in a refrigerator. The yellow exterior (autoxidized material) is much more soluble in acetic acid than is diindene itself.

The blue triboluminescence observed with solidified diindene appears compatible with two of the types which have been reported: (1) the nitrogen gas discharge spectrum type of triboluminescence attributed to a piezoelectric effect in noncentrosymmetric crystals[8] observed with crystalline sugars such as sucrose[8,9] or with aniline hydrochloride[10,11] or (2) triboinduced photoluminescence (probably related to the blue fluorescence associated with the liquid) observed with aromatic compounds[11] such as resorcinol,[11] coumarin,[11,12] and phenanthrene.[11] We have not carried out the studies required to make a distinction. Also, we have not examined (but hope that others will) the structure of the easily formed autoxidation product(s) of solidified diindene, which may have its (their) origin in the same crystal strain which gives rise to triboluminescence in this form of the dimer.

Experimental Section

Diindene [2-(2,3-Dihydro-1H-inden-1-yl)-1H-indene] (2a). The general procedure is that of Weissgerber,[2] except that vigorous sitrring was used and the reflux time was increased from 15 to 23 h. 1H-Indene (>97.5% purity, 200 g, 1.68 mol) was added to a solution of 85% phosphoric acid (200 mL) in water (200 mL), and the resulting two-phase mixture was refluxed with vigorous stirring for 23 h. The progress of the dimerization was followed by comparison of the refractive index of the indene–diindene layer with synthetic mixtures of the two prepared as calibration standards. At 4 h the dimerization was 52% complete (n^{24}_D 1.600), and at 23 h, when the reaction was terminated, it was 97.5% complete (n^{24}_D 1.621). The pale yellow organic layer was separated, dried (KOH), and fractionally distilled, giving 1H-indene as a colorless forerun (8.85 g, 4% recovery), bp 30–35 °C (0.9 mm), n^{24}_D 1.574, followed by diindene as a colorless but bright blue fluorescent (under UV light) liquid (156.0 g, 78%), bp 157–161 °C (0.9 mm), n^{24}_D 1.622, which solidified after being kept at room temperature or in a refrigerator to an extremely hard white crystalline solid, mp 42–52 °C. The solid emitted flashes of blue light when broken, struck, or scratched vigorously with a spatula: literature nearly quantitative,[2] 55%;[6b] bp 154 (0.4 mm),[13] 160–165 (1 mm),[4] 177 (1 mm),[13] 145–160 (1–2 mm),[6c] 210 (12 mm),[6b] 235–245 (16 mm),[2] and 355 °C (755 mm);[13] n^{20}_D 1.588;[14] mp 50–51,[3] 51,[2] and 53 °C.[6b] The NMR spectrum (in CDCl$_3$) was the same, except for an impurity peak at δ 0.98, as that reported (in CCl$_4$) below. Two recrystallizations from acetic acid gave white needles: mp 59–60 °C (lit. mp 55–56,[4] 56,[6c] 57–58,[3] and 57–59 °C[5]); UV (95% C$_2$H$_5$OH) λ$_{max}$ nm (log ε) 224 sh (4.17), 259 (4.21), 263 sh (4.18), 269 sh (4.11), 272 sh (3.99), 282 sh (3.32), 288 sh (2.84); IR (CCl$_4$) cm^{-1} 1610 m (C=C); NMR (17% w/v, CCl$_4$) δ 2.22 (m, $W_{1/2}$ = 47.5 Hz, 2 H, CH$_2$), 2.90 (m, $W_{1/2}$ = 24 Hz, 2 H, CH$_2$), 3.21 (br s, $W_{1/2}$ = 3 Hz, 2 H, CH$_2$), 4.25 (t, J = 8 Hz, 1 H, CH), 6.53 (br s, $W_{1/2}$ = 3.5 Hz, 1 H, =CH), 7.12 (m, major peak at δ 7.08, $W_{1/2}$ = 25 Hz, 8 H, aromatic H).

Anal. Calcd for C$_8$H$_{16}$ (232.31): C, 93.06; H, 6.94. Found: C, 92.96; H, 7.24.

3-Methylindene Dimer [3-Methyl-2-(2,3-dihydro-1-methyl-1H-inden-1-yl)-1H-indene] (2b). A mixture of 3-methyl-1H-indene (18.16 g, 139 mmol), 85% phosphoric acid (20 mL), and water (20 mL) was refluxed with vigorous stirring for 36 h. The progress of the dimerization was followed by rate of change of the refractive index of the monomer–dimer layer. When the dimerization appeared essentially complete (at 36 h), the yellow organic layer was separated, dried (KOH), and fractionally distilled, giving 3-methyl-1H-indene as a colorless forerun (2.07 g, 11% recovery), bp 40–49 °C (0.6 mm), n^{25}_D 1.552, followed by dimer **2b** as a yellow viscous oil (7.93 g, 44%), bp 140–150 °C (0.6 mm), n^{25}_D 1.6091. Redistillation of the product gave a viscous oil, collected in two fractions: (1) yellow (1.07 g, 6%), bp 156–162 °C (0.8 mm), n^{25}_D 1.6078; and (2) light yellow (2.89 g, 16%), bp 162–166 °C (0.8 mm), n^{25}_D 1.6088, which was used as an analytical sample; UV (95% C$_2$H$_5$OH) λ$_{max}$ nm (log ε) 227 sh (3.84), 261 (4.04), 272 sh (3.90); IR (neat) cm^{-1} 1595 m (C=C); NMR (38% w/v, CCl$_4$) δ 1.30 (s, 0.4 H, impurity), 1.52 (s, 3 H, CH$_3$), 1.66 (t, J = 1.7 Hz, 3 H, CH$_3$), 2.22 (m, $W_{1/2}$ = 38 Hz, 2 H, CH$_2$), 2.83 (q, J ≈ 8 Hz, 2 H, CH$_2$), 3.24 (incompletely resolved q, J = 1.7 Hz, 2 H, CH$_2$), 7.11 (m, major peak at δ 7.07, $W_{1/2}$ = 26 Hz, 8 H, aromatic H).

Anal. Calcd for C$_{20}$H$_{20}$ (260.36): C, 92.26; H, 7.74. Found: C, 91.97; H, 8.36.

References and Notes

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Synthesis of 1,2-Disubstituted Acenaphthylenes

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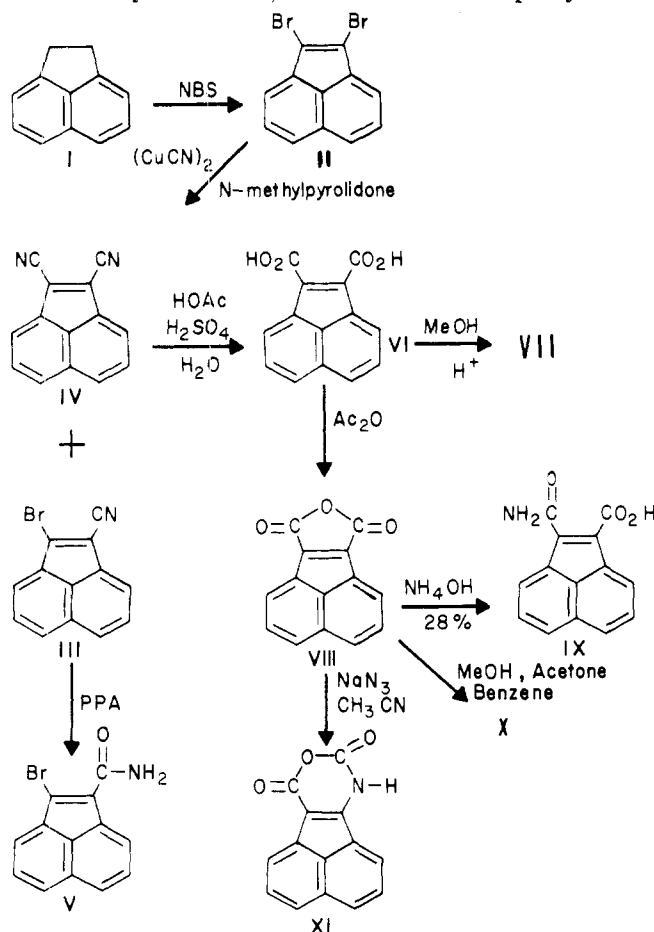
Our initial interest in the synthesis of 1,2-disubstituted acenaphthylenes stemmed from our need to prepare acenaphthylene-1-diazonium-2-carboxylate (XII). This high energy compound (XII) should in theory thermally or photochemically decompose to form acenaphthyne, a benzyne analogue which had eluded the intensive efforts of Rasheed.[3] Subsequently, our interest in the photochemical investigation of four of these 1,2-disubstituted acenaphthylenes[4] was encouraged by the works of (1) Bouas-Laurent et al.[5] (photodimerization of monosubstituted CN and CO$_2$H acenaphthylenes), (2) Michl et al.[6] (photodimerization of 1,2-dibromoacenaphthylene and acenaphthylene to form the mixed dimer), and (3) Cowan and Drisko[7] (investigations of mechanisms and heavy atom effects in the dimerization of acenaphthylene). Additionally, several of these compounds have been studied by magnetic circular dichroism,[8] and the 1,2-dicyanoacenaphthylene has been studied by electron spin resonance.[9] The syntheses of nine of these compounds are herein reported.

Results and Discussion

The synthetic route planned for the diazonium carboxylate precursor (XI) is shown in Scheme I; the synthesis, a sequence of five steps initiated with acenaphthene, is a straightforward procedure with a sequence yield of 29%. The byproduct of this sequence of reactions and any compounds prepared from main sequence products are discussed along with their main se-

Scheme I. Synthesis of 1,2-Disubstituted Acenaphthylenes

(X). VI was dehydrated to yield VIII, which in turn was converted into IX by reacting with an excess of aqueous ammonia. Additionally, using the method of Eliel and Burgstahler,[15] X was prepared in 99% yield from VIII by refluxing in a solution of methanol, benzene, and acetone.

1-N-Amido-2-acenaphthylenecarboxylic Anhydride (XI). Three methods were tried to introduce the amine functionality. The Hofmann and Schmidt degradations did not produce recoverable product. The Curtius rearrangement was successful in producing product in 99% yield. The reaction was run in refluxing acetonitrile for 2 h. The selection of acetonitrile as solvent was based on its high polarity and subsequent ability to increase the nucleophilicity of the azide ion.[16]

Further studies along the lines of hydrolysis and diazotization of XI to prepare XII are in progress.

Experimental Section

General Information. All melting points were taken on a Thomas Hoover Uni-Melt Model No. 6406-K capillary melting point apparatus and are corrected. IR spectra were obtained on a Perkin-Elmer Model 257 spectrometer. UV spectra were taken on either Unicam SP 800 B or Cary Model 14 ultraviolet spectrophotometers. Mass spectra were obtained on a Hitachi RMU-6E.

1-Bromo-2-cyanoacenaphthylene (III). 1,2-Dibromoacenaphthylene (17.9 g, 57.4 mmol) was heated with stirring to 170 °C in 50 mL of N-methyl-2-pyrrolidone and cuprous cyanide (90.4 g, 101.2 mmol) under a nitrogen atmosphere for 2 h. The reaction mixture was cooled and poured into a solution of sodium cyanide (20 g) in 600 mL of water. This solution was extracted with three 500-mL portions of benzene. The benzene was then washed with 800 mL of 10% aqueous sodium cyanide; the organic layer was separated, dried (MgSO$_4$), treated with Norit, and filtered. The benzene was removed under reduced pressure, yielding 12.5 g of crude material. This material was dissolved in methylene chloride, and 12.5 g of silica gel (Davison 950 grade, 60–200 mesh) was added to the solution. The methylene chloride was allowed to evaporate, leaving a dry powder. This was then placed on top of a 5 × 90 cm column of silica gel. The column was packed and eluted with benzene; 400-mL fractions were collected. Fractions 5 and 6 afforded 5.1 g of 1,2-dibromoacenaphthylene (28.5%, mp 113–115 °C); fractions 9–11 afforded 4.1 g of the desired 1-bromo-2-cyanoacenaphthylene (27.7%, mp 166–167 °C); fractions 16–20 afforded 2.5 g of 1,2-dicyanoacenaphthylene (21.4%, mp 242–243 °C). Recrystallization from absolute ethanol raised the melting point of III to 168–170 °C: UV max (CH$_2$Cl$_2$) 332 nm (ϵ 10 600); IR (KBr) 2215 cm^{-1} (C≡N); mass spectrum (70 eV), m/e (rel intensity) 257 (98), 255 (100), 176 (47), 175 (27), 149 (15). Anal. Calcd for C$_{13}$H$_6$BrN: C, 60.97; H, 2.34; N, 5.45. Found: C, 60.79; H, 2.27; N, 5.32.

1,2-Dicyanoacenaphthylene (IV). The method of Newman and Boden[11] was employed. Cuprous cyanide (9.9 g, 112.0 mmol) was heated to 160 °C in 55 mL of N-methyl-2-pyrrolidone; 1,2-dibromoacenaphthylene (8.5 g, 27.4 mmol) was then added. The reaction was continued with stirring under a nitrogen atmosphere for 2 h. The mixture was cooled before being poured into a solution of sodium cyanide (12 g) in 300 mL of water. The latter mixture was thoroughly shaken and then extracted three times with 350 mL of benzene, following which the organic and aqueous layers were filtered. The benzene layer was washed with 200 mL of 10% sodium cyanide solution and 200 mL of water. The solution was dried over anhydrous sodium sulfate, and the benzene was evaporated under reduced pressure, leaving 3.4 g of a brown solid. This was chromatographed on a 5 × 70 cm silica gel column (Davison silica gel, grade 950, 60–200 mesh), slurry packed in benzene.

The crude reaction mixture was dissolved in methylene chloride and mixed with silica gel (30 g). The resulting slurry was dried with a stream of dry nitrogen. The dried impregnated silica gel was placed on top of the prepared column, the column was eluted with benzene, and 250-mL fractions were collected. Fraction 3 afforded 1,2-dibromoacenaphthylene (40 mg, 0.5%, mp 112–114 °C); fraction 5 afforded 1-bromo-2-cyanoacenaphthylene (20 mg, 0.3%, mp 168–170 °C); fractions 9–14 afforded 1,2-dicyanoacenaphthylene (3.3 g, 59.5%, mp 240 °C). Recrystallization from benzene raised the melting point to 243 °C: UV max (CH$_2$Cl$_2$) 345 nm (ϵ 12 000); IR (KBr) 2240 cm^{-1} (C≡N); mass spectrum (68 eV), m/e (rel intensity) 202 (100), 175 (38), 174 (17), 100 (11), 76 (6). Anal. Calcd for C$_{14}$H$_6$N$_2$: C, 83.18; H, 2.89; N, 13.85. Found: C, 83.15; H, 2.99; N, 13.84.

quence precursors. Spectral data and elemental analyses are in agreement with assigned structures.

1,2-Dicyanoacenaphthylene (IV), 1-Bromo-2-cyanoacenaphthylene (III), and 1-Bromo-2-amidoacenaphthylene (V). The 1,2-dibromoacenaphthylene (II) was prepared according to the procedure of Trost and Britelli.[10] IV was prepared from II using the procedure developed by Newman and Boden[11] for replacement of aryl bromine atoms with nitrile groups. This method gave IV in 59.5% yield. Due to incomplete reaction with the CuCN, a small yield (0.3%) of III, in addition to IV, was also produced. The yield of III could be maximized by reducing the amount of CuCN used to a 2:1 mol CuCN/II ratio.

The hydrolysis of III with various concentrations of H$_2$SO$_4$, HOAc, and H$_2$O produced mainly mixtures of tar and water-soluble organics.[12] Conversely, polyphosphoric acid (PPA) has been reported as an excellent reagent for the hydrolysis of unhindered aromatic nitriles to amides. Hydrolysis of III in PPA gave a yield of 78.5% of V. This increased yield of product in PPA as opposed to conventional methods seems to indicate the exceptional stability of amides in this media.[13]

1,2-Acenaphthylenedicarboxylic Acid (VI) and 1,2-Acenaphthylenedicarboxylic Dimethyl Ester (VII). IV was easily hydrolyzed in excellent yield (85%) to VI with none of the undesired side reactions experienced with III. Subsequently, VI was converted to VII in an 87% yield by refluxing in absolute methanol with a catalytic amount of sulfuric acid. VII had previously been reported,[14] in 1969, as a product of trapping 1,8-dehydronaphthylene with dimethyl acetylenedicarboxylate.

1,2-Acenaphthylenedicarboxylic Anhydride (VIII), 1-Amido-2-acenaphthylenecarboxylic Acid (IX) and 1,2-Acenaphthylenedicarboxylic Acid Monomethyl Ester

1-Bromo-2-amidoacenaphthylene (V). The partial hydrolysis of 1-bromo-2-cyanoacenaphthylene (2.5 g, 97.6 mmol) in 60 mL of polyphosphoric acid was used to produce the desired product. The reaction mixture was stirred at 125 °C for 1 h and was then poured over 100 g of ice and stirred until the syrupy polyphosphoric acid mixture was dissolved in the aqueous phase. The suspended solid was extracted into 1600 mL of chloroform and washed with two 500-mL portions of 10% NaHCO$_3$ solution and then with 500 mL of water. The organic phase was dried (MgSO$_4$), treated with Norit, and filtered. The chloroform was removed under reduced pressure, yielding 2.1 g of product (78.5%, mp 254–256 °C). Recrystallization from 95% ethanol raised the melting point to 256–257 °C: UV max (CH$_2$Cl$_2$) 328 nm (ϵ 12 600); IR (KBr) 3385 and 3220 cm^{-1} (amide NH$_2$); mass spectrum (70 eV), m/e (rel intensity) 275 (94), 273 (94), 259 (99), 257 (100), 231 (16), 229 (19), 151 (25), 150 (86). Anal. Calcd for C$_{13}$H$_8$BrNO: C, 56.98; H, 2.94; N, 5.11. Found: C, 57.17; H, 2.94; N, 5.01.

1,2-Acenaphthylenedicarboxylic Acid (VI). 1,2-Dicyanoacenaphthylene (3.9 g, 19.4 mmol) in a solution of 125 mL of concentrated sulfuric acid, 125 mL of glacial acetic acid, and 125 mL of water was refluxed with stirring. The suspension dissolved after 1 h, and the reaction mixture turned bright red. After 1.5 h, an orange precipitate formed. The reaction was continued for 0.5 h after the appearance of the precipitate; the mixture was then poured into 500 g of ice and filtered. The orange filter cake was dissolved in 600 mL of 5% NaOH, washed with benzene, and filtered. The aqueous solution was acidified to pH 1, and the 1,2-acenaphthylenedicarboxylic acid precipitated. The solid was filtered and dried in vacuo over Drierite, yielding the 1,2-acenaphthylenedicarboxylic acid (3.94 g, 85%). Due to thermal decomposition, a melting point was not obtained. For elemental analysis, recrystallization was from 1,4-dioxane and water: UV max (CH$_2$Cl$_2$) 355 nm (ϵ 13 700); IR (KBr) 1705 cm^{-1} (acid C=O); mass spectrum (70 eV), m/e (rel intensity) 240 (100), 223 (29), 196 (59), 179 (83), 151 (40), 150 (41), 139 (55), 75 (30). Anal. Calcd for C$_{14}$H$_8$O$_4$: C, 70.00; H, 3.36; O, 26.64. Found: C, 69.95; H, 3.46; O, 26.42.

1,2-Acenaphthylenedicarboxylic Dimethyl Ester (VII). 1,2-Acenaphthylenedicarboxylic acid (1.0 g, 4.2 mmol) was refluxed in 10 mL of methanol with 0.1 mL of concentrated sulfuric acid for 1 h. The solution was cooled and the solid precipitate filtered, yielding 1.05 g of solid, which was then dissolved in 100 mL of benzene. The organic solution was washed rapidly with two 50-mL portions of ice-cold aqueous NaHCO$_3$ and then immediately dried (MgSO$_4$). The benzene was removed under reduced pressure, leaving 1,2-acenaphthylenedicarboxylic dimethyl ester (0.98 g, 87%, mp 105–106 °C). Recrystallization for elemental analysis was from methanol: UV max (CH$_2$Cl$_2$) 336 nm (ϵ 12 600); IR (KBr) 1740 cm^{-1} (ester C=O); mass spectrum (70 eV), m/e (rel intensity) 268 (65), 237 (100), 151 (35), 150 (38), 138 (22). Anal. Calcd for C$_{16}$H$_{12}$O$_4$: C, 71.64; H, 4.51. Found: C, 71.77; H, 4.72.

1,2-Acenaphthylenedicarboxylic Anhydride (VIII). 1,2-Acenaphthylenedicarboxylic acid (5.8 g, 24.2 mmol) was heated in a 250-mL flask with 115 mL of acetic anhydride until the mixture dissolved. The flask was covered and allowed to cool to room temperature and then cooled in a refrigerator for 4 h. The solid precipitate was filtered and washed with five 30-mL portions of anhydrous diethyl ether, yielding the 1,2-acenaphthylenedicarboxylic anhydride (4.5 g, 85.5%, mp 251–253 °C). Recrystallization from 1,4-dioxane/heptane raised the melting point to 252–253 °C: UV max (CH$_2$Cl$_2$) 350 nm (ϵ 11 000); IR (KBr) 1830 and 1770 cm^{-1} (anhydride C=O); mass spectrum (70 eV), m/e (rel intensity) 222 (84), 178 (100), 150 (47). Anal. Calcd for C$_{14}$H$_6$O$_3$: C, 75.68; H, 2.72. Found: C, 75.39; H, 2.66.

1-Amido-2-acenaphthylenecarboxylic Acid (IX). 1,2-Acenaphthylenedicarboxylic anhydride (500 mg, 2.25 mmol) and 30 mL of 28% aqueous ammonia were heated and stirred for 20 min in order to dissolve the suspended anhydride. This solution was stripped to dryness under reduced pressure, yielding 1-amido-2-acenaphthy-

lenecarboxylic acid (572 mg, 100%, mp 239–240 °C): UV max (CH$_2$Cl$_2$) 339 nm (ϵ 20 000); IR (KBr) 3380 and 3235 (amide NH$_2$), 1710 (acid C=O), 1645 (amide C=O) cm^{-1}; mass spectrum (70 eV), m/e (rel intensity) 239 (60), 223 (30), 195 (44), 179 (100), 151 (30), 139 (46). Anal. Calcd for C$_{14}$H$_9$NO$_3$: C, 70.29; H, 3.79; N, 5.85. Found: C, 70.21; H, 3.87; N, 5.63.

1,2-Acenaphthylenedicarboxylic Acid Monomethyl Ester (X). An extension of the method of Eliel and Burgstahler[15] was employed for the preparation of X. 1,2-Acenaphthylenecarboxylic anhydride (1.0 g, 45 mmol) was refluxed in a solution of 20 mL of absolute methanol, 10 mL of benzene, and 10 mL of acetone for 2 h. The solvent was removed under reduced pressure, yielding 1.14 g of the 1,2-acenaphthylenedicarboxylic acid monomethyl ester (1.14 g, 99%, mp 230–233 °C). Recrystallization from 1,4-dioxane/hexane raised the melting point to 232–233 °C: UV max (CH$_2$Cl$_2$) 324 nm (ϵ 13 000); IR (KBr) 1725 (ester C=O), 1650 (acid C=O) cm^{-1}; mass spectrum (70 eV), m/e (rel intensity) 254 (100), 223 (97), 179 (63), 151 (38), 150 (45), 139 (54). Anal. Calcd for C$_{15}$H$_{10}$O$_4$: C, 70.86; H, 3.96. Found: C, 70.56; H, 3.92.

1-N-Amido-2-acenaphthylenecarboxylic Anhydride (XI). 1,2-Acenaphthylenedicarboxylic anhydride (450 mg, 2.03 mmol) was refluxed with sodium azide (140 mg, 2.15 mmol) in 50 mL of acetonitrile for 2 h. The reaction was protected from atmospheric moisture with a calcium chloride drying tube. The solution was washed with 50 mL of water, and the solid was then filtered off, yielding the 1-N-amido-2-acenaphthylenecarboxylic anhydride (475 mg, 99%, melting point decomposition): UV max (CH$_2$Cl$_2$) 334 nm (ϵ 8200); IR (KBr) 3550 and 3490 (N–H), 1765 and 1723 (secondary amido anhydride) cm^{-1}; mass spectrum (70 eV), m/e (rel intensity) decomposed. Anal. Calcd for C$_{14}$H$_7$NO$_3$: C, 70.88; H, 2.97; N, 5.90; O, 20.23. Found: C, 70.71; H, 2.88; N, 5.88; O, 20.04.

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