The benzene ring is a slightly distorted regular hexagon. The average C–C distance is 1.382 Å. The deviations of the atoms of the ion from the least-squares plane defined by the six carbon atoms are shown in Fig. 6. The carbon ring is not significantly different from planar. The least-squares plane was calculated using a method described by Blow (1960). The orientation of the sulphonate group with respect to the carbon ring is such that atom O(1) is nearly in the plane of the ring. The angle between the least-squares plane and the plane defined by C(1), S and O(1) is 0.7° . The average O–S–O and C–S–O angles in the sulphonate group are 112·1 and $106\cdot2^{\circ}$ respectively. These angles are similar to those found in 2,5-dibromobenzenesulphonic acid



Fig. 6. Deviations of the atoms from the least-squares plane defined by the six carbon atoms of the 2,5-dichlorobenzene-sulphonate ion (Å).

trihydrate, 112.5 and 106.2° . The S–O distances found in the present compound 1.448, 1.426 and 1.419 Å are somewhat shorter than the S–O distances in the bromocompound, 1.463, 1.442 and 1.459 Å. (The distances are not corrected for thermal motion.)

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The Crystal Structures of Free Radical Salts and Complexes. I. (Morpholinium⁺)₂(7,7,8,8-Tetracyanoquinodimethane)₃²⁻

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Crystals of $(C_4H_{10}NO^+)_2(C_{12}H_4N_4)_3^2^-$ are monoclinic, belonging to the space group P_{21}/c , with lattice constants a = 7.04, b = 10.67, c = 26.51 Å, $\beta = 96.4^\circ$, and having Z = 2. The structure was solved from a three-dimensional Patterson synthesis and refined by block-diagonal least squares. It consists of columns of TCNQ molecules and anions packed plane-to-plane along the *b* axis. These columns are held together by the morpholinium ions through hydrogen bonding. The characteristic overlap and the short interplanar spacing of 3.25 Å indicate charge-transfer interaction between the TCNQ moieties.

Introduction

7,7,8,8-Tetracyanoquinodimethane (TCNQ) is a powerful electron acceptor and readily becomes the radical anion TCNQ⁻ by accepting an additional electron. As such, it forms salts with cations of many types, inorganic, organic and radical cations. Many of these salts exhibit large specific conductances (Melby, Harder, Hertler, Mahler, Benson & Mochel, 1962) for organic crystals. In addition, a number of complex salts containing extra molecules of formally neutral TCNQ have even lower resistivities. Crystal structure determinations have already been reported for a number of these simple and complex radical-ion salts but no general picture of the relationship between structure and electrical properties has emerged. To assist in the elucidation of this relationship a number of further structure determinations are being carried out. The present paper reports the work on the structure of the complex salt (morpholinium⁺)₂ (TCNQ)₃²⁻.

Experimental

Crystal data (C₄H₁₀NO)₂(C₁₂H₄N₄)₃, M.W.788. Monoclinic $a=7.04\pm0.02$, $b=10.67\pm0.04$, $c=26.51\pm0.06$ Å, $\beta=96.4\pm0.2^{\circ}$; U=1979.4 Å³; $D_m=1.317$ g.cm⁻³, Z=2, $D_c=1.322$ g.cm⁻³. F(000)=820 Mo K $\alpha(\lambda=0.7107$ Å), $\mu=0.94$ cm⁻¹. Systematic absences: h0l : l=2n+1 0k0 : k=2n+1Space group $P2_1/c$.

The material was prepared in the form of black lath-shaped crystals by mixing hot solutions in acetonitrile of TCNQ and morpholinium iodide. The space group and cell constants were deduced from oscillation and Weissenberg photographs with Cu $K\alpha$ radiation. The cell constants were refined subsequently on a Hilger and Watts linear diffractometer. Intensity data for 15 layers were collected with the crystal rotating about the *b* axis on the diffractometer with a scintillation counter and molybdenum $K\alpha$ radiation. Of the 4680 reflexions measured on the diffractometer significant counts were recorded for 2371. In view of the small size of the specimen, absorption corrections were deemed unnecessary. The intensities were corrected for Lorentz and polarization factors.

Structure determination

A three-dimensional Patterson map was computed using F_{o}^{2} as coefficients. Vectors typical of a TCNQ molecule were recognized round the origin and round large peaks at x = 0.000, y = 0.357, z = 0.027 and x =0.000, y = 0.280, z = -0.055. The former was taken to represent the combined vectors from a TCNQ moiety at the origin to one in a general position and the latter to represent combined vectors between a pair of centrosymmetrically related TCNQ moieties. (Two of the six TCNOs in the cell must lie in special positions on related centres of symmetry). The existence of a strong vector between the molecule at the origin and that in the general position suggests that these TCNQ moieties are packed parallel to each other. Thus the TCNQ at each centre of symmetry has a parallel centrosymmetrically related pair, one on either side of it, and the common orientation of the molecules in the group was derived from the vector pattern round the origin. Using the signs of the structure factors calculated from the coordinates of the TCNQ moieties so deduced, a

Table 1. Final positional and thermal parameters of non-hydrogen atoms ($\times 10^5$)

The figures in parentheses indicate the standard deviations.

Temperature factor = exp $[-(h^2B_{11}+k^2B_{22}+l^2B_{33}+hkB_{12}+hlB_{13}+klB_{23})]$

	x/a	y/b	z/c	B_{11}	B_{22}	B_{33}	B_{12}	B ₁₃	B_{23}
C(1)	- 19015 (28)	3830 (17)	253 (7)	1813 (44)	603 (14)	94 (3)	69 (42)	18 (17)	20 (10)
$\tilde{C}(2)$	-4919 (28)	4806 (16)	4605 (7)	2060 (46)	524 (14)	79 (3)	-173(40)	34 (17)	48 (9)
$\tilde{C}(3)$	14087 (27)	833 (17)	4145 (7)	1721 (43)	658 (15)	97 (3)	-271(40)	- 99 (17)	47 (10)
Č(4)	- 10034 (29)	9673 (17)	9128 (7)	2234 (49)	623 (15)	92 (3)	- 58 (44)	83 (18)	25 (10)
C(5)	3329 (33)	11022 (21)	13579 (8)	2773 (58)	872 (20)	93 (3)	27 (56)	36 (21)	-71(12)
C(6)	- 28974 (33)	13540 (19)	9697 (8)	2693 (59)	814 (18)	94 (3)	-216 (52)	247 (21)	-52(12)
C(7)	- 19064 (26)	39628 (17)	2613 (7)	1568 (40)	631 (15)	97 (3)	-60 (41)	57 (17)	-45 (10)
C(8)	-6261(26)	41191 (15)	7101 (6)	1757 (42)	567 (13)	73 (3)	- 348 (39)	26 (16)	10 (9)
C(9)	12874 (27)	37142 (16)	6998 (6)	1855 (43)	614 (15)	66 (3)	- 349 (40)	- 54 (16)	17 (9)
C(10)	18850 (27)	32182 (16)	2733 (7)	1636 (41)	635 (14)	75 (3)	- 69 (41)	- 55 (16)	-4 (10)
$\dot{C}(11)$	6043 (27)	30861 (15)	- 1784 (6)	1805 (42)	498 (13)	66 (2)	-105 (38)	-11 (16)	1 (9)
C(12)	-13110 (26)	34689 (16)	-1639 (6)	1666 (41)	638 (14)	60 (2)	-25 (38)	-61(15)	- 20 (9)
C(13)	12038 (28)	25705 (16)	-6252(7)	1783 (44)	588 (15)	76 (3)	-62 (40)	- 31 (17)	-11 (10)
C(14)	-1203 (29)	23842 (17)	- 10635 (7)	2050 (47)	643 (15)	88 (3)	26 (42)	169 (18)	- 26 (10)
C(15)	30712 (30)	21325 (18)	- 6584 (7)	2050 (48)	763 (17)	74 (3)	88 (44)	95 (18)	18 (10)
C(16)	-12639 (29)	46495 (17)	11514 (7)	2188 (48)	729 (16)	58 (2)	- 553 (45)	122 (16)	- 82 (10)
C(17)	-167 (30)	48419 (20)	16038 (7)	2016 (48)	917 (20)	95 (3)	- 690 (50)	171 (18)	-77 (12)
$\mathbf{C}(18)$	-31792 (29)	49962 (19)	11751 (7)	1924 (44)	844 (18)	85 (3)	- 446 (46)	185 (17)	-125 (11)
C(19)	65608 (39)	23767 (26)	23989 (9)	2828 (67)	1343 (28)	127 (4)	690 (70)	252 (25)	191 (16)
C(20)	67961 (37)	37684 (28)	24558 (11)	2545 (64)	1405 (31)	209 (5)	58 (69)	167 (27)	657 (20)
C(21)	38790 (35)	25198 (21)	28911 (9)	2529 (59)	843 (19)	154 (4)	27 (53)	206 (24)	142 (14)
C(22)	42128 (40)	39020 (21)	29240 (9)	3667 (73)	749 (20)	153 (4)	581 (62)	312 (26)	143 (14)
O(1)	50184 (25)	43540 (15)	24936 (7)	2983 (44)	945 (16)	196 (3)	300 (40)	157 (18)	497 (11)
N(1)	-44063 (32)	16700 (22)	10300 (8)	2857 (57)	1443 (26)	166 (4)	370 (62)	406 (23)	-81 (16)
N(2)	13431 (36)	12295 (24)	17204 (8)	4101 (73)	1420 (26)	126 (3)	-232 (68)	-216 (24)	-109 (14)
N(3)	-47285 (28)	52760 (20)	12086 (7)	2416 (48)	1143 (21)	144 (3)	- 357 (50)	238 (19)	-252 (13)
N(4)	9259 (31)	50372 (23)	19773 (7)	2749 (52)	1596 (26)	107 (3)	-771 (59)	-7 (19)	-186 (14)
N(5)	45809 (28)	17647 (20)	- 6953 (8)	2367 (50)	1220 (21)	156 (3)	551 (52)	207 (20)	20 (13)
N(6)	-11963 (30)	22293 (19)	- 14120 (7)	3026 (56)	1116 (20)	102 (3)	-209 (54)	- 308 (19)	-114 (12)
N(7)	56942 (26)	18683 (14)	28412 (6)	2468 (44)	620 (13)	106 (3)	-35 (39)	47 (16)	-15 (9)

three-dimensional Fourier electron-density distribution was computed. This confirmed the positions of the TCNQ moieties and showed the positions of all the atoms of the morpholinium ion. At this stage it was not certain which of the six atoms in the morpholinium ion were oxygen and nitrogen atoms so all were initially assumed to be carbon atoms and were given isotropic temperature factors of 3.00 Å^2 . Five cycles of block-



(b)

Fig. 1. Bond lengths (Å) and bond angles (°), with their standard deviations (in parentheses), for (a) the two independent TCNQ molecules (b) the morpholinium ion.



Fig. 2. The molecular arrangement, viewed along the *a* axis.

diagonal least-squares refinement of the positional parameters and isotropic thermal parameters gave an R value of 0.156. In the course of refinement two atoms of the morpholinium ion had consistently low temperature factors and the lowest also corresponded to the highest electron density peak in the Fourier election density map. It was therefore assumed to be the oxygen atom and the other, on the opposite side of the ring, was assumed to be nitrogen. This choice was subsequently confirmed by approximate equalization of all the morpholinium temperature factors and later by two short intermolecular contacts, interpreted as hydrogen bonds, made by what had been assumed to be the nitrogen atom.

The weighting scheme used was:

$$\forall w = 1$$
 if $|F_o| \le P_1$ or
= $\frac{P_1}{|F_o|}$ if $|F_o| > P_1$

H(1) H(2) H(3) H(4) H(5)

H(6)

H(7)

H(8) H(9)

H(10)

H(11) H(12)

H(13) H(14) H(15)

H(16)

where P_1 was given a value of 89.1 on the scale of Table 3.

After four further cycles of block-diagonal leastsquares refinement a difference Fourier synthesis showed peaks in plausible positions for all the hydrogen atoms. Two reflexions with largest F_c (102 and 102) were excluded from refinement because they were apparently affected by extinction. Further least-squares refinement on 2338 reflexions after excluding 31 weak reflexions, using anisotropic temperature factors for non-hydrogen atoms and constant isotropic temperature factors for hydrogen atoms, gave a final R value of 0.0645. The scattering factor curves of Cromer & Waber (1965) were used throughout. The final positional and thermal parameters of all the atoms, together with their standard deviations, are listed in Tables 1 and 2. The final calculated structure factors are compared with the observed values in Table 3. Least-squares planes were calculated through each of the molecules and are listed in Table 4.

 Table 2. Final positional and isotropic thermal parameters of hydrogen atoms

Positional	parameters	are	multiplied	by	103
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x/a	y/b	z/c	B(Å2)
-319(5)	58 (3)	6 (1)	3.42
238 (5)	21 (4)	67 (Ì)	3.56
316 (5)	294 (3)	29 (1)	3.19
227 (5)	380 (3)	101 (1)	3.16
-332(5)	416 (3)	28 (1)	3.42
-227(5)	336 (3)	-48 (1)	3.10
300 (6)	224 (4)	264 (1)	4.72
338 (6)	228 (4)	316 (2)	4.72
298 (6)	431 (4)	294 (2)	5.32
505 (6)	421 (4)	317 (2)	5.32
779 (6)	200 (4)	242 (2)	5.30
561 (6)	211 (4)	216 (2)	5.30
776 (6)	384 (5)	272 (2)	6.01
723 (6)	394 (4)	212 (2)	6.01
540 (5)	99 (4)	284 (1)	3.73
640 (5)	214 (3)	307 (1)	3.73

Description and discussion of the structure

Fig. 1(a) shows the dimensions of the two crystallographically independent types of TCNQ moiety. Aver-

age bond lengths of chemically similar bonds of both the TCNQ molecule and ion are summarized in Table 5. Though the averaged dimensions do not compare

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exactly with the previously published dimensions, a Table 3. Comparison of observed and final calculated structure factors - 201 - 162 - 421 - 152 - 155 - -44 205 251 -03 -03 -03 -03 -03

494

*•

-5. ..

13v 0 146 151 131 -247 -413 138 -299

Table 3 (cont.)

|--|--|--|--|

better indication of localization or delocalization of charge is obtained by comparing the differences between the two types of TCNQ moiety with the dif-



Fig. 3. Nearest neighbour overlap (a) molecules (A) and (B) (b) molecules (B) and (B').

ferences between TCNQ⁰ and TCNQ⁻. Some of the differences are individually only possibly significant, but they are all in consistent directions and approximately of the magnitude expected for the differences in dimensions between the ion and the molecule. A multivariate significance test (Cruickshank & Robertson, 1953) of the differences gives a value of $T^2 = 75.4$ which is highly significant for five degrees of freedom. This suggests that the centrosymmetric moiety (A) is TCNQ⁰ and the TCNQ moieties in the general positions (B) are TCNQ⁻. Molecule A is planar, but the molecule B does not have all its atoms coplanar due to the slight 2° twist of each C(CN)₂ plane from the plane of the quinonoid ring.

There is no ion with which the dimensions of the morpholinium ion, Fig. 1(b), may be compared directly. However the C-N distances averaging 1.479 Å may be compared with 1.497 Å in piperidinium chloride (Rérat, 1960*a*) and an average of 1.500 Å in piperazinium dichloride (Rérat, 1960*b*) and the C-N-C angle of 109.6° corresponds to the angles 112.3 and 110.6° in these two compounds respectively. The C-O distances averaging 1.414 Å and the C-O-C angle of 110.2°

may be compared with C–O distances of 1.388 and 1.428 Å in *trans*-2,5-dichloro-1,4-dioxan (Altona, Knobler & Romers, 1963) and 1.394 and 1.473 Å in *cis*-2,3dichloro-1,4-dioxan (Altona & Romers, 1963) and with the C–O–C angles 113·1° and 111·6° in these two compounds respectively. The C–C distances averaging 1.497 Å are a little shorter than the corresponding distances 1.507 Å in piperidinium chloride, 1.527 Å in piperazinium dichloride, 1.513 and 1.528 Å in the *trans*-2,5- and the *cis*-2,3-dichloro-1,4-dioxans, but the angles at the carbon atoms are very similar to those in the compounds cited.

The bond lengths and angles involving hydrogen atoms were calculated but are not considered sufficiently accurate to record. Within the rather large experimental error (σ for bond lengths approximately 0.04 Å and for bond angles approximately 2.7°) nearly all were normal.

Fig. 2. shows a general view of the structure in which the TCNQ moieties are seen almost edge-on, stacked very nearly parallel to each other (dihedral angle 1°28') along the *b* axis, which is 10.67 Å long. Fig. 2 also shows the hydrogen bonding which connects the morpholinium ions along the screw axis by NH····O hydrogen bonds of length 2.852 Å. This chain is also connected sideways to one nitrogen atom of TCNQ⁻ by NH····N hydrogen bonds of length 2.945 Å. Both these are normal hydrogen bond distances.

Fig. 3 shows the types of overlap of neutral TCNQ(A) projected onto TCNQ⁻(B) and for B' projected onto B (a centrosymmetrically related pair of TCNQ⁻ ions). The arrangements are very similar to those previously found (Fritchie & Arthur, 1966) for the only other 2:3 salt to have had its crystal structure determined, $(Cs^+)_2(TCNQ)_3^{2-}$. Both types of overlap minimize the direct overlap of atoms. These contacts, together with the other short intermolecular contacts, are listed in Table 6. The characteristic overlap and the short interplanar spacings of 3.25 Å (A-B) and 3.24 Å (B-B') indicate that there is charge transfer interaction between the TCNQ moieties.

Table 4. D	etails of	molecular	nlanes
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Х,	<i>Y,Z</i> are ort Molecule <i>A</i> (Equation to	hogonal atomic coordinates in Å w TCNQ°) the plane:	here X is along Molecule Fouation	a, Y is along b and Z is along c^* . B(TCNQ ⁻)
	0.2550X+0.	9224Y - 0.2900Z - 0.0000 = 0	0.2602X +	0.9132Y - 0.3136Z - 3.2502 = 0
	Atom	Distance from the plane (Å)	Atom	Distance from the plane (Å)
	C(1)	0.014	C(7)	0.025
	C(2)	-0.002	C(8)	0.007
	C(3)	-0.013	C(9)	-0.028
	C(4)	0.006	C(10)	-0.016
	C(5)	0.002	C(11)	0.029
	C(6)	-0.005	C(12)	0.037
	N(1)	-0.013	C(13)	0.040
	N(2)	0.007	C(14)	0.011
			C(15)	-0.015
			C(16)	0.008
			C(17)	0.016
			C(18)	-0.026
			N(3)	-0.068
			N(4)	0.041
			N(5)	-0.063
			N(6)	-0.022

Bond	а	b	с	d	е
* TCNQ° † TCNQ ^{1/2-} ‡ TCNQ ⁻ Δ (T ⁻ -T°) Molecule (<i>A</i>) Molecule (<i>B</i>) Δ (<i>B</i> - <i>A</i>)	$\begin{array}{c} 1.346 \ (4) \\ 1.354 \ (2) \\ 1.356 \ (6) \\ + 0.010 \\ 1.348 \ (3) \\ 1.355 \ (3) \\ + 0.007 \end{array}$	$\begin{array}{c} 1.448 (3) \\ 1.434 (2) \\ 1.425 (5) \\ -0.023 \\ 1.431 (3) \\ 1.419 (3) \\ -0.012 \end{array}$	$\begin{array}{c} 1.374 \ (4) \\ 1.396 \ (2) \\ 1.401 \ (6) \\ + \ 0.027 \\ 1.391 \ (3) \\ 1.415 \ (3) \\ + \ 0.024 \end{array}$	1.440 (3) 1.428 (3) 1.417 (5) -0.023 1.426 (3) 1.414 (3) -0.012	$\begin{array}{c} 1.138 \ (3) \\ 1.41 \ (3) \\ 1.156 \ (5) \\ + \ 0.018 \\ 1.141 \ (3) \\ 1.145 \ (3) \\ + \ 0.004 \end{array}$
	·		$\frac{b}{c}$		

Table 5. Comparison of mean bond lengths (uncorrected for libration)

* Long, Sparks & Trueblood (1965).

† Hanson (1968); Goldstein, Seff & Trueblood (1968).

‡ Hanson (1965); Fritchie & Arthur (1966); Fritchie (1966).

Table 6. Short intermolecular contacts

(a) Molecule A to Molecule B (stacking contacts of 3.400 Å or less) $C(1^{1}) -C(12^{1}) = 3.363$ Å

$C(1^{i}) - C(12^{i})$	3.363
$C(1^{i}) - C(15^{i})$	3.318
$C(2^{i}) - C(11^{i})$	3.389
$C(2^{i}) - C(13^{i})$	3.330
$C(2^{i}) - C(15^{i})$	3.400
$C(3^{i}) - C(10^{i})$	3.386
$C(3^{i}) - C(14^{i})$	3.326
$C(5^{i}) - C(9^{i})$	3.395

(b) Molecule B to molecule B' (stacking contacts less than $3\cdot300$ Å)

$C(7^{i}) - C(11^{iii})$	3·294 Å
$C(8^{i}) - C(11^{iii})$	3.299
$C(13^{i})-C(16^{iii})$	3.280
$C(14^{i})-C(17^{iii})$	3.294
$C(17^{i}) - N(6^{iii})$	3.294
$N(4^{i}) - N(6^{iii})$	3.294

(c) Hydrogen bonds

$O(1^{i}) - N(7^{iv})$	2·852 Å
$N(7^{i}) - N(6^{v})$	2.945

(d) Other non-stacking intermolecular contacts less than 3.400 Å.

$C(1^{i}) - N(5^{viii})$	3∙302 Å
$C(19^{i}) - N(4^{vii})$	3.383
$C(19^{i}) - N(6^{v})$	3.390
$C(20^{i}) - N(6^{v})$	3.345
$C(21^{i}) - N(3^{ix})$	3.386
$O(1^{i}) - N(4^{i})$	3.135
$N(3^{i}) - N(7^{v_{i}})$	3.174
$N(4^{i}) - N(7^{iv})$	3.075

Superscripts indicate equivalent positions as follows: -

i	x, y, z	vi	$\bar{x}, \frac{1}{2} + y, \frac{1}{2} - z$
ii	$ar{x}, ar{y}, ar{z}$	vii	$1-x, y-\frac{1}{2}, \frac{1}{2}-z$
iii	$\bar{x}, 1-y, \bar{z}$	viii	x - 1, y, z
iv	$1-x, \frac{1}{2}+y, \frac{1}{2}-z$	ix	$\vec{x}, y - \frac{1}{2}, \frac{1}{2} - z$
v	$1+x, \frac{1}{2}-y, \frac{1}{2}+z$		

Standard deviations are all within the range 0.002 to 0.003 Å

Confirmation of the fact that the TCNQ molecule and ion retain their separate identities in the complex salt is found in the solid state infrared spectrum which is virtually the superposition of bands due to TCNQ⁰, TCNQ⁻ and the morpholinium ion. The electronic spectrum also shows peaks characteristic of each of the TCNQ species as well as a charge-transfer band centred at about 8500 cm⁻¹ due to donor-acceptor interaction between TCNQ⁻ and TCNQ⁰.

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Structure Moléculaire et Cristalline du Tétraphényl-1,4,5,8 Naphtalène

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The structure of 1,4,5,8-tetraphenylnaphthalene has been determined by X-ray analysis. Space group $P2_1/c$, Z = 2, a = 6.455, b = 24.333, c = 8.017 Å, $\beta = 114^{\circ}3$. The molecule has 2/m point group symmetry. The naphthalene nucleus has a boat-like conformation. The maximum angle between the mean planes of the phenyl groups and that of the central naphthalene molecule is 58° .

Introduction

Ce travail fait partie d'un ensemble d'études expérimentales sur l'encombrement stérique intramoléculaire. Il fait suite à deux recherches publiées d'autre part (Evrard, Piret, Germain & Van Meerssche, 1971; Evrard, Piret & Van Meerssche, 1971) sur les structures du tétraphényl-1,2,3,4 cyclopentadiène et du bromo-5 tétraphényl-1,2,3,4 cyclopentadiène.

La détermination de la structure du tétraphényl-

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