Supporting Information

Efficient Photocatalytic Synthesis of Quinazolinones Using Nitrogen-Modulated Benzothiadiazole-Based Covalent Organic Frameworks

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Chemicals

4,4'-([1,2,5]thiadiazolo[3,4-c]pyridine-4,7-diyl)dibenzaldehyde (TPBA) was purchased from Jilin Chinese Academy of Science-Yanshen Technology Co., Ltd. 5-bromopicolinaldehyde, 6-bromonicotinaldehyde and 4,7-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzo[c][1,2,5]thiadiazole were purchased from Shanghai Bidepharm Co. Ltd. All the other chemicals were obtained from the chemical supplies and used without further purification.

Characterization

Powder X-ray diffraction (PXRD) patterns were recorded using Bruker D8 Advance X-ray diffractometer with Cu Kα radiation. Fourier transform infrared (FT-IR) spectra were recorded on a PerkinElmer spectrometer. The photoluminescent and UV-vis spectra were measured on FLS1000 spectrofluorometer (Edinburgh Instruments) and Shimadzu UV-3600 spectrophotometer, respectively. The TGA data were obtained using TGA 550 (TA Instruments) analyzer and the samples were heated from room temperature to 800 °C at a ramp rate of 10 °C / min. Scanning electron micrographs (SEM) images were taken using a JEOLJSM-IT800(SHL). Transmission electron microscope (TEM) was performed by JEOLJEM 2100F. Nuclear magnetic resonance (NMR) data was collected using 400 MHz JEOL JNM-ECZ400S. The solid-state ^{13}C cross polarization magic angle spinning NMR spectra were measured on a Bruker AVANCE III 600 M. Nitrogen sorption isotherms were collected by automated volumetric adsorption apparatus (BSD-660M). The electron paramagnetic resonance (EPR) measurements were performed by Bruker EMX PLUS. Gas chromatography-mass spectrometry (GC-MS) analysis was performed on an Agilent GC 8890 gas chromatograph equipped with an Agilent 5977B GC/MSD mass spectrometer using an

Agilent (HP-5 MS) capillary column (30 m × 250 μm × 0.25 μm). Photoelectrochemical measurements were operated on a CHI660E workstation (CH Instrument Corp, Shanghai).

DFT calculation

Density functional theory (DFT) calculations were carried out using the Gaussian 16. The PBE0 functional, along with the D3BJ dispersion correction, was employed[1]. For geometry optimization and frequency calculations for the COFs were conducted with the def2-SVP basis set[2], while single-point energy calculations were performed using the def2-TZVPD basis set[3]. The electrostatic potential (ESP) and HOMO/LUMO orbitals were visualized with the Multiwfn and VMD software packages[4, 5].

Photoelectrochemical Measurements

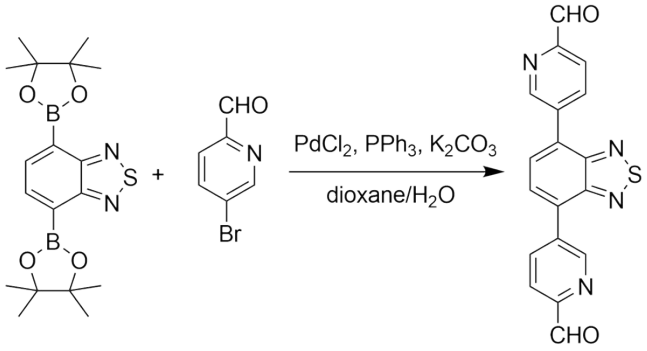
Photoelectrochemical measurements were conducted with a CHI660E (CH Instrument Corp, Shanghai) electrochemical workstation. Firstly, 5 mg COFs were added into a mixed solution of 1 mL of ethanol and 10 μL of 5 wt% Nafion, which was ultra-sonicated for two hours to get homogeneous suspension. Then the suspension was dropped on the surface of ITO glass and dried at room temperature. A standard three electrode system was used with the photocatalyst-coated ITO glass as the working electrode, Pt wire as the counter electrode and an Ag/AgCl as a reference electrode. 0.1 M Na_2SO_4 aqueous solution was used as the electrolyte. Mott-Schottky measurement was carried out at frequency of 1200, 1500 and 1800 Hz with amplitude of 5 mV.

General procedure for photocatalytic reaction

5 mg COFs and 10 μL (0.077 mmol) 1,2,3,4-tetrahydroisoquinoline and 10 mg (0.083 mmol) 2-aminobenzaldehyde were dispersed in 2 mL CH_3CN, then the mixture was stirred under air

at room temperature for 30 min in the dark. Then the above mixture was irradiated using 40 W white LED. After a certain period of time, a mixed solution of ethyl acetate and petroleum ether was used to diagnose whether the reaction was complete. The conversion and selectivity were determined by GC-MS. The purity of product was further confirmed by ^{1}H NMR spectroscopy.

Synthesis of 5,5'-(benzo[c][1,2,5]thiadiazole-4,7-diyl)dipicolinaldehyde (BTPA)



4,7-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzo[c][1,2,5]thiadiazole (5.0 mmol, 1.94 g), 5-bromopicolinaldehyde (10.4 mmol, 1.94 g), $PdCl_2$ (0.4 mmol, 70.0 mg), PPh_3 (0.8 mmol, 0.22 g) and K_2CO_3 (16.0 mmol, 2.20 g) were added in a solution containing 120 mL dioxane and 30 mL water. The reaction solution was degassed four times. Then the mixture was heated to reflux at 105 °C for 20 h under nitrogen atmosphere. Then the formed precipitates were filtrated to obtain 5,5'-(benzo[c][1,2,5]thiadiazole-4,7-diyl)dipicolinaldehyde (BTPA) as an orange solid (1.17 g, yield: 67.6%), which was directly used for COF synthesis without further purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H ppm 10.19 (s, 2H), 9.38 (s, 2H), 8.58 (d, J = 8.3 Hz, 2H), 8.17 (d, J = 7.6 Hz, 2H), 7.99 (s, 2H). We cannot obtain the ^{13}C NMR spectrum for BTPA due to its low solubility.

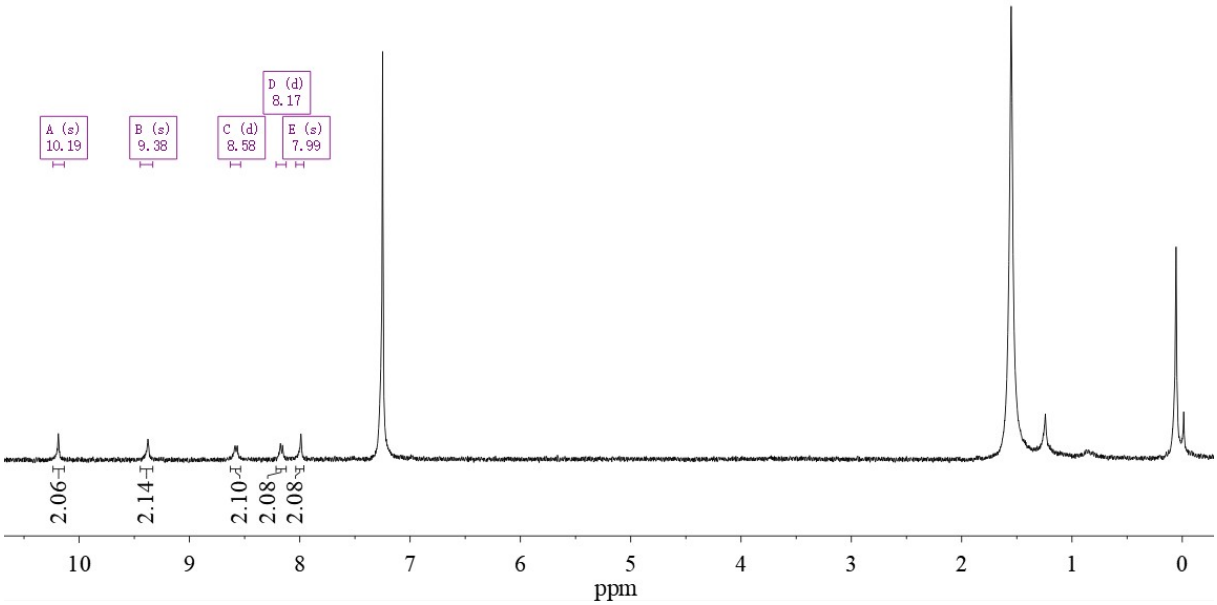


Figure S1. The ^{1}H NMR spectrum of BTPA in $CDCl_3$.

Synthesis of 6,6'-(benzo[c][1,2,5]thiadiazole-4,7-diyl)dinicotinaldehyde (BTNA)

4,7-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzo[c][1,2,5]thiadiazole (5.0 mmol, 1.94 g), 6-bromonicotinaldehyde (10.4 mmol, 1.94 g), $PdCl_2$ (0.4 mmol, 70.0 mg), PPh_3 (0.8 mmol, 0.22 g) and K_2CO_3 (16.0 mmol, 2.20 g) were added in a solution containing 120 mL dioxane and 30 mL water. The reaction solution was degassed four times. Then the mixture was heated to reflux at 105 °C for 20 h under nitrogen atmosphere. Then the formed precipitates were filtrated to obtain 6,6'-(benzo[c][1,2,5]thiadiazole-4,7-diyl)dinicotinaldehyde (BTNA) as an orange solid (1.21 g, yield: 69.2%), which was directly used for COF synthesis without further purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H ppm 10.21 (s, 2H), 9.25 (s, 2H), 9.06 (d, J = 8.4 Hz, 2H), 8.84 (s, 2H), 8.37 (d, J = 8.7 Hz, 2H). We cannot obtain the ^{13}C NMR spectrum for BTNA due to its low solubility.

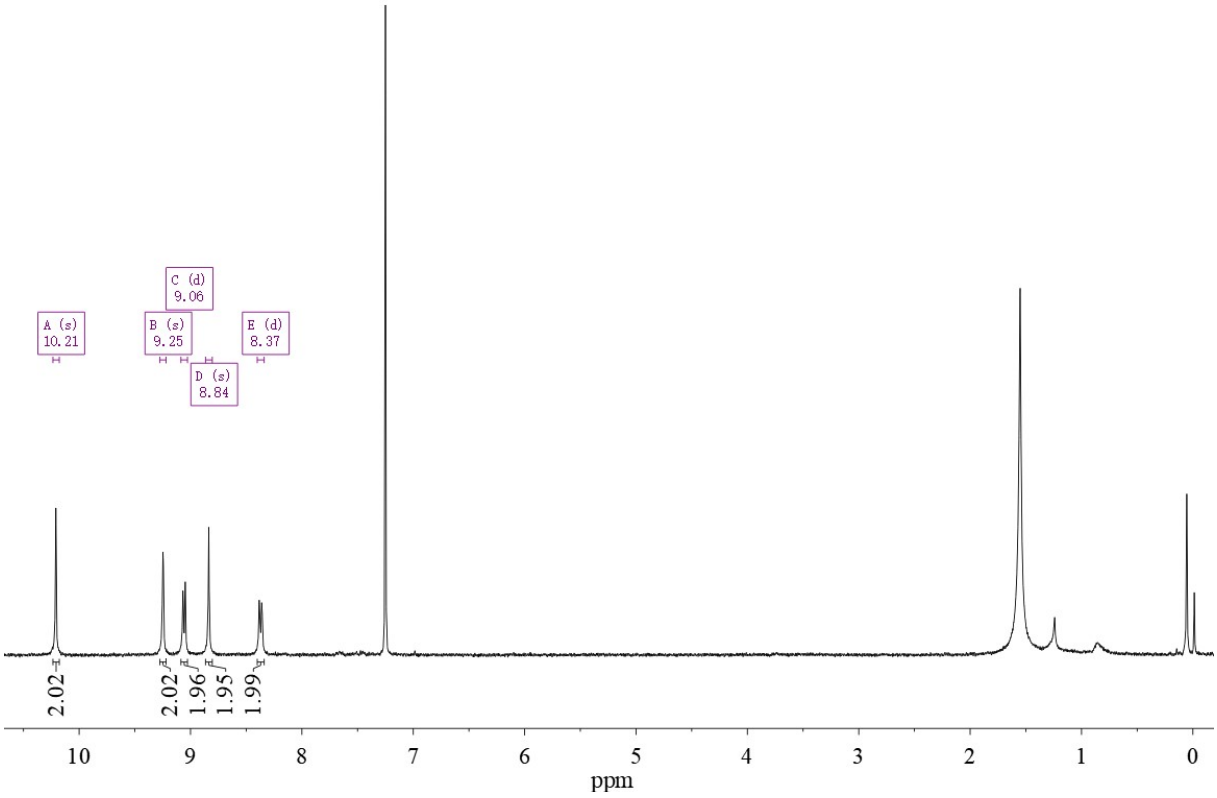
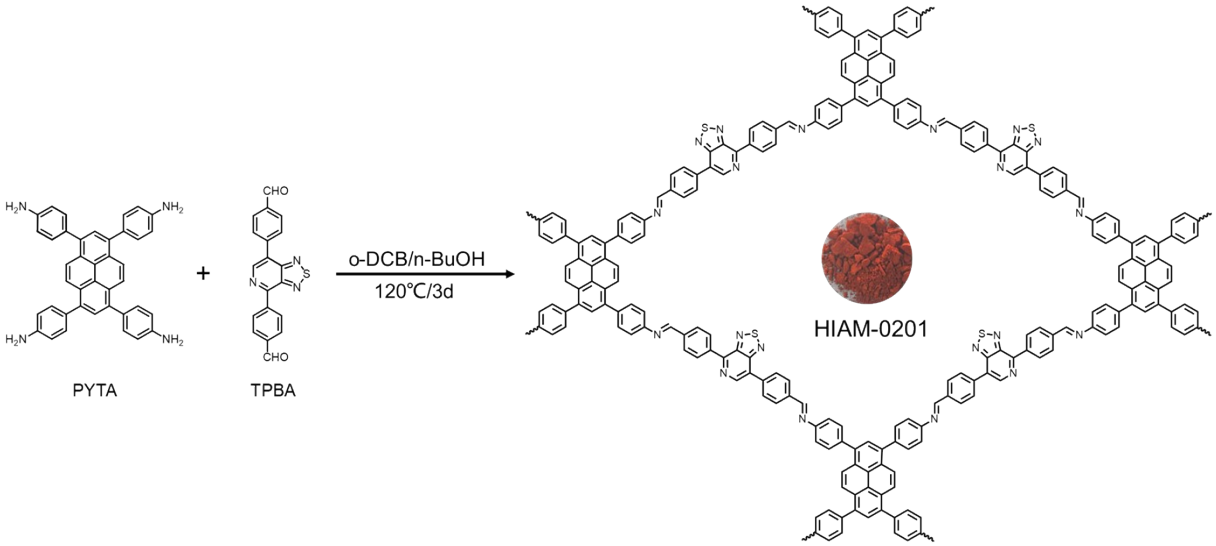


Figure S2. The ^{1}H NMR spectrum of BTNA in $CDCl_3$.

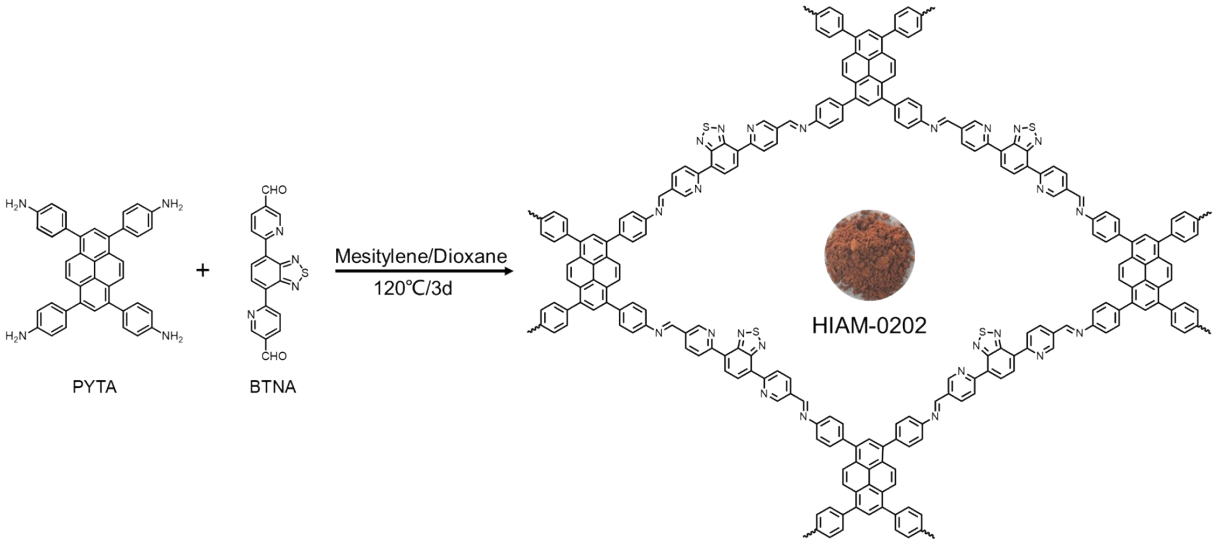
Synthesis of HIAM-0201

11.32 mg (0.02 mmol) of 4,4',4",4'"-(pyrene-1,3,6,8-tetrayl)tetraaniline (PYTA) and 13.80 (0.04 mmol) mg of 4,4'-([1,2,5]thiadiazolo[3,4-c]pyridine-4,7-diyl)dibenzaldehyde (TPBA) were added into a 10 mL Pyrex tube containing 0.5 mL of 1,2-dichlorobenzene (*o*-DCB) and 0.5 mL of *n*-butyl alcohol (*n*-BuOH), followed by adding 0.1 mL 6 M of aqueous acetic acid. This mixture was sonicated for 10 min to get a homogeneous dispersion. The tube was degassed through three freeze-pump-thaw cycles and kept at 120 °C for 3 days. Then the precipitate was collected by filtration and washed with THF for several times. The product was Soxhlet extracted in THF and dried under vacuum at 100 °C for 24 h to obtain red-orange powder (yield ~80%).



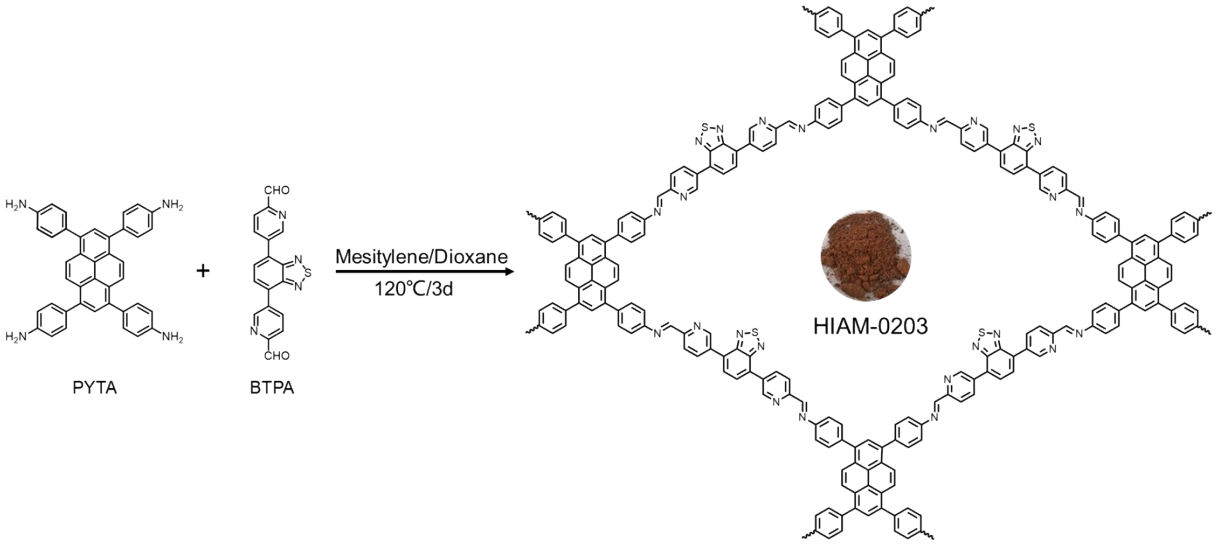
Synthesis of HIAM-0202

11.32 mg (0.02 mmol) of 4,4',4",4'"-(pyrene-1,3,6,8-tetrayl)tetraaniline (PYTA) and 13.84 mg (0.04 mmol) of 6,6'-(benzo[c][1,2,5]thiadiazole-4,7-diyl)dinicotinaldehyde (BTNA) were added into a 10 mL Pyrex tube containing 0.5 mL of dioxane and 0.5 mL of mesitylene, followed by adding 0.1 mL 12 M of aqueous acetic acid. This mixture was sonicated for 10 min to get a homogeneous dispersion. The tube was degassed through three freeze-pump-thaw cycles and kept at 120 °C for 3 days. Then the precipitate was collected by filtration and washed with THF for several times. The product was Soxhlet extracted in THF and dried under vacuum at 100 °C for 24 h to obtain yellow-brown powder (yield ~70%).



Synthesis of HIAM-0203

11.32 mg (0.02 mmol) of 4,4',4",4'"-(pyrene-1,3,6,8-tetrayl)tetraaniline (PYTA) and 13.84 mg (0.04 mmol) of 5,5'-(benzo[c][1,2,5]thiadiazole-4,7-diyl)dipicolinaldehyde (BTPA) were added into a 10 mL Pyrex tube containing 0.5 mL of dioxane and 0.5 mL of mesitylene, followed by adding 0.1 mL 6 M of aqueous acetic acid. This mixture was sonicated for 10 min to get a homogeneous dispersion. The tube was degassed through three freeze-pump-thaw cycles and kept at 120 °C for 3 days. Then the precipitate was collected by filtration and washed with THF for several times. The product was Soxhlet extracted in THF and dried under vacuum at 100 °C for 24 h to obtain yellow-brown powder (yield ~85%).



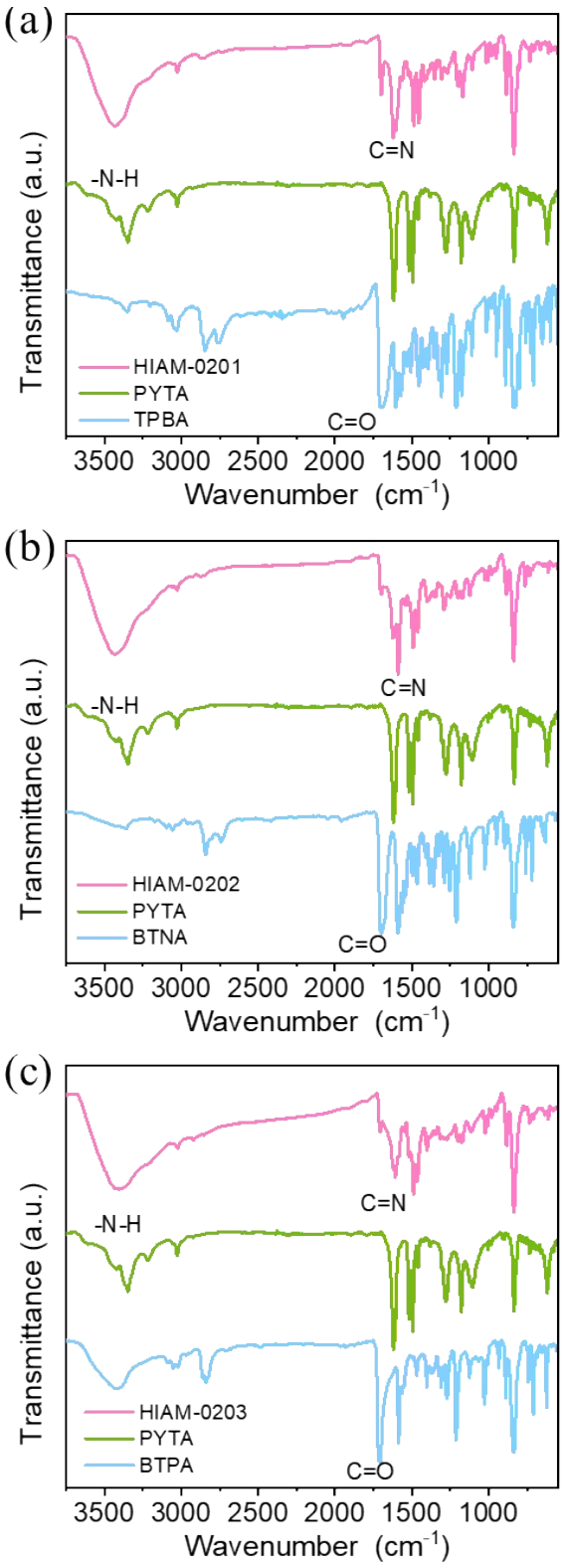


Figure S3. FT-IR spectra of HIAM-0201 (a), HIAM-0202 (b), HIAM-0203 (c) and the corresponding monomers.

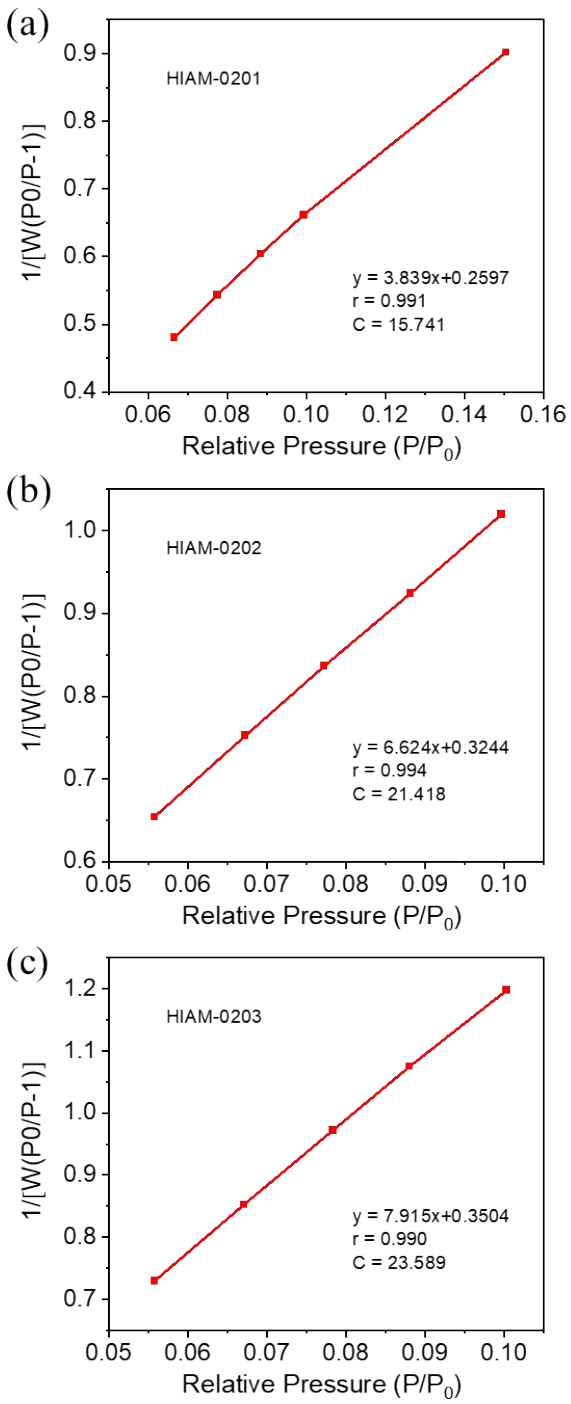


Figure S4. BET plots of (a) HIAM-0201, (b) HIAM-0202 and (b) HIAM-0203 calculated from N_2 adsorption data at 77K.

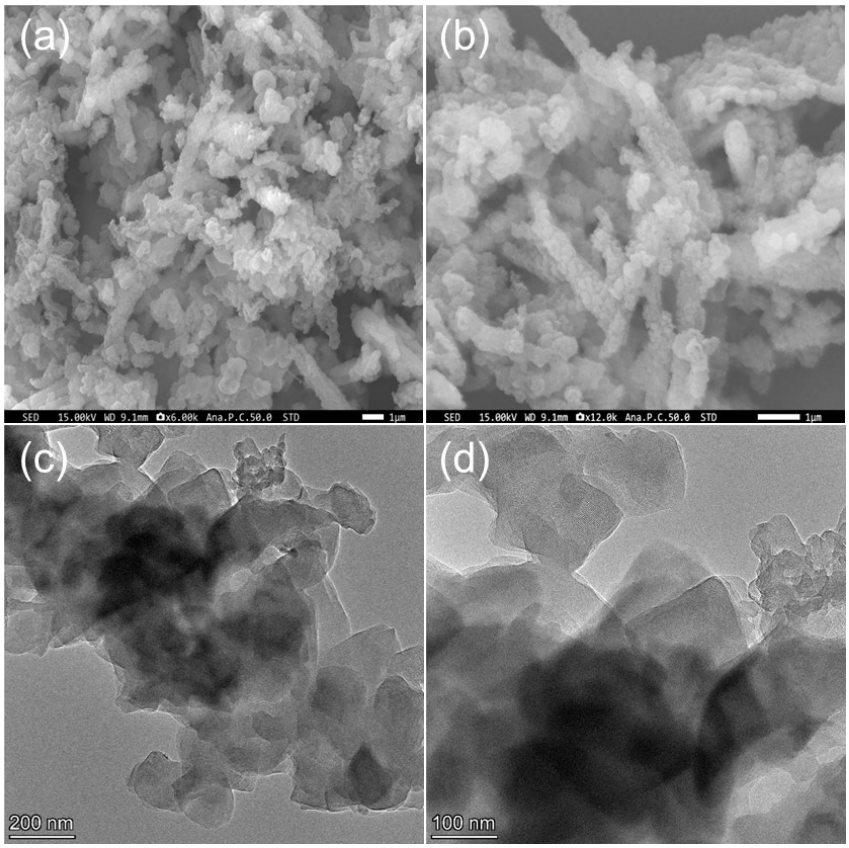


Figure S5. SEM images (a-b) and TEM images (c-d) of HIAM-0201.

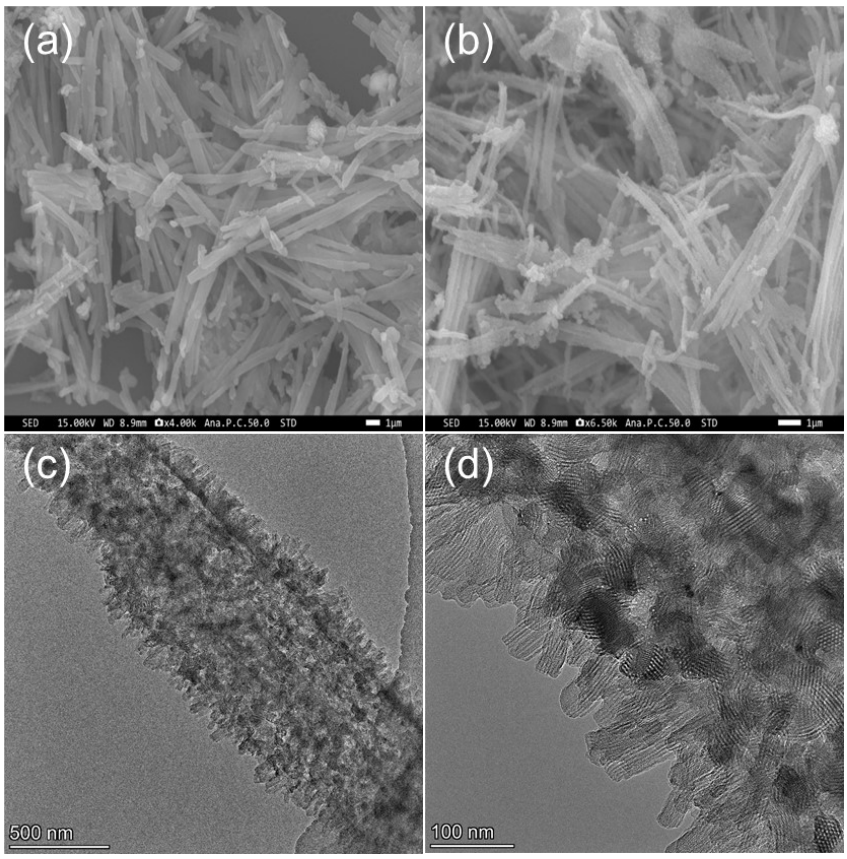


Figure S6. SEM images (a-b) and TEM images (c-d) of HIAM-0202.

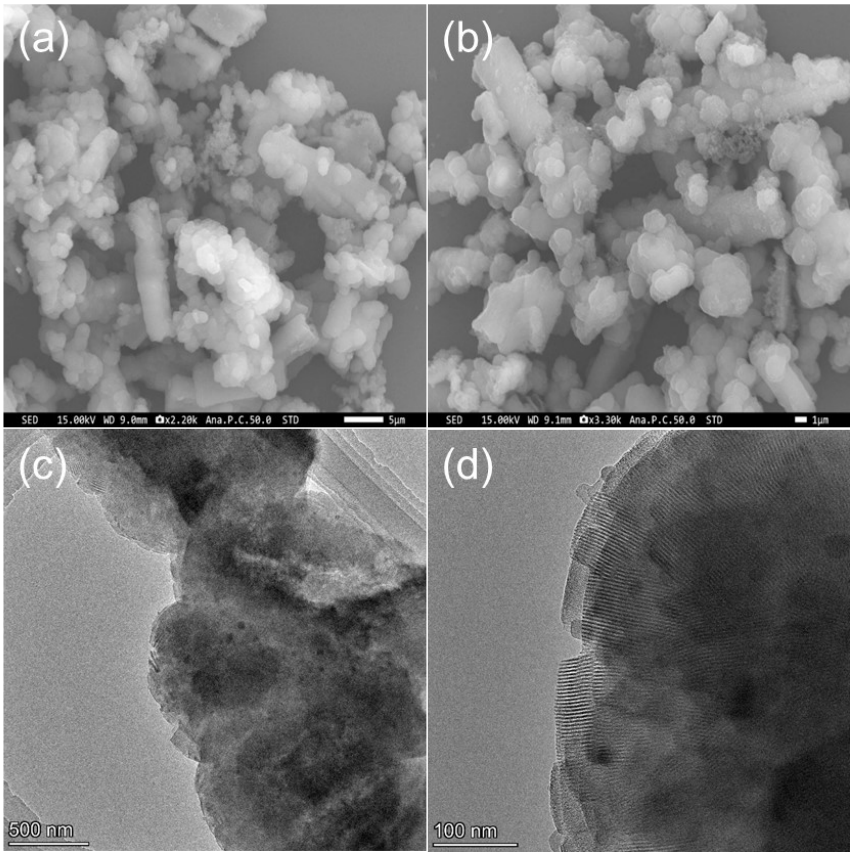


Figure S7. SEM images (a-b) and TEM images (c-d) of HIAM-0203.

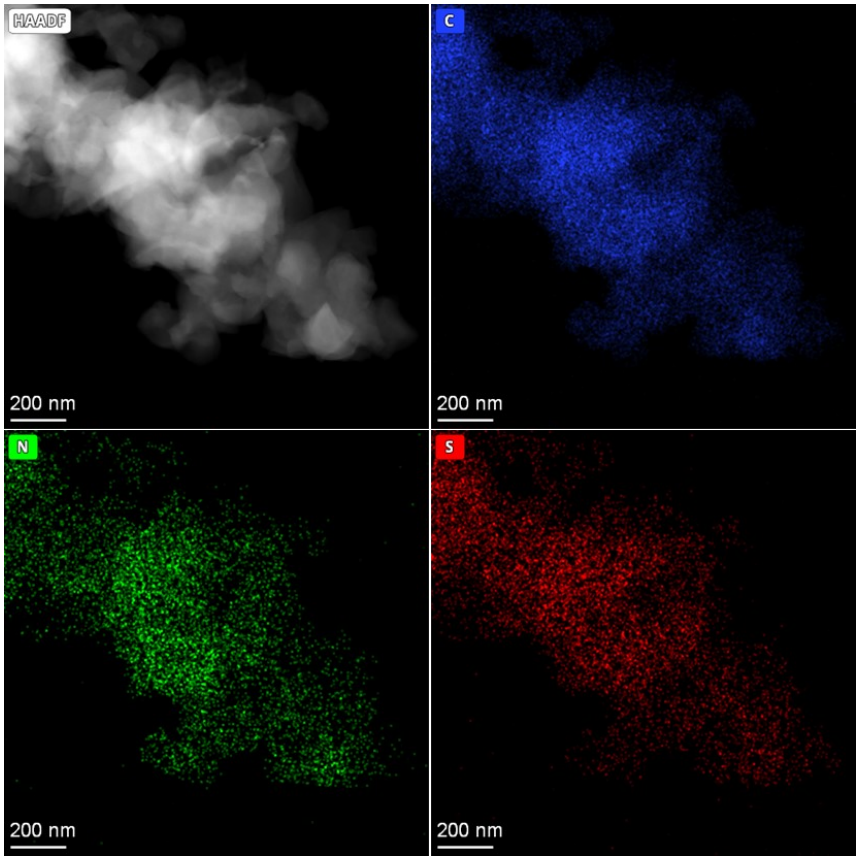


Figure S8. HAADF-STEM image and the corresponding STEM-EDS elemental mapping images of HIAM-0201.

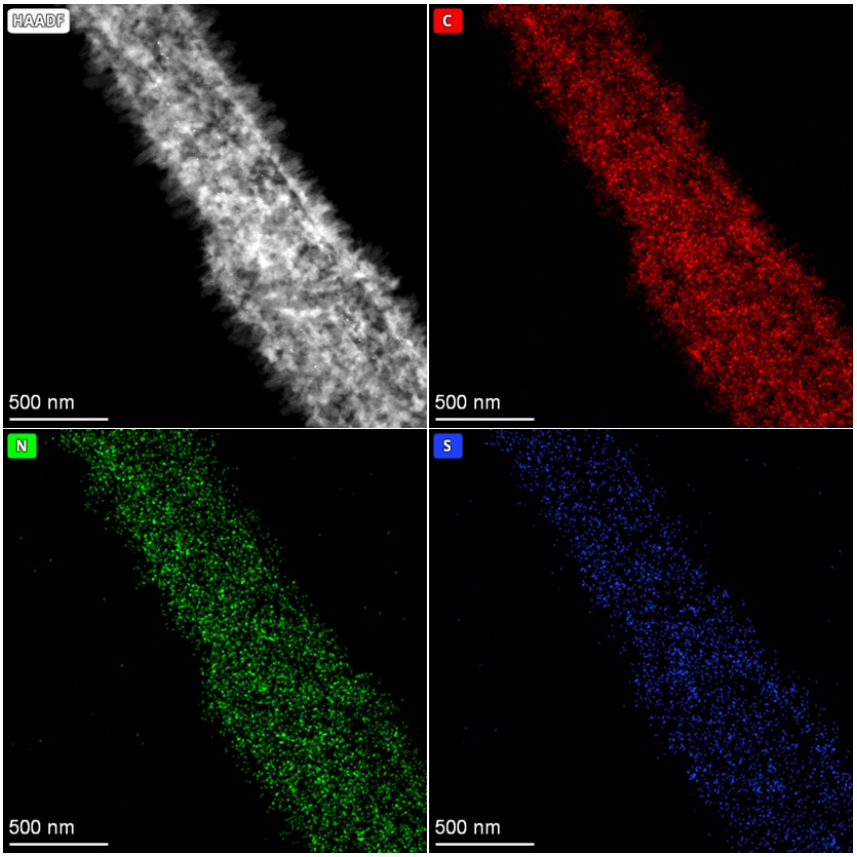


Figure S9. HAADF-STEM image and the corresponding STEM-EDS elemental mapping images of HIAM-0202.

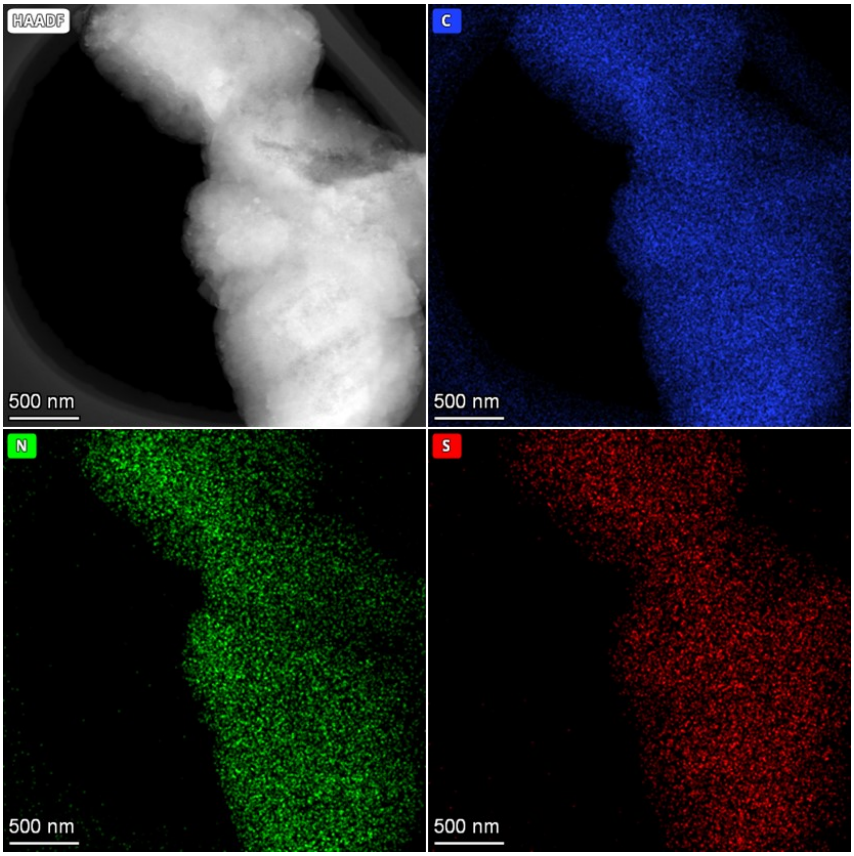


Figure S10. HAADF-STEM image and the corresponding STEM-EDS elemental mapping images of HIAM-0203.

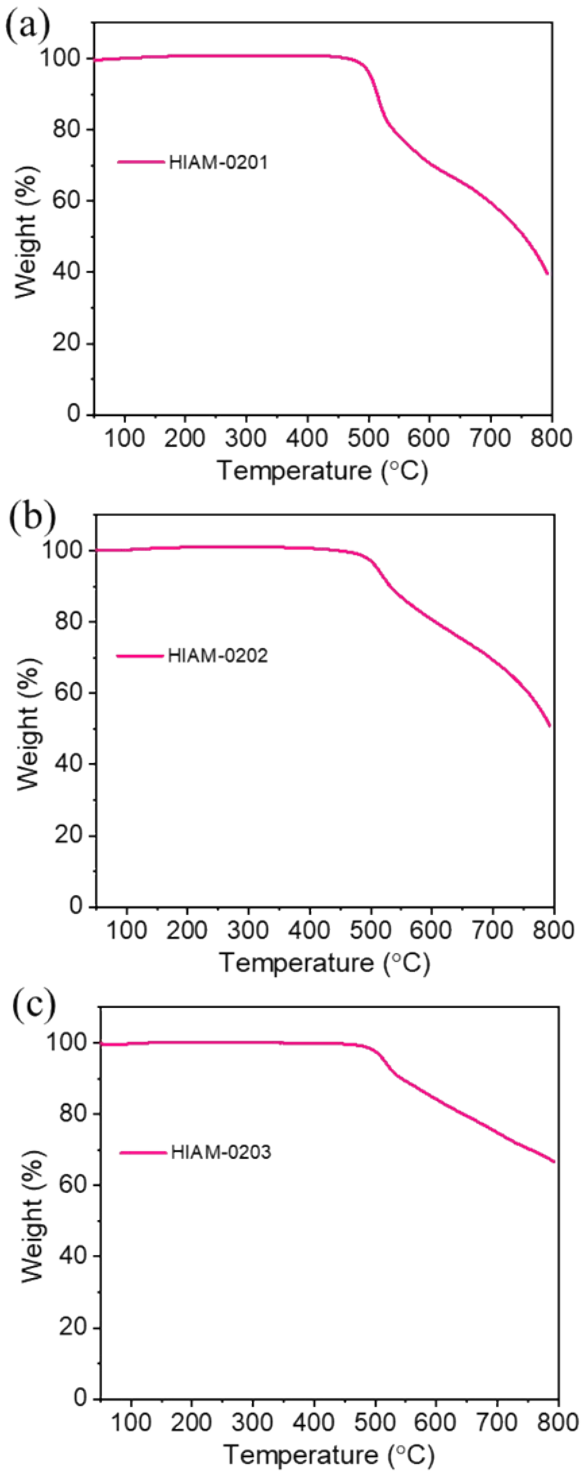


Figure S11. TGA curves of HIAM-0201 (a), HIAM-0202 (b), HIAM-0203 (c) under N_2 at 50-800 ℃.

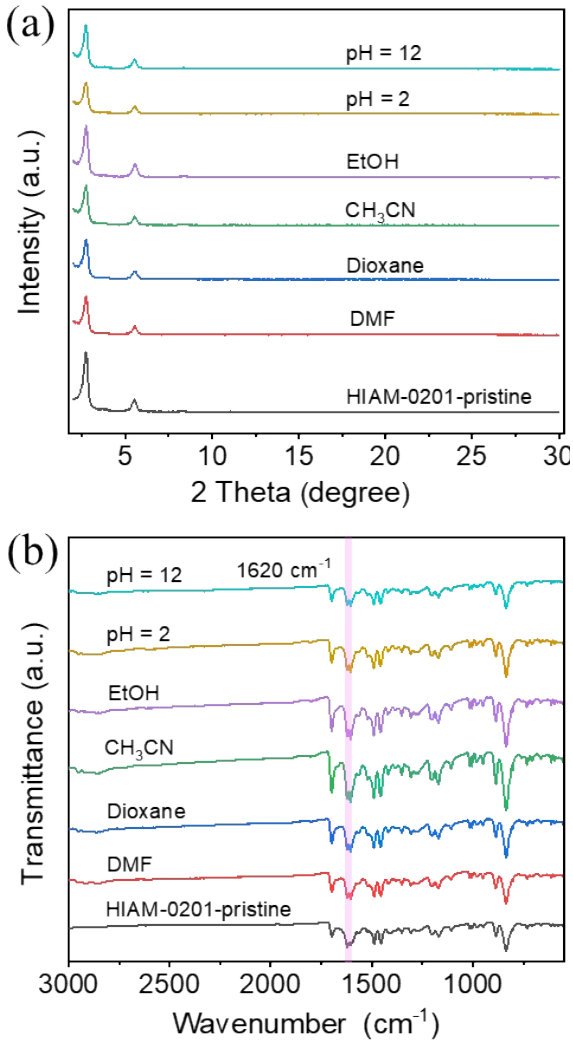


Figure S12. PXRD patterns (a) and FT-IR spectra (b) of HIAM-0201 after being immersed in different solutions for three days.

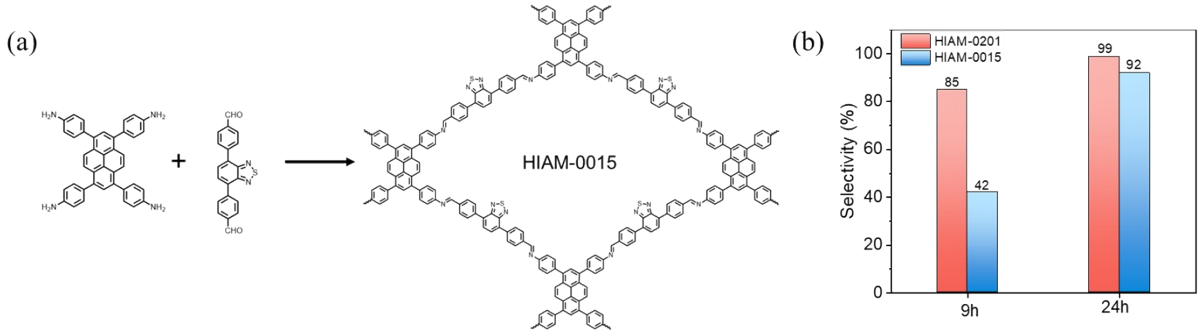


Figure S13. Schematic of the synthesis of HIAM-0015 (a), selectivity of photocatalytic synthesis of quinazolinones over HIAM-0201 and HIAM-0015 (b).

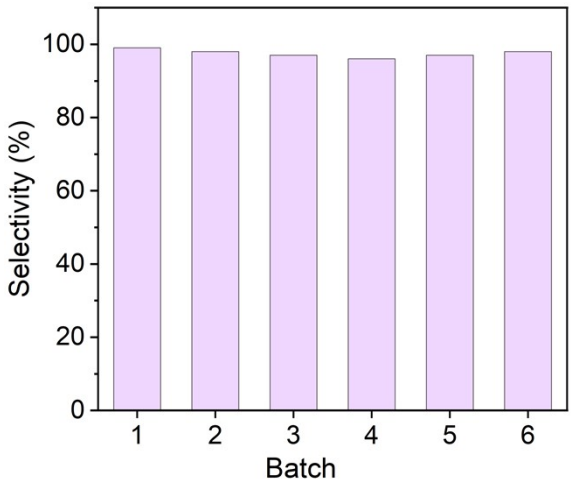


Figure S14. Repeatability test of HIAM-0201 for photocatalytic synthesis of quinazolinones.

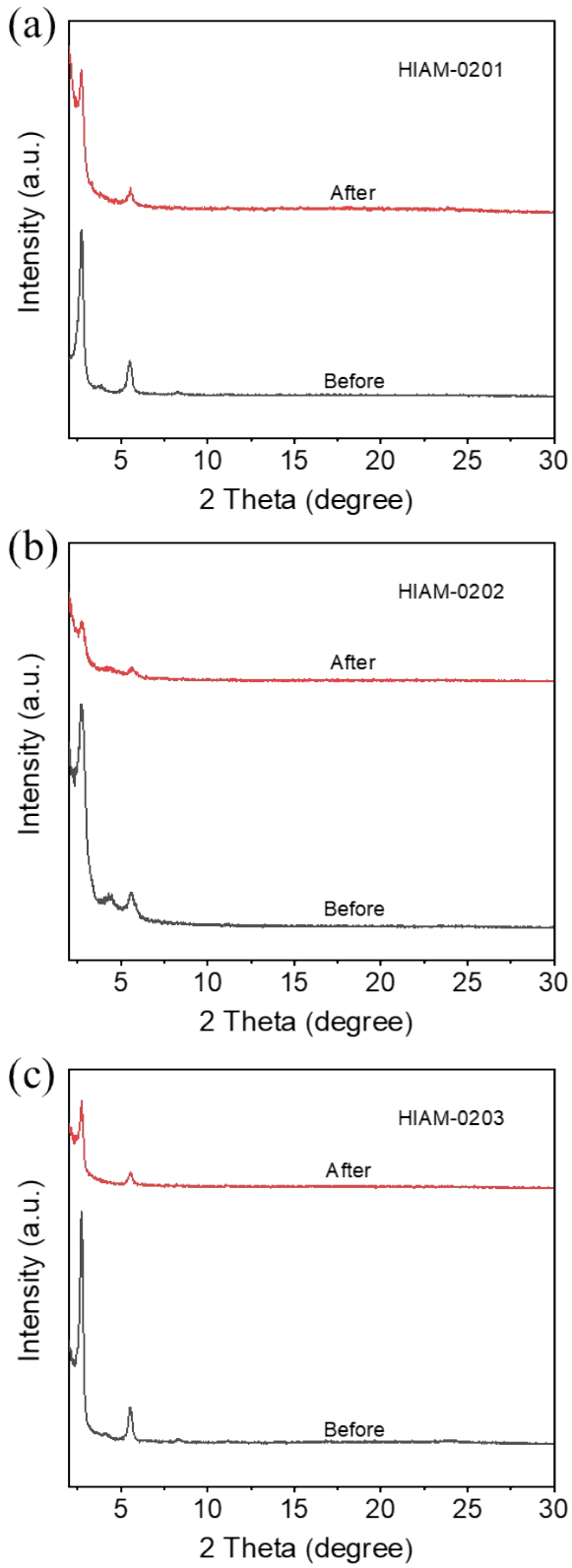


Figure S15. PXRD patterns of HIAM-0201 (a), HIAM-0202 (b), HIAM-0203 (c) before and after photocatalytic reaction.

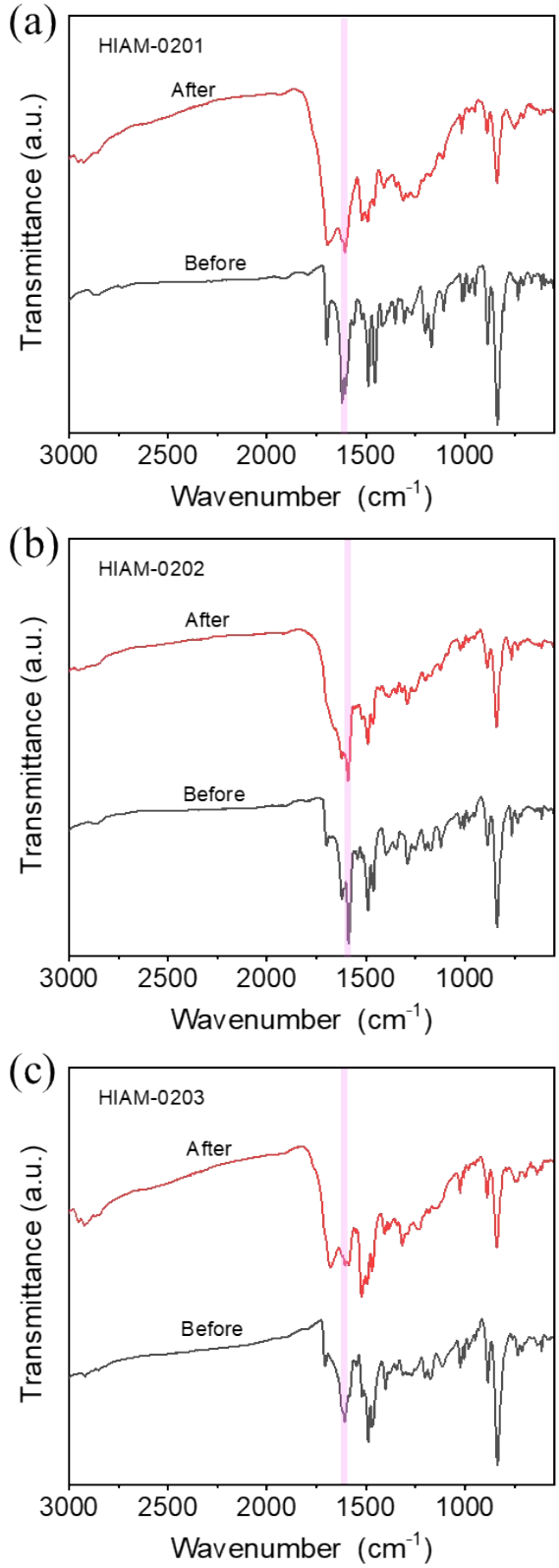


Figure S16. FT-IR spectra of HIAM-0201 (a), HIAM-0202 (b), HIAM-0203 (c) before and after photocatalytic reaction.

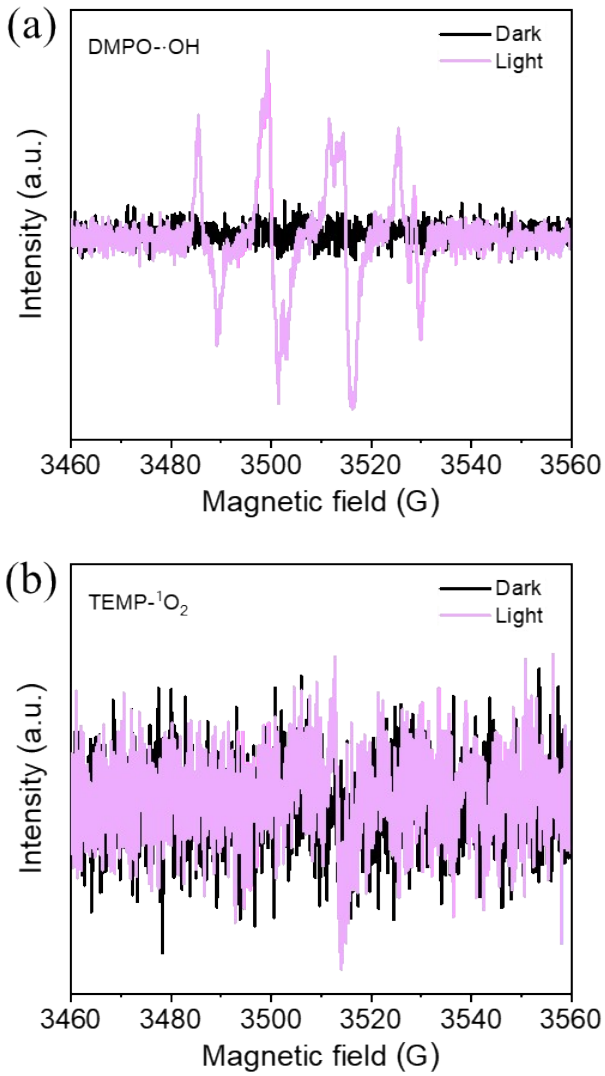


Figure S17. EPR spectra of HIAM-0201 and DMPO-·OH (a), TEMPO-$^1O_2$ (b) with and without visible light irradiation.

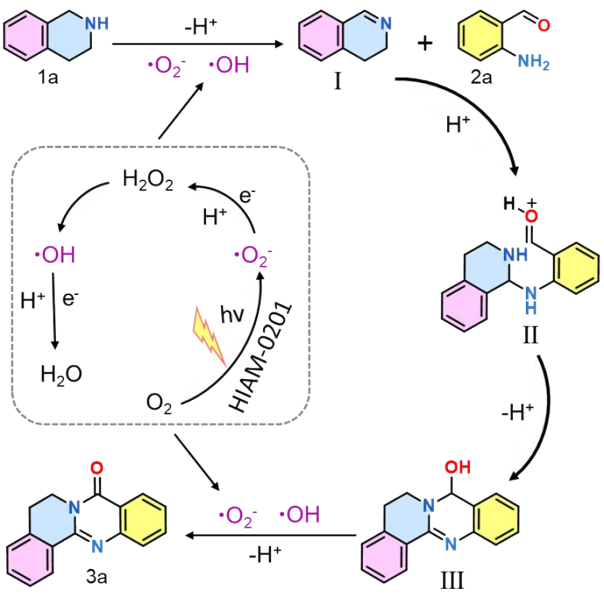


Figure S18. The probable reaction mechanism for photocatalytic synthesis of quinazolinones.

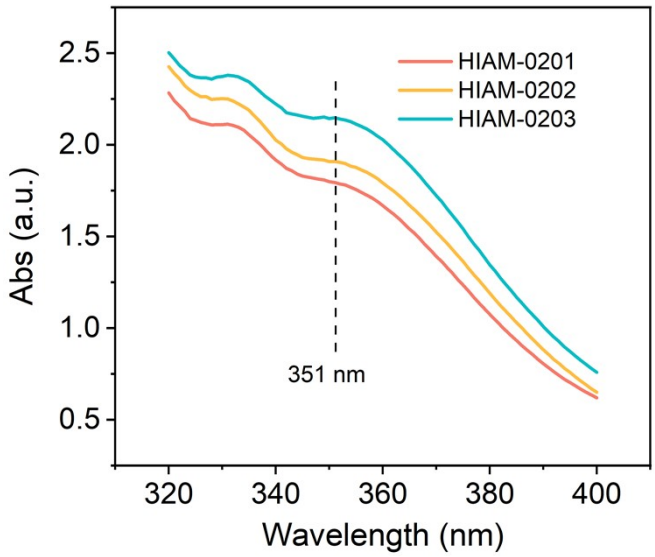


Figure S19. UV-vis absorption spectra of the iodimetry solution by HIAM-0201, HIAM-0202 and HIAM-0203 after photocatalytic reaction solution.

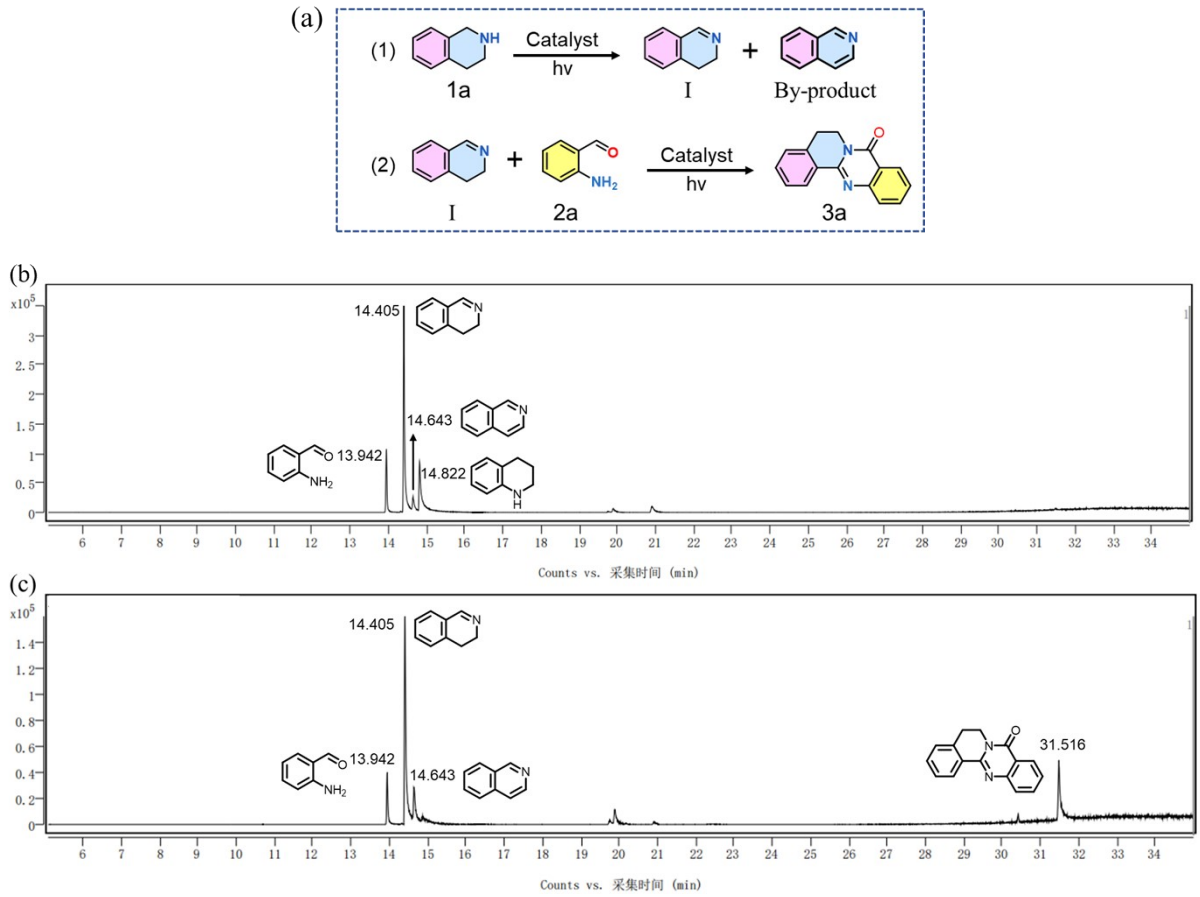


Figure S20. The photocatalytic synthesis of quinazolinones proceeds via a two-step reaction pathway (a). The first step involves the oxidation of tetrahydroisoquinoline (1a) to generate the key intermediate, 3,4-dihydroisoquinoline (I), along with minor by-products. In the second step, intermediate I reacts with 2a to yield the final product 3a. (b) and (c) depict the corresponding gas chromatograms of the reactants, intermediates, by-products and products for steps (1) and (2), respectively.

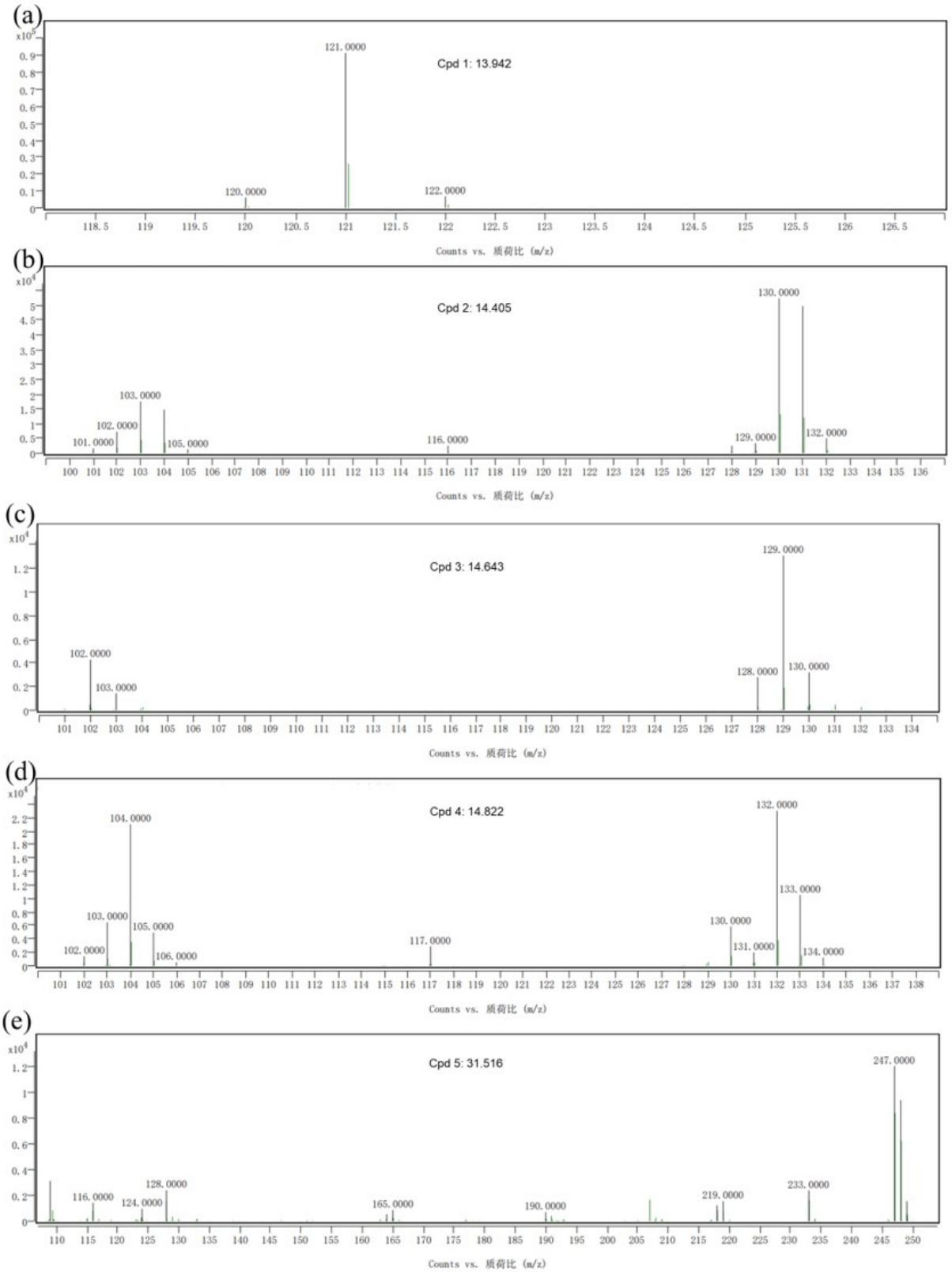


Figure S21. The mass spectra corresponding to the reactants (a, d), intermediates (b), by-product (c) and products (e) for the reaction steps depicted in panels (b) and (c) of Figure S20.

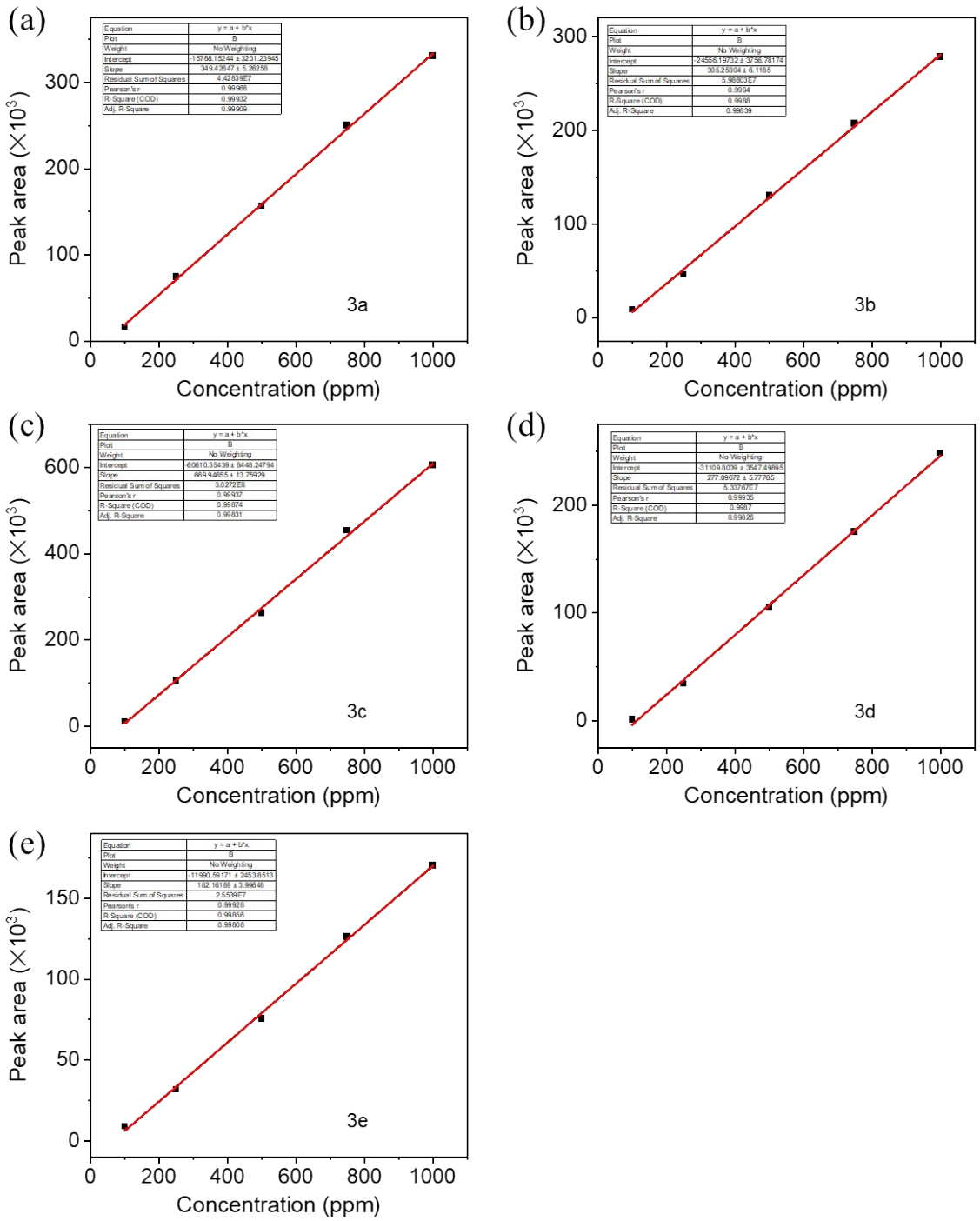
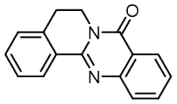


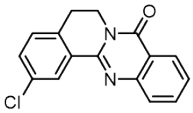
Figure S22.The linear relationship graph of products 3a (a), 3b (b), 3c (c), 3d (d) and 3e (e) between its peak area and concentration determined by external standard method.

^{1}H-NMR analysis of the photocatalytic products



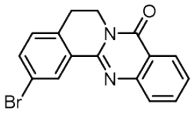
3a

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.48 (dd, J = 7.6, 1.7 Hz, 1H), 8.33-8.28 (m, 1H), 7.82-7.71 (m, 2H), 7.54-7.40 (m, 3H), 7.28 (dt, J = 7.0, 1.1 Hz, 1H), 4.45-4.38 (m, 2H), 3.10 (t, J = 6.5 Hz, 2H) ppm.



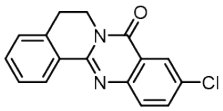
3b

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.47 (d, J = 2.3 Hz, 1H), 8.29 (d, J = 8.0 Hz, 1H), 7.78-7.74 (m, 2H), 7.50-7.39 (m, 2H), 7.22 (d, J = 8.1 Hz, 1H), 4.43-4.34 (m, 2H), 3.07 (t, J = 6.5 Hz, 2H) ppm.



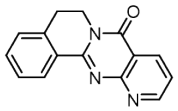
3c

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.62 (d, J = 2.1 Hz, 1H), 8.30 (dt, J = 7.9, 1.2 Hz, 1H), 7.78-7.74 (m, 2H), 7.58 (dd, J = 8.1, 2.1 Hz, 1H), 7.47 (ddd, J = 8.2, 5.0, 3.3 Hz, 1H), 7.16 (d, J = 8.1 Hz, 1H), 4.42-4.36 (m, 2H), 3.08-3.02 (m, 2H) ppm.



3d

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.45 (dd, J = 7.7, 1.6 Hz, 1H), 8.26 (d, J = 2.3 Hz, 1H), 7.76 -7.62 (m, 2H), 7.46 (dtd, J = 21.6, 7.4, 1.5 Hz, 2H), 7.30-7.27 (m, 1H), 4.45-4.34 (m, 2H), 3.10 (t, J = 6.5 Hz, 2H) ppm.



3e

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.97 (dd, J = 4.5, 2.0 Hz, 1H), 8.63 (ddd, J = 10.0, 7.8, 1.8 Hz, 2H), 7.49 (dd, J = 7.4, 1.6 Hz 1H), 7.45 (dd, J = 7.7, 1.4 Hz, 1H), 7.39 (dd, J = 7.9, 4.5 Hz, 1H), 7.28 (dd, J = 7.5, 1.3 Hz, 1H), 4.40 (dd, J = 7.1, 6.0 Hz, 2H), 3.12 (t, J = 6.5 Hz, 2H) ppm.

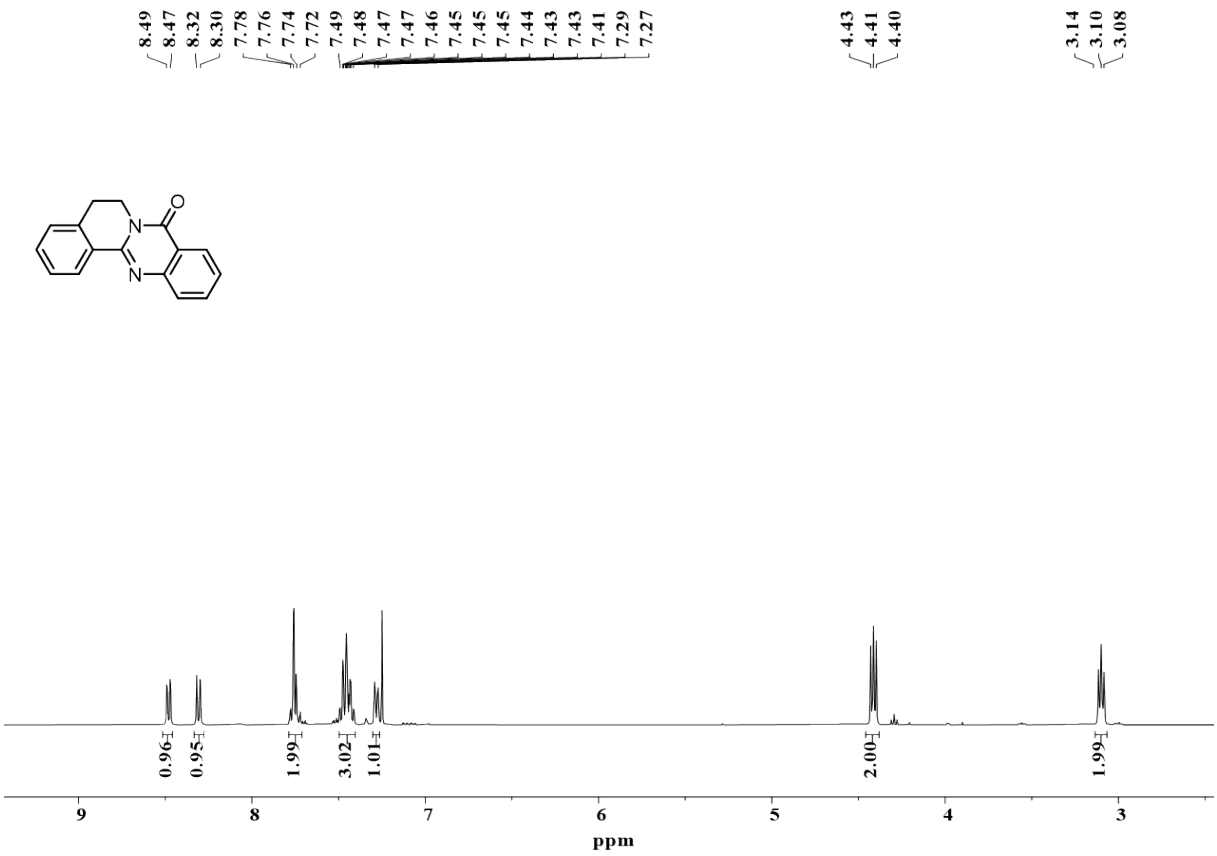


Figure S23. ^{1}H NMR spectrum of 3a in $CDCl_3$.

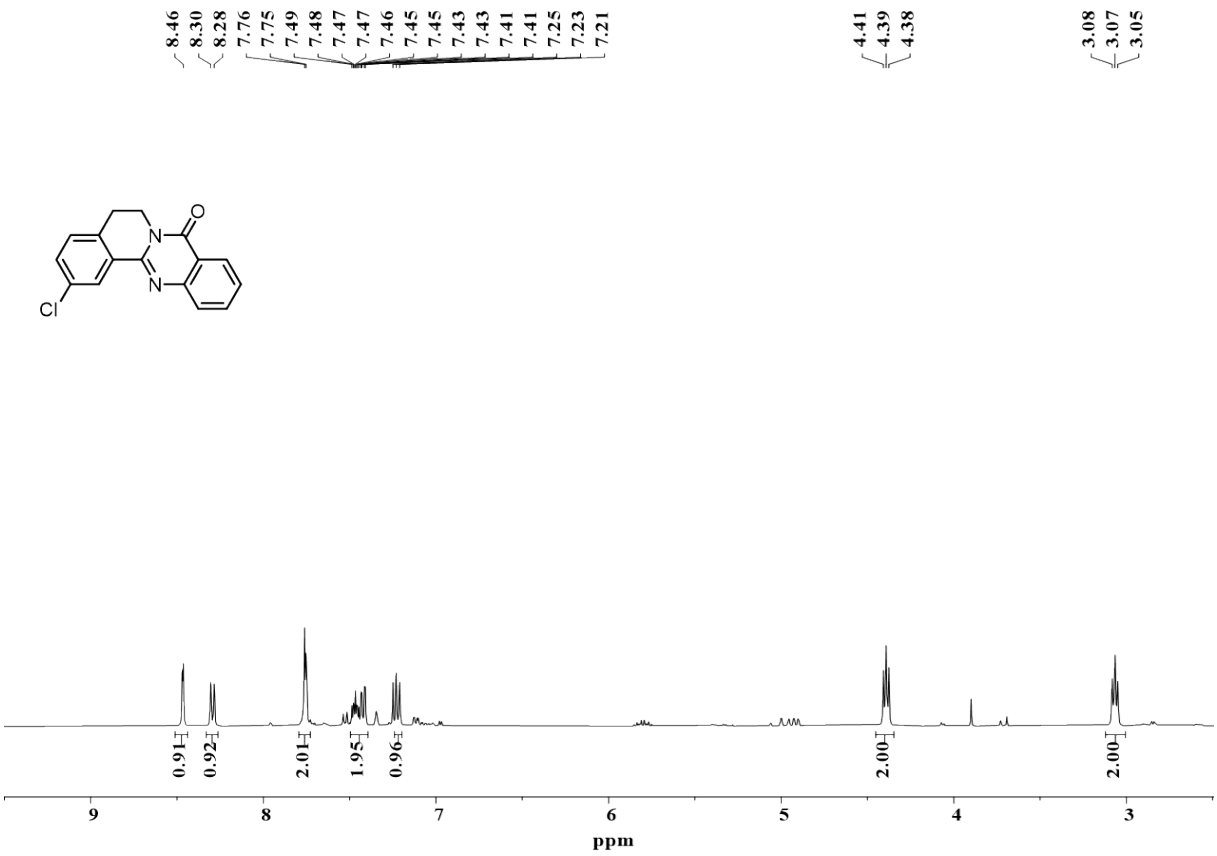


Figure S24. ^{1}H NMR spectrum of 3b in $CDCl_3$.

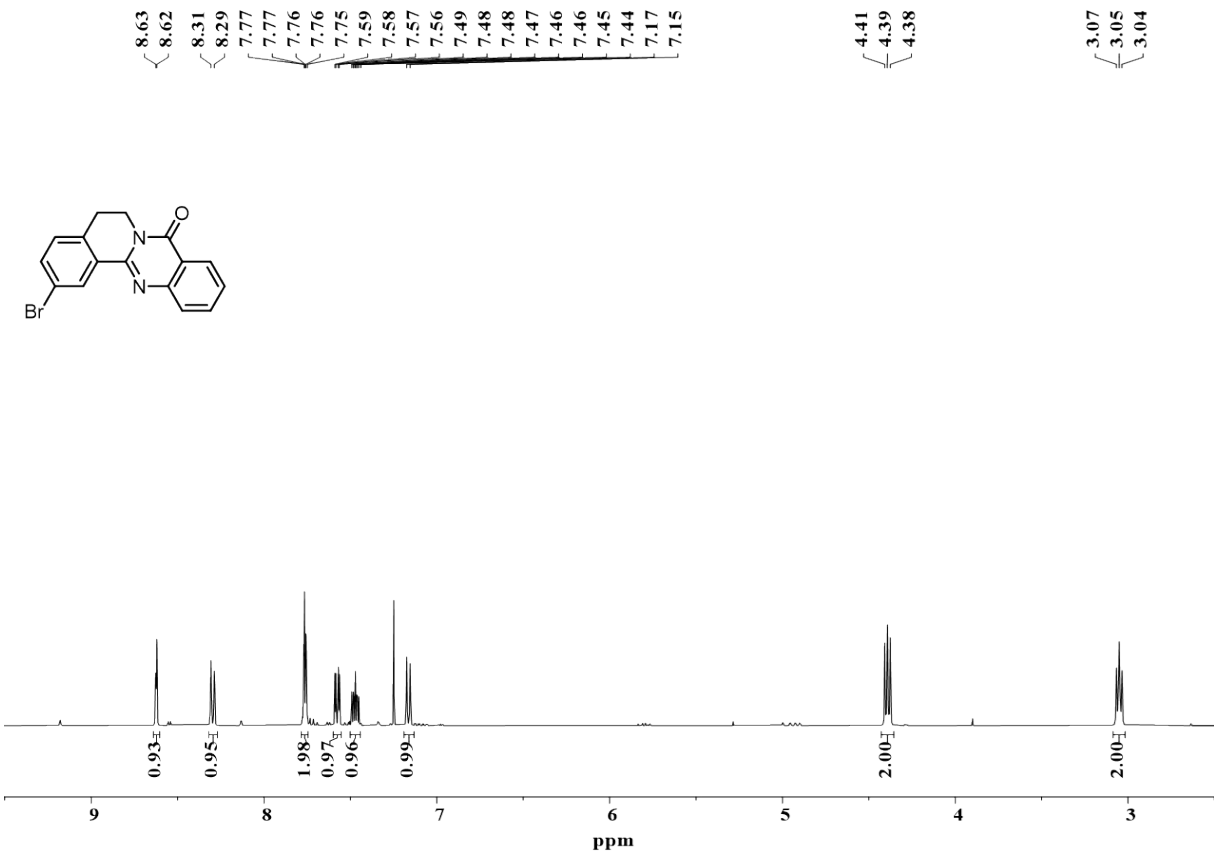


Figure S25. ^{1}H NMR spectrum of 3c in $CDCl_3$.

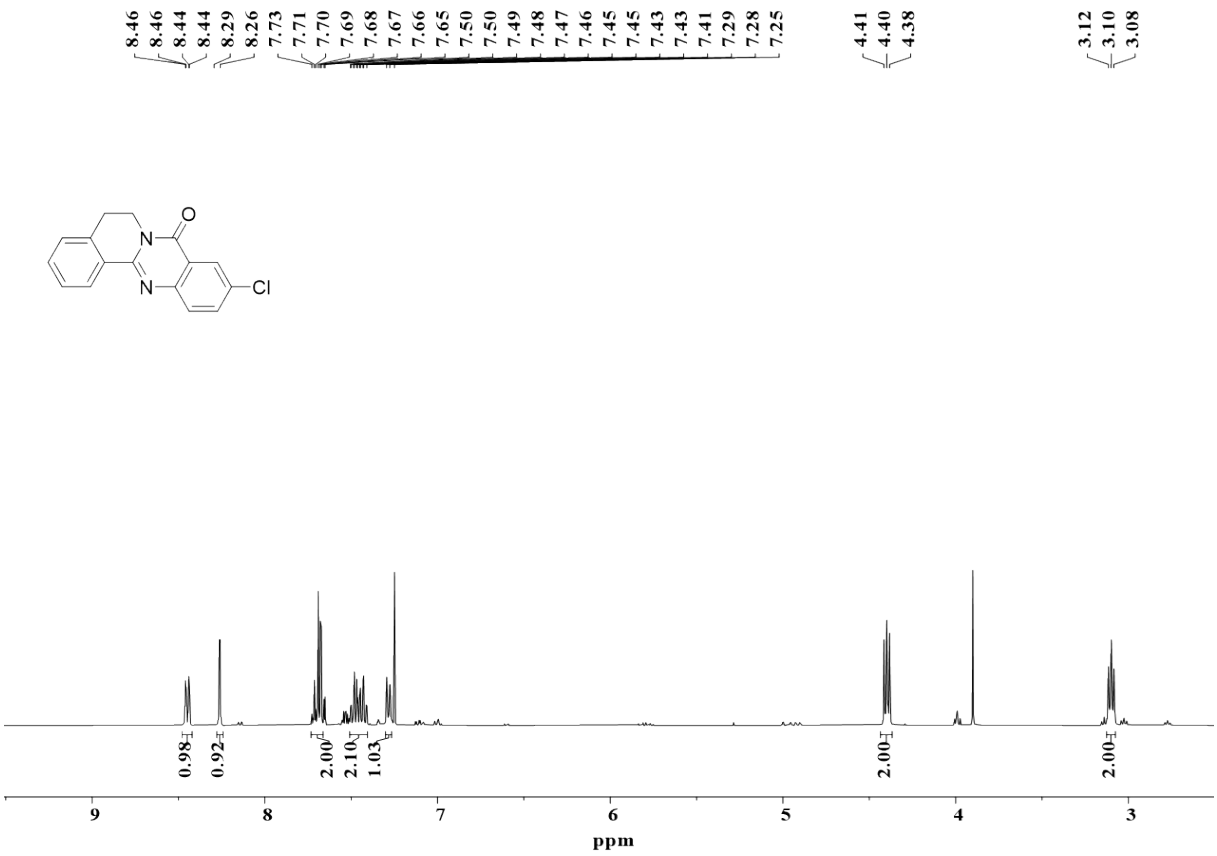


Figure S26. ^{1}H NMR spectrum of 3d in $CDCl_3$.

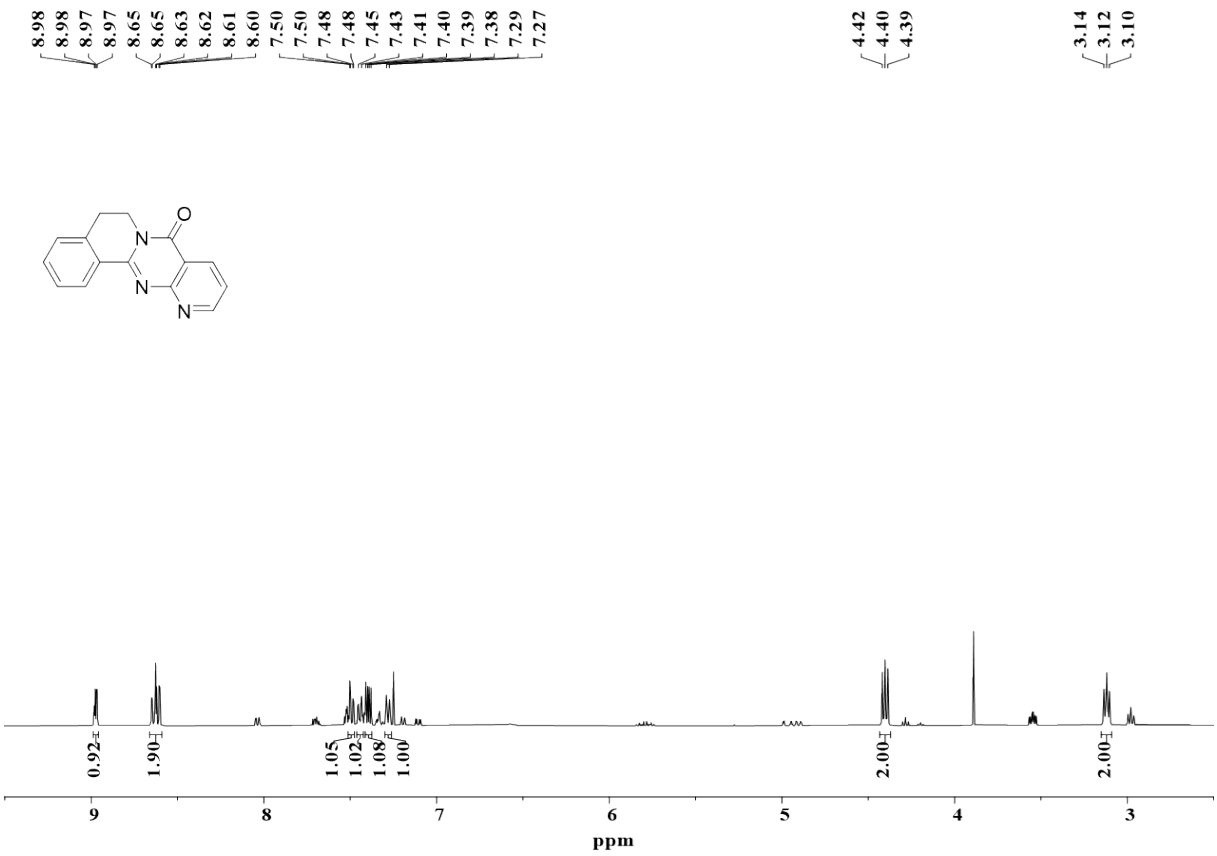


Figure S27. ^{1}H NMR spectrum of 3e in $CDCl_3$.

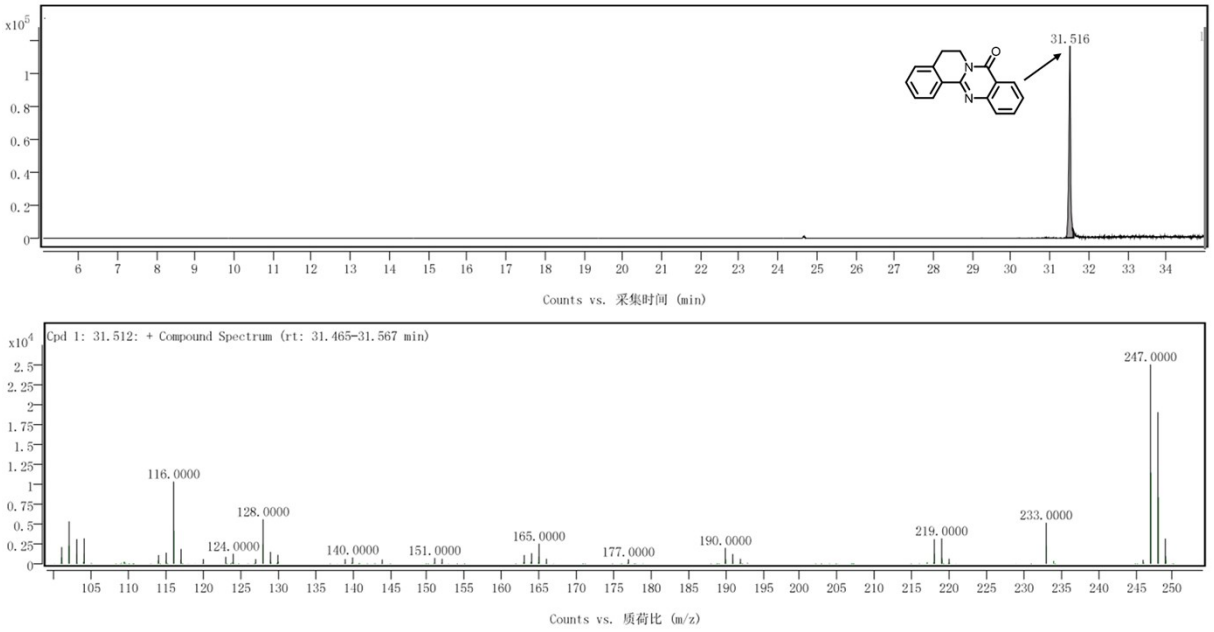


Figure S28. GC-MS spectra of 3a.

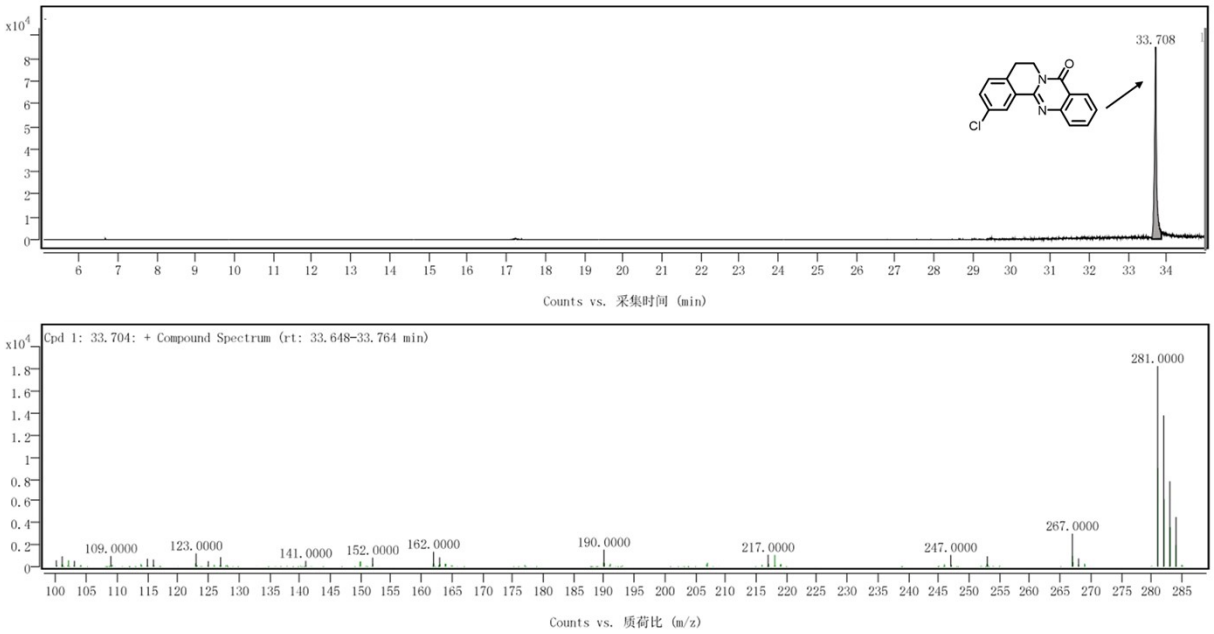


Figure S29. GC-MS spectra of 3b.

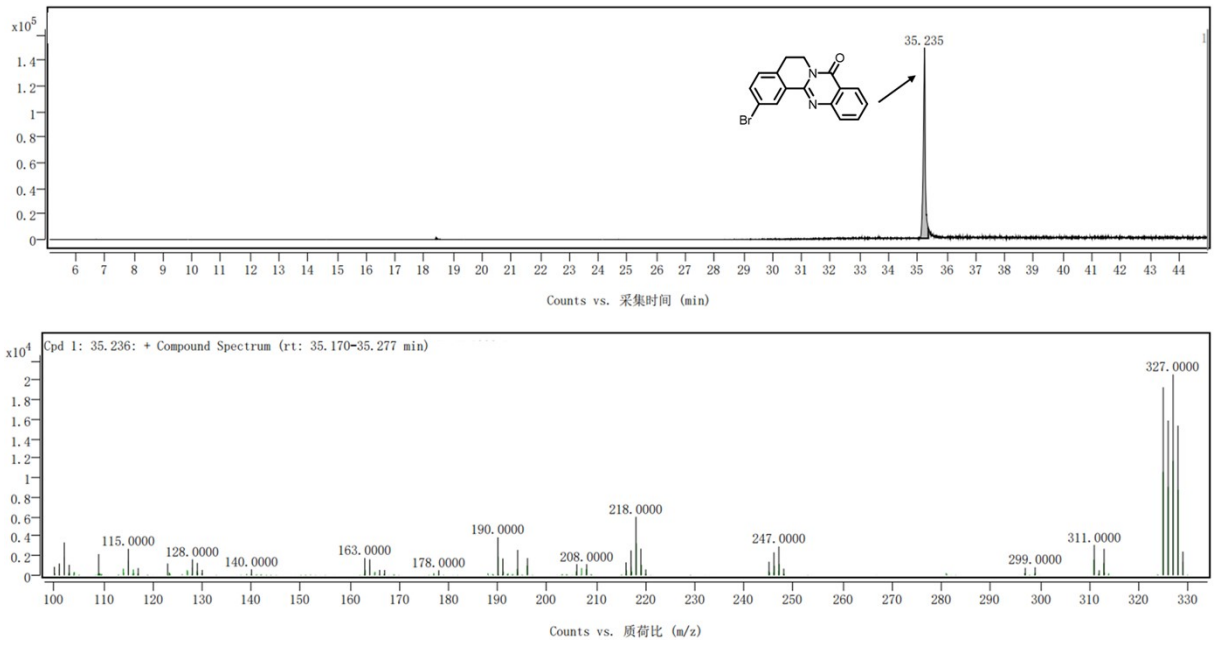


Figure S30. GC-MS spectra of 3c.

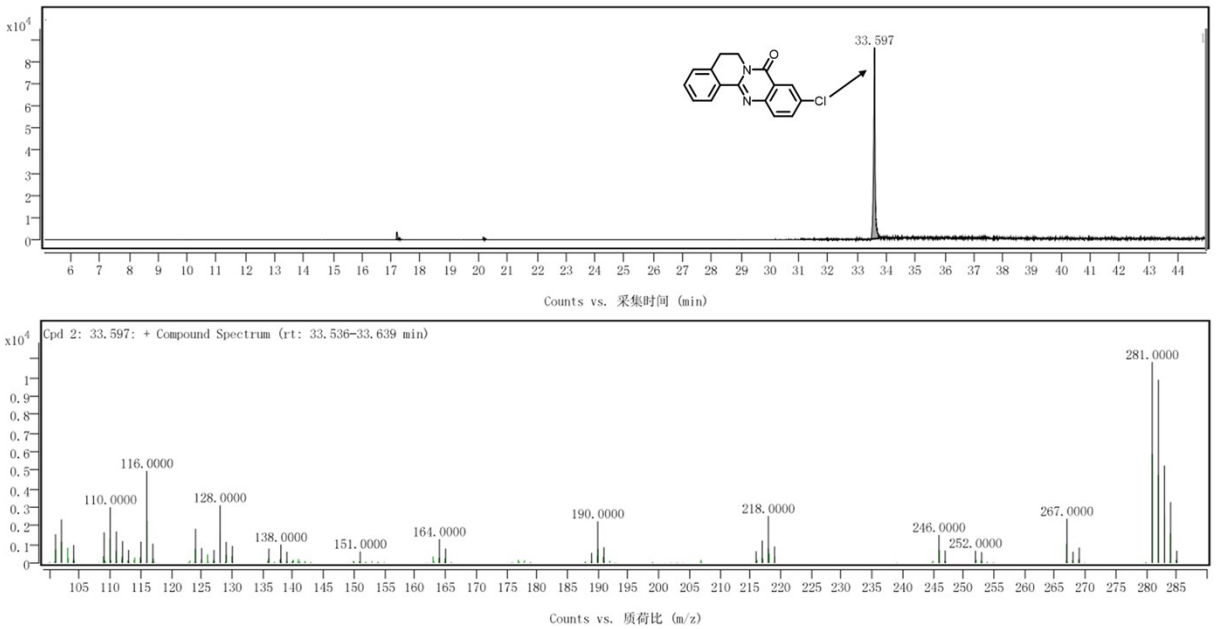


Figure S31. GC-MS spectra of 3d.

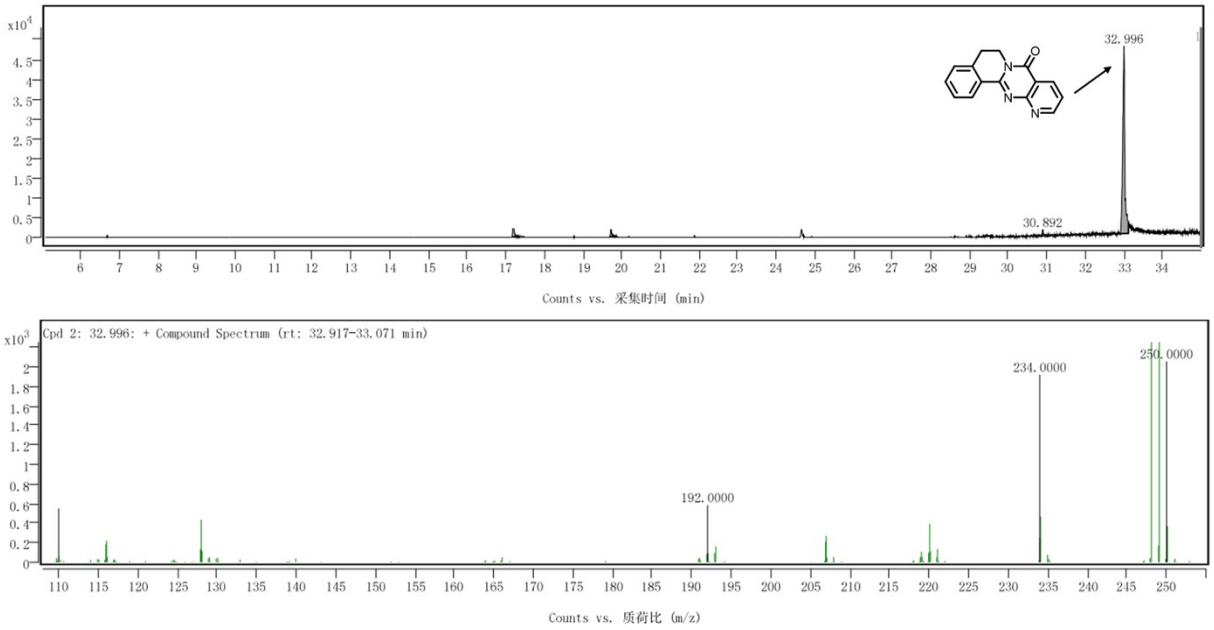


Figure S32. GC-MS spectra of 3e.

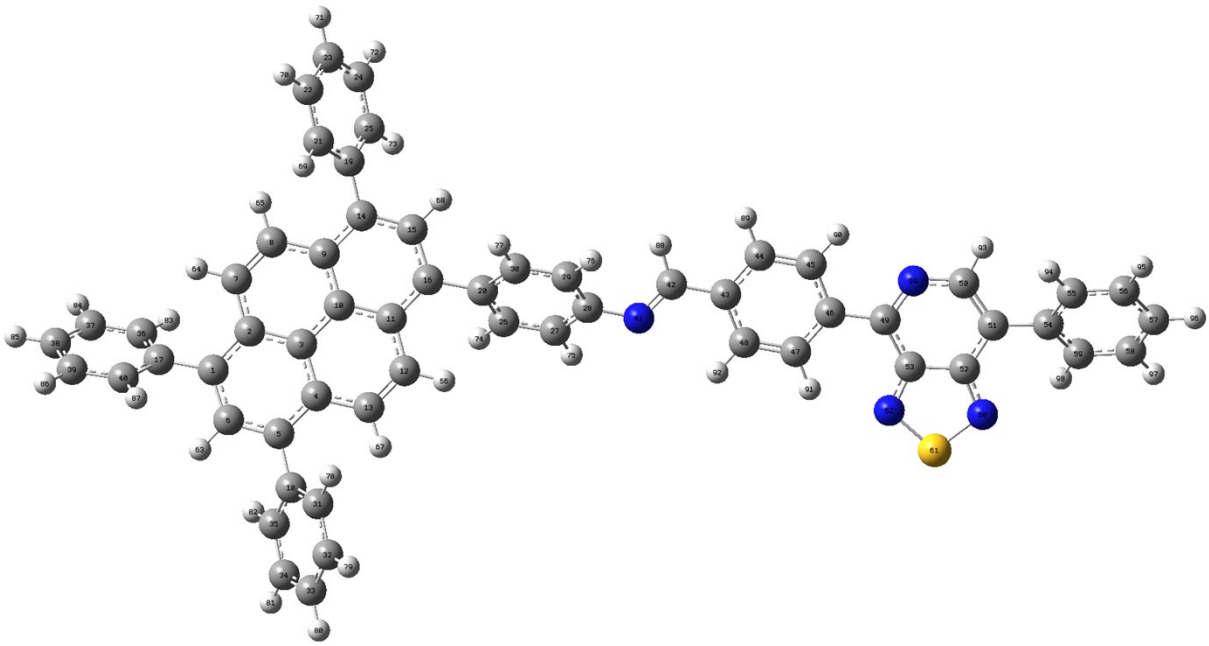


Figure S33. The calculated fragment of HIAM-0201. The white, grey, blue and yellow spheres refer to H, C, N and S, respectively.

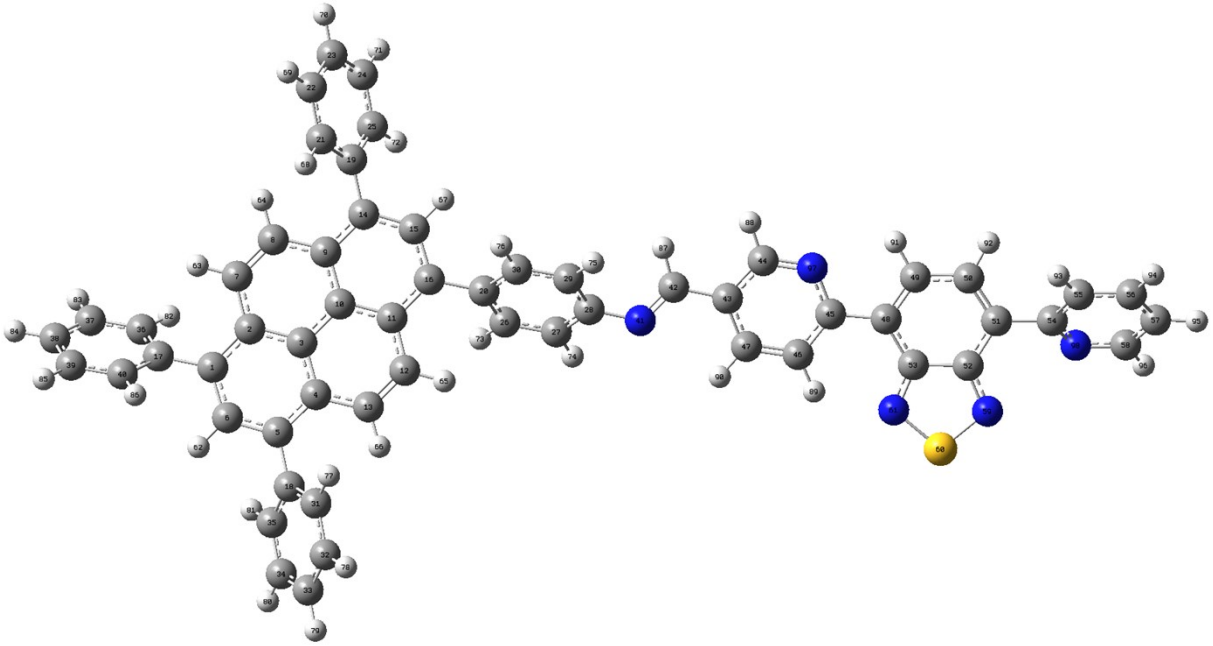


Figure S34. The calculated fragment of HIAM-0202. The white, grey, blue and yellow spheres refer to H, C, N and S, respectively.

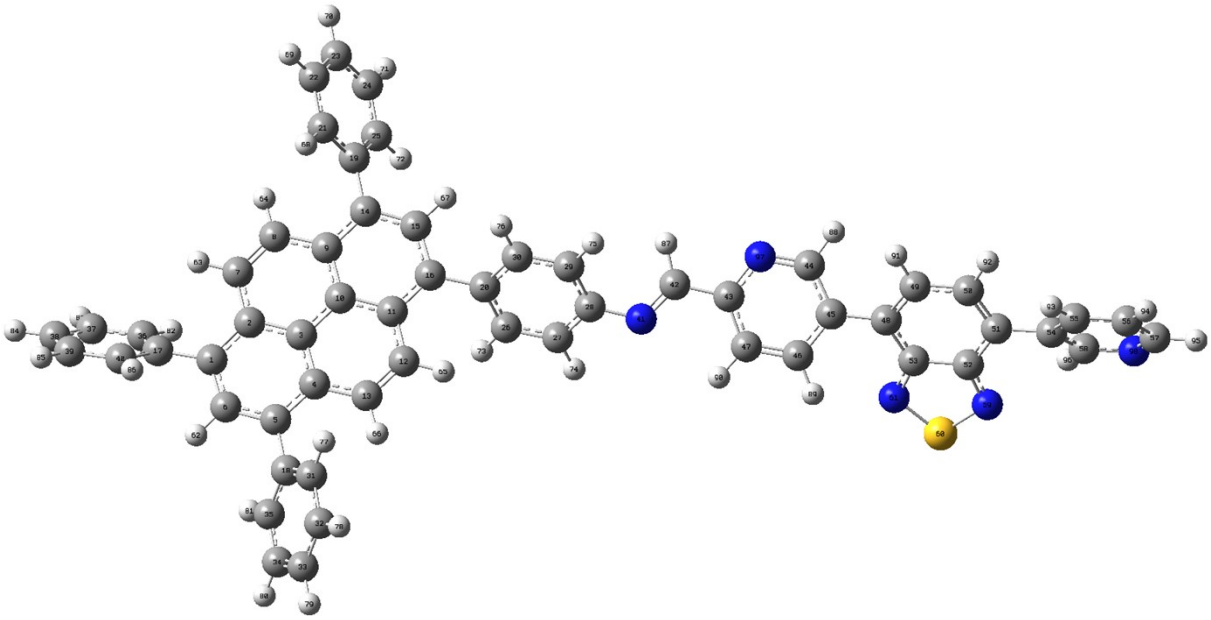


Figure S35. The calculated fragment of HIAM-0203. The white, grey, blue and yellow spheres refer to H, C, N and S, respectively.

Table S1. Unit cell parameters and fractional atomic coordinates for HIAM-0201.

Space group: *pm* a = 33.5343 Å, b = 3.9860 Å, and c = 33.8232 Å, α = γ = 90°, and β = 96.7239°				
N1	N	0.24764	-0.5	0.68844
C2	C	0.20806	-0.5	0.69641
C3	C	0.19532	-0.5	0.73565
C4	C	0.22657	-0.5	0.76535
C5	C	0.26694	-0.5	0.75615
C6	C	0.278	-0.5	0.71649
N7	N	0.22272	-0.5	0.80437
S8	S	0.26658	-0.5	0.83055
N9	N	0.29114	-0.5	0.78824
C10	C	0.31808	-0.5	0.70478
C11	C	0.15379	-0.5	0.74379
C12	C	0.354	-0.5	0.73109
C13	C	0.39378	-0.5	0.71933
C14	C	0.39916	-0.5	0.68059
C15	C	0.36322	-0.5	0.6541
C16	C	0.32338	-0.5	0.66593
C17	C	0.12008	-0.5	0.71443
C18	C	0.07878	-0.5	0.72217
C19	C	0.06953	-0.5	0.7598
C20	C	0.10304	-0.5	0.78918

C21	C	0.14425	-0.5	0.78133
C22	C	0.02857	-0.5	0.76896
C23	C	0.77432	-0.5	0.80595
C24	C	0.81439	-0.5	0.79747
C25	C	0.8242	-0.5	0.75808
C26	C	0.79074	-0.5	0.72596
C27	C	0.75036	-0.5	0.73214
C28	C	0.74286	-0.5	0.7719
C29	C	0.79608	-0.5	0.68734
C30	C	0.76443	-0.5	0.65473
C31	C	0.72534	-0.5	0.65874
C32	C	0.71767	-0.5	0.69863
C33	C	0.6772	-0.5	0.70475
C34	C	0.67187	-0.5	0.74343
C35	C	0.70371	-0.5	0.77594
C36	C	0.69427	-0.5	0.62439
C37	C	0.65388	-0.5	0.63281
C38	C	0.64382	-0.5	0.67249
C39	C	0.76837	-0.5	0.84801
C40	C	0.86688	-0.5	0.75325
C41	C	0.60108	-0.5	0.67775
C42	C	0.70166	-0.5	0.58233

C43	C	0.89384	-0.5	0.7831
C44	C	0.93677	-0.5	0.78066
C45	C	0.95432	-0.5	0.74747
C46	C	0.9277	-0.5	0.7165
C47	C	0.88498	-0.5	0.71971
C48	C	0.73717	-0.5	0.56814
C49	C	0.74405	-0.5	0.5265
C50	C	0.715	-0.5	0.49673
C51	C	0.6797	-0.5	0.51032
C52	C	0.67342	-0.5	0.55234
C53	C	0.73422	-0.5	0.86564
C54	C	0.7303	-0.5	0.90797
C55	C	0.76104	-0.5	0.93461
C56	C	0.79466	-0.5	0.91731
C57	C	0.79789	-0.5	0.87488
C58	C	0.57295	-0.5	0.64835
C59	C	0.53014	-0.5	0.65194
C60	C	0.51405	-0.5	0.68583
C61	C	0.54173	-0.5	0.71613
C62	C	0.58422	-0.5	0.71178
N63	N	0.7566	-0.5	0.97543
N64	N	0.47347	-0.5	0.69165

N65	N	0.7184	-0.5	0.45565
N66	N	0.99539	-0.5	0.7434
C67	C	0.43877	-0.5	0.66799
C68	C	0.75069	-0.5	0.43534
C69	C	0.78227	-0.5	0.00799
C70	C	0.7117	-0.5	0.32258
C71	C	0.71258	-0.5	0.36567
C72	C	0.74869	-0.5	0.39206
C73	C	0.78393	-0.5	0.37387
C74	C	0.78299	-0.5	0.33092
C75	C	0.79776	-0.5	0.1228
C76	C	0.80426	-0.5	0.08136
C77	C	0.77404	-0.5	0.04912
C78	C	0.73685	-0.5	0.05985
C79	C	0.73039	-0.5	0.10143
C80	C	0.74682	-0.5	0.3044
C81	C	0.76075	-0.5	0.1337
N82	N	0.68374	-0.5	0.17185
S83	S	0.64595	-0.5	0.202
N84	N	0.67688	-0.5	0.24325
C85	C	0.75498	-0.5	0.17567
C86	C	0.71802	-0.5	0.19221

C87	C	0.71391	-0.5	0.23411
C88	C	0.74705	-0.5	0.26137
N89	N	0.78203	-0.5	0.24452
C90	C	0.78702	-0.5	0.20402
H91	H	0.18785	-0.5	0.67322
H92	H	0.35165	-0.5	0.75906
H93	H	0.41879	-0.5	0.73951
H94	H	0.36565	-0.5	0.62578
H95	H	0.2982	-0.5	0.6459
H96	H	0.12518	-0.5	0.68701
H97	H	0.05556	-0.5	0.69996
H98	H	0.09784	-0.5	0.81681
H99	H	0.16721	-0.5	0.8036
H100	H	0.02395	-0.5	0.79618
H101	H	0.83772	-0.5	0.82125
H102	H	0.82327	-0.5	0.68201
H103	H	0.77078	-0.5	0.62764
H104	H	0.64447	-0.5	0.74882
H105	H	0.69752	-0.5	0.80276
H106	H	0.63073	-0.5	0.60886
H107	H	0.88256	-0.5	0.80751
H108	H	0.95483	-0.5	0.80341

H109	H	0.93939	-0.5	0.69172
H110	H	0.86737	-0.5	0.69738
H111	H	0.75864	-0.5	0.5881
H112	H	0.77017	-0.5	0.5184
H113	H	0.65819	-0.5	0.48961
H114	H	0.64764	-0.5	0.56073
H115	H	0.71203	-0.5	0.84797
H116	H	0.70519	-0.5	0.91939
H117	H	0.81727	-0.5	0.93516
H118	H	0.82267	-0.5	0.8639
H119	H	0.58331	-0.5	0.62359
H120	H	0.51105	-0.5	0.62966
H121	H	0.53104	-0.5	0.74132
H122	H	0.60268	-0.5	0.73382
H123	H	0.44124	-0.5	0.64018
H124	H	0.7779	-0.5	0.45209
H125	H	0.80929	-0.5	0.00275
H126	H	0.68508	-0.5	0.30466
H127	H	0.68648	-0.5	0.37751
H128	H	0.81079	-0.5	0.39175
H129	H	0.80918	-0.5	0.31927
H130	H	0.82054	-0.5	0.145

H131	H	0.83156	-0.5	0.07506
H132	H	0.71412	-0.5	0.03752
H133	H	0.70337	-0.5	0.10786
H134	H	0.8146	-0.5	0.19474

Table S2. Unit cell parameters and fractional atomic coordinates for HIAM-0202.

Space group: *pm* a = 33.6751 Å, b = 3.9428 Å, and c = 33.2775 Å, α = γ = 90°, and β = 96.6440°				
C1	C	0.24314	-0.5	0.66433
C2	C	0.20186	-0.5	0.66669
C3	C	0.18534	-0.5	0.70393
C4	C	0.21404	-0.5	0.73861
C5	C	0.25539	-0.5	0.7363
C6	C	0.27189	-0.5	0.69894
N7	N	0.20617	-0.5	0.77708
S8	S	0.24666	-0.5	0.81147
N9	N	0.27525	-0.5	0.77343
C10	C	0.3134	-0.5	0.69375
C11	C	0.14324	-0.5	0.70601
C12	C	0.346	-0.5	0.72468
C13	C	0.38565	-0.5	0.71517
C14	C	0.39413	-0.5	0.67466
C15	C	0.36113	-0.5	0.64469
N16	N	0.32365	-0.5	0.65536
C17	C	0.11434	-0.5	0.67158
C18	C	0.07348	-0.5	0.67556
C19	C	0.05969	-0.5	0.71386
C20	C	0.08875	-0.5	0.74774

N21	N	0.12819	-0.5	0.74237
C22	C	0.01597	-0.5	0.71538
C23	C	0.77175	-0.5	0.81344
C24	C	0.80994	-0.5	0.79898
C25	C	0.82238	-0.5	0.75973
C26	C	0.78764	-0.5	0.72961
C27	C	0.74652	-0.5	0.73783
C28	C	0.74016	-0.5	0.77983
C29	C	0.79091	-0.5	0.68844
C30	C	0.75971	-0.5	0.65799
C31	C	0.71928	-0.5	0.6629
C32	C	0.71278	-0.5	0.7049
C33	C	0.67166	-0.5	0.71298
C34	C	0.66824	-0.5	0.75429
C35	C	0.69961	-0.5	0.7849
C36	C	0.68827	-0.5	0.62885
C37	C	0.65008	-0.5	0.64298
C38	C	0.63733	-0.5	0.68226
C39	C	0.77083	-0.5	0.85794
C40	C	0.86614	-0.5	0.75649
C41	C	0.59353	-0.5	0.68492
C42	C	0.68976	-0.5	0.58437

C43	C	0.89706	-0.5	0.79056
C44	C	0.9389	-0.5	0.78877
C45	C	0.95491	-0.5	0.75183
C46	C	0.92666	-0.5	0.71756
C47	C	0.8855	-0.5	0.72042
C48	C	0.72454	-0.5	0.56336
C49	C	0.7258	-0.5	0.52152
C50	C	0.69095	-0.5	0.49422
C51	C	0.65516	-0.5	0.51185
C52	C	0.65501	-0.5	0.55433
C53	C	0.73653	-0.5	0.87989
C54	C	0.73665	-0.5	0.92209
C55	C	0.77236	-0.5	0.94767
C56	C	0.80765	-0.5	0.92928
C57	C	0.80636	-0.5	0.8869
C58	C	0.56361	-0.5	0.65004
C59	C	0.52182	-0.5	0.6504
C60	C	0.5051	-0.5	0.68689
C61	C	0.53169	-0.5	0.7221
C62	C	0.57325	-0.5	0.72056
N63	N	0.76972	-0.5	0.98785
N64	N	0.46565	-0.5	0.69118

N65	N	0.69068	-0.5	0.45323
N66	N	0.99529	-0.5	0.75067
C67	C	0.43314	-0.5	0.66347
C68	C	0.72278	-0.5	0.43202
C69	C	0.79837	-0.5	0.01965
C70	C	0.69091	-0.5	0.31967
C71	C	0.68784	-0.5	0.36158
C72	C	0.72242	-0.5	0.3898
C73	C	0.75947	-0.5	0.37402
N74	N	0.76076	-0.5	0.33387
C75	C	0.81144	-0.5	0.13216
C76	C	0.81996	-0.5	0.09195
C77	C	0.78904	-0.5	0.0597
C78	C	0.74955	-0.5	0.06992
N79	N	0.74275	-0.5	0.10919
C80	C	0.72884	-0.5	0.30494
C81	C	0.77139	-0.5	0.14155
N82	N	0.68706	-0.5	0.16254
S83	S	0.6433	-0.5	0.18206
N84	N	0.66885	-0.5	0.22836
C85	C	0.75997	-0.5	0.18145
C86	C	0.71954	-0.5	0.18983

C87	C	0.70866	-0.5	0.22921
C88	C	0.73729	-0.5	0.26393
C89	C	0.77767	-0.5	0.25542
C90	C	0.78854	-0.5	0.21609
H91	H	0.25164	-0.5	0.6359
H92	H	0.18443	-0.5	0.63972
H93	H	0.34211	-0.5	0.75438
H94	H	0.40833	-0.5	0.73833
H95	H	0.36468	-0.5	0.61469
H96	H	0.12173	-0.5	0.64289
H97	H	0.0539	-0.5	0.64968
H98	H	0.0808	-0.5	0.77634
H99	H	-0.00177	-0.5	0.68602
H100	H	0.83213	-0.5	0.81999
H101	H	0.81626	-0.5	0.67736
H102	H	0.76965	-0.5	0.63187
H103	H	0.64257	-0.5	0.76519
H104	H	0.68988	-0.5	0.81135
H105	H	0.62852	-0.5	0.6213
H106	H	0.89126	-0.5	0.81963
H107	H	0.95792	-0.5	0.81501
H108	H	0.93539	-0.5	0.6893

H109	H	0.87213	-0.5	0.69269
H110	H	0.75237	-0.5	0.57584
H111	H	0.75332	-0.5	0.51161
H112	H	0.6286	-0.5	0.49356
H113	H	0.6266	-0.5	0.56133
H114	H	0.70845	-0.5	0.86776
H115	H	0.71005	-0.5	0.93427
H116	H	0.83475	-0.5	0.94652
H117	H	0.83454	-0.5	0.87939
H118	H	0.56991	-0.5	0.62118
H119	H	0.50376	-0.5	0.62361
H120	H	0.52074	-0.5	0.74954
H121	H	0.5863	-0.5	0.74855
H122	H	0.43688	-0.5	0.63351
H123	H	0.75046	-0.5	0.44785
H124	H	0.8277	-0.5	0.01459
H125	H	0.66501	-0.5	0.3008
H126	H	0.66017	-0.5	0.37131
H127	H	0.78603	-0.5	0.39221
H128	H	0.83531	-0.5	0.15379
H129	H	0.84924	-0.5	0.08652
H130	H	0.72552	-0.5	0.04828

H131	H	0.80082	-0.5	0.27809
H132	H	0.8184	-0.5	0.21393

Table S3. Unit cell parameters and fractional atomic coordinates for HIAM-0203.

Space group: *pm* a = 32.8364 Å, b = 3.9874 Å, and c = 33.4562 Å, α = γ = 90°, and β = 96.0301°				
C1	C	0.22863	0.5	0.65622
C2	C	0.18675	0.5	0.66266
C3	C	0.1705	0.5	0.69928
C4	C	0.19913	0.5	0.72957
C5	C	0.24087	0.5	0.72337
C6	C	0.25681	0.5	0.68641
N7	N	0.19119	0.5	0.76638
S8	S	0.23285	0.5	0.79446
N9	N	0.26187	0.5	0.75568
C10	C	0.29855	0.5	0.679
C11	C	0.12765	0.5	0.7041
C12	C	0.33281	0.5	0.70818
N13	N	0.3724	0.5	0.70027
C14	C	0.38193	0.5	0.66474
C15	C	0.34903	0.5	0.63437
C16	C	0.30777	0.5	0.64158
C17	C	0.09654	0.5	0.67099
N18	N	0.05565	0.5	0.67533
C19	C	0.0414	0.5	0.71092
C20	C	0.07069	0.5	0.74525

C21	C	0.11344	0.5	0.7415
C22	C	-0.00184	0.5	0.71134
C23	C	0.75488	0.5	0.81066
C24	C	0.79419	0.5	0.80191
C25	C	0.80765	0.5	0.76232
C26	C	0.77691	0.5	0.72881
C27	C	0.73527	0.5	0.73577
C28	C	0.7253	0.5	0.7769
C29	C	0.78597	0.5	0.68765
C30	C	0.75523	0.5	0.6549
C31	C	0.71376	0.5	0.66066
C32	C	0.70393	0.5	0.70191
C33	C	0.66258	0.5	0.70906
C34	C	0.65578	0.5	0.74962
C35	C	0.68593	0.5	0.78255
C36	C	0.68272	0.5	0.62683
C37	C	0.64151	0.5	0.63597
C38	C	0.62988	0.5	0.67615
C39	C	0.74882	0.5	0.85288
C40	C	0.85101	0.5	0.75978
C41	C	0.58633	0.5	0.68047
C42	C	0.68998	0.5	0.58462

C43	C	0.88197	0.5	0.78831
C44	C	0.92485	0.5	0.78465
C45	C	0.93857	0.5	0.75105
C46	C	0.90872	0.5	0.72382
C47	C	0.86606	0.5	0.72924
C48	C	0.72358	0.5	0.57083
C49	C	0.73126	0.5	0.5295
C50	C	0.7042	0.5	0.4995
C51	C	0.66967	0.5	0.5124
C52	C	0.66309	0.5	0.55414
C53	C	0.71401	0.5	0.87012
C54	C	0.71003	0.5	0.91236
C55	C	0.74102	0.5	0.93908
C56	C	0.77569	0.5	0.92208
C57	C	0.77931	0.5	0.87994
C58	C	0.55916	0.5	0.65192
C59	C	0.51567	0.5	0.65279
C60	C	0.49738	0.5	0.68356
C61	C	0.52399	0.5	0.71381
C62	C	0.56736	0.5	0.71165
N63	N	0.7358	0.5	0.97963
N64	N	0.45571	0.5	0.68556

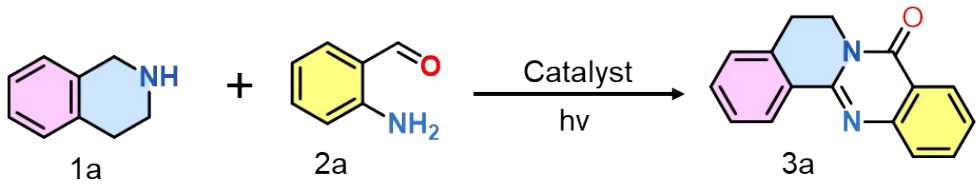
N65	N	0.70902	0.5	0.4589
N66	N	0.97981	0.5	0.74652
C67	C	0.42347	0.5	0.65818
C68	C	0.74051	0.5	0.43893
C69	C	0.76105	0.5	0.01145
C70	C	0.70493	0.5	0.33126
N71	N	0.70694	0.5	0.3727
C72	C	0.74013	0.5	0.39623
C73	C	0.77433	0.5	0.37667
C74	C	0.77324	0.5	0.3339
C75	C	0.77877	0.5	0.122
N76	N	0.78217	0.5	0.08127
C77	C	0.75244	0.5	0.05238
C78	C	0.71564	0.5	0.06541
C79	C	0.71107	0.5	0.10763
C80	C	0.73833	0.5	0.31009
C81	C	0.74287	0.5	0.13696
N82	N	0.66989	0.5	0.1779
S83	S	0.63494	0.5	0.20954
N84	N	0.66804	0.5	0.25019
C85	C	0.73988	0.5	0.17959
C86	C	0.70477	0.5	0.19751

C87	C	0.70369	0.5	0.23997
C88	C	0.73762	0.5	0.26704
C89	C	0.77223	0.5	0.24921
C90	C	0.77333	0.5	0.20646
H91	H	0.23833	0.5	0.62895
H92	H	0.16816	0.5	0.63993
H93	H	0.32853	0.5	0.73554
H94	H	0.35467	0.5	0.6071
H95	H	0.28444	0.5	0.61911
H96	H	0.10401	0.5	0.64313
H97	H	0.06138	0.5	0.77267
H98	H	0.13407	0.5	0.76658
H99	H	-0.01996	0.5	0.68388
H100	H	0.81455	0.5	0.8259
H101	H	0.81534	0.5	0.67978
H102	H	0.76454	0.5	0.62654
H103	H	0.62845	0.5	0.75613
H104	H	0.67813	0.5	0.81093
H105	H	0.61879	0.5	0.61188
H106	H	0.87373	0.5	0.81189
H107	H	0.9459	0.5	0.80584
H108	H	0.91776	0.5	0.70011

H109	H	0.84595	0.5	0.70999
H110	H	0.74306	0.5	0.5909
H111	H	0.75648	0.5	0.52178
H112	H	0.64924	0.5	0.49144
H113	H	0.63829	0.5	0.56204
H114	H	0.6912	0.5	0.85211
H115	H	0.68439	0.5	0.92361
H116	H	0.79888	0.5	0.93988
H117	H	0.80502	0.5	0.86925
H118	H	0.57069	0.5	0.62959
H119	H	0.49778	0.5	0.63076
H120	H	0.51182	0.5	0.73701
H121	H	0.58477	0.5	0.7328
H122	H	0.42921	0.5	0.63152
H123	H	0.76562	0.5	0.45517
H124	H	0.78821	0.5	0.00603
H125	H	0.67896	0.5	0.31593
H126	H	0.79997	0.5	0.39308
H127	H	0.79826	0.5	0.3203
H128	H	0.80275	0.5	0.14139
H129	H	0.69239	0.5	0.04474
H130	H	0.68447	0.5	0.11675

H131	H	0.79729	0.5	0.26727
H132	H	0.79914	0.5	0.19513

Table S4. Controlled experiments for photocatalytic synthesis of quinazolinone.



1a 2a 3a

Entry	Photocatalyst	Light	Atmosphere	Conv. (%)	Sel. (%)
1	HIAM-0201	White LED	Air	99	85
2	PYTA	White LED	Air	80	15
3	TPBA	White LED	Air	99	43
4	HIAM-0201	Dark	Air	80	/
5	-	White LED	Air	99	4
6	HIAM-0201	White LED	Nitrogen	82	4

Reaction condition: photocatalyst (5 mg), 1,2,3,4-tetrahydroisoquinoline 1a (10 μL, 0.077 mmol), 2-aminobenzaldehyde 2a (0.083 mmol), CH_3CN (2 mL), air, 9h, 40 W white LED, at room temperature.

Table S5. The performance comparison of covalent organic frameworks in photocatalytic cross-dehydrogenative reaction.

Type	Substrate	Catalyst	Solvent	Atmosphere	Light source	Time (h)	Yield (%)	Ref.
C-C bond	Tertiary aniline and maleimide	NKCOF-25-Ni	DMF	Air, rt	23 W fluorescent lamp	15	97	[6]
		H_2P-Bph-COF	$CHCl_3$	O_2, rt	Blue LEDs (λ= 450 nm)	14	72	[7]
		HIAM-0030	DMF	Air, rt	40 W white LED	18	99	[8]
	N-aryltetrahydroisoquinoline and CH_3NO_2	TFB-COF	-	O_2, rt	45W lamp	36	87	[9]
		TBPA-sp^2C-COF	MeOH	Air, rt	23W white LED	24	99	[10]
	N-phenyltetrahydroisoquinonline and CH_3NO_2	COF-JLU5	MeOH	O_2, rt	30 W blue LEDs with 460 nm	6	99	[11]
		COF-1	CH_3CN	Air, 40°C	440 nm LED	40	85	[12]
	2-phenyl-tetrahydroisoquinoline and CH_3NO_2	TTCOF	-	Air, rt	Blue LEDs	18	89	[13]
C-N bond	4-bromobenzotrifluoride and pyrrolidine	Tp-Acr COF	DMAc	-	440 nm LEDs	16	91	[14]
	1, 2, 3, 4-Tetrahydroisoquinoline and 2-2minobenzaldehyde	TAPP-Cu-An	CH_3CN	O_2, rt	Full light	6	99	[15]
		HIAM-0201	CH_3CN	Air, rt	40W white LED	24	99	This work

Table S6. The Hirshfeld charge of HIAM-0201, HIAM-0202, HIAM-0203.

HIAM-0201			HIAM-0202			HIAM-0203		
1	C	-0.000577	1	C	-0.000424	1	C	-0.000398
2	C	-0.004504	2	C	-0.004451	2	C	-0.004512
3	C	0.00137	3	C	0.001316	3	C	0.001334
4	C	-0.004414	4	C	-0.004399	4	C	-0.004374
5	C	-0.000442	5	C	-0.00033	5	C	-0.000412
6	C	-0.04147	6	C	-0.04129	6	C	-0.041335
7	C	-0.04154	7	C	-0.041314	7	C	-0.041259
8	C	-0.041538	8	C	-0.041541	8	C	-0.041367
9	C	-0.004592	9	C	-0.004505	9	C	-0.004436
10	C	0.001173	10	C	0.001284	10	C	0.001386
11	C	-0.004455	11	C	-0.004426	11	C	-0.004419
12	C	-0.041131	12	C	-0.041331	12	C	-0.041402
13	C	-0.041276	13	C	-0.041143	13	C	-0.041213
14	C	-0.000735	14	C	-0.00061	14	C	-0.000549
15	C	-0.041822	15	C	-0.04181	15	C	-0.04138
16	C	-0.000751	16	C	-0.00103	16	C	-0.001049
17	C	0.003517	17	C	0.003352	17	C	0.003459
18	C	0.003449	18	C	0.003379	18	C	0.003411
19	C	0.003443	19	C	0.003336	19	C	0.00342
20	C	-0.000575	20	C	-0.000017	20	C	0.001273
21	C	-0.034843	21	C	-0.03482	21	C	-0.034896
22	C	-0.038731	22	C	-0.038685	22	C	-0.03867
23	C	-0.040394	23	C	-0.040349	23	C	-0.040181
24	C	-0.038786	24	C	-0.038703	24	C	-0.038543
25	C	-0.037744	25	C	-0.03778	25	C	-0.037642
26	C	-0.033573	26	C	-0.033111	26	C	-0.033232
27	C	-0.039755	27	C	-0.0396	27	C	-0.038636
28	C	0.041604	28	C	0.040949	28	C	0.041311
29	C	-0.046758	29	C	-0.046811	29	C	-0.043951
30	C	-0.037609	30	C	-0.037269	30	C	-0.036509
31	C	-0.034729	31	C	-0.034818	31	C	-0.034806
32	C	-0.038585	32	C	-0.03849	32	C	-0.038525
33	C	-0.040212	33	C	-0.040038	33	C	-0.040207
34	C	-0.038505	34	C	-0.03853	34	C	-0.038496
35	C	-0.037706	35	C	-0.037605	35	C	-0.037583
36	C	-0.034784	36	C	-0.034736	36	C	-0.034819
37	C	-0.038745	37	C	-0.038671	37	C	-0.038546
38	C	-0.040287	38	C	-0.040252	38	C	-0.040234

39	C	-0.038686	39	C	-0.038536	39	C	-0.038539
40	C	-0.037666	40	C	-0.03766	40	C	-0.037642
41	N	-0.162172	41	N	-0.164254	41	N	-0.160193
42	C	0.061042	42	C	0.05931	42	C	0.061204
43	C	-0.003527	43	C	-0.013152	43	C	0.055958
44	C	-0.032696	44	C	0.037425	44	C	0.033164
45	C	-0.029353	45	C	0.068493	45	C	-0.000052
46	C	-0.001978	46	C	-0.036658	46	C	-0.014229
47	C	-0.033082	47	C	-0.005446	47	C	-0.025703
48	C	-0.025027	48	C	-0.004469	48	C	0.00083
49	C	0.075028	49	C	-0.022388	49	C	-0.029486
50	C	0.026488	50	C	-0.029898	50	C	-0.031805
51	C	-0.003582	51	C	0.005365	51	C	0.003077
52	C	0.050492	52	C	0.048535	52	C	0.047042
53	C	0.044731	53	C	0.048103	53	C	0.046931
54	C	-0.00171	54	C	0.065332	54	C	-0.004445
55	C	-0.03584	55	C	-0.038772	55	C	-0.019248
56	C	-0.037659	56	C	-0.018719	56	C	-0.040667
57	C	-0.034582	57	C	-0.039057	57	C	0.034678
58	C	-0.035583	58	C	0.036962	58	C	0.038745
59	C	-0.033285	59	N	-0.174388	59	N	-0.173915
60	N	-0.173446	60	S	0.231824	60	S	0.239933
61	S	0.248783	61	N	-0.175026	61	N	-0.176622
62	N	-0.159455	62	H	0.036337	62	H	0.03629
63	H	0.036213	63	H	0.035216	63	H	0.035243
64	H	0.035116	64	H	0.035165	64	H	0.03522
65	H	0.035116	65	H	0.035506	65	H	0.035382
66	H	0.035612	66	H	0.035385	66	H	0.035297
67	H	0.035338	67	H	0.035711	67	H	0.036105
68	H	0.035729	68	H	0.040266	68	H	0.040168
69	H	0.040211	69	H	0.040367	69	H	0.040322
70	H	0.040317	70	H	0.039631	70	H	0.039754
71	H	0.039526	71	H	0.040362	71	H	0.040453
72	H	0.040276	72	H	0.038998	72	H	0.039122
73	H	0.039015	73	H	0.041876	73	H	0.042006
74	H	0.041555	74	H	0.04271	74	H	0.042622
75	H	0.042585	75	H	0.037636	75	H	0.039994
76	H	0.037697	76	H	0.039918	76	H	0.040406
77	H	0.039681	77	H	0.040247	77	H	0.040256
78	H	0.04033	78	H	0.040454	78	H	0.040343
79	H	0.040344	79	H	0.039773	79	H	0.039711
80	H	0.039661	80	H	0.040378	80	H	0.040384
81	H	0.040267	81	H	0.039084	81	H	0.039094

82	H	0.039028	82	H	0.040235	82	H	0.040299
83	H	0.040226	83	H	0.040341	83	H	0.040336
84	H	0.040321	84	H	0.039707	84	H	0.039775
85	H	0.039573	85	H	0.040385	85	H	0.040254
86	H	0.040307	86	H	0.039035	86	H	0.039148
87	H	0.039022	87	H	0.035973	87	H	0.039492
88	H	0.034746	88	H	0.042069	88	H	0.041568
89	H	0.040371	89	H	0.035071	89	H	0.043372
90	H	0.037714	90	H	0.050053	90	H	0.047859
91	H	0.031403	91	H	0.04041	91	H	0.045475
92	H	0.042636	92	H	0.043528	92	H	0.044272
93	H	0.04398	93	H	0.043236	93	H	0.044875
94	H	0.038833	94	H	0.049085	94	H	0.046539
95	H	0.04176	95	H	0.046207	95	H	0.04551
96	H	0.041791	96	H	0.045882	96	H	0.039806
97	H	0.042189	97	N	-0.170587	97	N	-0.182749
98	H	0.035194	98	N	-0.173473	98	N	-0.185383
99	N	-0.164042						

Table S7. The Hirshfeld charge sum of the electron donor unit and the acceptor unit of HIAM-0201, HIAM-0202 and HIAM-0203.

Samples	Donor	Acceptor	Difference
HIAM-0201	-0.2036	0.1323	0.3359
HIAM-0202	-0.1646	0.1308	0.2953
HIAM-0203	-0.1521	0.1160	0.2681

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