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Some Diels–Alder reactions of trimethoxysilylpropylcyclopentadiene and the synthesis of a silica-supported 4,5-dicyanonorbornenepalladium complex

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Abstract

Trimethoxysilylpropylcyclopentadiene reacts with the electron-deficient acetylenes, $RO_2CC_2CO_2R$ (R = Me, Et) and olefins $R^1CH{=}CHR^2$ (R^1 = H, R^2 = SOPh; $R^1 = R^2 = P(O)Ph_2$; $R^1 = R^2$ = CN) to give the corresponding Diels–Alder adducts. The compounds CHCl=CHCl and E-$Ph_2P(S)CH{=}CHP(S)Ph_2$ failed to react under similar conditions. The adduct from fumarodinitrile has been used to synthesise a silica-anchored bis-nitrile palladium chloride complex, which catalyses the isomerisation of pent-1-ene.

Introduction

The first reports by Pitkethley et al. [1] of the synthesis of transition metal complexes of silica-supported monophosphines stimulated a considerable amount of research in this area [2,3]. It has become increasingly apparent, however, that transition metal catalysts bound to a silica surface by monodentate ligands have limited applicability in many cases as the metal can be leached from the support [4,5]. For this reason attention has turned towards the synthesis of more stable bidentate silica-anchored ligands, and several research groups [6–13] have recently reported silica-supported achiral [6–10] and chiral [11–13] diphosphinemetal complexes that exhibit similar catalytic activities and selectivities to those found with homogeneous counterparts. The synthesis of such ligands frequently requires multi-step sequences that are either not suitable or too expensive for larger scale operations. With this in mind it was of interest to explore the possibility of a simple, cheap route to silica-supported chelating ligands via the cyclopentadiene derivatives, $(MeO)_3Si(CH_2)_3C_5H_5$ (**1**), prepared in two steps [14] from commercially available $(MeO)_3Si(CH_2)_3Cl$.

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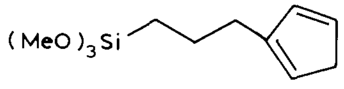
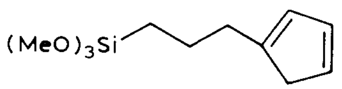
Results and discussion

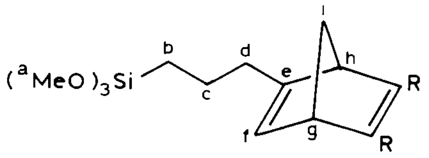
Compound **1**, which has been shown by ^{1}H and ^{13}C NMR spectroscopy to be an ca. 1/1 mixture of the isomers **a** and **b**, reacts with the acetylenes, $RO_2CC_2CO_2R$



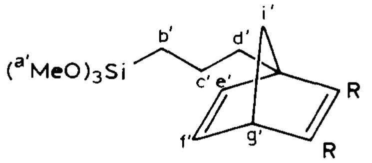
(1a)



(1b)

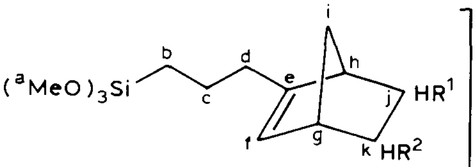


(2,3a)

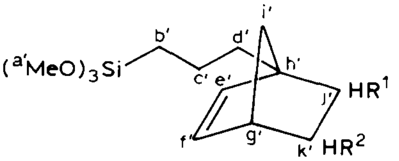


(2,3b)

(**2**: R = COOMe , **3** = COOEt)



(4-6a)



(4-6b)

(**4** : R^1 = H , R^2 = SOPh ; R^1 = SOPh , R^2 = H ;
5 : R^1 = R^2 = $Ph_2P(O)$; **6** : R^1 = R^2 = CN)

(R = Me, Et) in dichloromethane at room temperature during 24 h to give the cycloadducts **2** and **3** in 57 and 62% yields, respectively, as pale yellow oils. It is apparent from the complexity of the ^1H and ^{13}C NMR spectra that these products are also mixtures of two isomers, which are assumed to have the structures (**2a**,**2b**) and (**3a**,**3b**). The isomers could not be separated by TLC or flash chromatography. Reduction of the ester groups to the synthetically more versatile diol proved difficult owing to the hydrolytic and thermal sensitivity of the silyl group. Both $LiAlH_4$ and $NaBH_4$ reductions require an aqueous work up procedure, and attempts to reduce the ester groups of **2** using sodium in dry ethanol at reflux temperatures for 5 h was successful as evidenced by the disappearance of the ester carbonyl absorption in the IR spectrum, but decomposition to form a solid polysiloxane also occurred. Nevertheless, compounds **2** and **3** could prove to be useful intermediates for subsequent synthetic modification if they were anchored to silica prior to reduction.

trans-Dichloroethylene did not react with **1** at 100 °C during 6 days, and the only product isolated was the dimer of **1**, obtained as a deep yellow oil. Although compound **1** does not dimerise readily and can be stored at room temperature for several weeks, at higher temperatures there is a competition between dimer formation and the desired cycloaddition to another dienophile. The masked acetylene [15] phenyl vinylsulphoxide reacted only slowly with **1** at the reflux temperature of toluene during 24 h to give mainly the dimer of **1**, together with a 5% yield of an oil, that is believed to be the adduct **4**, formed as a mixture of isomers.

Reaction between **1** and *trans*-$Ph_2P(O)CH{=}CHP(O)Ph_2$ [16] in a sealed tube at 100 °C for 24 h gave the expected adduct **5** in 36% yield as a mixture of isomers. In contrast, the disulphide, *trans*-$Ph_2P(S)CH{=}CHP(S)Ph_2$ [17], did not react even in 20 h at 140 °C, and only the dimer of **1** was isolated from the reaction. Compound **5** is a useful intermediate for the synthesis of silica-supported diphosphine metal complexes, since several methods are available for the reduction of phosphine oxides to phosphines [18]. We hope to improve the yield of **5** by the use of high pressure techniques.

trans-1,2-Dicyanoethene reacted with **1** at 45 °C in dry toluene during 4 h to give **6** in 77% yield as a yellow oil. When **6** was heated with a suspension of Grace–Davison 952 silica in toluene for 16 h, followed by treatment with Me_3SiCl for 6 h, a supported dinitrile ligand was obtained which contained 0.9% N. A suspension of this ligand in benzene was stirred at room temperature with various concentrations of $[PdCl_2(PhCN)_2]$ for 20 h to give the samples **A**–**C** (Table 1). It can be seen from the table that for the high-loading sample **A**, the ratio of 2N/Pd/2Cl from microanalysis is consistent with a complex in which each Pd atom is bound to the

Table 1

Analytical data for loading levels of $[PdCl_2(PhCN)_2]$ on $(SIL\text{-}CH_2)_3C_7H_6(CN)_2]$ (SIL = silica)

Sample	Concentration [a] (μmol g^{-1})	Found (%)			Ratio Pd/N/Cl
		N	Pd	Cl	
A	250.0	0.7	1.3	1.6	1/2/2
B	178.5	0.5	1.5	0.9	1/1/1
C	142.8	0.4	0.4	0.4	1/4/2

[a] $PdCl_2(PhCN)_2$.

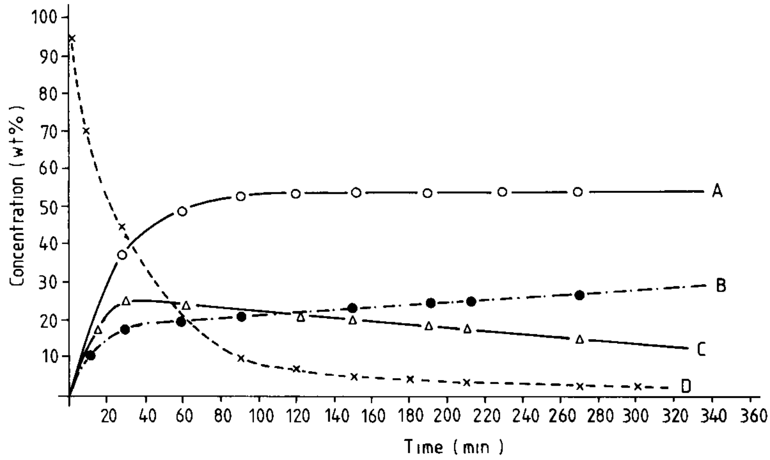


Fig. 1. Product accumulation curves as a function of time at 25°C and 1 atm H_2 for 1-pentene over catalyst C. (A) *trans*-2-pentene, (B) pentane, (C) *cis*-2-pentene, (D) 1-pentene.

cis-cyano ligands of the support, but this could not be confirmed by far IR spectroscopy as the absorptions in this region were too weak. In the lower-loading sample C, approximately 50% of the available cyano ligands are not bound to palladium.

A suspension of catalyst A in dry toluene was unaffected by an atmosphere of hydrogen, but as soon as pent-1-ene (catalyst/olefin mole ratio 1/500) was introduced at room temperature the catalyst turned black and hydrogen gas was taken up at a steady rate of 0.5 cm^3 min^{-1} for 30 min, and then there was no further uptake. Analysis of the mixture by GLC showed unreacted 1-pentene (3.5%), pentane (28.3%) *cis*- (22.3%) and *trans*-2-pentene (45.5%). Repetition of the reaction with catalyst C (catalyst/olefin ratio 1/1000) under similar conditions, and GLC analysis of aliquots removed at 30 min intervals throughout the reaction (Fig. 1) indicated that isomerization to *cis*- and *trans*-2-pentene is faster than hydrogenation to pentane, and the final mixture after 4 h consisted of 1-pentene (1.5%), pentane (27.2%), *trans*-2-pentene (52.1%) and *cis*-2-pentene (19.2%). The grey spent catalyst from these reactions was found to be inactive after filtration in air, but if fresh pent-1-ene was added to the catalyst while it was still under an atmosphere of hydrogen, catalysis did occur but the reaction was slower.

When the reaction of pent-1-ene was carried out using catalyst C (catalyst/olefin ratio 1/500) in the absence of hydrogen with toluene as solvent at room temperature, reaction was much slower and after 4 h the product distribution was 1-pentene (65.3%), pentane (7.0%), *trans*-2-pentene (16.0%) and *cis*-2-pentene (11.7%), and these percentages did not change during a further 3 h. Under these conditions the catalyst suspension turned black only after ca. 10 min, in contrast with the instantaneous colour change observed when hydrogen was present.

There have been many reports on the use of $[PdCl_2(PhCN)_2]$ as an alkene isomerisation catalyst [19], although the detailed mechanism and the nature of the true catalyst in these reactions is still open to question. There is little doubt that in the reactions described above involving silica-supported dicyanopalladium chloride as a catalyst the combination of alkene and hydrogen causes reduction of the palladium(II) to palladium metal.

Experimental

Trimethoxysilylpropylcyclopentadiene was prepared from commercially available (3-chloropropyl)trimethoxysilane as previously described [14]. *trans*-1,2-Bis(diphenylphosphinesulphide)ethane [17] and *trans*-1,2-bis(diphenylphosphineoxide)ethane [16] were prepared and purified by methods described previously, whereas dimethyl and diethyl acetylenedicarboxylate and fumaronitrile were commercial samples used without purification. All solvents were purified and dried by standard procedures, and except where stated all reactions were carried out under nitrogen purified by successive passage through Fieser's solution, concentrated sulphuric acid, KOH pellets, 4Å molecular sieves and self-indicating silica-gel.

IR spectra in the range 4000–665 cm^{-1} were recorded on a Perkin–Elmer Infracord 298 spectrophotometer with NaCl optics, and far IR spectra were determined on a Beckman IR 720M spectrophotometer. ^{1}H NMR spectra were recorded on a Perkin–Elmer R34 spectrometer at 220 MHz and ^{13}C NMR spectra on a Bruker WP 80 (20.1 MHz) instrument. Mass spectra were determined with a Kratos MS45 instrument.

Reaction of trimethoxysilylpropylcyclopentadiene

(a) With dimethyl acetylenedicarboxylate (DMAD). A solution of the diene **1** (1.80 g, 7.9 mmol) and DMAD (1.08 g, 7.6 mmol) in dry dichloromethane (55 cm^3) was stirred at room temperature for 16 h. Removal of the solvent under vacuum gave the product as a pale yellow oil (2.6 g), which was purified by flash chromatography (silica gel (40–32 μm); toluene/ethyl acetate (1/1 v/v)) to give **2** (1.6 g, 4.30 mmol, 57%) (Found: C, 54.8; H, 7.3. $C_{17}H_{26}O_7Si$ calcd.: C, 55.1; H, 7.0%). The ^{1}H and ^{13}C NMR spectra of this product indicated that it was a mixture of the isomers **2a** and **2b** in an approximate 1/1 ratio. IR (neat liquid): ν 3030vw (=C–H), 2950vs (CH_2), 2840vs (OCH_3), 1720vs (C=O), 1630s, 1610s (C=C), 1430s (C–H def.), 1270s (C–O str., CO_2Me str.), 1200-1100br.s (C–O str.), 810vs (C–H def.) cm^{-1}. ^{1}H NMR ($CDCl_3$, 220 MHz, Me_4Si ext. ref.) δ 0.56 (complex m, 4H, b,b′-CH_2), 1.40 (complex m, 4 H, c,c′-CH_2), 1.85, 2.00 (complex m, 4 H, i,i′-CH_2), 2.20 (complex m, 4 H, d,d′-CH_2), 3.45 (s, 18H, a,a′-OCH_3), 3.66, 3.70, 3.71, 3.73 (singlets, 12 H, OCH_3), 3.60 (brs, 1H, g or g′-CH), 3.76 (s, 1 H, h-CH), 3.85 (br.s, 1H, g or g′-CH), 6.25 (m, 1H, f-CH), 6.58 (d, 1H, e′-CH, $J_{e'f'}$ 5 Hz), 6.82 (dd, 1H, f′-CH, $J_{f'g'}$ 3 Hz) ppm. ^{13}C NMR ($CDCl_3$, 20.1 MHz, Me_4Si ext. ref.) δ 8.76 (d), 9.59 (d), 19.41 (t), 20.36 (t), 33.23 (m), 34.14 (m), 50.22 (q), 51.64 (q), 56.52 (d), 66.92 (s), 71.45 (t), 73.60 (t), 132.59 (d), 143.51 (d), 144.50 (d), 151.43 (s), 153.19 (s), 163.92 (s), 165.43 (s) ppm. MS: *m/z* 370 $[M]^+$ 2.5%.

(b) With diethyl acetylenedicarboxylate (DEAD). Reaction between **1** and DEAD (1.04 g, 6.12 mmol) in dichloromethane (55 cm^3) for 16 h at room temperature gave, after flash chromatography of the crude product (2.2 g), compound **3**, as a mixture of **3a** and **3b** in an approximate 1/1 ratio, as a colourless oil (1.5 g, 3.77 mmol, 62%). (Found: C, 57.0; H, 7.6. $C_{19}H_{30}O_7Si$ calcd.: C, 57.3; H, 7.5%). IR (neat liquid): ν 3025s (=CH), 2950s (CH_2), 2840s (OCH_3), 1720vs (C=O), 1630s, 1610ssh (C=C), 1460s (C–H) def.), 1390m, 1370s (CH_3 def.), 1260s, 1210m (C–O str.),

1190–1100 br.s (C–O str.), 810vs (C–H def.) cm^{-1}. ^1H NMR ($CDCl_3$, 220 MHz, Me_4Si ext. ref.): δ 0.50 (complex m, 4H, b,b′-CH_2), 1.10 (t, 12H, $4 \times CH_2CH_3$, J 7.0 Hz), 1.35 (complex m, 4H, c,c′-CH_2), 1.75, 1.90 (complex m, 4 H, i,i′-CH_2), 2.10 (complex m, 4H, d,d′-CH_2), 3.40 (s, 18H, a,a′-OCH_3), 3.50 (br.s, 1H, g or g′-CH), 3.65 (br.s, 1H, h-CH), 3.75 (br.s, 1H, g or g′-CH), 4.05 (q, 8H, $4 \times OCH_2$), 6.15 (m, 1 H, f-CH), 6.46 (d, 1H, e′-CH, $J_{e'f'}$ 5 Hz), 6.73 (dd, 1H, f′-CH, $J_{f'g'}$ 3 Hz) ppm. ^{13}C NMR ($CDCl_3$, 20.1 MHz, Me_4Si ext. ref.): δ 8.83 (d), 9.77 (d), 13.97 (q), 19.48 (t), 20.43 (t), 32.37 (m), 34.21 (m), 50.29 (q), 50.73 (s), 53.24 (d), 56.57 (d), 60.7 (t), 71.4 (t), 73.5 (t), 132.7 (d), 143.6 (d), 144.9 (d), 148.1 (s), 151.8 (s), 152.8 (s), 157.8 (s), 158.1 (s), 163.5 (s), 164.8 (s), 165.1 (s), 166.3 (s) ppm. MS: m/z 398 $[M]^+$ 3.0%.

(c) With phenylvinylsulphoxide. A solution of phenylvinylsulphoxide (0.59 g, 3.87 mmol) and **1** (1.22 g, 5.4 mmol) in toluene was kept at 100 °C for 24 h and gave a deep yellow oil after removal of the solvent. Flash chromatography (silica, 1/1 v/v ethyl acetate/toluene eluant) gave the dimer of **1** (0.02 g), unchanged phenylvinylsulphoxide (0.37 g), and the adduct **3** (0.1 g, 0.36 mmol, 5%) as a pale yellow oil. Compound **3** is assumed to be a mixture of the four possible isomers **3a** and **3b**. IR (neat liquid): ν 3050m (=CH), 2930vs (CH_3), 2830ssh (CH), 1620m (C=C), 1580m (C=C), 1440s (C–H def.), 800vs (C–H def.) cm^{-1}. ^1H NMR ($CDCl_3$, 220 MHz, Me_4Si ext. ref.): δ 0.5 (complex m, 4H, b,b′-CH_2), 1.05 (complex m, 4H, k,k′- or j,j′-CH_2), 1.40 (complex m, 4H, c,c′-CH_2), 1.8–2.3 (complex m, 6H, CHOSPh and i,i′-CH_2), 2.50 (complex m, 4H, d,d′-CH_2), 3.40 (br.s, ca. 21H, a,a′-OCH_3, and h,g,g′-CH), 5.70–6.05 (complex m, 3H, e′,f,f′-CH), 7.25 (complex m, 6H, aromatic), 7.45 (complex m, 4H, aromatic) ppm.

(d) With trans-1,2-bis(diphenylphosphineoxide)ethane. A mixture of **1** (0.89 g, 3.9 mmol) and *trans*-1,2-bis(diphenylphosphineoxide)ethane (0.23 g, 0.54 mmol) was heated in a thick-walled glass reaction tube at 140 °C for 20 h to give a yellow oil. Addition of hexane gave a yellow precipitate, which was found on flash chromatography (silica; 1/8 v/v ethanol/toluene eluant) to be a mixture of the dimer of **1**, and the adduct **5** (0.4 g, 0.6 mmol, 36%) as an approximate 1/1 mixture of isomers **5a** and **5b**, m.p. 150–152 °C (decomp.) (Found: C, 67.20; H, 6.30; P, 9.0. $C_{42}H_{42}O_5P_2Si$ calcd.: C, 67.85; H, 6.40; P. 9.45%). IR (Nujol/HCB mull): ν 3050m (=CH), 1620w (C=C), 1260vs, 1185s (P=O) cm^{-1}. ^1H NMR ($CDCl_3$, 220 MHz, Me_4Si ext. ref.) δ 0.5 (complex m, 4H, b,b′-CH_2), 1.10 (complex m, 1H, h-CH), 1.40 (complex m, 4H, c,c′-CH_2), 1.70 (br, 4H, i,i′-CH_2), 2.0 (complex m, 4H, d,d′-CH_2), 2.55 (br, 1H, g′-CH), 2.75 (br, 1H, g′-CH), 3.1 (complex m, 2H, j,k-CH), 3.35 (s, 9H, a or a′-OCH_3), 3.40 (s, 9H, a or a′-OCH_3), 6.85 (complex m, 3H, f,f′,e-CH), 7.15 (complex m, 8H, aromatic), 7.30 (complex m, 12H, aromatic), 7.50 (complex m, 12H, aromatic) ppm; ^{31}P NMR ($CDCl_3$, 32.4 MHz, BB decoupled): 26.06 (d, J 9 Hz), 28.96 (d, J 9 Hz) ppm.

(e) With trans-1,2-dicyanoethene. When **1** (1.74 g, 7.63 mmol) and *trans*-1,2-dicyanoethene (0.60 h, 7.68 mmol) were heated in dry toluene (30 cm^3) at 45 °C for 4 h the adduct **6** (1.8 g, 5.88 mmol, 77%) was obtained as a pale yellow oil after purification by flash chromatography (silica; 1/1 v/v toluene/ethyl acetate eluant). (Found: C, 58.9; H, 7.4; N, 9.5. $C_{15}H_{22}N_2O_3Si$ calcd.: C, 58.8, H, 7.2; N, 9.15%). ^1H and ^{13}C NMR spectroscopy indicates that **6** is a mixture of the isomers **6a** and **6b**. IR (neat liquid): ν 3050w (=C–H), 2940vs (-CH_2), 2930s (OCH_3), 2130vs (C≡N), 1620m, 1610w (C=C), 1460vs (C–H def.), 1410m, 1340m (CH_3 def.) 1260s (C–O str.), 1200s, 1100br.s (C–O str.), 810vs, 730m (C–H def.) cm^{-1}. ^1H NMR

($CDCl_3$, 220 MHz, Me_4Si ext. ref.): 0.50 (complex m, 4H, b,b′-CH_2), 1.40 (complex m, 4H, c,c′-CH_2), 1.80 (complex m, 4H, i,i′-CH_2), 2.05 (t, 2H, J 7 Hz, d′-CH_2), 2.15 (t, 2H, J 7 Hz, d-CH_2), 2.35 (complex m, 2H, g,g′-CH), 2.80 (d, 1H, J 15 Hz, h-CH), 3.05, 3.15 (complex m, 4H, j,k,j′,k′-CH), 3.35 (s, 18H, a,a′-CH_3O), 5.70 (d, 1H, $J_{e'f'}$ 9.8 Hz, e′-CH), 6.0 (dd, 1H, J 7 Hz, f′-CH), 6.20 (complex m, 1H, f-CH) ppm. ^{13}C NMR ($CDCl_3$, 20.1 MHz, Me_4Si ext. ref.) 8.4 (t), 9.3 (t), 19.0 (t), 19.3 (t), 19.7 (t), 31.9 (t), 33.2 (t), 35.1 (t), 38.5 (d), 45.7 (d), 46.4 (d), 46.9 (d), 48.0 (d), 48.9 (d), 49.6 (d), 50.6 (q), 58.8 (s), 118.6 (s), 118.9 (s), 119.4 (s), 119.8 (s), 126.9 (d), 127.7 (d), 135.7 (d), 136.8 (d), 138.5 (d), 139.2 (d), 150.5 (s), 151.4 (s) ppm. MS: m/z M^+ not seen, 276 $(M\text{-OMe})^+$ 1.4%.

Preparation of $SIL\text{-}CH_2CH_2CH_2C_7H_5(CN)_2PdCl_2$ *(SIL = silica)*

Silica-gel (Grace–Davison 952 grade) was heated at 300 °C for 24 h. It was then suspended in 10% aqueous hydrochloric acid for ca. 2 h, filtered off, resuspended in 10% aqueous hydrochloric acid, filtered off, and washed with water until neutral. It was dried at 120 °C and then at 170 °C in vacuo for 12 h.

In a typical preparation compound **6** (273 mg, 0.89 mmol) in dry toluene (5 cm^3) was added to a stirred suspension of the silica (1.02 g) in toluene (10 cm^3). The suspension was heated at the reflux temperature overnight, then cooled and filtered, and the solid collected was washed with toluene and dried in vacuo at 80 °C overnight. A suspension of the silylated dinitrile (1.0 g) in trimethylchlorosilane (20 cm^3) was stirred for 6 h, and then filtered. The solid collected was washed with toluene and dried in vacuo at 80 °C overnight (Found: N, 0.9%). A solution of bis(benzonitrile)palladium(II) chloride (0.13 g, 0.34 mmol) in benzene (10 cm^3) was added to a stirred suspension of the silylated dinitrile (0.5 g) in benzene (10 cm^3) and the mixture was stirred at room temperature for 24 h then filtered. The solid collected was washed with toluene and dried in vacuo at room temperature overnight. (Found: Pd, 1.3; N, 0.7; Cl, 1.6%).

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