensions show that this carboxyl group is partially ionized, although to a lesser extent than that of glycine.

4.5. Salt or molecular complex?

We have noted previously, in discussing the structure of histidinium trimesate (Herbstein & Kapon, 1979), that Johnson & Rumon's (1965) rule applies to that compound and many others, *i.e.* salts are found when $\Delta pK_a > 3.8$ and molecular complexes when $\Delta pK_a < 3.8$. In histidine the relevant pK_1 value is that of the imidazole nitrogen (which accepts a proton from one of

the TMA carboxyl groups), and the $H_3N^+-CH-COO^-$ zwitterion is not affected by the formation of the salt. In glycine.TMA.H₂O crystals the only possible proton acceptor in the glycine zwitterion is the ionized carboxyl group. TMA is a slightly stronger acid than glycine (pK_1 values, in dilute aqueous solution, are 2.12 and 2.34 respectively) and thus it is not surprising that the proton is shared almost equally between them. If these pK_1 values are relevant in the crystal, then one might expect the proton to be slightly closer to the glycine than to the TMA O atom; the converse is found in practice. The amino acids glycine, alanine, leucine, serine and glutamine all have pK_1 values for their carboxyl groups in the range 2.17–2.36 (and have no other ionizable groups). Thus, if these amino acids form complexes of analogous type with TMA, we might expect them to have nearly symmetrical hydrogen bonds like those found in glycine.TMA.H₂O.

Glycine.TMA.H₂O can be classified as an acid salt if we take the cation to be the NH_3^+ group of the glycine molecule (present as a zwitterion), while the two anions

are the partially ionized carboxyl groups of TMA and glycine, joined by a very short, nearly symmetrical, hydrogen bond.

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Structure of Bisadeninium Dinitrate Monohydrate, $(C_5H_6N_5^+ \cdot NO_3^-)_2 \cdot H_2O \cdot H_2O^* \dagger$

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Abstract

The title compound, $(C_5H_6N_5^+ \cdot NO_3^-)_2 \cdot H_2O$, is monoclinic, $P2_1/m$, with $a = 11.705$ (2), $b = 6.363$ (1), $c = 11.280$ (1) Å, $\beta = 104.241$ (6)°; $Z = 2$; $D_{calc} = 1.690$,

$D_{obs} = 1.70$ (2) Mg m⁻³. The structure was refined by a full-matrix least-squares method to $R(F) = 0.035$ for 1494 nonzero counter data to 0.84 Å. Analytical absorption and isotropic extinction corrections were applied. All hydrogen atoms were located and refined isotropically. E.s.d.'s of bond lengths and bond angles involving C, N, O atoms are 0.002 Å and 0.2°. The bases, and some of the other atoms, lie in the mirror planes at $y = \frac{1}{4}$ and $\frac{3}{4}$. The two independent adeninium ions are both protonated at N(1), and are part of an

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