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 MERCURATION OF α - AND γ -METHYLPYRYLIUM SALTS
 WITH MERCURIC ACETATE AND TRIFLUOROACETATE

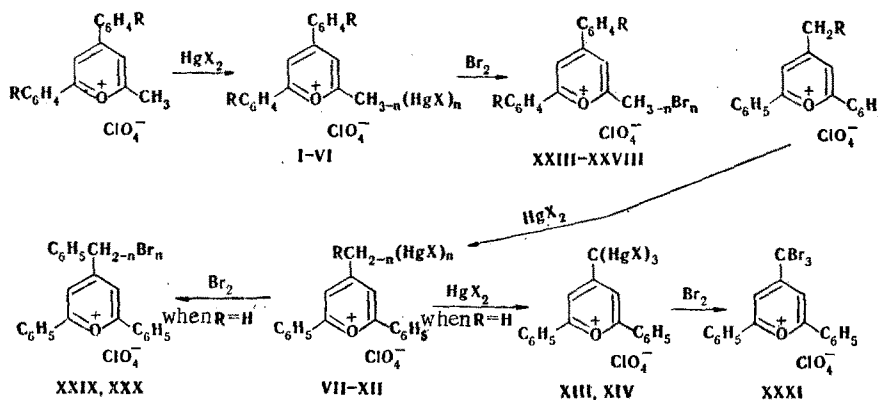
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Mercury-containing methylpyrylium perchlorates were synthesized by mercuration of α - and γ -methylpyrylium perchlorates with mercuric acetate and trifluoroacetate. A mechanism is proposed for the mercuration reaction. The salts obtained undergo decomposition upon reaction with mineral acids to give the starting methylpyrylium salts, while bromomethylpyrylium perchlorates are formed upon reaction with bromine as a result of cleavage of the C-Hg bond.

The high lability of the hydrogen atoms of the methyl groups in α - and γ -methylpyrylium salts is widely known. These salts readily undergo condensation with various aldehydes [1] to give styrylpyrylium salts, even with such inactive carbonyl-containing compounds as dimethylformamide (DMF) [2]; the hydrogen atoms of the methyl groups are rapidly exchanged by deuterium upon refluxing in D₂O [3] and also react smoothly via the methyl group with diazonium salts [4].

One might have assumed that methylpyrylium salts would react with mercury salts, which are known [5] to be effective mercuration agents of compounds with labile hydrogen atoms. As we established for the first time in the present research, 2-methyl-4,6-diphenylpyrylium and 2-methyl-4,6-(*p*-ethoxyphenyl)pyrylium perchlorates are readily mercured by mercuric acetate and trifluoroacetate to give products of replacement of the hydrogen atoms of the methyl group by HgX (X = CH₃COO and CF₃COO) in high yields. The reactions were carried out by brief refluxing of the substrates cited above in ethanol, and, depending on their ratio, products of mono- (I, II), di- (III, IV), and trimercuration (V, VI) were obtained, and the formation of acetic or trifluoroacetic acid, which we were able to isolate in the form of the ethyl ester, was also observed.

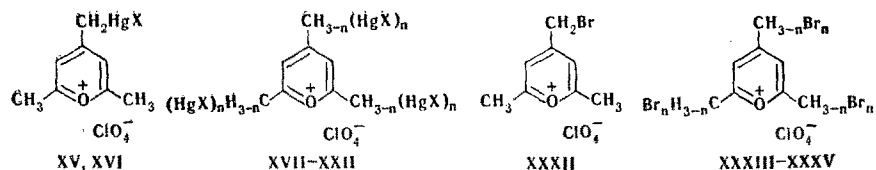


I, III, V, VII, VIII, X, XI, XXIII, XXV, XXVII R=H; II, IV, VI, XXIV, XXVI, XXVIII R=*p*-C₆H₄O; IX, XII R=Ph; I, III, V, VII, IX, X, XII, XIII X=CH₃COO; II, IV, VI, VIII, XI, XIV X=CF₃COO; I, II, VII-IX, XXIII, XXIV, XXIX n=1; III, IV, X-XII, XXV, XXVI, XXX n=2; V, VI, XXVII, XXVIII n=3

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4-Methyl- and 4-benzyl-2,6-diphenylpyrylium perchlorates are mercurated under similar conditions to give products of mono- (VII-IX), di- (X-XII), and trimercation (XIII, XIV); the ethyl esters of acetic and trifluoroacetic acids were also isolated in this case.

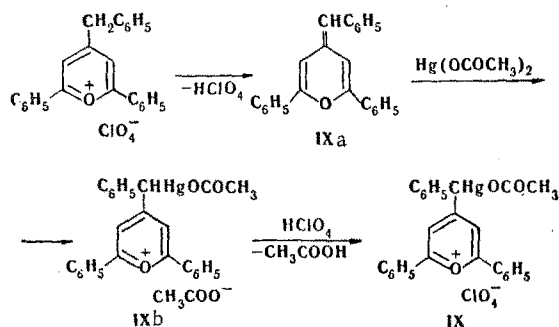
As a result of the mercuration of 2,4,6-trimethylpyrylium perchlorate with mercuric acetate and trifluoroacetate under the conditions presented above and at the corresponding molar ratios of the substrates we were able to obtain the perchlorates of the products of monomercuration (XV, XVI) and tri- (XVII, XVIII), hexa- (XIX, XX), and even nonamercuration (XXI, XXII) by replacing all of the hydrogen atoms of the methyl groups.



XV, XVII, XIX, XXI X=CH₃COO; XVI, XVIII, XX, XXII X=CF₃COO; XVII, XVIII, XXXIII n=1; XIX, XX, XXXIV n=2; XXI, XXII, XXXV n=3

It is known from the literature data [6, 7] that the methyl group in the γ position of the pyrylium ring of 2,4,6-trimethylpyrylium perchlorate has higher nucleophilic activity than the methyl groups in the α positions. In the monomercuration of this salt one should therefore have expected the formation of products XV and XVI with the structure precisely presented. From steric considerations, one may propose a structure for salts XVII-XX such as that represented in the scheme, although a different orientation of the HgX groups is also possible. Unfortunately, one cannot make a study of XV-XXII by PMR spectroscopy to determine the positions of the substituents in these compounds because of their very low solubilities in organic solvents.

It may be assumed that the mechanism of the mercuration reaction consists in the addition of the mercury salts to the double bond of the exocyclic methylene group of the corresponding methylenepyran (IXa), which is formed at the instant of the reaction and is the anhydro base of the starting methylpyrylium salt (for example, 4-benzyl-2,6-diphenylpyrylium perchlorate).



The fact that the liberation of free perchloric acid, which can act as the most likely catalyst for the esterification of the resulting acetic and trifluoroacetic acids by ethanol, is proposed in the reaction process, is a confirmation of the assumptions relative to this mechanism for the mercuration of methylpyrylium salts. As we have already emphasized, we isolated the ethyl esters of these acids. A second fact that confirms the reaction mechanism presented above is the ability of pyran IXa to add mercuric acetate [8] under the conditions of the mercuration of methylpyrylium salts. The resulting monomercuration product (IXb) [8] under the influence of perchloric acid readily exchanges the acetate anion for ClO₄⁻ to give IX, samples of which that were synthesized by various methods proved to be identical with respect to their physicochemical properties and IR spectra.

Mercurated salts I-XXII are colored crystalline substances that are slightly soluble in organic solvents. Compounds I-XXII undergo decomposition to give the starting methylpyrylium salts under the influence of concentrated aqueous solutions of strong mineral and organic acids (hydrochloric, hydrobromic, perchloric, and trifluoroacetic acids).

In the IR spectra of salts I-XXII (Table 1) the variations of the pyrylium cation appear at 1600-1630 and 1500 cm⁻¹ [9], while the stretching vibrations of the ClO₄⁻ ion appear at

TABLE 1. Mercury-Containing Methylpyrylium Salts (I-XXII)

Com- pound	Reaction time, min	mp, °C	IR spectrum, cm ⁻¹	Empirical formula	Hg, %		Yield, %
					found	calc.	
1	2	3	4	5	6	7	8
I	15	261—262	1620, 1590, 1573, 1508, 1487, 1415, 1225, 1183, 1095, 1000, 950, 920, 865, 840, 767, 720	C ₂₀ H ₁₇ ClHgO ₇	32,9	33,1	91
II	10	244—245	1682, 1620, 1582, 1508, 1300, 1241, 1175, 1080, 1032, 918, 825, 730, 717	C ₂₃ H ₂₂ ClF ₃ HgO ₉	27,6	27,3	99
III	20	281—282	1650, 1610, 1570, 1490, 1418, 1320, 1257, 1226, 1195, 1165, 1090, 1000, 925, 875, 768, 720, 680	C ₂₂ H ₁₉ ClHg ₂ O ₉	46,8	46,4	92
IV	15	256—257 ^a	1690, 1620, 1582, 1566, 1510, 1305, 1245, 1176, 1110, 1083, 1035, 960, 916, 826, 727, 716	C ₂₅ H ₂₁ ClF ₆ Hg ₂ O ₁₁	38,1	38,3	86
V	30	270 ^b	1655, 1615, 1575, 1490, 1335, 1250, 1190, 1100, 1015, 940, 780, 735	C ₂₄ H ₂₁ ClHg ₃ O ₁₁	53,9	53,6	89
VI	30	288—290 ^a	1684, 1615, 1582, 1508, 1302, 1240, 1177, 1080, 1032, 917, 827, 728, 715	C ₂₇ H ₂₀ ClF ₉ Hg ₃ O ₁₃	44,4	44,2	70
VII	15	253—254	1615, 1573, 1518, 1485, 1310, 1270, 1199, 1160, 1100, 1000, 950, 925, 880, 840, 778, 727	C ₂₀ H ₁₇ ClHgO ₇	32,9	33,1	82
VIII	15	275 ^b	1685, 1615, 1575, 1520, 1487, 1310, 1275, 1198, 1165, 1090, 1000, 950, 924, 882, 835, 785, 730	C ₂₀ H ₁₄ ClF ₃ HgO ₇	30,3	30,4	83
IX	15	208—209	1680, 1612, 1568, 1500, 1300, 1260, 1200, 1140, 1085, 990, 960, 940, 920, 880, 835, 750, 712	C ₂₆ H ₂₁ ClHgO ₇	29,4	29,4	73
X	20	268 ^b	1610, 1571, 1518, 1480, 1315, 1275, 1200, 1100, 1002, 955, 930, 877, 845, 780, 725, 683	C ₂₂ H ₁₉ ClHg ₂ O ₉	47,0	46,4	92
XI	20	275 ^b	1685, 1610, 1570, 1518, 1483, 1265, 1190, 1090, 1000, 950, 925, 875, 850, 775, 720, 680	C ₂₂ H ₁₃ ClF ₆ Hg ₂ O ₉	41,5	41,3	77
XII	25	278—279	1620, 1555, 1500, 1305, 1220, 1165, 1080, 1010, 915, 880, 840, 745, 710	C ₂₈ H ₂₃ ClHg ₂ O ₉	42,1	42,7	74
XIII	30	285 ^b	1695, 1607, 1572, 1512, 1480, 1316, 1274, 1238, 1200, 1185, 1100, 1000, 955, 935, 873, 842, 778, 720, 683	C ₂₄ H ₂₁ ClHg ₃ O ₁₁	53,7	53,6	85
XIV	30	285 ^b	1660, 1617, 1578, 1515, 1200, 1185, 1090, 1000, 950, 933, 872, 800, 775, 718	C ₂₄ H ₁₂ ClF ₉ Hg ₃ O ₁₁	46,2	46,8	58
XV	10	252—253	1650, 1635, 1540, 1510, 1320, 1250, 1145, 1095, 980, 910, 882, 730	C ₁₀ H ₁₃ ClHgO ₇	42,2	41,7	99
XVI	10	214—215	1685, 1645, 1540, 1510, 1317, 1250, 1183, 1150, 1095, 1025, 972, 937, 900, 825, 720	C ₁₀ H ₁₀ ClF ₃ HgO ₇	37,4	37,5	93
XVII	15	270 ^b	1620, 1576, 1510, 1310, 1198, 1150, 1095, 970, 915, 865, 730	C ₁₄ H ₁₇ ClHg ₃ O ₁₁	60,7	60,3	99
XVIII	15	270 ^b	1680, 1620, 1500, 1320, 1240, 1187, 1150, 1080, 975, 920, 850, 786, 720	C ₁₄ H ₈ ClF ₉ Hg ₃ O ₁₁	52,3	51,8	90
XIX	30	250 ^b	1620, 1575, 1315, 1185, 1150, 1080, 925, 732, 720, 687	C ₂₀ H ₂₃ ClHg ₅ O ₁₇	68,2	67,8	96
XX	30	210—215 ^c	1670, 1615, 1575, 1410, 1200, 1160, 1085, 930, 860, 805, 785, 740, 727	C ₂₀ H ₅ ClF ₁₈ Hg ₆ O ₁₇	57,3	57,4	88
XXI	120	265 ^b	1690, 1620, 1575, 1508, 1310, 1196, 1085, 960, 925, 870, 755, 718, 680	C ₂₀ H ₂₉ ClHg ₉ O ₂₃	70,8	70,8	96
XXII	120	210—215 ^c	1675, 1610, 1580, 1410, 1200, 1155, 1085, 930, 855, 785, 735	C ₂₆ H ₂ ClF ₂₇ Hg ₉ O ₂₃	60,2	59,4	89

^aWith decomposition. ^bDecompose explosively. ^cDecompose without melting.

TABLE 2. Bromine-Containing Methylpyrylium Salts (XXIII-XXXV)

Compound	mp, °C	IR spectrum, cm ⁻¹	Empirical formula	Br+Cl, %		Yield, %
				found	calc.	
XXIII	204—205	1620, 1579, 1500, 1488, 1400, 1243, 1185, 1080, 988, 950, 880, 800, 762, 710, 682, 657	C ₁₈ H ₁₄ BrClO ₅	26,3	27,1	71
XXIV	184—185 ^a	1630, 1580, 1520, 1493, 1292, 1238, 1170, 1080, 1025, 940, 910, 832, 800, 711, 654, 1	C ₂₂ H ₂₂ BrClO ₇	22,0	22,5	97
XXV	241—242 ^b	1618, 1580, 1500, 1486, 1400, 1244, 1184, 1085, 987, 948, 882, 800, 761, 710, 655	C ₁₈ H ₁₃ Br ₂ ClO ₅	38,6	38,7	94
XXVI	164—165	1626, 1580, 1520, 1490, 1300, 1240, 1171, 1190, 1022, 945, 910, 867, 832, 800, 713	C ₂₂ H ₂₁ Br ₂ ClO ₇	32,5	33,0	74
XXVII	228—229 ^a	1735, 1615, 1574, 1500, 1480, 1238, 1180, 1075, 980, 945, 915, 875, 760, 715, 685, 656	C ₁₈ H ₁₂ Br ₃ ClO ₅	46,8	47,2	83
XXVIII	151—152 ^a	1735, 1630, 1579, 1520, 1490, 1400, 1300, 1264, 1240, 1173, 1100, 1039, 1020, 950, 915, 865, 835, 800, 754, 713, 655	C ₂₂ H ₂₀ Br ₃ ClO ₇	41,1	41,0	99
XXIX	239—240 ^a	1622, 1582, 1565, 1500, 1330, 1295, 1200, 1197, 1155, 1080, 985, 945, 922, 830, 800, 770, 710, 690, 668	C ₁₈ H ₁₄ BrClO ₅	26,4	27,1	86
XXX	214—215 ^c	1612, 1585, 1562, 1331, 1300, 1260, 1200, 1152, 1075, 985, 946, 920, 832, 800, 772, 710, 667	C ₁₈ H ₁₃ Br ₂ ClO ₅	38,0	38,7	99
XXXI	277—278	1730, 1609, 1580, 1564, 1485, 1415, 1250, 1200, 1154, 1075, 985, 944, 920, 860, 830, 770, 745, 710, 690, 665	C ₁₈ H ₁₂ Br ₃ ClO ₅	46,3	47,2	98
XXXII	151—152	1700, 1638, 1530, 1505, 1308, 1244, 1085, 1018, 968, 930, 900, 865, 710, 655	C ₈ H ₁₀ BrClO ₅	38,0	38,3	98
XXXIII	137—138 ^a	1700, 1638, 1600, 1535, 1300, 1240, 1200, 1140, 1080, 1015, 910, 710, 655	C ₈ H ₈ Br ₃ ClO ₅	58,8 ^d	59,9	99
XXXIV	153—154 ^a	1600, 1300, 1200, 1135, 1080, 912, 910, 750, 710, 651	C ₈ H ₈ Br ₅ ClO ₅	72,9 ^d	74,0	99
XXXV	180—182 ^a	1605, 1300, 1130, 1070, 962, 915, 750, 711, 656	C ₈ H ₂ Br ₉ ClO ₅	80,0 ^d	81,0	99

^aWith decomposition. ^bAccording to the data in [11], this compound has mp 230–231°C. ^cAccording to the data in [11], this compound has mp 239–240°C. ^dThe analyses were performed by the method in [13].

1100 cm⁻¹. Numerous absorption bands at 1450–1600 and 650–1000 cm⁻¹ characterize the vibrations of the aromatic rings and the out-of-plane deformation vibrations of the C–H bonds of these rings [10]. The absorption bands at 1680 cm⁻¹ correspond to the stretching vibrations of the C=O group, while the bands at 1170–1200 cm⁻¹ correspond to the vibrations of the C–O bond.

The mercury-containing pyrylium salts I-XXII that we obtained react with electrophilic reagents such as halogens. As a result of bromination of I-XXII in glacial acetic acid, the C–Hg bond is cleaved to give the corresponding bromomethyl-substituted pyrylium perchlorates (XXIII–XXXV) and mercury salts (BrHgX); a halogen atom takes the place of each

mercury-containing substituent (depending on the amount of bromine used in the reaction). Thus, considering the accessibility of the starting I-XXII, the simplicity of the experiment, and the high yields of desired products XXIII-XXXV, this reaction can be recommended as a convenient method for the preparation of difficult-to-obtain [11] bromomethyl-substituted pyrylium salts.

Absorption bands that are characteristic for the pyrylium cation, the perchlorate anion, and phenyl rings are present in the IR spectra of bromides XXIII-XXXV (Table 2), whereas the absorption band at 1680 cm^{-1} that is related to the vibrations of the C=O group is absent. The presence of a strong absorption band at $650\text{--}655\text{ cm}^{-1}$ is characteristic for the vibrations of C-Br bonds [10] in XXIII-XXXV.

EXPERIMENTAL

The IR spectra of mineral oil suspensions of the compounds were recorded with a Specord 71 IR spectrometer.

Mercury-Containing Methylpyrylium Salts (I-XXII, Table 1). A mixture of 1 mmole of the methylpyrylium perchlorate and the necessary amount of the mercury salt (1, 2, 3, 6, or 9 mmole) in 5-10 ml of ethanol was refluxed with stirring for the time indicated in Table 1, and the resulting precipitated mercurated salts were removed by filtration, washed with ether, dried, and crystallized from glacial acetic acid. In the isolation of XX and XXII the solvent was removed by vacuum distillation, while the solid residues were purified as indicated above. The ethyl esters of acetic or trifluoroacetic acid were isolated from the combined filtrates obtained from five to six parallel experiments. For the isolation of ethyl acetate, 40-50 ml of a saturated aqueous solution of NaCl was added to the filtrate (25-50 ml), and the mixture was stirred vigorously for 5 min. It was then filtered and allowed to stand in a separatory funnel for 1 h. The upper part of the liquid (2-3 ml) was separated, 1-1.5 g of anhydrous Na_2SO_4 was added, and the mixture was allowed to stand overnight. It was then distilled from a small flanged flask with collection of the fraction with bp $77\text{--}78^\circ\text{C}$ to give 0.2-0.8 ml (35-50%) of ethyl acetate.

In the isolation of ethyl trifluoroacetate the filtrate was distilled slowly with a 50-cm long rod-and-disk type fractionating column with collection of the fraction boiling up to 70°C (1.5-2.5 ml). This liquid was then distilled from a small flanged flask with collection of the fraction with bp $61\text{--}62^\circ\text{C}$ to give 0.3-1 ml (45-60%) of ethyl trifluoroacetate.

2,6-Diphenyl-4-(α -acetomercuribenzyl)pyrylium Acetate (IXb). A mixture of 0.32 g (1 mmole) of 2,6-diphenyl-4-benzylidene-4H-pyran (IXa) [12] and 0.32 g (1 mmole) of mercuric acetate in 5 ml of ethanol was refluxed for 15 min, after which it was cooled, and the resulting precipitate was removed by filtration, washed with ether, and dried to give 0.5 g (78%) of IXb with mp $202\text{--}203^\circ\text{C}$ (mp $201\text{--}202^\circ\text{C}$ [8]).

2,6-Diphenyl-4-(α -acetomercuribenzyl)pyrylium Perchlorate (IX). A 0.1-m (0.68 mmole) sample of 70% perchloric acid was added to a solution of 0.32 g (0.5 mmole) of acetate IXb in 5 ml of glacial acetic acid. After 30 min, the precipitate was removed by filtration, washed with ether, dried, and crystallized from glacial acetic acid to give 0.18 g (53%) of IX with mp $208\text{--}209^\circ\text{C}$; no melting-point depression was observed for a mixture with IX obtained by other methods.

Reaction of Mercury-Containing Pyrylium Salts (I-XXII) with Acids. A 3-ml sample of a concentrated aqueous solution of HCl was added to 0.5 mmole of I-XXII, and the mixture was refluxed for 10 min. It was then cooled and treated with 0.2 ml (1.36 mmole) of 70% perchloric acid. The precipitated pyrylium perchlorates were removed by filtration, washed with 5 ml of water, dried, and crystallized from suitable solvents. The pyrylium salts obtained had the same melting points as the starting salts, and no melting-point depressions were observed for mixtures with them.

The reactions with concentrated hydrobromic acid, 56% perchloric acid, and anhydrous trifluoroacetic acid were carried out similarly.

Bromine-Containing Methylpyrylium Salts (XXIII-XXXV, Table 2). The necessary amount of bromine (1 mmole of bromine for each HgX group) was added with stirring to a hot solution of 1 mmole of the corresponding mercury-containing pyrylium salt (I-XXII) in 10-20 ml of glacial acetic acid, and the mixture was heated to the boiling point and filtered. The

filtrate was cooled and treated with 20 ml of ether, and the precipitated bromomethylpyrylium salts XXIII-XXXV were removed by filtration, washed with ether, and crystallized from glacial acetic acid.

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SYNTHESIS AND PROPERTIES OF BIS(PYRIDYLMETHYLENE)MERCURY

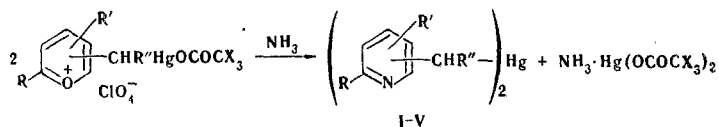
DERIVATIVES*

V. I. Boev and A. V. Dombrovskii

UDC 547.828.254.9

The action of ammonia on α - and γ -monomercurimethylenepyrylium perchlorates gave bis(pyridylmethylen)mercury derivatives, which undergo decomposition to the corresponding pyridine derivatives and mercuric chloride.

In our preceding communication [1] we described the syntheses of mercury-containing methylpyrylium perchlorates, which are of definite interest for the synthesis of various heteroorganic heterocyclic systems. It is known that under the influence of ammonia pyrylium salts are readily converted to the corresponding nitrogen-containing heterocyclic compounds [2]. We have found that bis(pyridylmethylen)mercury derivatives (I-V) can be obtained by the action of dry ammonia on α - and γ -monomercurimethylenepyrylium perchlorates [1] in anhydrous chloroform; in addition to conversion of the pyrylium cation to a pyridine ring, the starting substrates undergo symmetrization under the influence of ammonia [3] to give completely substituted organomercury compounds (I-V).



X=H, F; I R=CH₃, R'=2-C₆H₅, R''=H; II R=C₆H₅, R'=2-C₆H₅, R''=H; III R=C₆H₅, R'=2-C₆H₅, R''=C₆H₅; IV R=C₆H₅, R'=4-C₆H₅, R''=H; V R=p-C₆H₄OC₂H₅, R''=H

Compounds I-V are colorless or slightly colored crystalline substances that are quite soluble in organic solvents and mineral acids. The structure of I-V is confirmed by data

*See [1] for our preceding communication.

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