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Photochemical synthesis of isomeric (*E*/*Z*)-3-alkylidene-3*H*-isobenzofuranones

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Abstract—The synthesis of isomeric (*E*/*Z*)-3-alkylidene-3*H*-isobenzofuranones by photoisomerization of 2-aroyl-2-methyl/benzylindan-1,3-diones in high yields is described.

1. Introduction

There is significant interest in the synthesis[1] of naturally occurring (*E*/*Z*)-3-alkylidene-3*H*-isobenzofuranones[2] because of their wide range of biological properties[3] and use as valuable intermediates[4] in the synthesis of a variety of ring systems. It has been reported that natural and unnatural (*Z*)-3-alkylidene-3*H*-isobenzofuranones and 3-substituted isocoumarins can be obtained regioselectively by cyclization of 2-(1-alkynyl)benzoic acid by a transition metal-catalyzed reaction.[5] Furthermore, the synthesis of (*E*)-3-alkylidene-3*H*-isobenzofuranones may be accomplished by Jones oxidation of disubstituted alkynes (obtained from 2-iodobenzylalcohol and acetylenic carbinols) in the presence of bis(triphenylphosphine)palladium (II) chloride.[6]

Recently, a facile synthesis of (*E*/*Z*)-3-alkylidene-3*H*-isobenzofuranones has been described, which uses the DABCO-catalyzed Baylis–Hillman reaction of 2-carboxybenzaldehyde with some activated vinyl compounds.[7] We have also reported the stereoselective synthesis of (*Z*)-3-alkylidene-3*H*-isobenzofuranones through photo-reorganization of the corresponding 2-alkoxy-2-arylindane-1,3-dione.[8] In continuation of these studies, we report herein the photoisomerization of 2-aroyl-2-methyl/benzylindan-1,3-diones (**2**) that resulted in the formation of a mixture of isomeric (*E*/*Z*)-3-alkylidene-3*H*-isobenzofuranones (**3** and **4**) in 86–91% yields.

2. Results and discussion

2-Aroyl-2-methyl/benzylindan-1,3-diones (**2**), required for photolysis, were prepared by C-alkylation of 2-aroylindan-1,3-dione (**1**) with methyl iodide/benzyl chloride in the presence of anhydrous potassium carbonate and potassium iodide in refluxing dry acetone in 41–55% yields. Photoirradiation of **2** in dry benzene with a 450 W Hanovia mercury vapor lamp under a nitrogen atmosphere in a pyrex reactor resulted in the formation of mixture of two isomeric products i.e. (*E*)-3-alkylidene-3*H*-isobenzofuranones (**3**) (42–46%) and (*Z*)-3-alkylidene-3*H*-isobenzofuranones (**4**) (44–46%), which were separated by column chromatography (Scheme 1).

The mechanism may be easily rationalized in terms of α-cleavage (Norrish type I reaction) to generate a biradical intermediate, which reorganizes in two ways perhaps almost equally and readily followed by cyclization resulting in the formation of **3** and **4** (Scheme 2).

The structures of **2**–**4** were established from their physical and spectroscopic data. The stereochemistry of *E*- and *Z*-isomers was based on the IR and ^{1}H NMR spectra as well as by analogy with similarly constituted compounds.[7] The occurrence of the lactone carbonyl group at 1785 cm^{-1} and the other conjugated carbonyl group at 1655 cm^{-1} and similarly bands at 1779 and 1655 cm^{-1} were attributed to **3a** and **4a**, respectively. The ^{1}H NMR spectrum of **3a** demonstrated that the doublet of doublets due to C_4-H is shielded (δ 7.27) probably due to the anisotropic effect of the aryl group in comparison to C_4-H of **4a** in which there is no signal in between δ 7.2 and 7.4. The spectra of **3b** and **3c** also illustrated that C_4-H is shielded in comparison to C_4-H of **4b** and **4c** probably due to the anisotropic effect of phenyl group

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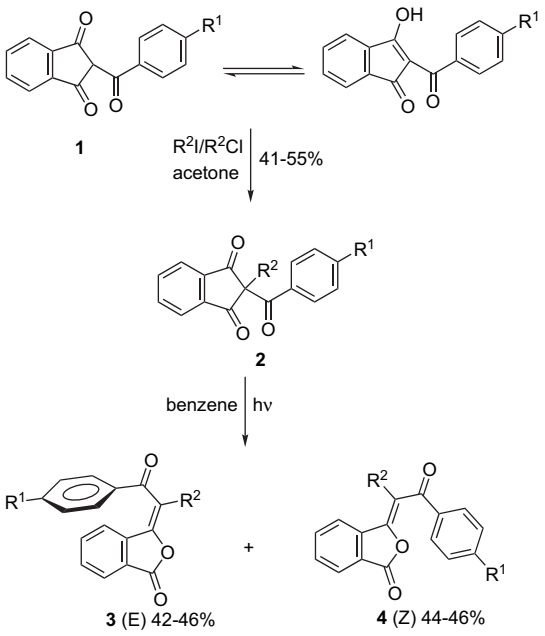
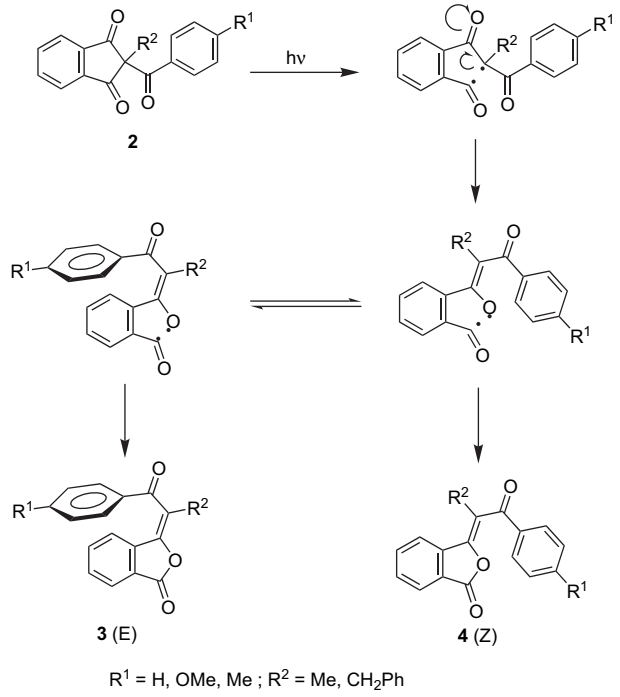


Scheme 1.



Scheme 2.

(vide experimental). Both isomers exist in the more stable *s*-trans conformation rather than the *s*-cis conformation as indicated by the position of the carbonyl group bands in IR spectra and is well supported by ^{1}H NMR spectroscopic data. There was no substantial difference in the mass spectra of **3** and **4**. However, it was hard to explain how the signal due to C_4-H emerged at δ 8.96–9.07 in (*E*)-3-alkylidene-3*H*-isobenzofuranones as claimed by the authors.[6] It was also mentioned that *E*-isomers were transformed completely to *Z*-isomers on treatment with concd H_2SO_4. In contrast, in our case when photolysis of the *E* or *Z*-isomer was carried out under similar conditions, as employed in their synthesis, we observed the formation of a mixture of *E*/*Z*-isomers in almost equal ratio as indicated by the ^{1}H NMR spectra of the crude photolysate and comparison with authentic samples through TLC.

To study the effect of solvent, the photolysis of **2** was also carried out in methanol. A mixture of two isomeric (*E*/*Z*)-3-alkylidene-3*H*-isobenzofuranones (**3** and **4**) was obtained. The only difference was that longer irradiation time (7–10 h) was required and the reaction did not proceed to completion. This may be due to the formation of intermolecular hydrogen bonding, between the starting material (**2**) and methanol, resulting in an increase in the energy gap between the ground state and the excited state and which slows down the rate of these transformations.

In conclusion, the present photochemical method allows convenient synthesis of important isomeric (*E*/*Z*)-3-alkylidene-3*H*-isobenzofuranones (**3** and **4**) in high yields utilizing simple and inexpensive starting materials and is much better than the previous methods that involve distinctive starting materials, metal-catalyzed reaction, and more cumbersome experimental work-up.[5–7]

3. Experimental

3.1. General

Melting points were observed in open capillaries and are uncorrected. UV spectra were taken in methanol solution on U-2000 (Hitachi) UV spectrophotometer. IR spectra were scanned in Nujol on Perkin–Elmer 842 IR spectrophotometer. NMR spectra were recorded in $CDCl_3$ using TMS as an internal standard on 200 MHz Bruker AM-200 spectrometer and 90 MHz Perkin Elmer spectrometer. Mass spectra were recorded at 70 eV using a VG-70S instrument. Silica gel (100–200 mesh) was used for column chromatography. The columns were packed with silica gel in light-petroleum (60–80 °C) and left overnight before separation. Solvents were dried using standard literature procedures. 2-Aroylindan-1,3-dione (**1**) required for alkylation was prepared according to a literature procedure.[9]

3.2. 2-Aroyl-2-methyl/benzylindan-1,3-dione (2): general procedure

A mixture of 2-aroylindan-1,3-dione (**1**) (0.02 mol), methyl iodide/benzyl chloride (0.08 mol), anhydrous potassium carbonate (11.0 g, 0.08 mol), and potassium iodide (0.5 g, 0.003 mol) was refluxed in dry acetone (200 mL) for 10 h. The reaction mixture was cooled and the inorganic salts filtered. Excess of acetone was evaporated and the reaction mixture dispensed into water and extracted with ether (3×50 mL). The combined ether extracts were dried over anhydrous magnesium sulfate, filtered, and ether distilled off. The crude product thus obtained was crystallized from benzene–light-petroleum mixture to yield **2**.

Compound **2a**; 2.16 g, 41%; colorless crystals, mp 127–128 °C; UV: λ_{max}=239, 382 nm; IR: 1742, 1700,

1677 cm^{-1}; ^1H NMR (90 MHz): δ 1.73 (s, 3H, CH_3), 7.20–7.50 (m, 3H, H-3′, H-4′, H-5′), 7.69 (dd, J=8.0, 2.5 Hz, 2H, H-2′, H-6′), 7.83–8.17 (m, 4H, H-4, H-5, H-6, H-7); Anal. Calcd for $C_{17}H_{12}O_3$: C, 77.24; H, 4.54. Found C, 77.31; H, 4.48.

Compound **2b**; 2.94 g, 50%; colorless crystals, mp 125–126 °C; IR: 1749, 1710, 1652 cm^{-1}; ^1H NMR (90 MHz): δ 1.68 (s, 3H, CH_3), 3.75 (s, 2H, OCH_3), 6.75 (d, J=8.5 Hz, 2H, H-3′, H-5′), 7.65 (d, J=8.5 Hz, 2H, H-2′, H-6′), 7.77–8.13 (m, 4H, H-4, H-5, H-6, H-7); Anal. Calcd for $C_{18}H_{14}O_4$: C, 73.46; H, 4.76. Found C, 73.59; H, 4.66.

Compound **2c**; 2.55 g, 46%; colorless crystals, mp 180–181 °C; UV: λ_{max}=215 nm; IR: 1749, 1710, 1675 cm^{-1}; ^1H NMR (90 MHz): δ 1.67 (s, 3H, CH_3), 2.25 (s, 3H, CH_3), 7.02 (d, J=8.0 Hz, 2H, H-3′, H-5′), 7.51 (d, J=8.5 Hz, 2H, H-2′, H-6′), 7.72–8.10 (m, 4H, H-4, H-5, H-6, H-7); Anal. Calcd for $C_{18}H_{14}O_3$: C, 77.69; H, 5.03. Found C, 77.80; H, 5.11.

Compound **2d**; 3.46 g, 51%; colorless crystals, mp 140–141 °C; UV: λ_{max}=225, 367 nm; IR: 1741, 1701, 1670 cm^{-1}; ^1H NMR (90 MHz): δ 3.70 (s, 2H, $CH_2C_6H_5$), 6.90 (s, 5H, $CH_2C_6H_5$), 7.12–7.42 (m, 3H, H-3′, H-4′, H-5′), 7.50–7.91 (m, 6H, H-4, H-5, H-6, H-7, H-2′, H-6′); Anal. Calcd for $C_{23}H_{16}O_3$: C, 81.17; H, 4.70. Found C, 81.29; H, 4.61.

Compound **2e**; 3.84 g, 52%; colorless crystals, mp 152–153 °C; UV: λ_{max}=209, 224 nm; IR: 1741, 1703, 1674 cm^{-1}; ^1H NMR (90 MHz): δ 3.70 (s, 2H, $CH_2C_6H_5$), 3.73 (s, 3H, OCH_3), 6.71 (d, J=8.5 Hz, 2H, H-3′, H-5′), 6.91 (s, 5H, $CH_2C_6H_5$), 7.50–7.90 (m, 6H, H-4, H-5, H-6, H-7, H-2′, H-6′); Anal. Calcd for $C_{24}H_{18}O_4$: C, 77.83; H, 4.86. Found C, 77.65; H, 4.64.

Compound **2f**; 3.89 g, 55%; colorless crystals, mp 167–168 °C; UV: λ_{max}=210, 313 nm; IR: 1741, 1707, 1674 cm^{-1}; ^1H NMR (90 MHz): δ 2.27 (s, 3H, CH_3), 3.72 (s, 2H, $CH_2C_6H_5$), 6.92 (s, 5H, $CH_2C_6H_5$), 7.03 (d, J=8.0 Hz, 2H, H-3′, H-5′), 7.50 (d, J=8.0 Hz, 2H, H-2′, H-6′), 7.58–7.99 (m, 4H, H-4, H-5, H-6, H-7); Anal. Calcd for $C_{24}H_{18}O_3$: C, 81.35; H, 5.08. Found C, 81.57; H, 5.22.

3.3. (*E* and *Z*)-3-Alkylidene-3*H*-isobenzofuranones (3 and 4): general procedure

A solution of 2-aroyl-2-methyl/benzylindan-1,3-dione (**2**, 500 mg), in dry benzene (80 mL) was deoxygenated by bubbling dry nitrogen gas for 15 min. The solution was then irradiated with a 450 W Hanovia mercury vapor lamp in a pyrex reactor under nitrogen atmosphere. The reaction progress was monitored by TLC (benzene–EtOAc 19:1) on aliquots withdrawn from the reaction mixture at different intervals of time. After 3 h of irradiation, TLC showed complete absence of starting compound. The solvent was distilled off and the resulting viscous photolysate was chromatographed over a column of silica gel (30 g, packed in light-petroleum). The column was then eluted initially with light-petroleum followed by benzene–light-petroleum (1:1) affording firstly **3**, followed by subsequent elution with benzene only provided **4** in high yields.

3.3.1. *E*-3-Alkylidene-3*H*-isobenzofuranones (3). Compound **3a**; 210 mg, 42%; colorless crystals, mp 78–79 °C; UV: λ_{max}=235, 334 nm; IR: 1785, 1655 cm^{-1}; ^1H NMR (200 MHz): δ 2.32 (s, 3H, CH_3), 7.27 (dd, J=8.6, 3.4 Hz, 1H, H-4), 7.46–7.70 (m, 5H, H-5, H-6, H-3′, H-4′, H-5′), 7.91 (dd, J=8.0, 3.2 Hz, 1H, H-7), 8.01 (d, J=7.3 Hz, 2H, H-2′, H-6′); MS: m/z (%) 264 (M^+, 21), 187 (5), 159 (6), 131 (4), 105 (100), 103 (7), 77 (38), 76 (9), 51 (9); Anal. Calcd for $C_{17}H_{12}O_3$: C, 77.24; H, 4.54. Found C, 77.36; H, 4.45.

Compound **3b**; 220 mg, 44%; colorless crystals, mp 119–120 °C; UV: λ_{max}=263, 295 nm; IR: 1778, 1655 cm^{-1}; ^1H NMR (200 MHz): δ 2.32 (s, 3H, CH_3), 3.85 (s, 3H, OCH_3), 6.94 (d, J=8.9 Hz, 2H, H-3′, H-5′), 7.20–7.30 (m, 1H, H-4), 7.30–7.55 (m, 2H, H-5, H-6), 7.84 (dd, J=8.0, 2.1 Hz, 1H, H-7), 7.95 (d, J=8.9 Hz, 2H, H-2′, H-6′); MS: m/z (%) 294 (M^+, 18), 263 (10), 136 (13), 135 (100), 107 (11), 92 (12), 77 (23); Anal. Calcd for $C_{18}H_{14}O_4$: C, 73.46; H, 4.76. Found C, 73.59; H, 4.63.

Compound **3c**; 210 mg, 42%; colorless crystals, mp 119–120 °C; UV: λ_{max}=263, 311 nm; IR: 1780, 1650 cm^{-1}; ^1H NMR (200 MHz): δ 2.32 (s, 3H, CH_3), 2.44 (s, 3H, CH_3), 7.25 (dd, J=8.4, 2.1 Hz, 1H, H-4), 7.31 (d, J=8.2 Hz, 2H, H-3′, H-5′), 7.47–7.56 (m, 2H), 7.89–7.93 (m, 3H); MS: m/z (%) 278 (M^+, 11), 263 (13), 120 (12), 119 (100), 105 (12), 91 (39), 77 (13), 65 (14); Anal. Calcd for $C_{18}H_{14}O_3$: C, 77.69; H, 5.03. Found C, 77.73; H, 5.01.

Compound **3d**; 215 mg, 43%; colorless crystals, mp 128–129 °C; UV: λ_{max}=248, 314 nm; IR: 1784, 1665 cm^{-1}; ^1H NMR (200 MHz): δ 4.13 (s, 2H, CH_2), 7.10–7.26 (m, 6H), 7.34–7.63 (m, 5H), 7.83 (dd, J=7.7, 2.0 Hz, 2H, H-2′, H-6′), 7.92 (dd, J=7.6, 2.1 Hz, 1H, H-7); MS: m/z (%) 340 (M^+, 58), 235 (88), 207 (13), 178 (18), 105 (100), 91 (36), 77 (62); Anal. Calcd for $C_{23}H_{16}O_3$: C, 81.17; H, 4.70. Found C, 81.15; H, 4.65.

Compound **3e**; 230 mg, 46%; colorless crystals, mp 147–148 °C; UV: λ_{max}=266, 297 nm; IR: 1782, 1654 cm^{-1}; ^1H NMR (200 MHz): δ 3.82 (s, 3H, OCH_3), 4.12 (s, 2H, CH_2), 6.85 (d, J=8.8 Hz, 2H, H-3′, H-5′), 7.10–7.25 (m, 6H), 7.37–7.48 (m, 2H), 7.81 (d, J=8.8 Hz, 2H, H-2′, H-6′), 7.91 (dd, J=6.4, 2.0 Hz, 1H, H-7); MS: m/z (%) 370 (M^+, 14), 339 (15), 135 (100), 107 (13), 105 (27), 104 (39), 92 (13), 91 (97), 77 (35), 65 (19); Anal. Calcd for $C_{24}H_{18}O_4$: C, 77.83; H, 4.86. Found C, 77.99; H, 4.65.

Compound **3f**; 220 mg, 44%; colorless crystals, mp 137–138 °C; UV: λ_{max}=262, 311 nm; IR: 1775, 1655 cm^{-1}; ^1H NMR (200 MHz): δ 2.38 (s, 3H, CH_3), 4.12 (s, 2H, CH_2), 7.12–7.26 (m, 8H), 7.37–7.50 (m, 2H), 7.74 (d, J=8.1 Hz, 2H, H-2′, H-6′), 7.91 (dd, J=6.6, 2.1 Hz, 1H, H-7); MS: m/z (%) 354 (M^+, 29), 340 (22), 339 (78), 235 (28), 119 (100), 91 (51), 65 (13); Anal. Calcd for $C_{24}H_{18}O_3$: C, 81.35; H, 5.08. Found C, 81.40; H, 5.00.

3.3.2. *Z*-3-Alkylidene-3*H*-isobenzofuranones (4). Compound **4a**; 220 mg, 44%; colorless crystals, mp 124–125 °C; UV: λ_{max}=234, 334 nm; IR: 1779, 1655 cm^{-1}; ^1H NMR (200 MHz): δ 2.46 (s, 3H, CH_3), 7.42–7.68 (m, 4H),

7.78–8.00 (m, 5H); MS: *m/z* (%) 264 (M^+, 18), 187 (4), 159 (3), 131 (4), 105 (100), 103 (8), 77 (41), 76 (10), 51 (10); Anal. Calcd for $C_{17}H_{12}O_3$: C, 77.24; H, 4.54. Found C, 77.41; H, 4.42.

Compound **4b**; 225 mg, 45%; colorless crystals, mp 161–162 °C; UV: λ_{max}=262, 290 nm; IR: 1778, 1655 cm^{-1}; ^1H NMR (200 MHz): δ 2.43 (s, 3H, CH_3), 3.85 (s 3H, OCH_3), 6.93 (d, *J*=8.9 Hz, 2H, H-3′, H-5′), 7.62 (dd, *J*=8.5, 2.5 Hz, 1H, H-4), 7.76–7.97 (m, 5H, H-5, H-6, H-7, H-2′, H-6′); MS: *m/z* (%) 294 (M^+, 18), 263 (9), 136 (11), 135 (100), 107 (8), 92 (10), 77 (17); Anal. Calcd for $C_{18}H_{14}O_4$: C, 73.46; H, 4.76. Found C, 72.53; H, 4.69.

Compound **4c**; 230 mg, 46%; colorless crystals, mp 128–129 °C; UV: λ_{max}=264, 312 nm; IR: 1779, 1655 cm^{-1}; ^1H NMR (200 MHz): δ 2.42 (s, 3H, CH_3), 2.44 (s, 3H, CH_3), 7.26 (d, *J*=8.2 Hz, 2H, H-3′, H-5′), 7.65 (dd, *J*=8.4, 2.1 Hz, 1H, H-4), 7.77–7.86 (m, 3H, H-5, H-6, H-7), 7.97 (d, *J*=8.3 Hz, 2H, H-2′, H-6′); MS: *m/z* (%) 278 (M^+, 14), 263 (14), 120 (13), 119 (100), 105 (7), 91 (36), 77 (9), 65 (14); Anal. Calcd for $C_{18}H_{14}O_3$: C, 77.69; H, 5.03. Found C, 77.81; H, 5.11.

Compound **4d**; 220 mg, 44%; colorless crystals, mp 147–148 °C; UV: λ_{max}=250, 314 nm; IR: 1779, 1654 cm^{-1}; ^1H NMR (200 MHz): δ 4.30 (s, 2H, CH_2), 7.17–7.42 (m, 7H), 7.50 (dd, *J*=7.3, 2.1 Hz, 1H, H-4′), 7.60–7.83 (m, 4H), 7.94–8.04 (m, 2H); MS: *m/z* (%) 340 (M^+, 54), 235 (76), 207 (11), 178 (15), 105 (100), 91 (22), 77 (56); Anal. Calcd for $C_{23}H_{16}O_3$: C, 81.17; H, 4.70. Found C, 81.19; H, 4.68.

Compound **4e**; 225 mg, 45%; colorless crystals, mp 126–127 °C; UV: λ_{max}=265, 292 nm; IR: 1777, 1647 cm^{-1}; ^1H NMR (200 MHz): δ 3.83 (s, 3H, OCH_3), 4.28 (s, 2H, CH_2), 6.84 (d, *J*=8.8 Hz, 2H, H-3′, H-5′), 7.17–7.35 (m, 5H), 7.60–7.76 (m, 2H), 7.80 (d, *J*=8.8 Hz, 2H, H-2′, H-6′), 7.96–8.03 (m, 2H); MS: *m/z* (%) 370 (M^+, 24), 339 (30), 135 (100), 107 (13), 105 (25), 104 (7), 92 (12), 91 (11), 77 (23), 65 (8); Anal. Calcd for $C_{24}H_{18}O_4$: C, 77.83; H, 4.86. Found C, 77.92; H, 4.68.

Compound **4f**; 225 mg, 45%; colorless crystals, mp 145–146 °C; UV: λ_{max}=262, 313 nm; IR: 1775, 1655 cm^{-1}; ^1H NMR (200 MHz): δ 2.36 (s, 3H, CH_3), 4.28 (s, 2H, CH_2), 7.13–7.30 (m, 7H), 7.59–7.80 (m, 4H), 7.96–8.02 (m, 2H); MS: *m/z* (%) 354 (M^+, 27), 340 (25), 339 (80), 235 (31), 119 (100), 91 (60), 65 (18); Anal. Calcd for $C_{24}H_{18}O_3$: C, 81.35; H, 5.08. Found C, 81.26; H, 5.21.

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