

Supplementary Information

Multiple Hydrogen Bonding Enables Large-Area Doped Phosphorescent Glasses with Robust Stability and High-Temperature Afterglow

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Supplementary Methods

Synthesis and characterization of the TFH (Supplementary Fig. 2):

To a DMF solution (120 mL) of hexakis(bromomethyl)benzene (5.0 g, 7.87 mmol) was successively added Dimethyl 2-butyndioate (2.90 mL, 23.6 mmol) and NaI (17.7 g, 118 mmol) at 0 °C, and the mixture was stirred at 85 °C for 36 h. The reaction mixture was allowed to cool to room temperature and evaporated to dryness under reduced pressure to afford a brown solid substance. The residue was poured into water and extracted with CHCl₃. A combined organic extract was washed successively with a saturated aqueous solution of Na₂S₂O₃ and brine, and an organic phase separated was dried over anhydrous MgSO₄ and then evaporated to dryness under a reduced pressure. The residue was subjected to column chromatography (SiO₂, CHCl₃) and recrystallized from MeOH and then a mixture of EtOH/CH₂Cl₂ (1/1 v/v), affording 1 as white solid (0.65 g, 1.12 mmol) in 14% yield.

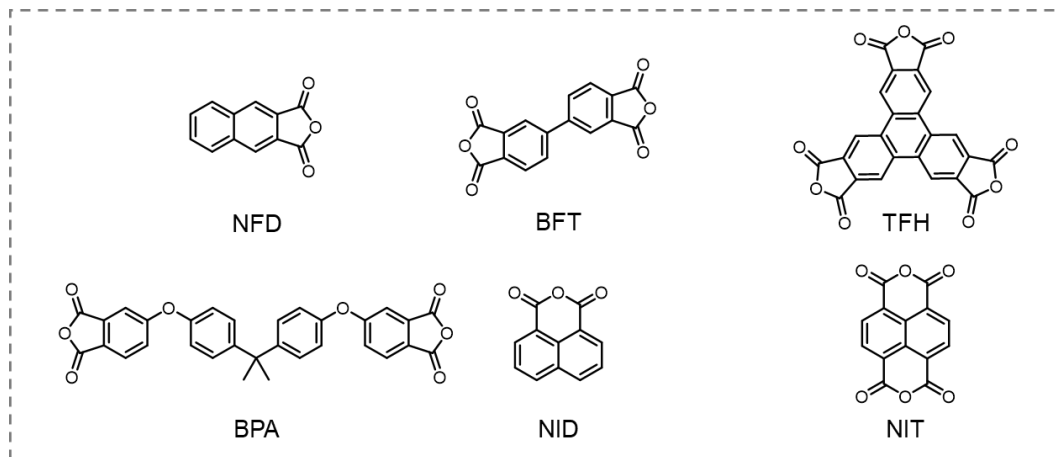
To a toluene solution (50 mL) of 1 (300 mg, 0.51 mmol) was added MnO₂ (300 mg, 3.45 mmol), and the mixture was stirred at 65 °C for 10 h under Ar. The reaction mixture was allowed to cool to room temperature, diluted with CHCl₃, filtered off from an insoluble fraction by passing through a celite pad and then a membrane filter, and washed with CHCl₃. The filtrate was evaporated to dryness under a reduced pressure. The residue was recrystallized from MeOH, affording 2 as white solid (280 mg, 0.49 mmol) in 95% yield.

To a THF solution (37.5 mL) of 2 (700 mg, 1.06 mmol) was added an aqueous solution of KOH (1M, 37.5 mL), and the mixture was stirred at 65 °C for 12 h under Ar. The reaction mixture was poured into acidified water, and white precipitates formed were collected by centrifugation, washed with water, and dried under a reduced pressure, affording 3 as white solid (509 mg, 1.04 mmol) in 98% yield.

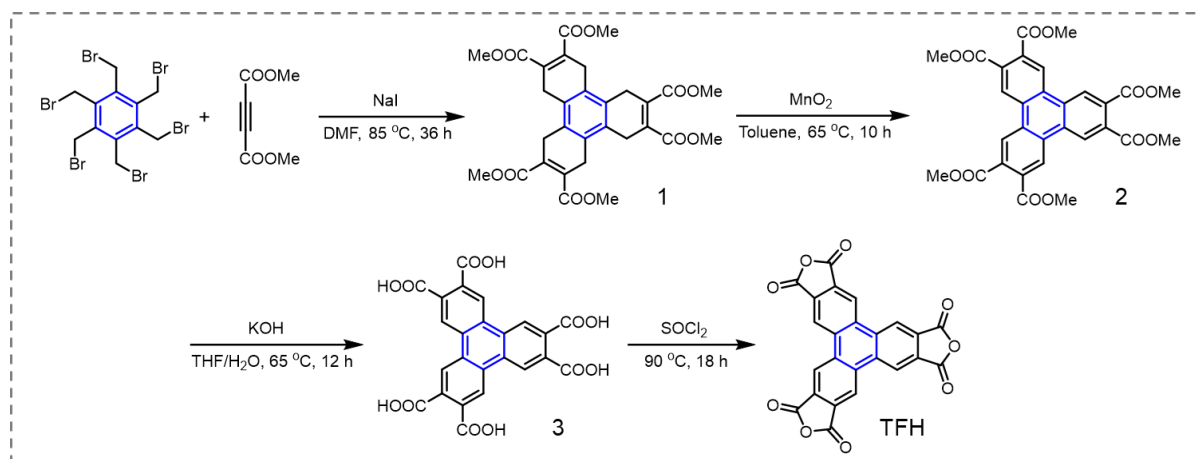
A dried 50 mL two-neck flask was charged with a stir bar, hexacarboxylic acid 3 (279 mg, 0.567 mmol, 1.00 eq.) and thionyl chloride (28 mL). The reaction mixture was stirred under reflux at 90 °C oil bath temperature for 18 h. The solvent was removed under reduced pressure. The solid residue was washed with DCM and acetone via centrifugation and dried under high vacuum overnight. The product TFH (247 mg, 0.564 mmol, 99%) was obtained as white solid and used without further purification.

TFH: white solid. ^1H NMR (500 MHz, $\text{DMSO}-d_6$) δ (ppm) 9.67 (s, 6H). ^{13}C NMR (125 MHz, $\text{DMSO}-d_6$) δ (ppm) 163.4, 135.5, 131.8, 126.6. High-resolution MS (ESI): m/z calcd. for $\text{C}_{24}\text{H}_6\text{O}_9$: 438.00063 $[\text{M}]^+$, found 438.00153.

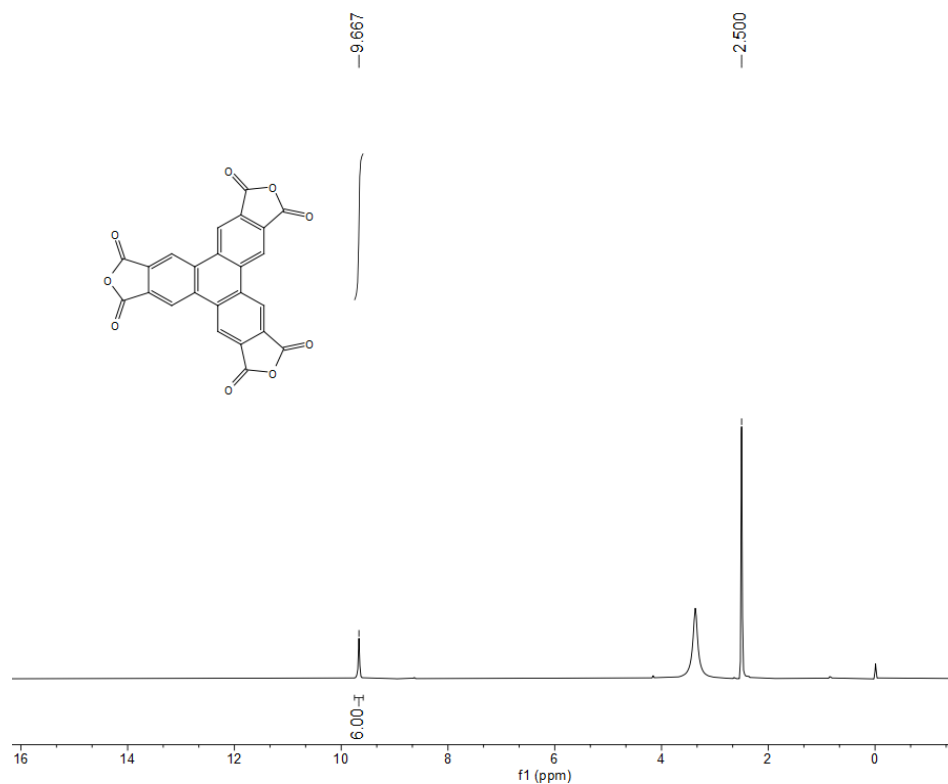
Supplementary Figures



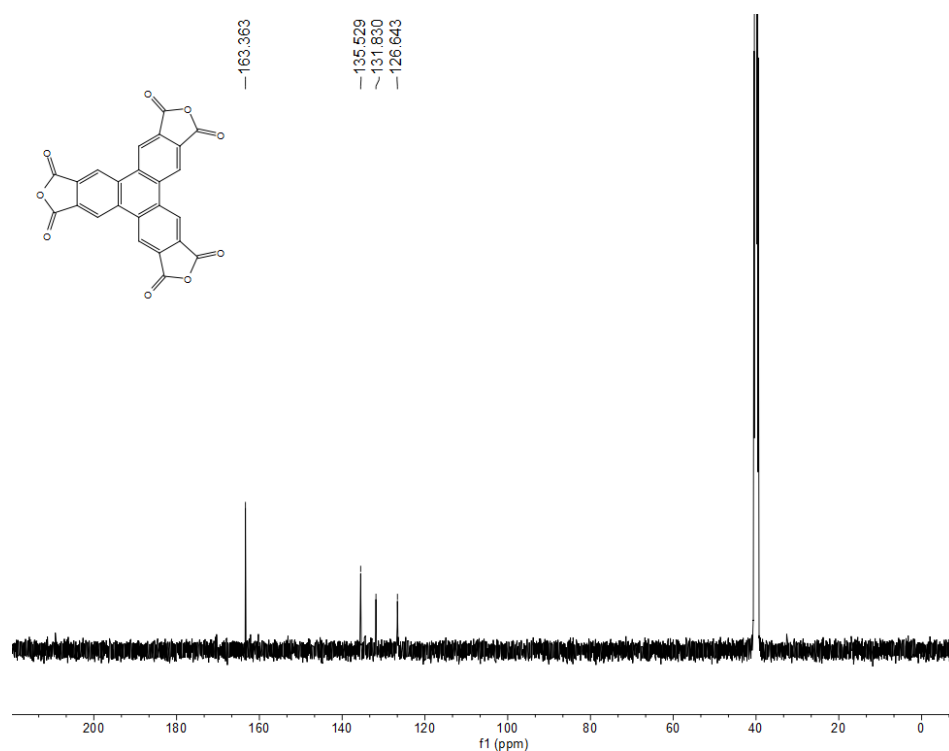
Supplementary Figure 1. Molecular structure of anhydride guests.



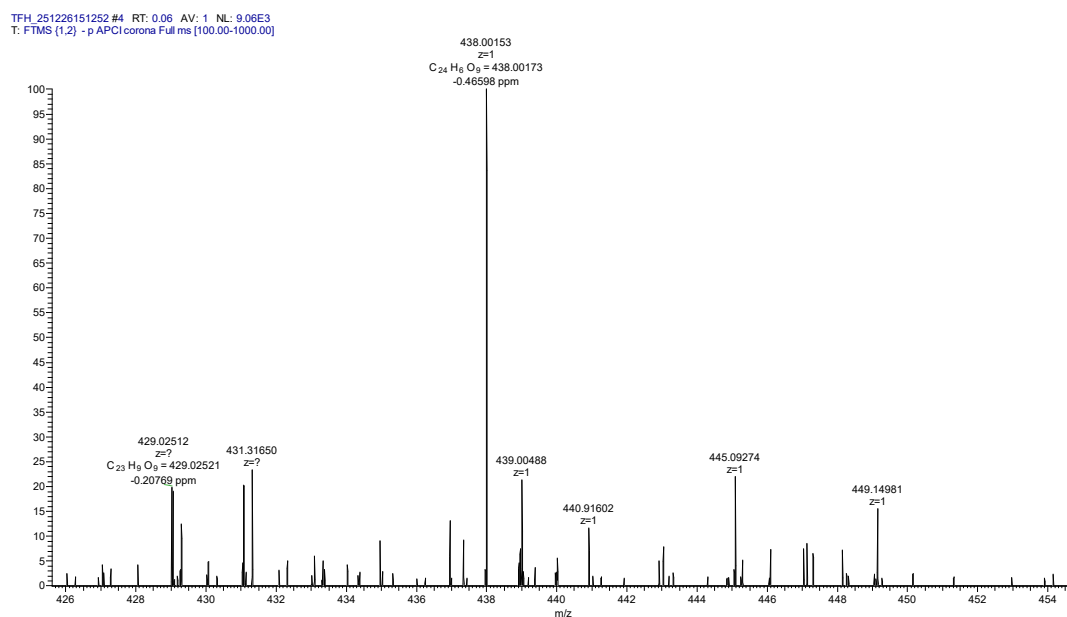
Supplementary Figure 2. Synthetic routes and molecular structure of the TFH guest^[1-3].



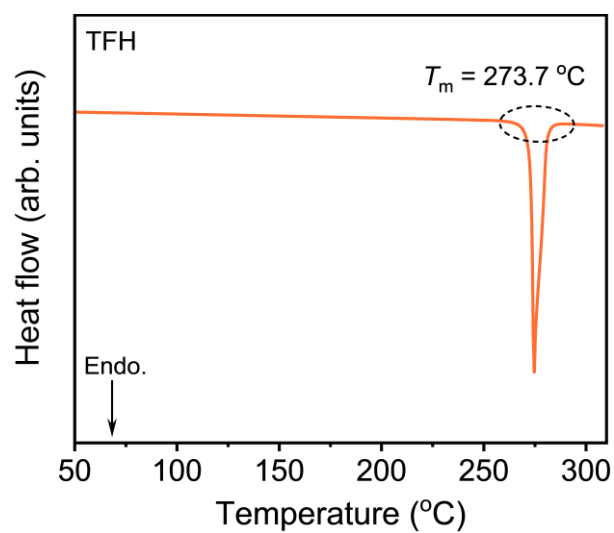
Supplementary Figure 3. The ^1H NMR spectrum of TFH (DMSO- d_6 , 500 MHz).



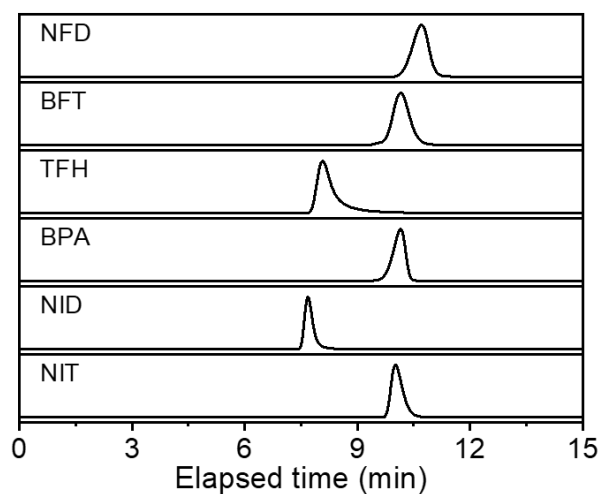
Supplementary Figure 4. The ^{13}C NMR spectrum of TFH (DMSO- d_6 , 125 MHz).



Supplementary Figure 5. The high-resolution MS of TFH.



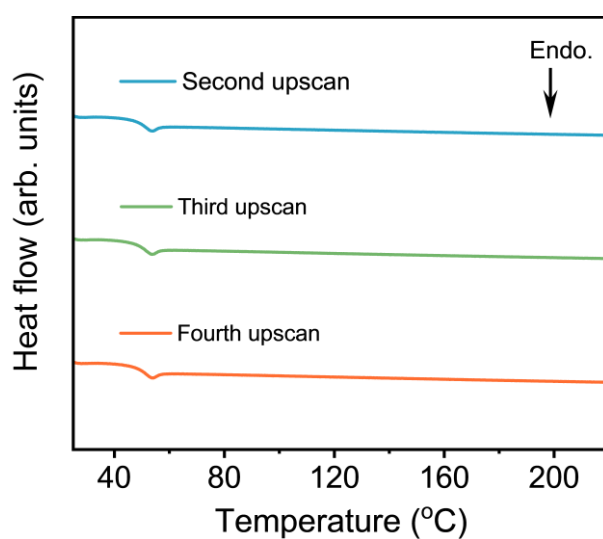
Supplementary Figure 6. DSC trace of TFH powder.



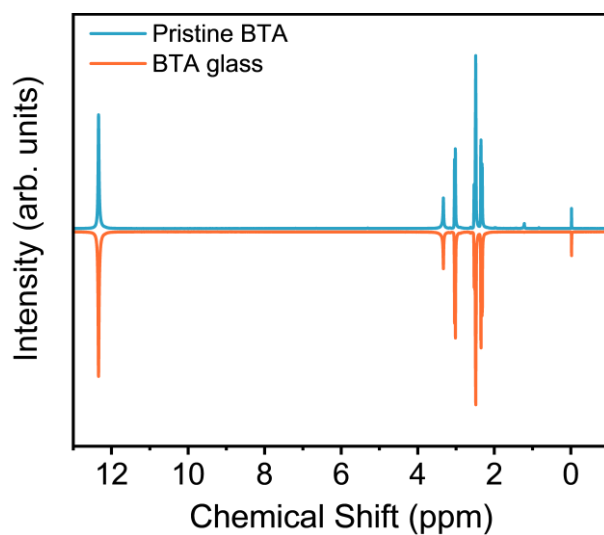
Supplementary Figure 7. High performance liquid chromatography of the all anhydride guests (Conditions: EtOH/H₂O = 70%:30%).



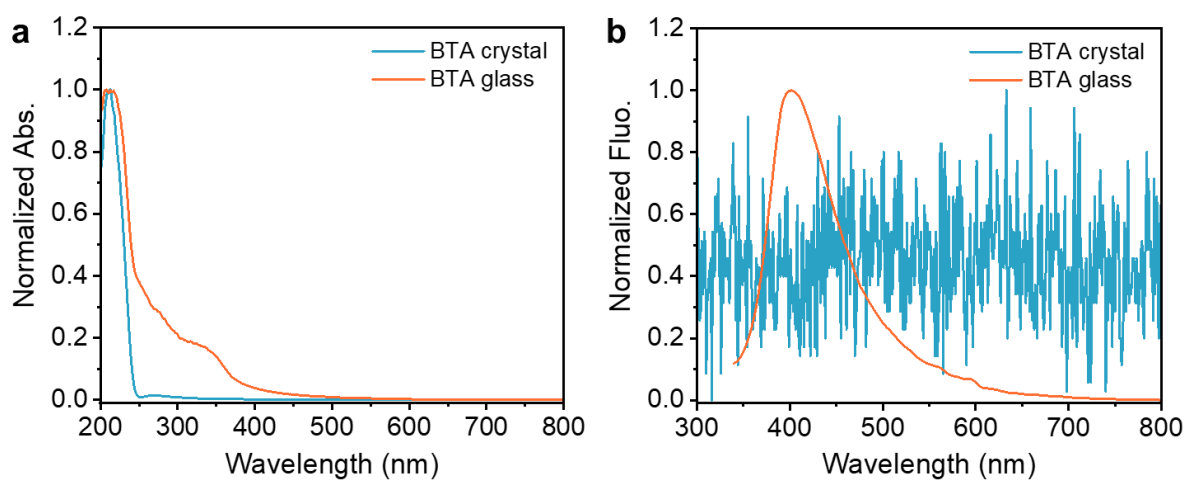
Supplementary Figure 8. The preparation process and state picture of BTA glass and crystal.



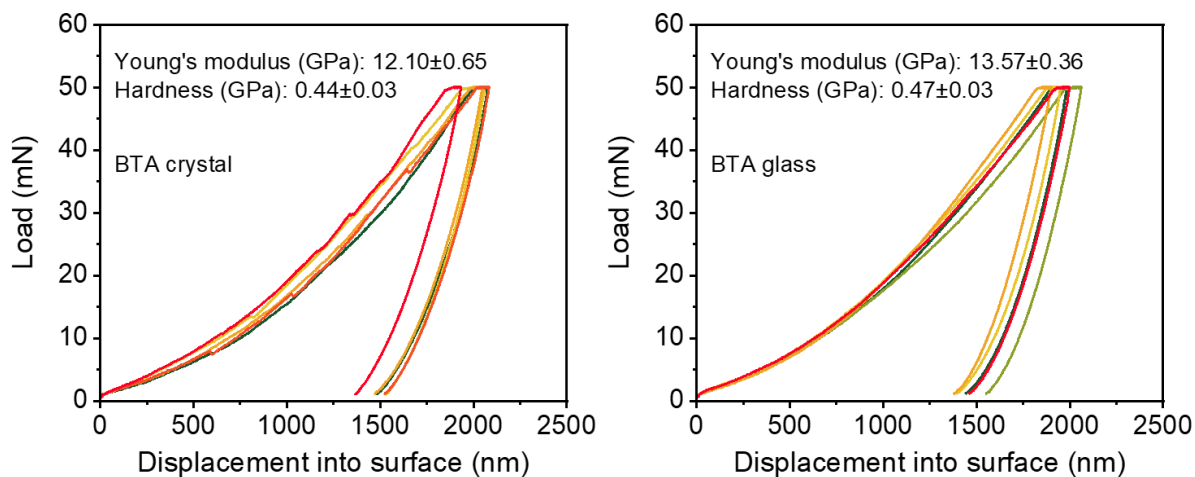
Supplementary Figure 9. DSC curves of different heating cycles of BTA glass.



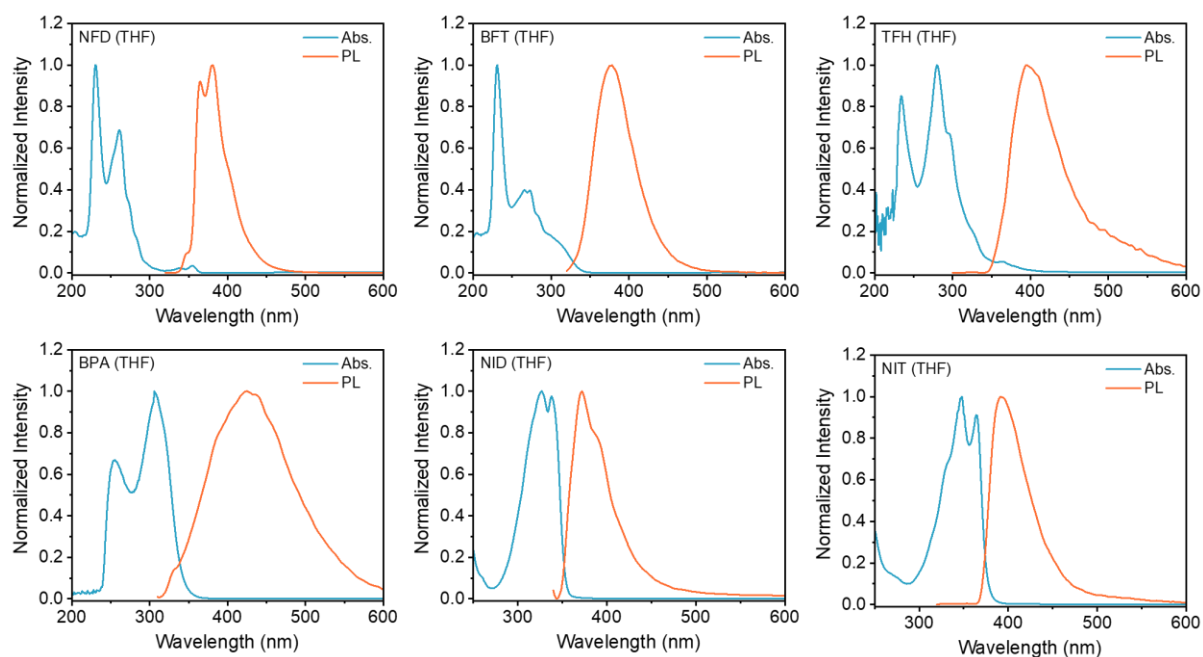
Supplementary Figure 10. ^1H NMR spectra of pristine BTA and BTA glass.



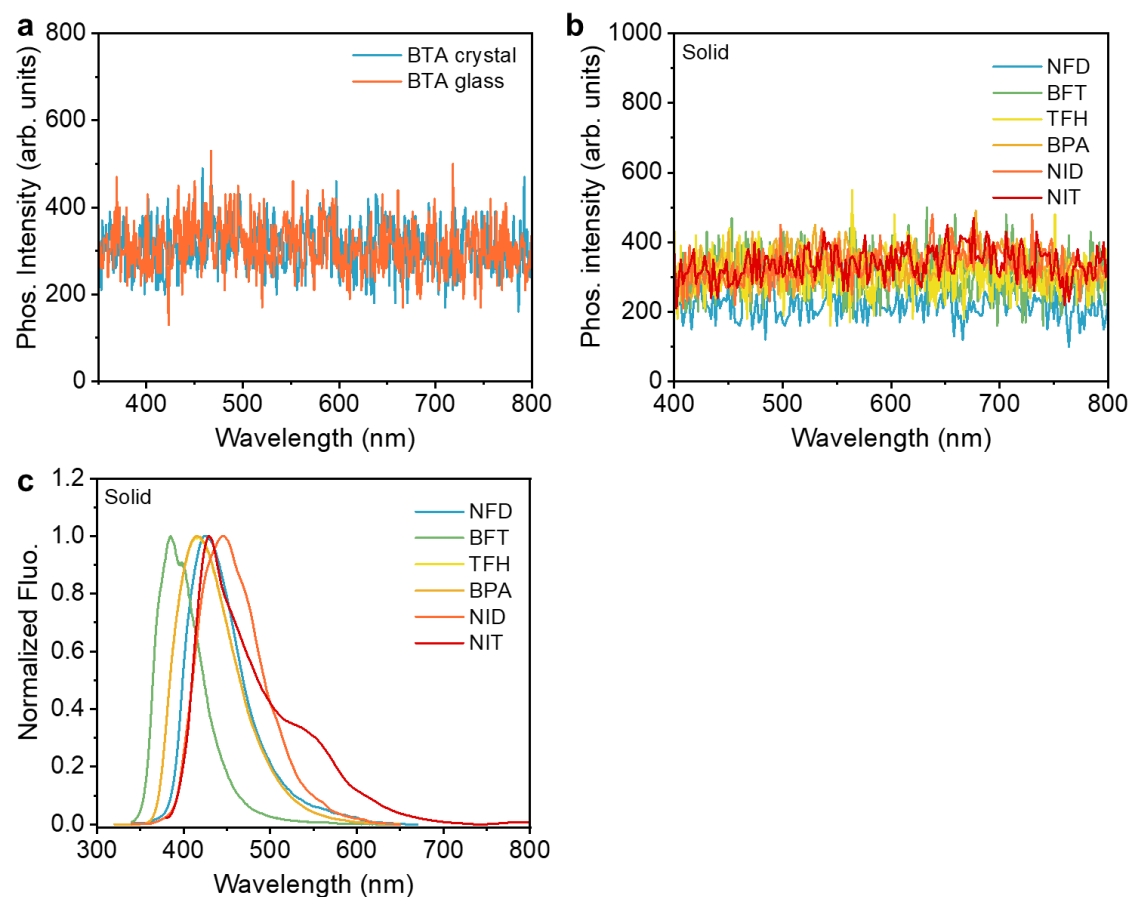
Supplementary Figure 11. (a) Normalized UV-vis absorption (Abs.) and (b) steady-state PL spectra of BTA crystal and glass.



Supplementary Figure 12. Nanoindentation tests of BTA crystal and glass with the load-displacement curves.

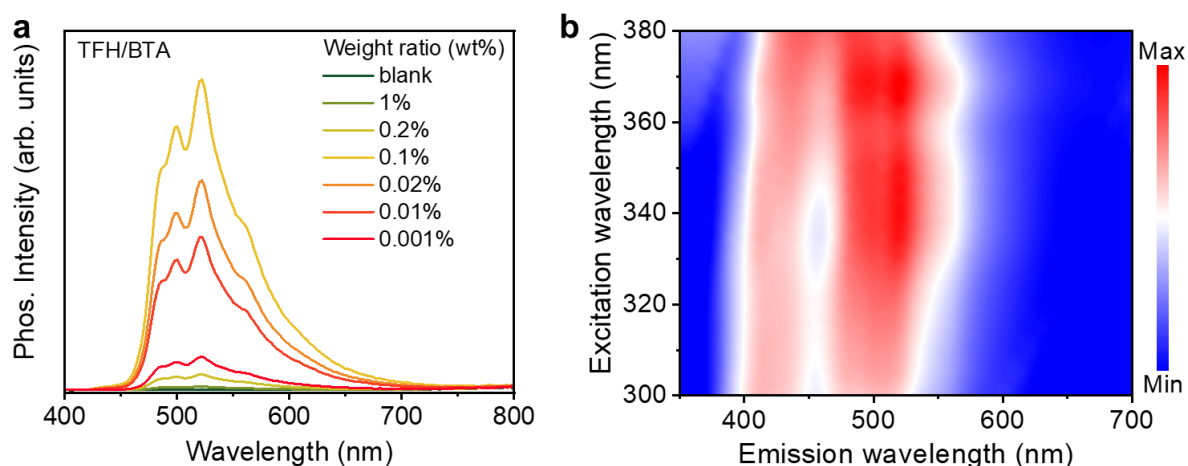


Supplementary Figure 13. Normalized UV-vis absorption (Abs.) and steady-state PL spectra of all the guests in THF solution (1.0×10^{-5} mol/L).



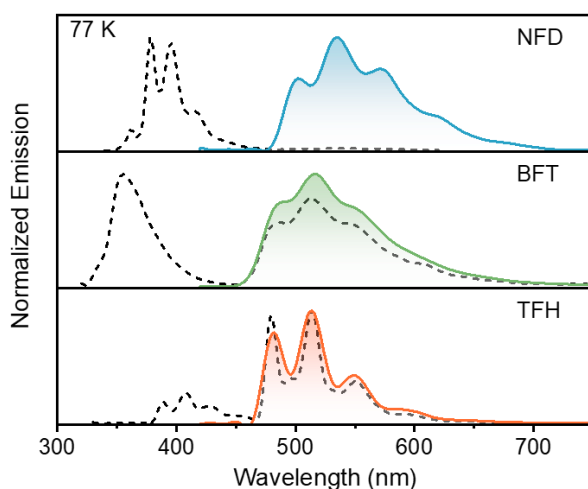
Supplementary Figure 14. Delayed (delayed time: 50 μ s) emission spectra of (a) BTA crystal and glass, (b) all the guests in solid state. (c) Normalized steady-state PL spectra of all the guests in solid state.

Supplementary Note 1. No obvious RTP emission could be observed for host and all guests in the solid state. Therefore, RTP was only achieved via a host-guest doped system.

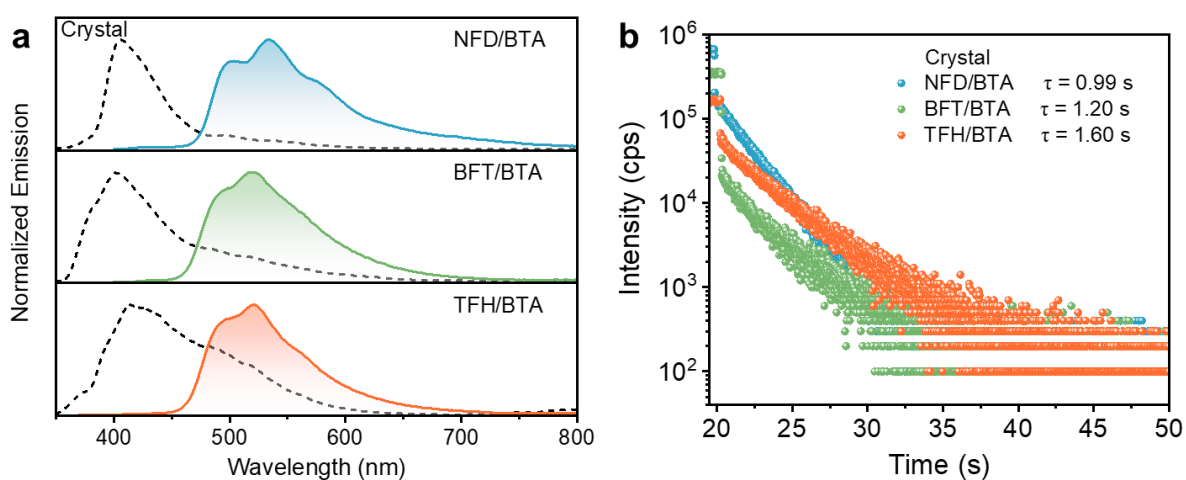


Supplementary Figure 15. (a) Delayed (delayed time: 50 μ s) emission spectra of the TFH/BTA doped SGs at different weight ratios. (b) Steady-state excitation-photoluminescence mapping of the TFH/BTA doped SGs.

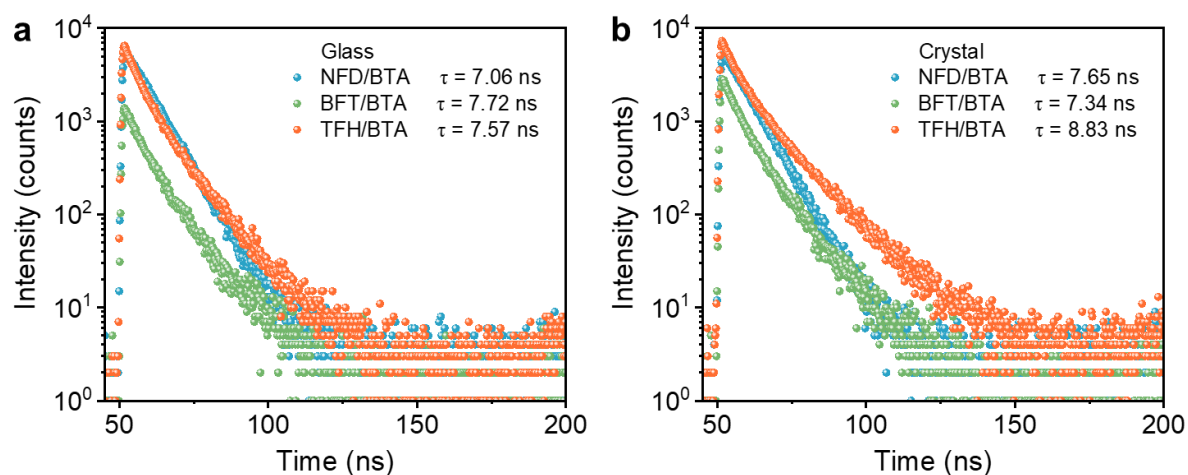
Supplementary Note 2. The long-wavelength phosphorescence emission remained fixed with a dominant peak at 522 nm, even as the excitation wavelengths varied from 300 to 380 nm (Supplementary Fig. 13b), implying the dopant guest should be uniformly dispersed in the BTA glass matrix.



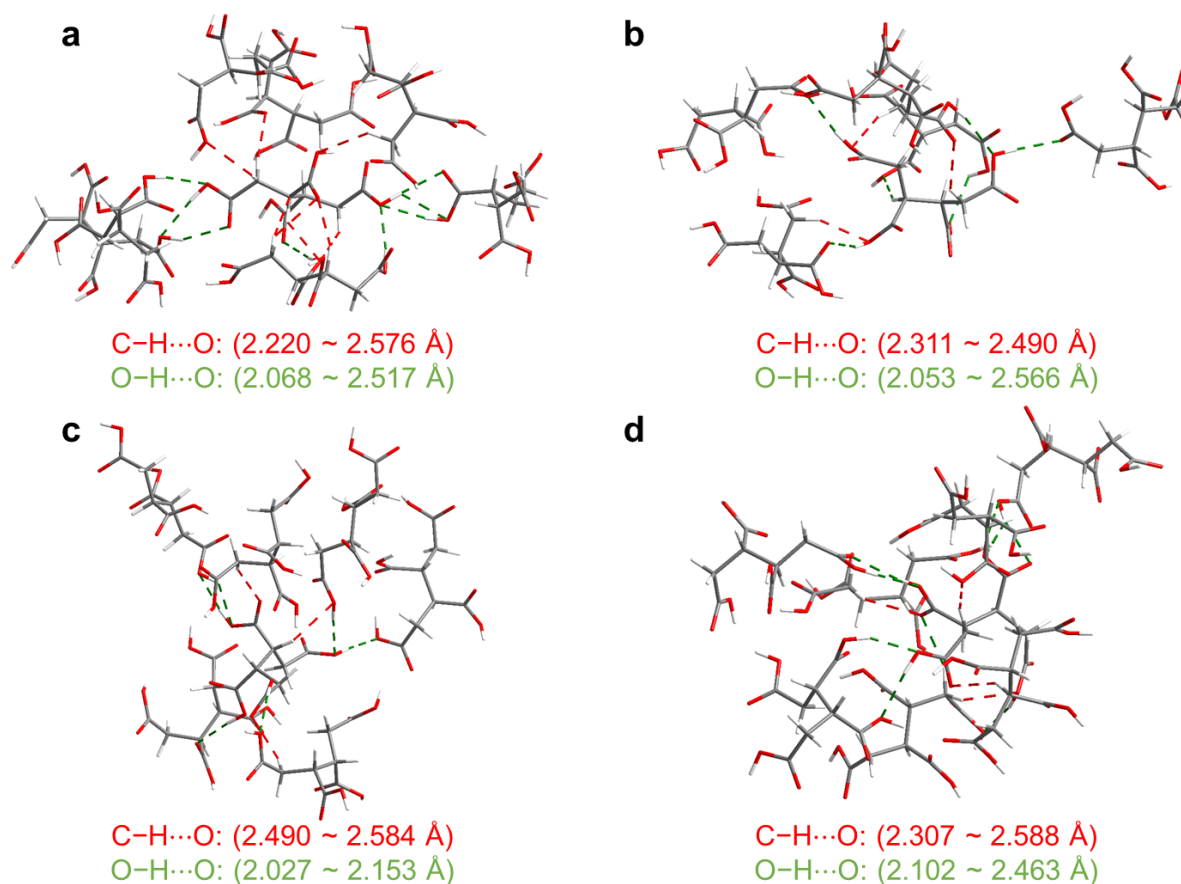
Supplementary Figure 16. Normalized steady-state PL and delayed (delayed time: 50 μ s) emission spectra of the guests in 2-MeTHF at 77 K (concentration: 1×10^{-5} mol/L).



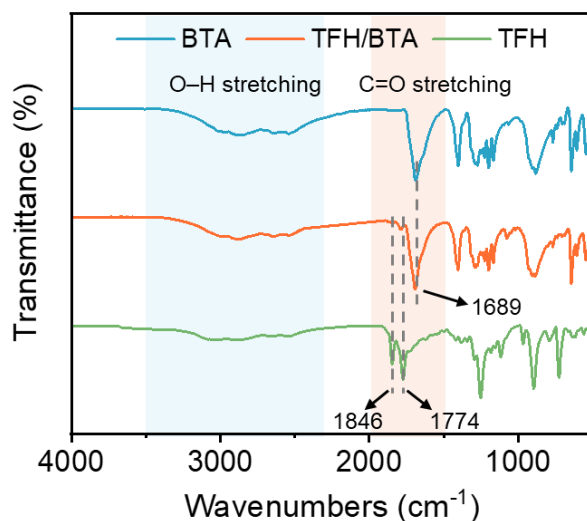
Supplementary Figure 17. (a) Normalized steady-state PL (dotted line) and delayed (solid line, delayed time: 50 μ s) emission spectra, and (b) phosphorescence decay curves of BTA-based doped materials in crystal state at room temperature.



Supplementary Figure 18. The fluorescence decay curves of the BTA-based doped (a) SGs and (b) crystal materials at room temperature.

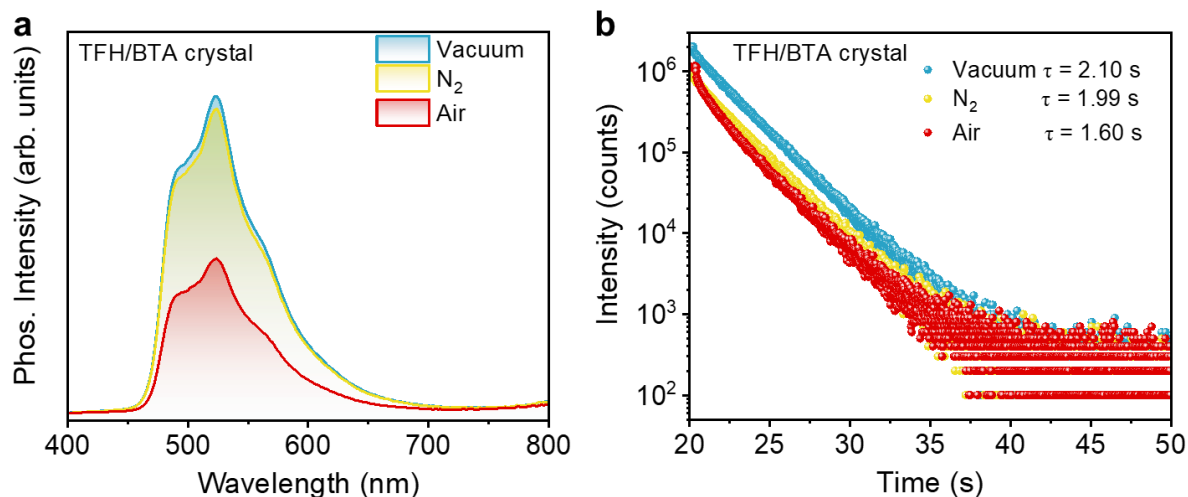


Supplementary Figure 19. (a-d) Typical molecular clusters representing the local arrangements within BTA glass. The hydrogen-bonding interaction distance of the C–H···O is red and the O–H···O is green.

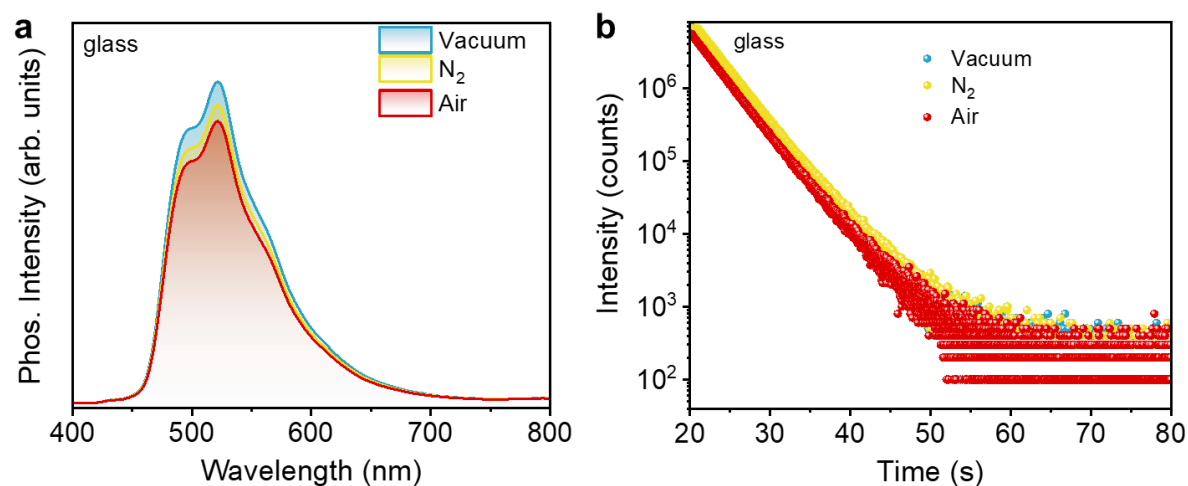


Supplementary Figure 20. Fourier-transform infrared (FT-IR) spectra of TFH/BTA doped glass together with pristine BTA glass and TFH powder.

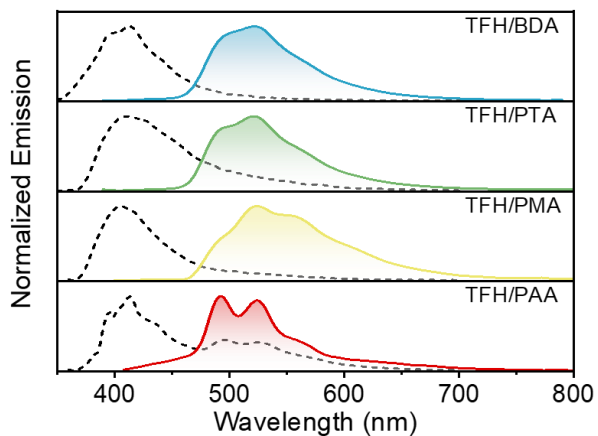
Supplementary Note 3. The FT-IR spectrum of BTA glass displays a broad, unstructured O–H stretching vibration peak between 2500 and 3500 cm^{-1} , suggesting the existence of multiple disorder intra- and intermolecular hydrogen bonding within such glass. The peaks located at 1689 cm^{-1} is attributed to the stretching vibrations of C=O in carboxyl groups. Additionally, compared to the BTA glass, the FT-IR spectrum of the TFH/BTA doped glass reveals new peaks at 1846 and 1774 cm^{-1} , corresponding to the C=O stretching vibrations of the anhydride group. This indicates that the BTA glass matrix and the guest TFH maintain structural integrity in the doped glass.



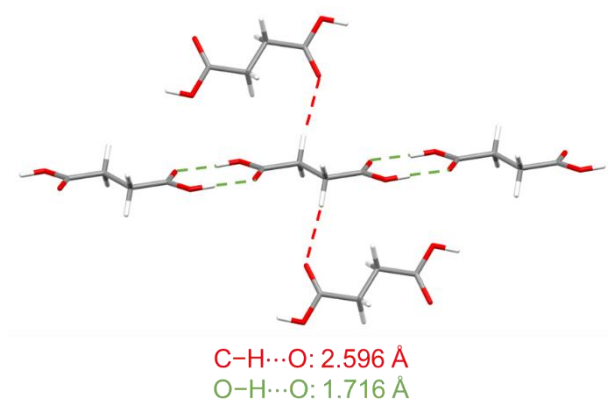
Supplementary Figure 21. (a) Delayed (delayed time: 50 μ s) emission spectra and (b) phosphorescence decay curves of the TFH/BTA doped crystal at different atmosphere conditions.



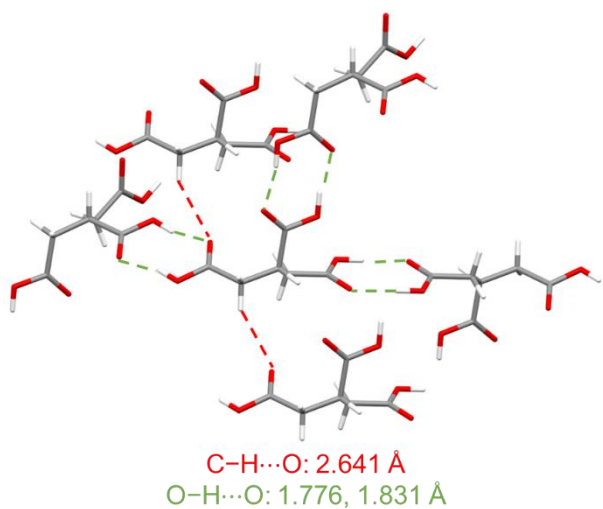
Supplementary Figure 22. (a) Normalized delayed (delayed time: 50 μ s) emission spectra and (b) phosphorescence decay curves of the TFH/BTA doped SGs at different atmosphere conditions.



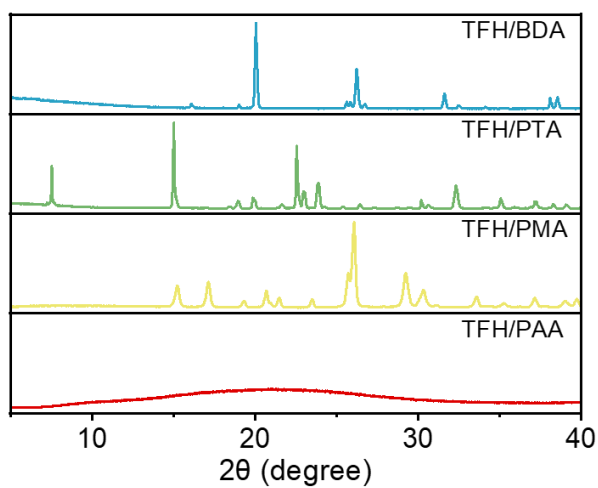
Supplementary Figure 23. Normalized steady-state PL (dash line) and delayed (solid line, delayed time: 50 μ s) emission spectra of reference doped materials under ambient conditions.



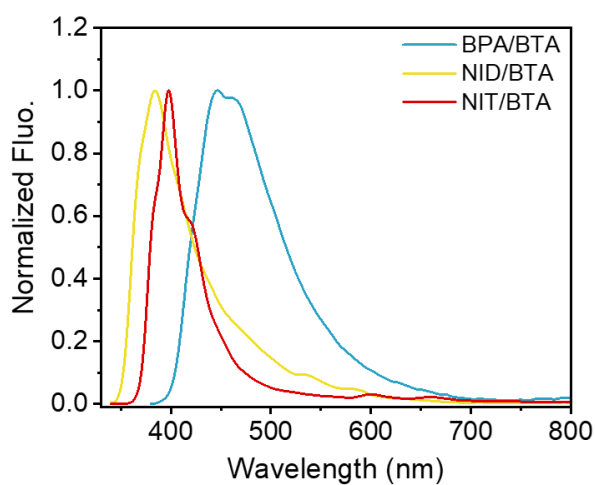
Supplementary Figure 24. Single-crystal packing patterns of BDA crystal. The hydrogen-bonding interaction distance of the C-H...O is red and the O-H...O is green.



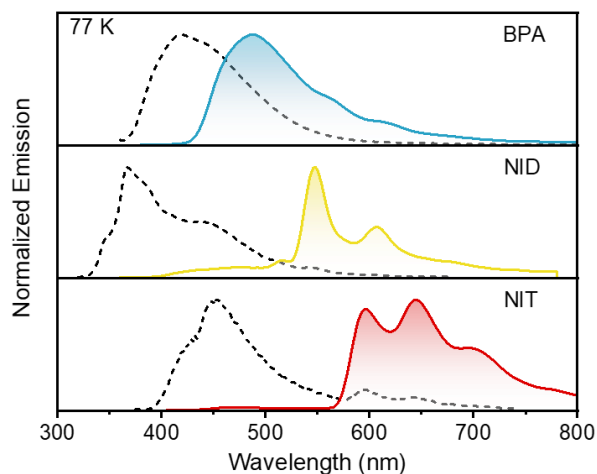
Supplementary Figure 25. Single-crystal packing patterns of PTA crystal. The hydrogen-bonding interaction distance of the C–H···O is red and the O–H···O is green.



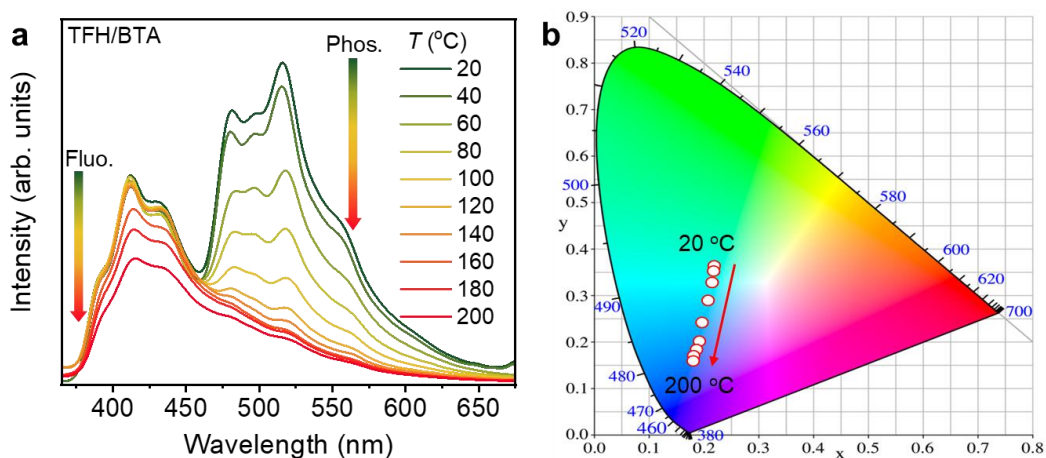
Supplementary Figure 26. PXRD patterns of reference doped materials.



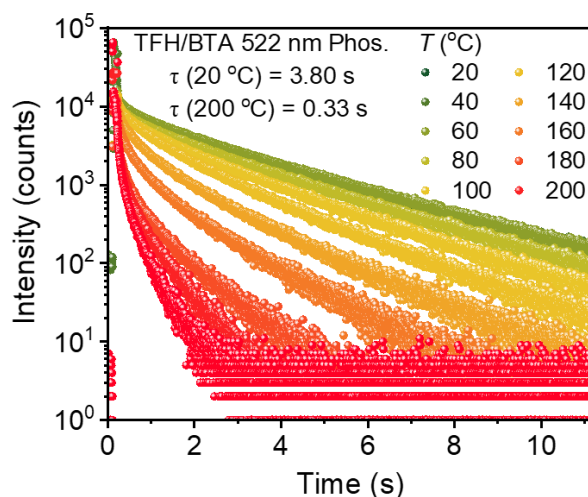
Supplementary Figure 27. Normalized steady-state PL emission spectra of BPA/BTA, NID/BTA, and NIT/BTA doped SGs at room temperature.



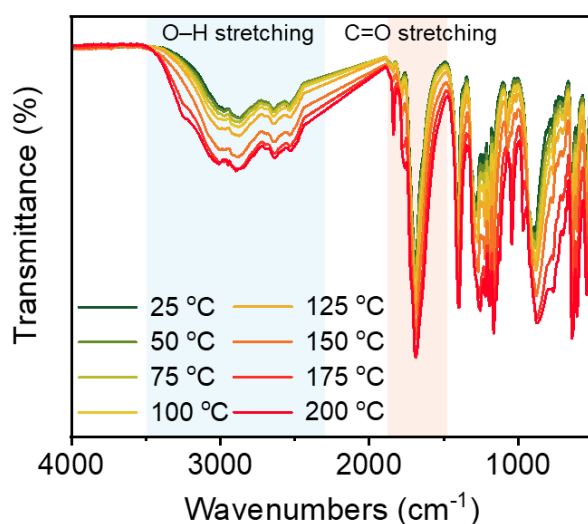
Supplementary Figure 28. Normalized steady-state PL and delayed (delayed time: 50 μs) emission spectra of the anhydride derivative guests in 2-MeTHF at 77 K (concentration: 1×10^{-5} mol/L).



Supplementary Figure 29. (a) Steady-state PL emission spectra and (b) CIE coordinates of the TFH/BTA glass at different temperatures.

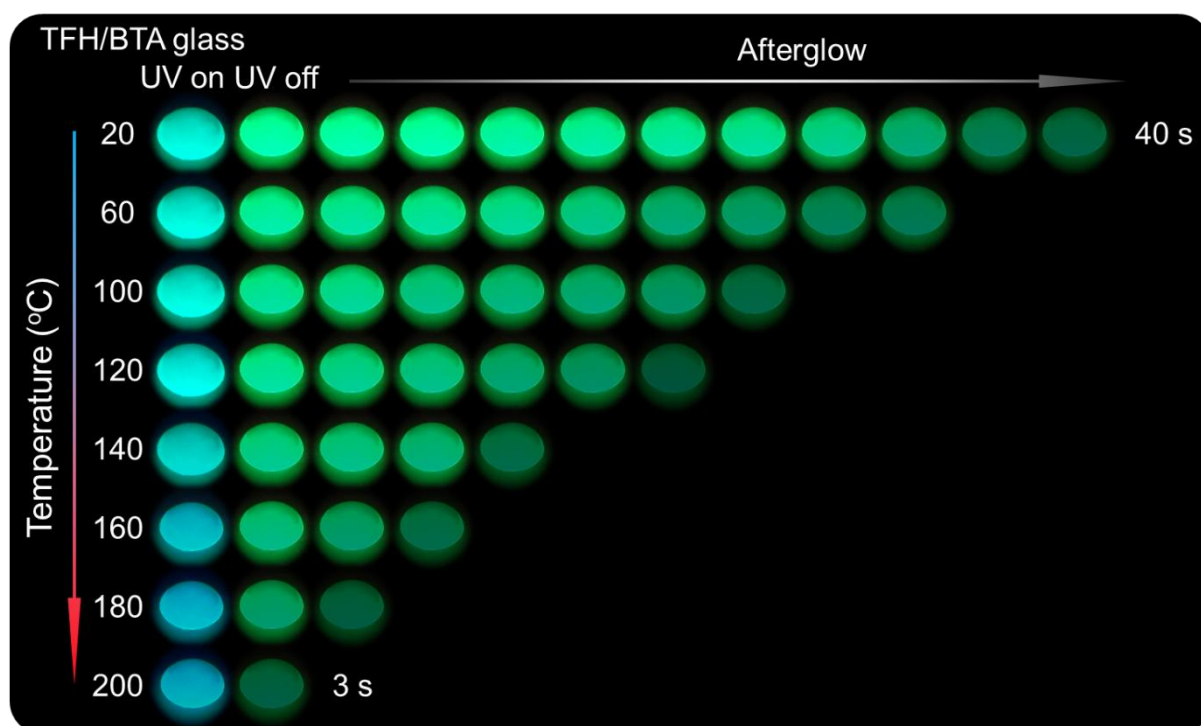


Supplementary Figure 30. Phosphorescence decay curves of the TFH/BTA doped SGs at different temperature.

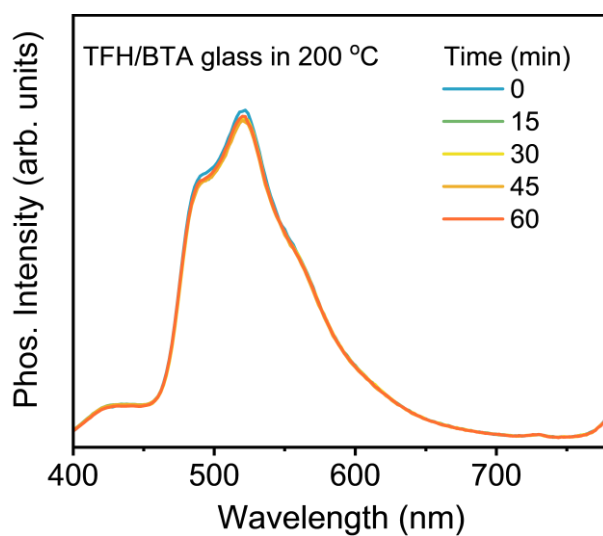


Supplementary Figure 31. Temperature-dependent FT-IR spectra of TFH/BTA doped glass.

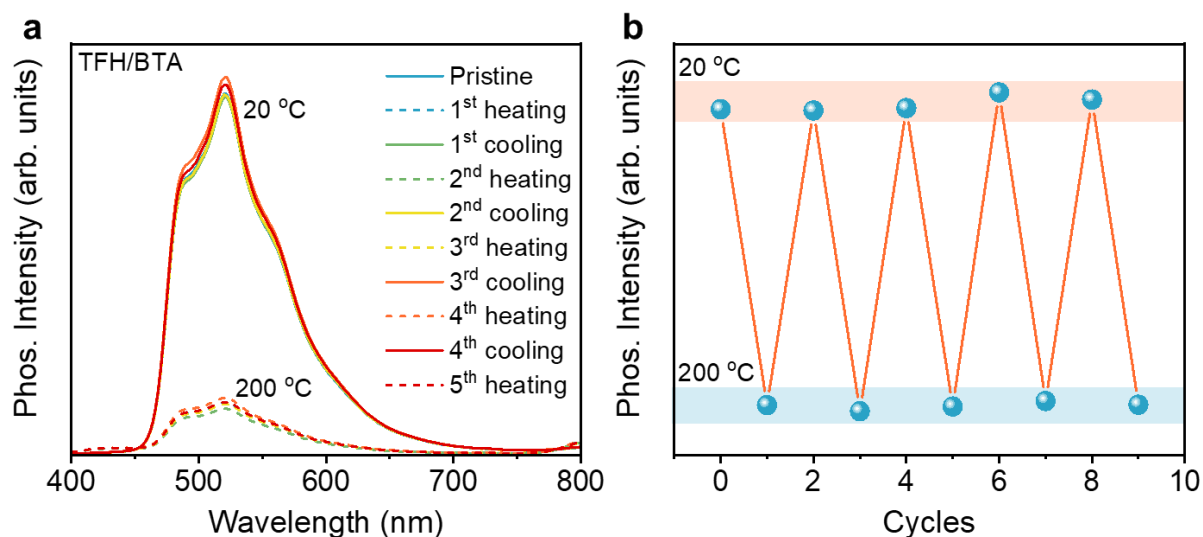
Supplementary Note 4. During the heating processes, the signal intensity shows minimal change in the range from 25 to 125 °C. As the temperature increases further, all of the vibration signals significantly increase in intensity and gradually broaden. At 175 °C, a new feature appears at 3240 cm^{-1} , indicating partial disruption of the $\text{C}=\text{O}\cdots\text{H}-\text{O}$ hydrogen bonds. These results suggest that as the temperature rises, the intermolecular hydrogen bonding interactions in the doped glass weaken, leading to enhanced non-radiative transitions, which ultimately results in reduced luminescence efficiency and shortened lifetimes at elevated temperatures.



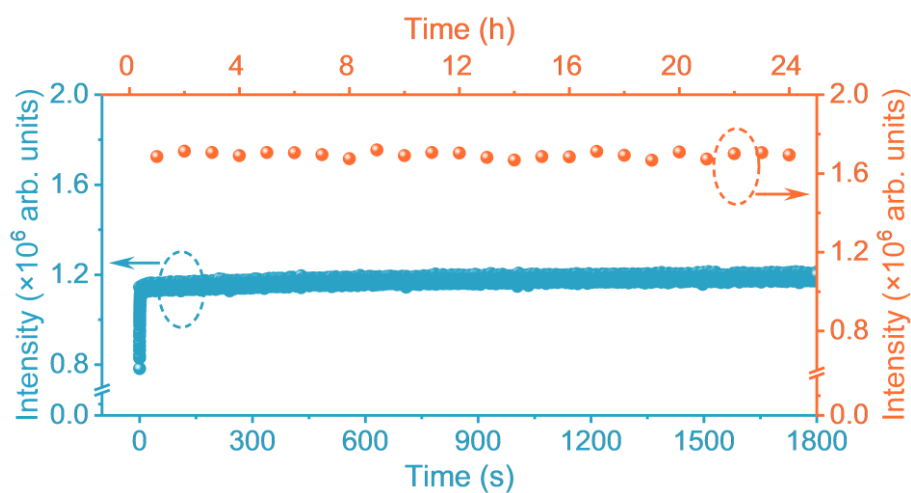
Supplementary Figure 32. Photographs of the TFH/BTA doped SGs at different temperature under 365 nm UV light on and off.



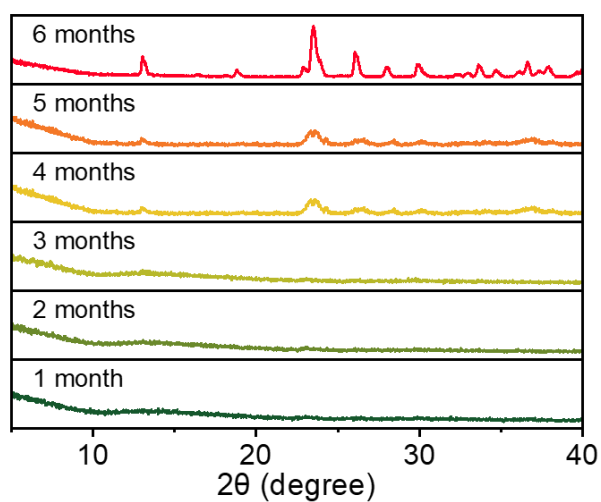
Supplementary Figure 33. Delayed (delayed time: 50 μ s) emission spectra of the TFH/BTA glass at high temperatures (200 °C) for different durations.



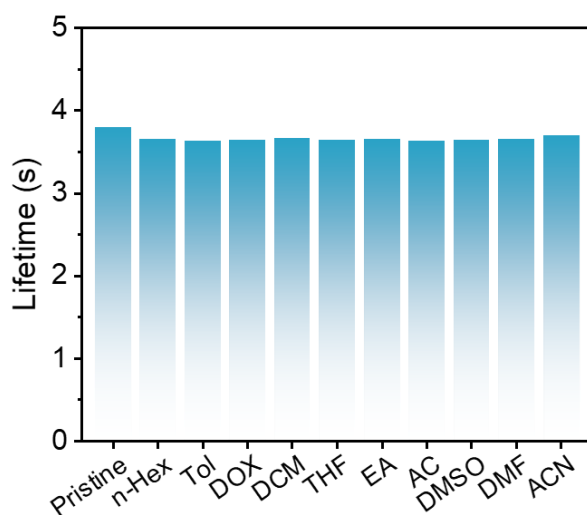
Supplementary Figure 34. Phosphorescence intensity of the TFH/BTA glass after many heating/cooling cycles.



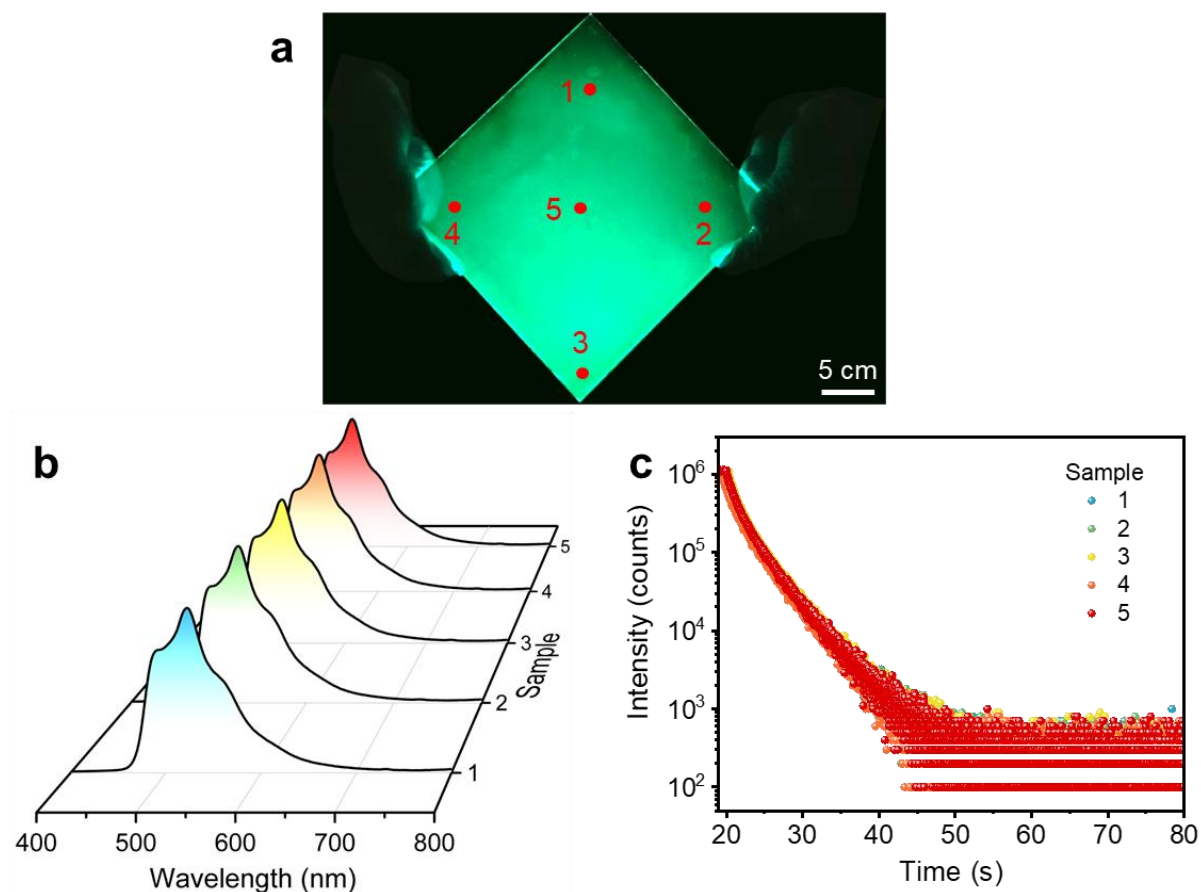
Supplementary Figure 35. Photostability of TFH/BTA glass upon irradiation of 365 nm UV light (power: $30 \mu\text{W}/\text{cm}^2$) under ambient conditions monitored by measuring the phosphorescence intensity at 522 nm.



Supplementary Figure 36. PXRD patterns of TFH/BTA doped SGs for 6 months under ambient conditions.



Supplementary Figure 37. Phosphorescence lifetimes of TFH/BTA doped SGs after being soaked in different solvents for 1 month (Note: a bar chart column represents a single measurement).



Supplementary Figure 38. (a) Afterglow photograph of large-size TFH/BTA doped SGs plate prepared under dark conditions (scale bar = 5 cm). (b) Delayed (delayed time: 50 μ s) emission spectra and (c) phosphorescence decay curves of the large-area TFH/BTA doped SGs plate at different regions.

Supplementary Note 5. The uniformity of phosphorescence performance across the 25 cm $\times$ 25 cm panel was confirmed by measuring the intensity and lifetime at different regions of the panel. The phosphorescence intensity and decay curves of five different points across the large-area TFH/BTA glass panel are nearly identical. The results confirm the scalability and reproducibility in large-scale fabrication.

Supplementary Tables

Supplementary Table 1. Photophysical properties of guest molecules in THF and solid state.

Sample	λ_{THF} [nm]	Φ_{THF} [%]	τ_{THF} [ns]	λ_{Solid} [nm]	Φ_{Solid} [%]	τ_{Solid} [ns]
NFD	381	19.98	7.27	424	6.91	7.19
BFT	378	5.63	2.75	385	42.81	4.00

TFH	395	27.98	3.18	414	24.10	3.56
BPA	425	5.49	3.30	416	71.36	5.57
NID	372	22.17	1.54	445	12.31	4.13
NIT	392	5.15	1.03	430	9.52	2.47

Supplementary Table 2. Photophysical properties of host–guest doping materials.

Sample		τ_F [ns]	Φ_F [%]	τ_P [s]	Φ_P [%]	k_r^F [s ⁻¹]	Φ_{ISC} [%]	k_{ISC} [s ⁻¹]	k_r^P [s ⁻¹]	k_{nr}^P [s ⁻¹]
NFD/BTA	Crystal	7.65	11.34	0.99	3.72	1.48×10^7	88.66	4.86×10^6	0.038	0.97
	Glass	7.06	22.97	1.62	5.72	3.25×10^7	77.03	8.10×10^6	0.035	0.58
BFT/BTA	Crystal	7.34	14.75	1.20	13.11	2.01×10^7	85.25	1.79×10^7	0.11	0.72
	Glass	7.72	15.45	2.82	23.88	2.00×10^7	84.46	3.09×10^7	0.10	0.26
TFH/BTA	Crystal	8.83	15.05	1.60	27.61	1.70×10^7	84.95	3.13×10^7	0.17	0.45
	Glass	7.57	11.45	3.80	56.82	1.51×10^7	88.55	7.51×10^7	0.15	0.11

Note: k_r^F , k_{ISC} , k_r^P , k_{nr}^P are the radiative rate constant of prompt fluorescence, rate constant of ISC, radiative rate constant of phosphorescence, and non-radiative rate constant of phosphorescence of T₁. $k_r^F = \Phi_F/\tau_F$; $\Phi_{ISC} = 1 - \Phi_F - \Phi_{IC} \approx 1 - \Phi_F$; $k_{ISC} = \Phi_P/\tau_F$; $k_r^P = \Phi_P/\tau_P$; $k_{nr}^P = (1 - \Phi_P)/\tau_P$.

Supplementary Table 3. Photophysical properties of host–guest doping materials.

Sample		τ_F [ns]	Φ_F [%]	τ_P [s]	Φ_P [%]	k_r^F [s ⁻¹]	Φ_{ISC} [%]	k_{ISC} [s ⁻¹]	k_r^P [s ⁻¹]	k_{nr}^P [s ⁻¹]
TFH/BDA		3.95	20.80	1.12	1.70	5.27×10^7	79.20	4.30×10^6	0.015	0.88
TFH/PTA		4.50	27.48	1.31	2.89	6.11×10^7	72.52	6.42×10^6	0.022	0.74
TFH/PMA		2.77	45.21	0.81	2.38	1.63×10^8	54.79	8.59×10^6	0.029	1.20
TFH/PAA		5.46	10.61	1.30	4.03	1.94×10^7	89.39	7.38×10^6	0.031	0.74

Note: k_r^F , k_{ISC} , k_r^P , k_{nr}^P are the radiative rate constant of prompt fluorescence, rate constant of ISC, radiative rate constant of phosphorescence, and non-radiative rate constant of phosphorescence of T₁. $k_r^F = \Phi_F/\tau_F$; $\Phi_{ISC} = 1 - \Phi_F - \Phi_{IC} \approx 1 - \Phi_F$; $k_{ISC} = \Phi_P/\tau_F$; $k_r^P = \Phi_P/\tau_P$; $k_{nr}^P = (1 - \Phi_P)/\tau_P$.

Supplementary Table 4. A summary of high-temperature (≥ 373 K) afterglow materials was reported in literatures and our work, including the maximum emission wavelength, phosphorescence quantum yield, lifetime and the highest temperature that afterglow can be observed by naked-eyes.

	Materials	Φ_p [%]	$\tau_{\text{afterglow}}$ [ms]	T [K]	Reference
Hybrid organic- inorganic	BA@CE	/	2410	423	[4]
	Cd-IDB-IPA	21.6%	322.8	380	[5]
	CDs@Paper	51.1%	390.6	403	[6]
	PACs/B ₂ O ₃	15.87%	2224	673	[7]
	Pphen@BA	36.3%	1870	398	[8]
Organic	RB/TPP-6OC ₄ Cs	5.43%	124.6	413	[9]
	BCZ/PVP	22.4%	4180	433	[10]
	CC1	4.5%	2160	423	[11]
	MCTPA	/	453.3	476	[12]
	MCATMA	65%	1300	473	[13]
	NPR/WPU	4.41%	762.3	433	[14]
	P4	4.30%	1990	373	[15]
	PF-H/PAA	14.3%	905.0	373	[16]
	COR-MF	22.7%	4833	440	[17]
	Py-BA/COC	5.31%	363.0	378	[18]
	Py/Xan	2.32%	410.0	423	[19]
	triamino-s-triazine derivative	19.3%	474.7	397	[20]
	CoTAK/BAK	38.7%	2700	673	[21]
	PSSNa	2.40%	2139	433	[22]
	Cdp/DMAP	4.04%	768.0	383	[23]
	PMPF ₆ /phCB[6]	2.1%	1347	373	[24]
	Cs-C ₅ OD	27.04%	426.7	403	[25]
	PPCzPy-2	13%	3540	413	[26]
	TN/TPBi	/	> 24 h	400	[27]
	NDOH/POOH	1.6%	127.1	373	[28]
	TFH/BTA	56.82%	3800	473	This work

Supplementary References

- [1] Zenka M, Preinl J, Pertermann E, Lützen A, Tiefenbacher K. A Water- and Base-Stable Iminopyridine-Based Cage That Can Bind Larger Organic Anions. *Eur. J. Inorg. Chem.* **26**, e202300110 (2023).
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