which was recrystallized from hot ethanol: mp 311–313°; mass spectrum *m/e* 406 (for the cation part of the molecule, the molecular ion peak at 441 was absent), 405 (M^+ − HCl); ^{31}P absorption, δ −13.8 (C_2H_5OH, from 85% H_3PO_4).

2,3,4,5-Tetrahydro-8-methyl-2-phenyl-1*H*-phosphorino[4,3-*b*]indole (3b) and Formation of the Oxide 5b.—The compound **3b** was prepared from *p*-tolylhydrazine hydrochloride (2.55 g, 0.016 mol) and ketone **1** (3.08 g, 0.016 mol) by the above procedure. The crude product, on fractional recrystallization with aqueous ethanol, gave **3b**, 2.1 g (48%), mp 155–156°, *m/e* 279 (M^+), and **5b**, 0.4 g (10%), mp 203–204°, *m/e* 295 (M^+). Other data for **3b** are ir 3470 cm^{-1} (NH); nmr ($DCCl_3$) δ 2.09 (m, 2, CH_2), 2.45 (s, 3, CH_3), 2.60 (m, 2, CH_2), 3.11 (m, 2, CH_3), and 7.16 (m, 8, ArH and NH) (apparently oxidation of **3b** to **5b** occurred during recrystallization). The phosphine **3b** formed the phosphonium salt **6b** by the previously described method in quantitative yield, mp 299–301°, mass spectrum *m/e* 420 (for the cation part of the molecule, the molecular ion peak at 455 was absent), 419 (M^+ − HCl).

2,3,4,5-Tetrahydro-8-hydroxy-2-phenyl-1*H*-phosphorino[4,3-*b*]indole (3c) and Formation of the Oxide 5c.—The compound **3c** was prepared from *p*-methoxyphenylhydrazine hydrochloride (**2c**, 4.3 g, 0.024 mol) and ketone **1** (4.7 g, 0.024 mol) by the above procedure, except that the extraction of the reaction mixture was done with chloroform and recrystallization with chloroform–hexane to give **3c**: 2.5 g (36%); mp 100–102° (see footnote *a*, Table I); ir 3470 (NH), 3350 cm^{-1} (OH); nmr ($DCCl_3$) δ 2.4 (m, 2, CH_2), 2.72 (m, 2, CH_2), 3.06 (m, 2, CH_2), 4.80 (s, 1, OH), and 7.12 (m, 9, ArH and NH); *m/e* 281 (M^+).

The phosphine **3c** formed the phosphonium salt **6c** by the previously described method in 91% yield, mp 265–267°, mass spectrum *m/e* 422 (for the cation part of the molecule, the molecular ion peak at *m/e* 457 was absent), 421 (M^+ − HCl).

The oxide **5c** was formed (by air oxidation) when **3c** was recrystallized using methanol–ether, mp 274–275°, *m/e* 297 (M^+).

2,3,4,5-Tetrahydro-8-fluoro-2-phenyl-1*H*-phosphorino[4,3-*b*]indole (3d) and Formation of the Oxide 5d.—The compound **3d** was prepared from 4-fluorophenylhydrazine hydrochloride (**2d**, 5.42 g, 0.033 mol) and ketone **1** (6.4 g, 0.033 mol) by the above procedure. The crude product on fractional recrystallization from ether–hexane gave **3d**, 6.9 g (73%), mp 113–114°, *m/e* 283 (M^+), and **5d**, 0.3 g (3%), mp 184–186°, *m/e* 299 (M^+). Other data for **3d** are ir 3470 cm^{-1} (NH); nmr ($DCCl_3$) δ 1.96 (m, 2, CH_2), 2.48 (m, 2, CH_2), 2.93 (m, 2, CH_2), and 7.02 (m, 9, ArH and NH). The phosphine **3d** formed the phosphonium salt **6d** by the previously described method in quantitative yield, mp 276–278°, mass spectrum *m/e* 424 (for the cation part of the molecule, the molecular ion peak at *m/e* 459 was absent), 423 (M^+ − HCl).

Attempted Preparation of 2,3,4,5-Tetrahydro-8-chloro-2-phenyl-1*H*-phosphorino[4,3-*b*]indole (3e). Formation of the Oxide 5e.—The reaction of 4-chlorophenylhydrazine hydrochloride (**2e**, 3.0 g, 0.017 mol) and ketone **1** (3.2 g, 0.017 mol) was done, as in the general procedure, except that the reaction mixture was extracted with chloroform. The product did *not* contain any indole **3e**, but only the oxide **5e**, 2.4 g (46%), mp 220–224°, *m/e* 315 (M^+). Recrystallization from chloroform–ether gave the analytical sample: mp 223–225°; ir 3470 cm^{-1} (NH); nmr (acetone-d_6) δ 2.62 (m, 2, CH_2), 3.09 (m, 2, CH_2), 3.62 (m, 2, CH_2), and 7.25 (m, 9, ArH and NH). Again apparently **3e** was oxidized during purification.

Attempted Preparation of 2,3,4,5-Tetrahydro-8-bromo-2-phenyl-1*H*-phosphorino[4,3-*b*]indole (3f). Formation of the Oxide 5f.—The reaction of 4-bromophenylhydrazine hydrochloride (**2f**, 5.6 g, 0.025 mol) and ketone **1** (4.8 g, 0.025 mol) was done as in the general procedure. The product did not contain any indole **3f** but only the oxide **5f**, 4.7 g (52%), mp 229–230°, *m/e* 361 and 359 (M^+). Recrystallization from methanol–H_2O gave the analytical sample: mp 230–232°; ir 3470 cm^{-1} (NH); nmr ($DCCl_3$) δ 2.26 (m, 2, CH_3), 2.98 (m, 2, CH_2), 3.46 (m, 2, CH_2), 7.39 (m, 8, ArH), and 8.98 (s, 1, NH).

Attempted Preparation of 2,3,4,5-Tetrahydro-8-nitro-2-phenyl-1*H*-phosphorino[4,3-*b*]indole (3g). Formation of the Oxide 5g.—The reaction of 4-nitrophenylhydrazine (**2g**, 0.5 g, 0.003 mol) and ketone **1** (0.64 g, 0.003 mol) was done, as in the above procedure, except that the reaction mixture was extracted with chloroform. The product did not contain any indole **3g**, but only the oxide **5g**, 0.2 g (21%), *m/e* 326 (M^+). It was purified by chromatographing through neutral alumina column and eluting with chloroform, giving a deep orange solid: mp 220–222° dec; ir 3470 cm^{-1} (NH); nmr ($DCCl_3$) δ 1.78 (m, 2, CH_2), 2.50 (m, 2, CH_2), 2.98 (m, 2, CH_2), and 7.12 (m, 9, ArH and NH).

The 4-nitrophenylhydrazone **4** (R = NO_2) was isolated by boiling (6 hr) 4-nitrophenylhydrazine (0.6 g, 0.004 mol) and ketone **1** (0.64 g, 0.003 mol) in ethanol and diluting (H_2O). The solid product was collected by filtration: 0.72 g (66%, based on the amount of ketone); mp 150–151°; nmr ($DCCl_3$) δ 2.39 (m, 8, alicyclic H), 7.03 (d, 2, *J* = 9 Hz, ArH), 8.10 (d, 2, *J* = 9 Hz, ArH), 7.37 (m, 5, ArH), and 7.72 (s, 1, NH); *m/e* 327 (M^+). *Anal.* Calcd for $C_{17}H_{18}N_3O_2P$: N, 12.84. Found: N, 12.62.

Registry No.—**3a**, 36720-80-6; **3b**, 36720-81-7; **3c**, 36720-82-8; **3d**, 36720-83-9; **4** (R = NO_2), 36720-84-0; **5b**, 36720-85-1; **5c**, 36720-86-2; **5d**, 36720-87-3; **5e**, 36720-88-4; **5f**, 36720-89-5; **5g**, 36720-90-8; **6a**, 36720-91-9; **6b**, 36763-71-0; **6c**, 36720-92-0; **6d**, 36720-93-1.

2-Carbomethoxycyclopent-2-enone[1]

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Although many substituted cyclopentenones are known, no simple derivatives containing only an electron-withdrawing substituent in the 2 position seem to have been reported. Such compounds would be expected to be relatively unstable, since the substituent would polarize the enone system further, and probably enhance the tendency toward polymerization shown by cyclopentenone itself. An unsuccessful attempt to synthesize 2-acetylcyclopent-2-enone (Ia) has been reported.[3] A recently reported[4] method for synthesizing 3-alkyl-2-carboalkoxycyclopentenones failed for 3-methyl-2-carbethoxycyclopent-2-enone (Ib), the simplest case investigated, although Ib had been prepared by Yates[5] previously by a similar route.

We now report the synthesis of 2-carbomethoxycyclopent-2-enone (Ic), a compound of much potential value for natural products synthesis. The compound can be obtained in *ca.* 45% yield (nmr analysis) by oxidation of 2-carbomethoxycyclopentanone (IIa) with selenium dioxide in refluxing dioxane. Dichlorodicyanoquinone (DDQ) oxidation also gives the compound, but in low yield (5–10%), as it is polymerized under the reaction conditions. Ic is fairly stable in dioxane solution, but attempted purification by any of several methods leads to rapid polymerization. Fractions containing colored, moderately pure material (nmr analysis) were obtained by very rapid silica gel chromatography, but the material polymerized fairly rapidly. However, the compound could be trapped by adding dienes to the reaction mixture. 2,3-Dimethylbutadiene reacted smoothly at 100° to give the adduct III, and cyclopentadiene at 25° gave a 1:1 mixture of the endo and exo adducts IV, which were separated by silica gel chromatography. Pyrolysis of either isomer or

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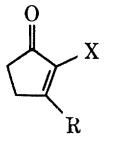
the mixture of IV at 438° and trapping at −10° gave a pure sample of 2-carbomethoxycyclopent-2-enone (Ic). In contrast, adduct III was stable under these conditions.

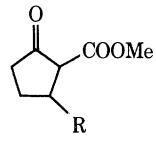
All spectral data on compound Ic are in accord with the assigned structure. The vinyl proton signal in the nmr spectrum appears as a triplet (J = 2.5 Hz) at δ 8.38. This is a very deshielded value for a vinyl proton and reflects the great polarity in the enone chromophore of the compound.

Pure 2-carbomethoxycyclopent-2-enone (Ic) polymerizes within several days at −10° but can be stored for prolonged periods in dilute solution at −10°. It polymerizes very readily with a variety of acidic and basic protic reagents (*e.g.*, anhydrous HBr,[6] diethylamine, NaOMe in MeOH, semicarbazide). Reaction with lithium dimethylcopper,[7] however, proceeds smoothly to give 3-methyl-2-carbomethoxycyclopentanone (IIb), whose semicarbazone was identical with an authenic sample.[5]

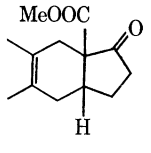
Some other attempts to obtain 2-carbomethoxycyclopent-2-enone (Ic) are of interest. Bromination of 2-carbomethoxycyclopentanone (IIa) in CCl_4 gave



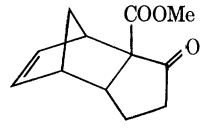
Ia, X = COOEt, R = Me
b, X = COMe, R = H
c, X = COOMe, R = H



IIa, R = H
b, R = Me



III



IV

exclusively the 5-bromo isomer. However, bromination of IIa in water containing $Cu(NO_3)_2$ gave the 2-bromo isomer in good yield, as suggested by a mechanistic study of the reaction.[8] However, on attempted dehydrobromination, rearrangement of the 2-bromo isomer to the 5-bromo isomer evidently occurred since either isomer gave only 5-carbomethoxycyclopent-2-enone and much polymer, but no Ic, under any dehydrobromination conditions investigated.

Experimental Section

2-Carbomethoxycyclopentenone–Cyclopentadiene Diels–Alder Adduct (IV).—To a solution of 14.2 g (0.10 mmol) of 2-carbomethoxycyclopentanone (IIa) in 25 ml of reagent grade dioxane was added 12.2 g (0.11 mol) of SeO_2. No oxidation occurred in dioxane which had been distilled from $LiAlH_4$. The mixture was refluxed for 25 min, and the black Se was removed by filtration. Then 66 g of cyclopentadiene was added and the dark brown solution stirred at room temperature overnight. Distillation of all volatile materials at 0.25 mm was followed by absorption of the residue on 25 g of silica gel and chromatography on 600 g of silica gel packed in petroleum ether (bp 40–60°). After elution with 3 l. of petroleum ether, a 1:1 mixture of the two stereoisomeric adducts IV (2.92 g) was obtained by elution with 10% ether in petroleum ether (2:1). More careful rechromatography of the combined fractions under the same conditions with 5% ether in petroleum ether gave, in early fractions, one pure stereoisomer of the adduct IV: ir (film) 3000, 1760, 1730 cm^{-1}; nmr (CCl_4) δ 0.9–3.4, complex absorption, 3.63 (3 H, s, COOMe) 6.28 (2 H, t, J = 2 Hz, vinyl); 2,4-dinitrophenylhydrazone, mp 164–166°. *Anal.* Calcd for $C_{18}H_{18}N_4O_6$: C, 55.96, H, 4.70. Found: C, 55.87; H, 4.65.

Further elution with the same solvent mixture gave the same isomer contaminated with increasing amounts of its stereoisomer, which was obtained essentially pure in later fractions: ir (film) 3000, 1760, 1730 cm^{-1}; nmr (CCl_4) δ 0.8–3.4 (complex absorption), 3.70 (3 H, s, COOMe), 6.20 (2 H, t, J = 2 Hz, vinyl H's).

2-Carbomethoxycyclopentenone–2,3-Dimethylbutadiene Adduct (III).—To a total SeO_2 oxidation mixture from 1.42 g of 2-carbomethoxycyclopentanone as described above was added 0.74 g of 2,3-dimethylbutadiene and the mixture heated in a sealed tube for 6 hr at 100°. The adduct III was isolated (256 mg) as the only monomeric product by chromatography over silica gel: ir (CCl_4) 1760, 1730 cm^{-1}; nmr (CCl_4) δ 1.65 (6 H, broadened s, Me's), 1.8–2.5 (mult), 3.70 (3 H, s, COOMe); simicarbazone, mp 207–208° (ethanol). *Anal.* Calcd for $C_{14}H_{21}N_3O_3$: C, 60.20; H, 7.58. Found: C, 60.45; H, 7.95. This compound was recovered unchanged upon attempted pyrolysis at 438°.

Pyrolysis of Adduct IV. 2-Carbomethoxycyclopent-2-enone (Ic).—A 1:1 mixture of the two isomeric adducts IV (676 mg, 3.3 mmol) was heated at 60–65° and carried by a slow stream (2 ml/min) of N_2 through a heated inlet system into a 50-ml pyrolysis chamber heated to 438° with a lead bath (contact time 20–30 sec). The exit gases were condensed in a U-tube cooled in an ice–salt bath, conditions which allowed the cyclopentadiene to escape. After 3 days, the tube contained 263 mg (57%) of pure 2-carbomethoxycyclopent-2-enone (Ic): λ_{max}^{EtOH} 220 nm (ϵ >8000); nmr ($CDCl_3$) δ 2.70 (4 H, mult), 3.92 (3 H, s, Me), 8.38 (1 H, t, J = 2.5 Hz, vinyl H). The compound polymerized on standing a few hours neat at room temperature or after several days in a refrigerator but can be kept for extended periods in dry ether solution in a refrigerator. It also polymerized during attempts to form a crystalline derivative or to effect reaction with a number of nucleophiles in protic media, as mentioned in the text.

2-Carbomethoxy-3-methylcyclopentanone (IIb).—To a suspension of 114 mg (0.50 mmol) of pure dry CuI in 10 ml of dry ether was added by syringe 26 mg (1.2 mmol) of methyllithium (0.67 ml of 1.8 M ethereal solution). The resulting pale yellow solution was cooled to −78° (Dry Ice–acetone) and 150 mg (1.06 mmol) of 2-carbomethoxycyclopent-2-enone in 10 ml of ether was added dropwise. The mixture was allowed to warm to room temperature during 1.5 hr, then poured into a saturated NH_4Cl solution. Concentrated NH_4OH was added until solution was complete. Then the ether layer was washed and dried ($MgSO_4$) and the ether evaporated to give an oil (100 mg) showing one spot on tlc and one peak on vpc: ir (CCl_4) 1765 and 1735 cm^{-1} (ketone and ester of keto form) and 1665 and 1630 (ester and double bond of enol form); nmr (CCl_4) δ 1.22, 1.24 (3 H total, d, J = 6 Hz, C-3 Me's of enol and keto form), 2.3–2.9 (complex absorption), 3.81 (3 H, s, COOMe); semicarbazone, mp 168–169.5° (H_2O), no depression on admixture with a sample kindly supplied by Professor Yates;[5] 2,4-dinitrophenylhydrazone ($EtOH–H_2O$), mp 127.5–128.5°. *Anal.* Calcd for $C_{14}H_{16}N_4O_6$: C, 50.00; H, 4.80. Found: C, 50.05; H, 4.48.

2-Bromo-2-carbomethoxycyclopentanone.—To an aqueous solution (250 ml) which was 0.10 M in KBr, 0.15 M in HNO_3, and 0.10 M in $Cu(NO_3)_2$ was added 14.2 g of 2-carbomethoxycyclopentanone (0.10 mol). An aqueous solution containing 16.0 g of Br_2 (0.10 mol) was added during 0.5 hr. Ether extraction gave 20.8 g (94%) of 2-bromo-2-carbomethoxycyclopentanone, homogeneous by tlc: nmr (CCl_4) δ 2.31 (6 H, mult), 3.86 (3 H, s, COOMe), absence of absorption at 2.8–3.2 due to the C-2 H of starting material.

5-Bromo-2-carbomethoxycyclopentanone.—Treatment of 1.42 g of 2-carbomethoxycyclopentanone in 10 ml of CCl_4 with 1.60 g of Br_2 in 10 ml of CCl_4 by dropwise addition with stirring and then washing and drying ($MgSO_4$) gave a quantitative yield of the 5-bromo isomer, homogeneous by tlc: nmr (CCl_4) δ 2.50 (5 H, mult), 3.89 (3 H, s, COOMe), 4.88 (1 H, mult, C-5 H).

5-Carbomethoxycyclopent-2-enone.—To 1.1 g of 5-bromo-2-carbomethoxycyclopentanone in 10 ml of DMF under N_2 was added 3 g of powdered $CaCO_3$, and the mixture was refluxed 10 min. The cooled mixture was filtered, diluted with water, and

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(8) K. J. Pederson, *Acta. Chem. Scand.*, **2**, 385 (1948).

extracted with ether. After washing, drying, and removal of the ether, 460 mg of oily 5-carbomethoxycyclopent-2-enone was obtained: nmr (CCl_4) δ 2.0–3.6 (mult), 3.82 (3 H, s, COOMe), 6.45 (1 H, mult, C-2 H), 8.17 (1 H, mult, C-3 H). No carbonyl derivative could be obtained.

Substitution of 1.2 g of 2-bromo-2-carbomethoxycyclopentanone for the 5-bromo isomer in the above procedure gave (nmr analysis) 0.4 g of an oil containing mostly 5-carbomethoxycyclopent-2-enone and some 2-carbomethoxycyclopentanone (contaminant in the starting material), but no 2-carbomethoxycyclopent-2-enone (Ic), since the signal at δ 8.38 was absent.

Other dehydrobromination reagents whose action on the above compounds was investigated include Li_2CO_3–LiBr–DMF; 1,5-diazabicyclo[5.4.0]undec-5-ene (DBU); 5-ethyl-2-methylpyridine; and KOBu-*t*. These reagents gave only polymeric material.

Registry No.—Ic, 36601-73-7; IIb, 18067-33-9; IIb DNP, 36601-75-9; III, 36601-76-0; exo-IV, 36601-77-1; *endo*-IV, 36622-61-4; IV DNP, 36596-59-5; 2-bromo-2-carbomethoxy cyclopentanone, 36596-60-8; 5-bromo-2-carbomethoxycyclopentanone, 36596-61-9; 5-carbomethoxycyclopent-2-enone, 36596-62-0.

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Carbon Tetrachloride Dimerization of 2-Nitropropane Anion. An Electron-Transfer Process

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The lithium or potassium salts of 2-nitropropane react exothermically with carbon tetrachloride in DMSO saturated with nitrogen to yield 2,3-dinitro-2,3-dimethylbutane in approximately 50% yield. Although a variety of procedures[1,2] are available for producing the synthetically useful vicinal dinitroalkanes[2] in high yields, the carbon tetrachloride oxidative dimerization of nitroalkyl anions reported in this note may prove to be a synthetically useful reaction because of its greater simplicity and speed, especially with *in situ* preparation of the anion by use of commercial potassium *tert*-butoxide.

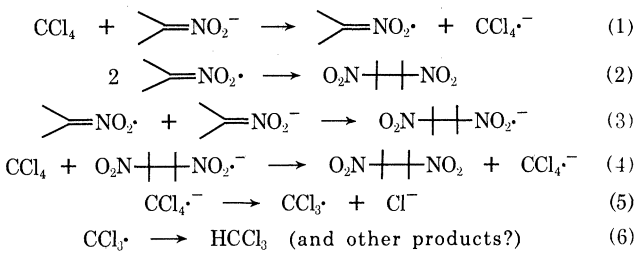
Table I lists the yields of products obtained under several reaction conditions. Since both carbon tetrachloride and the 2-nitropropyl anion are stable to oxygen, the reduction in dimer yield and the formation of acetone when the reaction solution is saturated with oxygen instead of nitrogen suggest that the reaction proceeds *via* 2-nitropropyl radicals which are trapped by oxygen.[3] Quenching the reaction with 6 *N* HNO_3 and Volhard titration of the liberated chloride indicated that the reaction is complete within 1–2 min with DMSO or 2:1 DMSO–cyclohexene as solvent. No detectable reaction occurs in 1 hr with ether, cyclohexene, carbon tetrachloride, ethanol, or 10:2 *tert*-butyl alcohol–water as solvent. Because CCl_4 undergoes nucleophilic substitution with carbanions[4] and is also a good acceptor of electrons, with the intermediate carbon tetrachloride radical anion exothermically decomposing to trichloromethyl radical and chloride anion,[5] two mechanisms for the reaction are immediately conceivable: mechanism A, consisting of nucleophilic substitution on carbon tetrachloride by 2-nitropropyl anion to form trichloromethyl carbanion and 2-chloro-2-nitropropane, which reacts with 2-nitropropyl anion by a radical anion process to form the dimer,[3] and mechanism B (eq 1–6).

TABLE I

REACTION OF CCl_4 WITH 2-NITROPROPYL ANION IN DMSO

$[CCl_4]$, *M*	[2-Nitropropane], *M*	[*t*-BuOK], *M*	Gas	Products, mol % Dimer	Cl^-	Other
1.8	0.3 (Li salt)		N_2	50	70	60 ($CHCl_3$)
1.8	0.3 (Li salt)		O_2	30	40	$CHCl_3$, acetone
1.8[a]	0.3 (Li salt)		N_2	40	70	50 ($CHCl_3$)
0.4	0.22	0.22	N_2	50	80	
0.22	0.22	0.22	N_2	50	80	
0.4	0.22	0.22	O_2	30	40	

[a] Solvent: 2:1 DMSO–cyclohexene.



We tentatively favor mechanism B as the mechanism of the reaction for the following reasons. (a) Mechanism A involves the formation of trichloromethyl carbanion, which should rapidly decompose to dichlorocarbene which is trappable by cyclohexene.[4] When the reaction was conducted in 2:1 DMSO–cyclohexene solvent no dichloronorcarane was detectable. (b) The yield of dimer in the carbon tetrachloride reaction was reduced from 50 to 30% when the reaction was carried out in the presence of oxygen instead of nitrogen (Table I). In mechanism A dimer is formed *via* the reaction of 2-nitropropyl anion with 2-chloro-2-nitropropane. When this reaction was conducted in oxygen- instead of nitrogen-saturated DMSO solutions, the yield of dimer decreased from 65 to $\leq$5% at 22° and from 80 to 10% at 60° (Table II) (see also ref 3). It therefore appears unlikely that 2-chloro-2-nitropropane is an intermediate in the carbon tetrachloride mediated dimerization of 2-nitropropyl anion as required by mechanism A. (c) The dimerization of 2-nitropropyl

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