Supporting Information

An Effective Approach towards Conjugated Porous Organic Frameworks based on Phenanthrene Building Blocks. Metal-free Heterogeneous photocatalysts.

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Materials
Solvents were dried by standard methods or by elution using a PureSolv Innovative Technology drying system. All reagents were commercially available and used without further purification unless otherwise indicated. The microwave ovens used for the polymer synthesis were Discover SP® CEM Corporation 3100 and ANTON PAAR MONOWAVE 300.

Characterization Methods
Microanalyses were made with a Carlo Erba EA1108 elemental analyzer (C, H, N). Proton Nuclear Magnetic Resonance (1H NMR) spectra were recorded with a VARIAN INOVA 300 MHz for liquids and a Bruker AV400 WB spectrometer (Larmor frequencies of 400 and 100 MHz), using 4 mm MAS probes spinning at 10 kHz rate for 13C solid-state MAS-NMR measurements. The 13C CP-MAS spectra were obtained using 3.5 ms contact time 4 s relaxation time and 1024 scans. Mass spectrometer was operated in electronic impact (EI) hp MASS SELECTIVE DETECTOR 5973 - (SIS) DIRECT INSERTIVE PROBE and hp HEWLETT PACKARD SERIES 1100MSD for electrospray (ES). Infrared (FTIR) and ATR-FTIR spectra were recorded on an appliance type PerkinElmer Spectrum One or Two spectrometer with a Fourier equipped with a diamond internal element, using an accumulation of 4 runs and the same pressure of the crystal on the surface in each sample. Infrared adsorption isotherms where measured at 77 K using a Micromeritics ASAP 2020 M surface and porosity analyzer. Prior to measurement, the samples were degassed at 100°C for 12 h. Specific surface areas were determined by BET technique and the pore size average by DFT methods. Thermogravimetric and differential thermal analyses (TGA-DTA) were conducted in an air stream with a TA Instruments Model TA-Q500 analyzer in the temperature range between 40 and 800 ºC with a heating rate of 10 ºC min⁻¹.

UV-Visible studies were carried out on a PerkinElmer Lambda XLS+ and a JASCO V-730 Spectrophotometers. Fluorescence spectra were recorded on an Aminco SLM 8000 spectrophotometer. Ultraviolet–visible diffuse reflectance spectra (UV–Vis DRS) were carried out on a Shimadzu UV-2401 PC UV-Visible Spectrometer. The reflectance values (R) were transformed to the Kubelka–Munk function F(R) according to the eqn (1): \( F(R) = \frac{(1-R)^2}{2R} \). The band gap was estimated by a Tauc plot of \((h\nu F(R))^{1/n}\) against \(h\nu\) (h is Planck's constant, \(\nu\) is the frequency of vibration and \(n = 1/2\) for direct transitions).

Cyclic voltamperometry (CV) measurements were obtained on an Autolab PGSTAT302N potentiostat/galvanostat station in a three electrodes cell (Pt working electrode) at room
temperature, under nitrogen atmosphere. As electrolyte, we have used recrystallized tetra-n-butylammonium hexafluorophosphate 0.1 M (TBAPF₆) in acetonitrile. A slurry was prepared mixing 2 mg of each material with 25 µL of Nafion perflorinated resin solution (Sigma-Aldrich) and 250 µL of isopropanol. Then, it was placed on the platinum working electrode and dried. Potentials were measured against Ag/AgCl as reference electrode.

The photoreactor used for aza-Henry reactions was lab-made and has one or two LED blue lamps of 50 W. The reaction progress was followed by Gas Chromatographic (GC) using a KONIK HRGC 5000B and a KAP-S capillary column (15 m, 0.25 mm, 0.25 µm).
Synthetic Procedures and Characterization Data

Synthesis of monomers

1,3,5-Trifluoro-2,4,6-tris[(trimethylsilyl)ethynyl]benzene

A solution of 1,3,5-trifluoro-2,4,6-triiodobenzene (398 mg, 0.78 mmol, 1.0 eq.), CuI (7.42 mg, 39 µmol, 0.05 eq.) in diisopropylamine (DIPA) (4 mL) were degassed with Argon. Then, Pd(PPh3)2Cl2 (40 mg, 58 µmol, 0.075 eq.) and trimethylsilylacetylene (649 µL, 4.68 mmol, 6.0 eq.) were added. The reaction was performed under microwave activation at 100 W, 100 °C, 12 bar, 30 min and monitored by TLC using heptane as eluent. The catalyst was filtered through celite® and silica gel. Finally, the solvents were removed under vacuum. Yield (90 %). 1H NMR (300 MHz, Chloroform-d) δ (ppm): 0.26 (s, 27H).

1,3,5-triethynyl-2,4,6-trifluorobenzene

1,3,5-Trifluoro-2,4,6-tris[(trimethylsilyl)ethynyl]benzene (370 mg, 0.88 mmol, 1.0 eq.) and K2CO3 (243 mg, 1.76 mmol, 2.0 eq.) were dissolved in a mixture of tert-BuOH (9.25 mL) and H2O (0.22 mL). The reaction was heated under reflux 5 hours and monitored by TLC using heptane as eluent. 500 mg more of K2CO3 were added and the mixture was maintained at reflux overnight. It was cooled to room temperature and the solvents were removed under reduced pressure. The solid was purified by silica gel column chromatography eluting with heptane. Yield (50 %). 1H NMR (300 MHz, Chloroform-d) δ (ppm): 3.53 (s, 3H).

2',7'-diiododispiro[[1,3]dioxolane-2,9'-phenanthrene-10',2''-[1,3]dioxolane]

Camphor-10-sulfonic acid (70 mg, 0.300 mmol, 0.15 eq.) and 2,7-diiodophenanthrene-9,10-dione (920 mg, 2.0 mmol, 1 eq.) were dissolved in ethylene glycol (11.1 mL) and MeOH (2.2 mL). The mixture was deaerated with argon and heated at 120 °C overnight. The reaction was monitored by TLC using Hep/AcOEt (2:1). The solid was filtered and it was washed with water and MeOH. Yield (83 %). 1H NMR (300 MHz, Chloroform-d) δ (ppm): 8.07 (d, J = 2.1 Hz, 2H), 7.79 (dd, J = 8.6, 2.1 Hz, 2H), 7.58 (d, J = 8.6 Hz, 2H), 4.34 – 4.13 (m, 4H), 3.81 – 3.54 (m, 4H).

Synthesis of Conjugated Porous Polymers

CPPs-Sonogashira

General method: CPPs were synthesized through a microwave-activated Sonogashira-type coupling reaction. The corresponding derivative of biphenyl and CuI were dissolved in a dry mixture of DIPA
and DMF (1:1). The solution was deaerated with argon for 15 minutes. After that, the catalyst Pd(PPh₃)₂Cl₂ and the corresponding acetylene-derivate were added. The reaction was performed in two steps: 1) 120 °C, 100 W, 30 min; 2) 140 °C, 100 W, 30 min. The crude was filtered and washed with methanol. The solid was stirring with H₂O and KCN overnight to remove the Pd(0) residues. Then, it was filtered under vacuum and washed several times with water, methanol and diethyl ether to obtain the final product.

**Hydrolysis of CPPs-Sonogashira-acetal to give CPPs-Sonogashira-diketone.**

General method: A suspension of the corresponding CPP-Sonogashira-dioxolane (200 mg) in a mixture of acetonitrile/water (1:1, 2-4 mL) was deaerated with argon for 15 minutes. After that, trifluoromethanesulfonic acid (20 eq.) was added and the reaction activated with MW. The conditions were 150 °C, 2 hours and strong stirring. The crude was filtered by a membrane and washed with water, ACN, MeOH and diethyl ether. The solid was dried under vacuum.

**Synthesis of CPPs-Sonogashira-Benzimidazole⁵.**

General method: A suspension of the corresponding CPP-Sonogashira-O2 (50 mg, 1.0 eq.) and ammonium acetate (127 mg, 10 eq.) in a hot mixture of DCM and EtOH (1:1, 8 mL). After 10 minutes, 2,4-difluoro-6-hydroxybenzaldehyde (10.4 mg, 0.2 eq.) and few drops of acetic acid (2-3 drops) were added to the reaction mixture. The reaction was monitored by TLC using Hep/AcOEt (2:1) until no aldehyde was detected in the supernatant liquid. The solid was filtrated and washed with water and methanol, and dried in vacuum to give the compound.

**CPP-So1**

4,4'-Diiodobiphenyl (145 mg, 0.357 mmol, 1.5 eq.); Cul (1.01 mg, 5.35 µmol, 0.045 eq.); 1,3,5-tri(4-ethynylphenyl)benzene¹⁶ (90 mg, 0.238 mmol, 1.0 eq.); Pd(PPh₃)₂Cl₂ (7.51 mg, 10.7 µmol, 0.09 eq.); DIPA (2 mL); DMF (2 mL).

**BET Surface Area (m²/g):** 554. **ATR-FTIR (cm⁻¹):** 3032, 1596, 1511, 1438, 1391, 1181, 1109, 1003, 886, 819, 732, 608, 542. **Elemental analysis:** % C: 83.2; % H: 4.6.

**CPP-So1-O2**

2,7-diiodophenanthrene-9,10-dione (164 mg, 0.357 mmol, 1.5 eq.); Cul (1.01 mg, 5.35 µmol, 0.045 eq.); 1,3,5-tri(4 ethynylphenyl)benzene (90 mg, 0.238 mmol, 1.0 eq.); Pd(PPh₃)₂Cl₂ (7.51 mg, 10.7 µmol, 0.09 eq.); DIPA (2 mL); DMF (2 mL).

**BET Surface Area (m²/g):** 177. **ATR-FTIR (cm⁻¹):** 3032, 2325, 2188,
1681, 1595, 1505, 1468, 1392, 1263, 887, 824, 725, 648, 607, 541, 471. **Elemental analysis:** % C: 73.2; % H: 3.6.

CPP-So1-acetal

2',7'-diiododispiro[[1,3]dioxolane-2,9'-phenanthrene-10',2''-[1,3]dioxolane (196 mg, 0.357 mmol, 1.5 eq.); CuI (0.95 mg, 5.0 µmol, 0.0225 eq.); 1,3,5-tris(4-ethynylphenyl)benzene (90 mg, 0.238 mmol, 1.0 eq.); Pd(PPh₃)₂Cl₂ (7.5 mg, 10.7 µmol, 0.045 eq.); DIPA (2 mL); DMF (2 mL). **BET Surface Area (m²/g):** 525.  
**ATR-FTIR (cm⁻¹):** 2963, 2868, 1596, 1513, 1477, 1383, 1264, 1216, 1184, 1105, 1089, 1047, 1015, 991, 967, 911, 888, 822, 753, 733, 705, 601, 543. **Elemental analysis:** % C: 76.3; % H: 4.4.

Hydrolysis of CPP-So1-acetal (CPP-So1-O2H)

Following the general method. **BET Surface Area (m²/g):** 384. **ATR-FTIR (cm⁻¹):** 3032, 2968, 2869, 1682, 1596, 1510, 1471, 1435, 1408, 1388, 1316, 1280, 1261, 1219, 1203, 1148, 1109, 1045, 912, 826, 710, 538, 478. **Elemental analysis:** % C: 75.2; % H: 3.9.

CPP-So1-Phlm-2F

Following the general method. 92 % Yield. **BET Surface Area (m²/g):** 456. **FTIR ν (KBr, cm⁻¹):** 3434, 3031, 2925, 2200, 1909, 1708, 1682, 1595, 1504, 1473, 1466, 1437, 1391, 1363, 1220, 1175, 1109, 1016, 999, 887, 826, 725, 607, 576, 541. **Elemental analysis:** % C: 73.5; % H: 3.9; % N: 2.4.

CPP-So2

4,4'-Diiodobiphenyl (175 mg, 0.432 mmol, 2.0 eq.); CuI (2.5 mg, 13.2 µmol, 0.013 eq.); tetra(4-ethynylphenyl)methane₁₆ (90 mg, 0.216 mmol, 1.0 eq.); Pd(PPh₃)₂Cl₂ (18.2 mg, 26 µmol, 0.026 eq.); DIPA (2 mL); DMF (2 mL). **BET Surface Area (m²/g):** 1313. **ATR-FTIR (cm⁻¹):** 3032, 1601, 1504, 1401, 1192, 1004, 818, 738, 539, 406. **Elemental analysis:** % C: 79.3; % H: 4.9.

CPP-So2-O2

2,7-diiodophenanthrene-9,10-dione (177 mg, 0.384 mmol, 2.0 eq.); CuI (2.0 mg, 12 µmol, 0.06 eq.); tetra(4-ethynylphenyl)methane (80 mg, 0.192 mmol, 1.0 eq.); Pd(PPh₃)₂Cl₂ (16 mg, 23 µmol, 0.12 eq.); DIPA (2 mL); DMF (2 mL). **BET Surface Area (m²/g):** 431. **ATR-FTIR (cm⁻¹):** 3036, 2325, 2165, 2051, 1982,
1683, 1600, 1501, 1217, 1044, 1018, 915, 823, 768, 725, 582, 529. **Elemental analysis**: % C: 69.6; % H: 4.2.

**CPP-So2-acetal**

2',7'-diiododispiro[1,3]dioxolane-2,9'-phenanthrene-10',2''-[1,3]dioxolane (211 mg, 0.384 mmol, 2.0 eq.); Cul (2 mg, 12 µmol, 0.06 eq.); tetra(4-ethynlyphenyl)methane (80 mg, 0.192 mmol, 1.0 eq.); Pd(PPh₃)₂Cl₂ (16 mg, 23 µmol, 0.12 eq.); DIPA (2 mL); DMF (2 mL). **BET Surface Area (m²/g)**: 1003.

**ATR-FTIR (cm⁻¹)**: 2964, 2877, 1597, 1502, 1474, 1280, 1213, 1189, 1110, 1089, 1047, 1015, 993, 969, 913, 822, 729, 586, 522. **Elemental analysis**: % C: 70.4; % H: 4.7.

**Hydrolysis of CPP-So2-acetal (CPP-So2-O2H)**

Following the general method. **BET Surface Area (m²/g)**: 816. **ATR-FTIR (cm⁻¹)**: 2974, 2900, 1684, 1596, 1502, 1474, 1280, 1214, 1153, 1109, 1098, 1051, 1017, 910, 823, 774, 743, 721, 697, 580, 503, 472. **Elemental analysis**: % C: 72.5; % H: 4.5.

**CPP-So2-PhIm-2F**

It was prepared following following the general method. 93% yield. **BET Surface Area (m²/g)**: 684. **FTIR ν(KBr, cm⁻¹)**: 3431, 2346, 1700, 1623, 1402, 1227, 1117, 824, 547. **Elemental analysis**: % C: 66.6; % H: 4.6 % N: 4.4. **Elemental analysis**: % C: 77.6; % H: 4.1 % N: 3.3.

**CPP-So3**

4,4'-Diodobiphenyl (239 mg, 0.588 mmol, 1.5 eq.); Cul (1.7 mg, 8.82 µmol, 0.045 eq.); 1,3,5-triethynyl-2,4,6-trifluorobenzene (80 mg, 0.392 mmol, 1.0 eq.); Pd(PPh₃)₂Cl₂ (12.4 mg, 18 µmol, 0.09 eq.); DIPA (4 mL). **BET Surface Area (m²/g)**: 131. **ATR-FTIR (cm⁻¹)**: 3300, 3036, 2325, 2220, 1599, 1495, 1452, 1350, 1181, 1102, 1003, 949, 819, 724, 628, 526. **Elemental analysis**: % C: 66.3; % H: 2.5.

**CPP-So3-O2**

2,7-diiodophenanthrene-9,10-dione (203 mg, 0.441 mmol, 1.5 eq.); Cul (1.3 mg, 6.6 µmol, 0.045 eq.); 1,3,5-triethynyl-2,4,6-trifluorobenzene (60 mg, 0.294 mmol, 1.0 eq.); Pd(PPh₃)₂Cl₂ (9.3 mg, 13 µmol, 0.09 eq.); DIPA (4 mL). **BET Surface Area (m²/g)**: 26. **ATR-FTIR (cm⁻¹)**: 3302, 2325, 2207, 2114, 1674, 1600, 1452, 1286, 1262, 1204, 1109, 996, 905, 825, 752, 825, 752, 690, 630, 486, 449. **Elemental analysis**: % C: 56.3; % H: 1.8.
CPP-So3-acetal

\[ \text{2',7'-diiododispiro[[1,3]dioxolane-2,9'-phenanthrene-10',2''-[1,3]dioxolane]} \]

(202 mg, 0.368 mmol, 1.5 eq.); Cul (2.09 mg, 11 \(\mu\)mol, 0.045 eq.); 1,3,5-triethynyl-2,4,6-trifluorobenzene (50 mg, 0.245 mmol, 1.0 eq.); \(\text{Pd(PPh}_3\text{)Cl}_2\) (15.4 mg, 22 \(\mu\)mol, 0.09 eq.); DIPA (2 mL); DMF (2 mL). BET Surface Area (m\(^2\)/g): 727. ATR-FTIR (cm\(^{-1}\)): 2967, 2873, 1601, 1482, 1446, 1280, 1221, 1185, 1110, 1098, 1047, 1015, 995, 957, 916, 823, 752, 694, 660, 591, 522. Elemental analysis: % C: 71.1; % H: 4.2; % N: 1.7.

Hydrolysis of CPP-So3-acetal (CPP-So3-O2H)

Following the general method. BET Surface Area (m\(^2\)/g): 694. ATR-FTIR (cm\(^{-1}\)): 2969, 2879, 1691, 1591, 1476, 1448, 1415, 1287, 1225, 1187, 1091, 1049, 997, 957, 931, 823, 755, 719, 691, 565, 589, 515, 482. Elemental analysis: % C: 64.7; % H: 2.5.

CPP-So3-PhIm-2F

It was prepared following the general method. 90% Yield. BET Surface Area (m\(^2\)/g): 568. FTIR \(\nu\) (KBr, cm\(^{-1}\)): 3435, 2925, 2206, 1617, 1453, 1109, 617. Elemental analysis: % C: 60.0; % H: 2.3; % N: 2.5.

General method for aza-Henry reaction\(^5\)

2-phenyl-1,2,3,4-tetrahydroisoquinoline (20 mg, 95.7 \(\mu\)mol, 1.0 eq.), nitrocompound (2 mL) and photocatalyst (1 %) were irradiated with standard white (15w) or blue LED light (50 or 100 W) in darkness. It was monitored by gas chromatography. The catalyst was separated by centrifugation and product purified by silica gel flash chromatography using Hep/AcOEt (20/1).
Scheme S1. Synthetic route for CPP-So1 polymers.

Scheme S2. Synthetic route for CPP-So2 polymers.
Scheme S3. Synthetic route for CPP-So3-polymers.
Characterization data of conjugated porous polymers

**Figure S1.** $^{13}$C NMR spectra of CPP-So1-polymers

**Figure S2.** $^{13}$C NMR spectra of CPP-So2-polymers
Figure S3. $^{13}$C NMR spectra of CPP-So3-polymers.
Figure S4a. FT-ATR and FT-IR spectra of CPP-So1- (left) and CPP-So2-materials (right).

Figure S4b. FT-ATR and FT-IR spectra of CPP-So2- (left) and CPP-So3-materials (right).
Figure S5a. TGA of CPP-So1-materials.

Figure S5b. TGA of CPP-So3-materials.
Figure S5c. TGA of CPP-So3-materials.
Figure S6a. SEM images of CPP-So1-PhIm-2F.

Figure S6b. SEM images of CPP-So2-PhIm-2F.
Figure S6c. SEM images of CPP-So3-PhIm-2F.
Figure S7a. Pore distribution by N\textsubscript{2}-DFT method of CPPs-Sonogashira.

Figure S7b. Nitrogen adsorption/desorption isotherms of CPPs-Sonogashira.

Figure S7c. Nitrogen adsorption-desorption isotherms (left) and pore distribution (N\textsubscript{2}-DFT method) (right) of CPPs-So2PhIm2F after aza-Henry reaction.
Characterization data of aza-Henry Products.

1-(nitromethyl)-2-phenyl-1,2,3,4-tetrahydroisoquinoline (2a)

![Chemical Structure](image)

**Figure S8a.** $^1$H-NMR spectrum of 2a.

![NMR Spectrum](image)

**Figure S8b.** MS spectrum of 2a.
1-(1-nitroethyl)-2-phenyl-1,2,3,4-tetrahydroisoquinoline (2b)

Figure S9a. $^1$H-NMR spectrum of 2b.

Figure S9b. MS spectrum of 2b.
1-(1-nitropropyl)-2-phenyl-1,2,3,4-tetrahydroisoquinoline (2c)

**Figure S10a.** $^1$H-NMR spectrum of 2c.

**Figure S10b.** MS spectrum of 2c.
2-Phenyl-1,2,3,4-tetrahydroisoquinoline-1-carbonitrile (2d).
Prepared following the general method for aza-Henry reaction using 2.5 eq of TMSCN and acetonitrile (2 mL) as solvent.

![Chemical structure of 2d]

Figure S11a. $^1$H-NMR spectrum of 2d.

![MS spectrum of 2d]

Figure S11b. MS spectrum of 2d.
Dimethyl 2-(2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)malonate (2e)

Figure S12a. $^1$H-NMR spectrum of 2e.

Figure S12b. MS spectrum of 2e.
Methyl 2-cyano-2-(2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)acetate (2f)

Figure S13a. $^1$H-NMR spectrum of 2f.

Figure S13b. MS spectrum of 2f.
Photophysical properties.

**Figure S14.** Electronic absorption spectra of CPPs in Solid state and acetonitrile suspension.

**Figure S15.** Emission spectra of CPPs in acetonitrile suspension.

**Figure S16.** Solid-state UV-Vis Diffuse reflectance (UV-DRS).
Figure S17. The tauc plot of the Kubelka-Munk function of polymers.

Figure S18. Cyclic voltammetry of imidazole-polymers deposited on platinum (electrolyte: acetonitrile/0.1M TBAPF₆), Pt as working electrode (scan rate was 50 mVs⁻¹).
Table S1. Literature data for photocatalyzed aza-Henry reaction.  

<table>
<thead>
<tr>
<th>Cat.</th>
<th>Catalytic amount</th>
<th>Photo-conditions</th>
<th>Conv. (h)</th>
</tr>
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<tr>
<td>CPP-Suzuki-PhIm-2F10</td>
<td>1 mg</td>
<td>Standard lamp, 15 W, air</td>
<td>85 (48)</td>
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<tr>
<td>Ru-CP11</td>
<td>0.2 mol %</td>
<td>Standard lamp, 26 W, air</td>
<td>97 (8h)</td>
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<td>UiO-68Se12</td>
<td>4 mg</td>
<td>Blue LEDs ($\lambda_{\text{max}} = 450$ nm, 3 W), air</td>
<td>100 (4h)</td>
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<td>EY-POP-113</td>
<td>10 mg</td>
<td>household bulb, 14 W, in air</td>
<td>98 (24)</td>
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<tr>
<td>COF-JLU514</td>
<td>4 mg</td>
<td>blue LEDs ($\lambda = 460$ nm), 30 W, O2 (1 bar)</td>
<td>99 (6h)</td>
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<td>TFB-COF15</td>
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<td>energy-saving lamp, 45 W, O2 (1 atm)</td>
<td>71 (32h)</td>
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<td>85—90</td>
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<tr>
<td>CMBDP17</td>
<td>10 mg</td>
<td>LED light (15 W) 82—96</td>
<td>82—96</td>
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<tr>
<td>CPOP-2018</td>
<td>2 mol%</td>
<td>fluorescent lamp (23 W)</td>
<td>69—91</td>
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<td>19 mg</td>
<td>bulb (60 W)</td>
<td>80—97 (12 h)</td>
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<td>CNP3</td>
<td></td>
<td>Energy-saving bulb, O2</td>
<td>&gt;90 (22-34h)</td>
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<td>Different catalysts20</td>
<td></td>
<td>common fluorescent lamp</td>
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<td>Silica/Rose Bengal21</td>
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<td>NITTP22</td>
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<td>Blue LED (18w), air</td>
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<td>2 mg</td>
<td>30W blue LED lamp (460 nm) O2 (1.0 bar),</td>
<td>99 (1h)</td>
</tr>
</tbody>
</table>

References

9 Savateev, A.; Antonietti, M. Heterogeneous Organocatalysis for Photoredox Chemistry. ACS Catalysis 2018, 8, 9790–9808 and references therein.