DIALKYLPHOSPHINOBENZOIC ACIDS AND SOME OF THEIR DERIVATIVES

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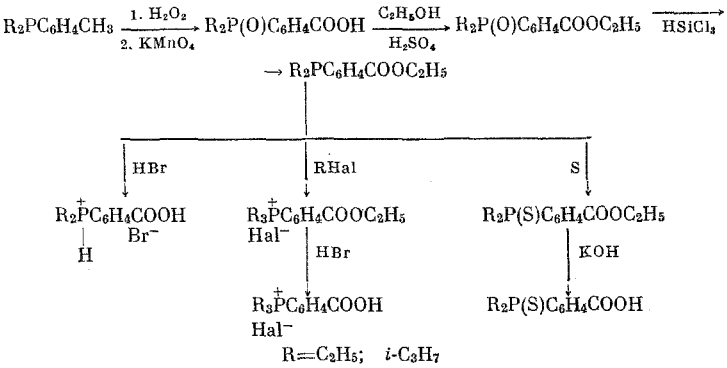
In a previous communication [1] we described a method for the preparation of the m- and p-dimethylphosphinobenzoic acids and their analogs that contain pentavalent phosphorus. In the present paper we synthesized the m- and p-phosphino-, phosphinyl-, thiophosphinyl-, and phosphoniobenzoic acids that contain ethyl and isopropyl radicals on the phosphorus atom. These compounds were needed for a study of the electronic effects of phosphorus-containing groupings. Of the compounds in which we were interested only p-diethylphosphinylbenzoic acid is known [2, 3]. To synthesize the m- and p-dialkylphosphinobenzoic acids and their analogs that contain pentavalent P we employed the scheme described in [1]



The dialkyl-m- and dialkyl-p-tolylphosphine oxides served as the starting compounds, which were converted by oxidation with $KMnO_4$ [4] to the m- and p-dialkylphosphinylbenzoic acids, and then to their esters. The reduction of the latter with silicochloroform [1] led to the phosphinobenzoic acid esters, which by treatment with sulfur or alkyl halides were converted to the corresponding phosphine sulfides and phosphonium derivatives. The alkaline or acid hydrolysis of the obtained phosphorus-substituted benzoic acid esters, respectively, led to the m- and p-dialkylphosphinyl-, dialkylphosphino-, dialkylthiophosphinyl-, and trialkylphosphoniobenzoic acids. The phosphinobenzoic acids were isolated as the hydrobromides.

The starting dialkyltolylphosphine oxides were synthesized mainly from the corresponding dialkylchlorophosphines and the m- and p-bromotoluenes via the organomagnesium compounds, and subsequent oxidation of the intermediate tertiary phosphines. In addition, p-tolyldichlorophosphine was used as the starting compound to synthesize the diethyl- and diisopropyl-p-tolylphosphines [1].

The ethyl ester of p-diethylphosphinobenzoic acid was also obtained from p-carbethoxyphenyldichlorophosphine, the synthesis of which was worked out by us previously [5]

$$Cl_2PC_6H_4COOC_2H_5 \xrightarrow{C_2H_5MgBr} (C_2H_5)_2PC_6H_4COOC_2H_5$$

However, this method lacks any advantages over that described above. The yields, constants, and analytical data of the obtained compounds are given in Table 1.

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TABLE 1. Yields, Constants, and Analyses of Obtained Compounds

Compound	Formula	Yield, %	Bp, °C (p, mm of Hg)	Mp, °C	n_D^{20}	d_4^{20}	MR found	MR calculated	Found, % C	Found, % H	Found, % P	Empirical formula	Calculated, % C	Calculated, % H	Calculated, % P
(I)	p-$(C_2H_5)_2PC_6H_4CH_3$ [6—9]	90	108—110(13)		1,5435	0,9333	60,91	59,64				$C_{11}H_{17}P$	73,3	9,5	17,2
(II)	m-$(C_2H_5)_2PC_6H_4CH_3$ [10]	76	113—115(12)		1,5416	0,9385	60,46		73,3	9,6	17,0				
(III)	p-$(C_2H_5)_2P(O)C_6H_4CH_3$ [2]	95	148—151(5)	73—74								$C_{11}H_{17}OP$	67,3	8,7	15,8
(IV)	m-$(C_2H_5)_2P(O)C_6H_4CH_3$	90	153—155(3)	56—58					67,0	8,8	15,6				
(V)	p-$(C_2H_5)_2P(O)C_6H_4COOH$ [2]	74		202—203 (water)								$C_{11}H_{15}O_3P$	58,4	6,7	13,7
(VI)	m-$(C_2H_5)_2P(O)C_6H_4COOH$	73		148—149 (water)					58,4	6,8	13,9				
(VII)	p-$(C_2H_5)_2P(O)C_6H_4COOC_2H_5$	92	202—203(15)	101—102 heptane-benzene, 2 : 1)					61,5	7,5	12,3	$C_{13}H_{19}O_3P$	61,4	7,5	12,2
(VIII)	m-$(C_2H_5)_2P(O)C_6H_4COOC_2H_5$	77	179—180(4)	43—45 (heptane—CCl_4, 1 : 1)					61,6	7,5	12,2				
(IX)	p-$(C_2H_5)_2PC_6H_4COOC_2H_5$ [11]	a) 98 b) 80	131—132(4)		1,5461	1,0333	73,03	70,53				$C_{13}H_{19}O_2P$	65,5	8,0	13,0
(X)	m-$(C_2H_5)_2PC_6H_4COOC_2H_5$	94	129—130(2)		1,5384	1,0311	72,32		65,6	8,0	13,0				
(XI)	p-$(C_2H_5)_2P(S)C_6H_4COOC_2H_5$	77		48,5—49,5 (hexane)					57,6 57,7	6,9 7,1	11,3 11,3	$C_{13}H_{19}O_2PS$	57,8	7,1	11,5
(XII)	m-$(C_2H_5)_2P(S)C_6H_4COOC_2H_5$	87	190—192(4)		1,5697	1,1276	78,64	76,59	58,0	7,0	11,8				
(XIII)	p-$(C_2H_5)_2P(S)C_6H_4COOH$	70		186—187benzene)					54,9	6,1	12,7	$C_{11}H_{15}O_2PS$	54,5	6,2	12,8
(XIV)	m-$(C_2H_5)_2P(S)C_6H_4COOH$	69		135—136 (water—alcohol, 20 : 1)					54,5	6,2	12,8				
(XV)	p-$(C_2H_5)_3\overset{+}{P}C_6H_4COOC_2H_5$ $\bar{I}$	80		148—148,5 (methylethylketone)					45,8	6,2	7,9	$C_{15}H_{24}IO_2P$	45,7	6,1	7,9
(XVI)	m-$(C_2H_5)_3\overset{+}{P}C_6H_4COOC_2H_5$ $\bar{I}$	76		120—121 (ethyl acetate—methyl ethyl ketone 1:1)					45,6	6,0	8,0				
(XVII)	p-$(C_2H_5)_3\overset{+}{P}C_6H_4COOH$ Br^-	42		198—199 (water)					48,8	6,2	9,6	$C_{13}H_{20}BrO_2P$	48,9	6,3	9,7
(XVIII)	m-$(C_2H_5)_3\overset{+}{P}C_6H_4COOH$ Br^-	40		196—197 (isopropanol, methyl ethyl ketone)					48,9	6,2	9,8				
(XIX)	p-$(C_2H_5)_2PC_6H_4COOH \cdot HBr$	48		196—197 (acetonitrile—water 20 : 1)					45,6	5,7	10,8	$C_{11}H_{16}BrO_2P$	45,	5,5	0,6
(XX)	m-$(C_2H_5)_2PC_6H_4COOH \cdot HBr$	67		194—195 (acetonitrile—water 20 : 1)					45,5	5,5	10,8				
(XXI)	p-$(i\text{-}C_3H_7)_2PC_6H_4CH_3$	a) 80 b) 90	122—123(9)		1,5360	0,9269	70,07	68,87	74,9	10,1	14,9	$C_{13}H_{21}P$	75,0	10,2	14,9
(XXII)	m-$(i\text{-}C_3H_7)_2PC_6H_4CH_3$	91	137—139(22)		1,5350	0,9238	70,20		74,9	10,0	14,8				
(XXIII)	p-$(i\text{-}C_3H_7)_2P(O)C_6H_4CH_3$	74	123—124(2)	45—47					69,8	9,5	13,6	$C_{13}H_{21}OP$	69,6	9,4	13,8
(XXIV)	m-$(i\text{-}C_3H_7)_2P(O)C_6H_4CH_3$	92	135—138(3)	30—31					69,5	9,5	13,8				
(XXV)	p-$(i\text{-}C_3H_7)_2P(O)C_6H_4COOH$	77		245—246 (water—alcohol, 1 : 1)					61,6	7,5	12,1	$C_{13}H_{19}O_3P$	61,6	7,5	12,2
(XXVI)	m-$(i\text{-}C_3H_7)_2P(O)C_6H_4COOH$	75		180—181 (water—alcohol, 2 : 1)					61,5	7,6	12,1				
(XXVII)	p-$(i\text{-}C_3H_7)_2P(O)C_6H_4COOC_2H_5$	62	180—182(2)	70—71 (cyclohexane)					64,1	8,3	10,8	$C_{15}H_{23}O_3P$	63,8	8,2	11,0
(XXVIII)	m-$(i\text{-}C_3H_7)_2P(O)C_6H_4COOC_2H_5$	56	180—181(2)	28—30					64,1	8,2	11,0				
(XXIX)	p-$(i\text{-}C_3H_7)_2PC_6H_4COOC_2H_5$	66	146—147(3)		1,5362	1,0123	82,14	79,76	67,6	8,6	11,5	$C_{15}H_{23}O_2P$	67,6	8,7	11,6
(XXX)	m-$(i\text{-}C_3H_7)_2PC_6H_4COOC_2H_5$	93	135—136(1)		1,5321	1,0092	81,80		67,5	9,0	11,3				
(XXXI)	p-$(i\text{-}C_3H_7)_2PC_6H_4COOH \cdot HBr$	50		224—225,5 (acetonitrile—water, 20:1)					48,9	6,2	9,9	$C_{13}H_{20}BrO_2P$	48,9	6,3	9,7
(XXXII)	m-$(i\text{-}C_3H_7)_2PC_6H_4COOH \cdot HBr$	50		204—205 (acetonitrile—water, 20:1)					48,9	6,4	9,9				

TABLE 2. NMR Spectral Data for Derivatives of p-Dialkylphosphinobenzoic Acids (δ, ppm, and J, Hz)

Compound	Protons of aromatic ring (Y– 2 3 –P)					Protons of substituents on phosphorus atom							$COOCH_2CH_3$		
	2		3		$J_{2\,3}$ HH	CH_3		CH_2		CH		J	δ_{CH_2}	δ_{CH_3}	$J_{CH_2CH_3}$
	δ	J_{HP}	δ	J_{HP}		δ	J	δ	J	δ	J				
$(CH_3)_2PC_6H_4COOC_2H_5$	7,63	1,5	7,10	6,5	8,0	1,30	CH_3P 3,3						4,40	1,38	7,0
$(C_2H_5)_2PC_6H_4COOC_2H_5$	8,02	~1,0	7,51	5,9	7,8	0,89	CH_3CP 15,3	1,57	CH_2P 7,3			CH_2CH_3 7,3	4,35	1,42	7,3
$(i\text{-}C_3H_7)_2PC_6H_4COOC_2H_5$	7,97	~1,0	7,49	6,3	8,3	0,88*	CH_3CP 8,7			2,07	CHP ⩽3	$CHCH_3$ 6,9	4,34	1,35	7,2
						1,10*	CH_3CP 14,8								
$(C_2H_5)_2PC_6H_4COOH \cdot HBr$ in CF_3COOH	8,14	3,3	7,75	13,3	8,0	1,02	CH_3CP 21,5	2,38				CH_2CH_3 8,0			
$(i\text{-}C_3H_7)_2PC_6H_4COOH \cdot HBr$ in CF_3COOH	8,13	2,5	7,76	11,9	8,0	1,14*	CH_3CP			2,70		CH_3CH			
						1,03*	19,0					6,7			

*The splitting is due to the diastereotopicity of the isopropyl groups.

TABLE 3. NMR Spectral Data for Derivatives of m-Dialkylphosphinobenzoic Acids (δ, ppm, and J, Hz)

Compound	Protons of aromatic ring (Y– 6 5 4 2 P)							Protons of substituents on phosphorus atom						$COOCH_2CH_3$		
	2	4		5		6		CH_3		CH_2	CH		J	δ_{CH_2}	δ_{CH_3}	$J_{CH_2CH_3}$
	δ	δ	$J_{H^4H^5} = J_{H^4P}$	δ	$J_{H^5H^6}$	δ	J_{H^2P}	δ	J	δ	δ	J				
$(CH_3)_2PC_6H_4COOC_2H_5$	8,03	7,46	7,3	7,20	7,0	7,81	6,5	1,22	CH_3P 3,3					4,29	1,28	7,3
$(C_2H_5)_2PC_6H_4COOC_2H_5$	8,26	7,62	7,0	7,34	7,0	7,99	6,5	0,99	CH_3CP 21,2	1,56			CH_2CH_3 7,3	4,30	1,18	7,1
$(i\text{-}C_3H_7)_2PC_6H_4COOC_2H_5$	8,31	7,73	6,9	7,46	7,8	8,13	6,9	0,79*	CH_3CP 8,4		2,11	CH—P ⩽3,0	$CHCH_3$ 7,0	4,36	1,31	7,0
								1,01*	CH_3CP 12,3							
$(C_2H_5)_2PC_6H_4COOH$-HBr в CF_3COOH	Multiplet in 7.40-8.30 region							1,02	CH_3CP 21,8	2,43			CH_2CH_3 ~7,5			
$(i\text{-}C_3H_7)_2PC_6H_4COOH$-HBr в CF_3COOH	Multiplet in 7.40-8.50 region							1,20*	CH_3CP		2,71		$CHCH_3$			
								1,08*	19,7				7,1			

*The splitting is due to the diastereotopicity of the isopropyl goups.

The structure of the compounds (including those described previously [1]) was confirmed by the NMR method. In the NMR spectra of the ethyl esters and hydrobromides of the p-dialkylphosphinobenzoic acids the protons of the phenyl ring appear as a multiplet that is characteristic for the AB portion of the ABX system, where X is the nucleus of the phosphorus atom (Table 2). More complex spectra are observed for the derivatives of the m-series. In the spectra of the ethyl esters and hydrobromides of the m-dialkylphosphinobenzoic acids the protons of the phenyl ring give a multiplet that is characteristic for the ABCD portion of the ABCDX system, where X is the nucleus of the phosphorus atom. The compounds of the m-series were analyzed employing double heteronuclear ^1H-$\{^{31}P\}$ suppression (Table 3).

EXPERIMENTAL METHOD

The operations with the P(III) compounds were run in an argon atmosphere, including the recrystallization of the hydrobromides of the p- and m-dialkylphosphinobenzoic acids. The NMR spectra were taken on an RN-2305 NMR spectrometer (60 MHz) using HMDS as the internal standard.

Diethyl-p-tolylphosphine (I). With stirring, to the Grignard reagent (from 15.0 g of Mg and 68.0 g of C_2H_5Br) in 350 ml of absolute ether was added in drops, at 0 to −10°, 50.0 g of p-tolyldichlorophosphine [1], and then 200 ml of saturated NH_4Cl solution and 50 ml of water were added at 20° until all of the precipitate had dissolved. The layers were separated, and the aqueous layer was extracted with benzene (3 × 300 ml). The combined extract was dried over Na_2SO_4, evaporated in vacuo, and the residue was distilled to give 42.1 g of (I).

Diethyl-m-tolylphosphine (II). To 15.3 g of Mg turnings, activated with water, in 300 ml of absolute THF was added a solution of 77.4 g of m-bromotoluene in 20 ml of THF in drops. The mixture was refluxed for 1 h. To the solution at 0° was added 53.0 g of diethylchlorophosphine [10] in 30 ml of THF, and then 60 ml of saturated NH_4Cl solution, 200 ml of ether, and 100 ml of water were added at 20° until all of the precipitate had dissolved. The layers were separated, and the aqueous layer was extracted with benzene (2 × 100 ml). The combined extract was dried over Na_2SO_4, evaporated in vacuo, and the residue was distilled. We obtained 58.3 g of diethyl-m-tolylphosphine (II).

Diethyl-p-tolylphosphine Oxide (III) and Diethyl-m-tolylphosphine Oxide (IV). Oxides (III) and (IV) were obtained in the same manner as the p- and m-dimethyltolylphosphine oxides [1] by the oxidation of the appropriate phosphines with H_2O_2 in acetone.

p-Diethylphosphinylbenzoic Acid (V). With stirring, to a solution of 8.2 g of oxide (III) in 40 ml of pyridine and 80 ml of water, heated to 90°, was added 32.0 g of $KMnO_4$ in 1 h. The mixture was refluxed for 2 h, after which were added 10 ml of methanol, 100 ml of conc. HCl (until acid), and saturated $NaNO_2$ solution until the reaction mixture was completely colorless (several drops). The solution was evaporated in half, and extracted with $CHCl_3$ (4 × 200 ml). The extract was dried over Na_2SO_4 and evaporated in vacuo; the residue was recrystallized from water. The yield of (V) was 7.0 g.

m-Diethylphosphinylbenzoic Acid (VI). Acid (VI) was obtained in the same manner as the p-isomer from diethyl-m-tolylphosphine oxide (IV).

Ethyl Ester of p-Diethylphosphinylbenzoic Acid (VII). A solution of 3.0 g of acid (V) and 1.5 ml of conc. H_2SO_4 in 50 ml of absolute alcohol was refluxed for 5 h. The excess alcohol was distilled off, the residue was treated with 30 ml of water, and the mixture was extracted with benzene (3 × 50 ml). The extract was washed with 30 ml of saturated $NaHCO_3$ solution, dried over Na_2SO_4, and evaporated in vacuo; the residue was distilled. The yield of (VII) was 3.1 g.

Ethyl Ester of m-Diethylphosphinylbenzoic Acid (VIII). Ester (VIII) was obtained in the same manner as the p-isomer from m-diethylphosphinylbenzoic acid (VI). The compound is hygroscopic.

Ethyl Ester of p-Diethylphosphinobenzoic Acid (IX). Ester (IX) was obtained by two methods. a) In the same manner as the ethyl ester of p-dimethylphosphinobenzoic acid [1] from the ethyl ester of p-diethylphosphinylbenzoic acid (VII) by reduction with trichlorosilane.

b) The Grignard reagent (from 2.3 g of Mg and 9.9 g of C_2H_5Br) in 150 ml of absolute ether was added in drops, at −50 to −60°, to 10.0 g of p-carbethoxyphenyldichlorophosphine [5] in 50 ml of absolute ether and 15.3 g of pyridine. After adding 30 ml of water (20°) the layers were separated, the aqueous layer was extracted with benzene (3 × 100 ml), and the combined extract was dried over Na_2SO_4 and evaporated in vacuo; the residue was distilled. The yield of (IX) was 7.6 g. The ethyl ester of m-diethylphosphinobenzoic acid (X) was obtained in a similar manner using method a).

Ethyl Ester of p-Diethylthiophosphinylbenzoic Acid (XI). A solution of 4.1 g of the ethyl ester of p-diethylphosphinobenzoic acid (IX) in 100 ml of absolute benzene was refluxed for 2 h with 0.55 g of sulfur. The solvent was removed in vacuo, and the residue was recrystallized. The yield of (XI) was 3.5 g.

The ethyl ester of m-diethylthiophosphinylbenzoic acid (XII) was obtained in a similar manner from the ethyl ester of m-diethylphosphinobenzoic acid (X).

p-Diethylthiophosphinylbenzoic Acid (XIII). A solution of 1.5 g of ethyl ester (XI) in 10 ml of 50% alcohol was refluxed for 3 h with 0.35 g of KOH, evaporated in vacuo, and acidified with AcOH (or HCl). The obtained acid was extracted with 100 ml of $CHCl_3$, the extract was dried over Na_2SO_4, evaporated in vacuo, and the residue was recrystallized from benzene. The yield of (XIII) was 1.0 g.

m-Diethylthiophosphinylbenzoic acid (XIV) was obtained in a similar manner from the ethyl ester of m-diethylthiophosphinylbenzoic acid (XII).

Ethyl Ester of p-Triethylphosphoniobenzoic Acid Iodide (XV). A solution of 3.4 g of ethyl ester (IX) in 20 ml of absolute benzene was refluxed for 2 h with 2.7 g of C_2H_5I. The obtained precipitate was filtered and recrystallized from a 1:1 ethyl acetate–methyl ethyl ketone mixture, and then from methyl ethyl ketone. The yield of (XV) was 4.0 g.

The ethyl ester of m-triethylphosphoniobenzoic acid iodide (XVI) was obtained in a similar manner from the ethyl ester of m-diethylphosphinobenzoic acid (X).

p-Triethylphosphoniobenzoic Acid Bromide (XVII). A mixture of 7.3 g of ethyl ester (IX), 15 ml of alcohol, and 6.7 g of C_2H_5Br was heated for 5 h at 100° in a glass ampule in an autoclave. The solution was evaporated in vacuo, the residue was refluxed for 2 h with 30 ml of 40% HBr solution, the mixture was evaporated in vacuo, and the residue was recrystallized from water. The yield of (XVII) was 3.6 g.

m-Triethylphosphoniobenzoic acid bromide (XVIII) was obtained in a similar manner from the ethyl ester of m-diethylphosphinobenzoic acid (X).

p-Diethylphosphinobenzoic Acid Hydrobromide (XIX) and m-Diethylphosphinobenzoic Acid Hydrobromide (XX). Hydrobromides (XIX) and (XX) were obtained by the previously-described method [1] in the same manner as the hydrobromides of the p- and m-dimethylphosphinobenzoic acids. The principal absorption bands in the IR spectrum (cm^{-1}) (in Nujol) are: for the p-isomer (XIX): 1240 s (CO), 1500 w, 1510 w, 1570 w (aromatic ring), 1720 s (C=O); for the m-isomer (XX): 1225 s (CO), 1590 w, 1600 w (aromatic ring), 1720 s (C=O).

Diisopropyl-p-tolylphosphine (XXI). Compound (XXI) was obtained by two methods: a) in the same manner as diethyl-p-tolylphosphine (I) from p-tolyldichlorophosphine [1] and isopropylmagnesium bromide; b) from diisopropylchlorophosphine [12] and p-tolylmagnesium bromide.

Diisopropyl-m-tolylphosphine* (XXII) was obtained in the same manner as the p-isomer by method b).

p- and m-Diisopropyltolylphosphine Oxides (XXIII) and (XXIV), p- and m-Diisopropylphosphinylbenzoic Acids (XXV) and (XXVI), Ethyl Esters of p- and m-Diisopropylphosphinylbenzoic Acids (XXVII) and (XXVIII), Ethyl Esters of p- and m-Diisopropylphosphinobenzoic Acids (XXIX) and (XXX), and Also the p- and m-Diisopropylphosphinobenzoic Acid Hydrobromides (XXXI) and (XXXII). The compounds were obtained in the same manner as the similar compounds with ethyl radicals on the phosphorus atom. The principal absorption bands in the IR spectrum (cm^{-1}) (in Nujol) of the diisopropylphosphinobenzoic acid hydrobromides (XXXI) and (XXXII) are: for the p-isomer (XXXI): 1240 s (CO), 1510 w, 1570 w (aromatic ring), 1720 s (C=O); for the m-isomer (XXXII): 1270 s (CO), 1580 w, 1600 w (aromatic ring), 1720 s (C=O).

CONCLUSIONS

1. The m- and p-diethyl-, and the m- and p-diisopropylphosphinylbenzoic acids, and their ethyl esters were synthesized by the oxidation of the corresponding dialkyltolylphosphine oxides with potassium permanganate and subsequent esterification of the formed acids.

* In order to remove halogen-containing impurities and acidic compounds the phosphine was heated with potassium for 4 h at 90°.

2. The reduction of the dialkylphosphinylbenzoic acid esters with $HSiCl_3$ gave the dialkylphosphinobenzoic acid esters, which on hydrolysis with hydrobromic acid gave the hydrobromides of the corresponding dialkylphosphinobenzoic acids. The ethyl ester of p-diethylphosphinobenzoic acid was also obtained from p-carbethoxyphenyldichlorophosphine and ethylmagnesium bromide.

3. The diethylthiophosphinyl- and triethylphosphoniobenzoic acids were obtained by treating the diethylphosphinobenzoic acid esters with sulfur or ethyl halide and subsequent alkaline or acid hydrolysis.

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