

Electronic Supplementary Information for

The influence of the π - π stacking on the room temperature phosphorescence of phenothiazine 5,5-dioxide derivatives

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3. Supplementary References

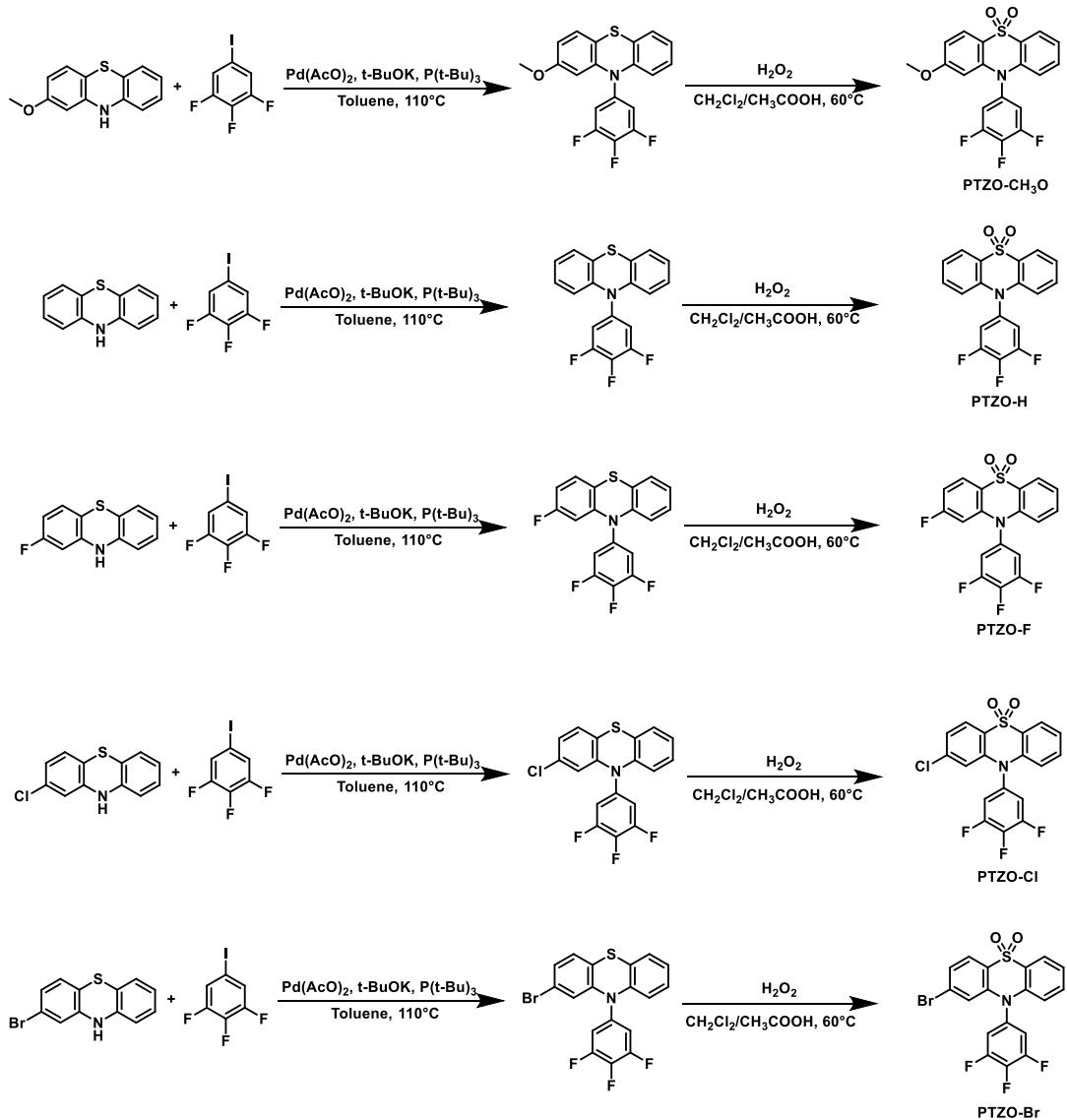
1. General Information

Syntheses and Characterization

¹H NMR and ¹³C NMR spectra were recorded on a 600 MHz Bruker Ascend spectrometer. Mass spectra were measured on a UHPLC/Q-TOF MS spectrophotometer. UV-Vis absorption spectra were measured on a Shimadzu UV-2600. Photoluminescence spectra were performed on a Hitachi F-4600 fluorescence spectrophotometer. Powder X-ray diffraction (PXRD) patterns were recorded by Rigaku Smartlab9KW. The single-crystal X-ray diffraction data of these samples were collected in XtaLAB SuperNova X-ray diffractometer. Photoluminescence quantum yields and lifetimes were determined with FLS1000 spectrometer.

The Gaussian 09 program was utilized to perform TD-DFT calculations with m062x/6-31g* method. Non-covalent interactions of intermolecular interactions analyses were carried out by Multiwfn with reduced density gradient (RDG)^[1]. The NCI results were plotted via VMD software.

The synthetic routes of compounds PTZO-CH₃O, PTZO-H, PTZO-F, PTZO-Cl, PTZO-Br were described in Scheme S1. Compounds phenothiazine, 2-Methoxy-10H-phenothiazine and 2-Chloro-10H-phenothiazine were purchased commercially. Compounds 2-Fluoro-10H-phenothiazine and 2-Bromo-10H-phenothiazine were synthesized on the grounds of the previous literature.^[2-5]



Scheme S1. The synthetic routes of PTZO-CH₃O, PTZO-H, PTZO-F, PTZO-Cl, PTZO-Br.

General procedure for the syntheses of PTZO-CH₃O, PTZO-H, PTZO-F, PTZO-Cl, PTZO-Br.

PTZO-CH₃O: 2-Methoxy-10H-phenothiazine (2.30 g, 10 mmol), 3,4,5-trifluoriodobenzene (2.58 g, 10 mol), potassium *tert*-butoxide (1.68 g, 15 mmol), palladium acetate (0.11 g, 0.5 mmol) and tri-*tert*-butylphosphine solution (0.5 mL, 0.25 mmol) were dissolved in toluene (100 mL) in a Schlenk tube. The resultant mixture was refluxed for 12 hours under nitrogen, then extracted with water. The combined organic extracts were dried over anhydrous Na₂SO₄ and concentrated by rotary evaporation. The crude product was purified by column chromatography on silica gel using petroleum ether as eluent. Then the collected product was dissolved in dichloromethane (90 mL), acetic acid (45 mL) and H₂O₂ (2 mL). After reacting for another 24 hours at 60 °C, the reaction mixture was extracted with dichloromethane and further purified by column chromatography using petroleum dichloromethane/petroleum ether (2:1 v/v) as eluent to afford a white solid in a yield of 65%. ¹H-NMR (600 MHz, CDCl₃): δ 8.18-8.15 (m, H), 8.12-8.10 (d, H), 7.46-7.42 (t, H), 7.32-7.28 (t, H), 7.14-7.11 (t, 2H), 6.87-6.84 (t, H), 6.61-6.59 (d, H), 6.05-6.04 (d, H), 3.78 (s, 3H);

¹³C-NMR (150 MHz, CDCl₃): δ 163.2, 154.1, 151.5, 141.9, 140.2, 134.1, 132.9, 125.92, 123.7, 122.8, 116.6, 116.3, 116.0, 115.9, 115.8, 115.7, 108.6, 102.3, 100.055.7; MS (EI), m/z: [M⁺], calcd. for C₁₉H₁₂F₃NNaO₃S, 414.0398; found, 414.0382.

PTZO-H: Phenothiazine (1.99 g, 10 mmol), 3,4,5-trifluoriodobenzene (2.58 g, 10 mol), potassium *tert*-butoxide (1.68 g, 15 mmol), palladium acetate (0.11 g, 0.5 mmol) and tri-*tert*-butylphosphine solution (0.5 mL, 0.25 mmol) were dissolved in toluene (100 mL) in a Schlenk tube. The resultant mixture was refluxed for 12 hours under nitrogen, then extracted with water. The combined organic extracts were dried over anhydrous Na₂SO₄ and concentrated by rotary evaporation. The crude product was purified by column chromatography on silica gel using petroleum ether as eluent. Then the collected product was dissolved in dichloromethane (90 mL), acetic acid (45 mL) and H₂O₂ (2 mL). After reacting for another 24 hours at 60 °C, the reaction mixture was extracted with dichloromethane and further purified by column chromatography using petroleum dichloromethane/petroleum ether (2:1 v/v) as eluent to afford a white solid in a yield of 62%. ¹H-NMR (600 MHz, CDCl₃): δ 8.20-8.17 (m, 2H), 7.49-7.45 (m, 2H), 7.34-7.30 (t, 2H), 7.16-7.12 (t, 2H), 6.67-6.64 (d, 2H); ¹³C-NMR (150 MHz, CDCl₃): δ 154.1, 154.0, 151.6, 151.5, 151.4, 142.3, 140.1, 139.7, 134.0, 133.1, 123.8, 123.2, 122.9, 116.6, 116.0, 115.8; MS (EI), m/z: [M⁺], calcd. for C₁₈H₁₀F₃NNaO₂S, 384.0287; found, 384.0277.

PTZO-F: 2-Fluoro-10H-phenothiazine (2.17 g, 10 mmol), 3,4,5-trifluoriodobenzene (2.58 g, 10 mol), potassium *tert*-butoxide (1.68 g, 15 mmol), palladium acetate (0.11 g, 0.5 mmol) and tri-*tert*-butylphosphine solution (0.5 mL, 0.25 mmol) were dissolved in toluene (100 mL) in a Schlenk tube. The resultant mixture was refluxed for 12 hours under nitrogen, then extracted with water. The combined organic extracts were dried over anhydrous Na₂SO₄ and concentrated by rotary evaporation. The crude product was purified by column chromatography on silica gel using petroleum ether as eluent. Then the collected product was dissolved in dichloromethane (90 mL), acetic acid (45 mL) and H₂O₂ (2 mL). After reacting for another 24 hours at 60 °C, the reaction mixture was extracted with dichloromethane and further purified by column chromatography using petroleum dichloromethane/petroleum ether (2:1 v/v) as eluent to afford a white solid in a yield of 66%. ¹H-NMR (600 MHz, CDCl₃): δ 8.19-8.17 (t, 2H), 7.50-7.47 (t, H), 7.36-7.33 (t, H), 7.15-7.13 (t, 2H), 7.04-7.01 (t, H), 6.65-6.63 (d, H), 6.34-6.32 (t, H); ¹³C-NMR (150 MHz, CDCl₃): δ 166.0, 164.3, 153.9, 152.2, 142.2, 140.0, 133.3, 126.8, 123.8, 123.7, 123.5, 119.7, 116.8, 115.9, 115.8, 115.7, 111.1, 111.0, 104.0, 103.8; MS (EI), m/z: [M⁺], calcd. for C₁₈H₉F₄NNaO₂S, 402.0198; found, 402.0182.

PTZO-Cl: 2-Chloro-10H-phenothiazine (2.34 g, 10 mmol), 3,4,5-trifluoriodobenzene (2.58 g, 10 mol), potassium *tert*-butoxide (1.68 g, 15 mmol), palladium acetate (0.11 g, 0.5 mmol) and tri-*tert*-butylphosphine solution (0.5 mL, 0.25 mmol) were dissolved in toluene (100 mL) in a Schlenk tube. The resultant mixture was refluxed for 12 hours under nitrogen, then extracted with water. The combined organic extracts were dried over anhydrous Na₂SO₄ and concentrated by rotary evaporation. The crude product was purified by column chromatography on silica gel using petroleum ether as eluent. Then the collected product was dissolved in dichloromethane (90 mL), acetic acid (45 mL) and H₂O₂ (2 mL). After reacting for another 24 hours at 60 °C, the reaction mixture was extracted with dichloromethane and further purified by column chromatography

using petroleum dichloromethane/petroleum ether (2:1 v/v) as eluent to afford a white solid in a yield of 68%. $^1\text{H-NMR}$ (600 MHz, CDCl_3): δ 8.18-8.15 (m, H), 8.11-8.09 (d, H), 7.51-7.46 (t, H), 7.36-7.32 (t, H), 7.29-7.27 (t, H), 7.15-7.12 (t, 2H), 6.65-6.63 (d, H), 6.62-6.62 (d, H); $^{13}\text{C-NMR}$ (150MHz, CDCl_3): δ 154.2, 154.1, 151.7, 151.6, 151.5, 142.5, 141.0, 140.0, 139.9, 139.5, 133.4, 125.3, 123.7, 123.5, 123.4, 123.3, 121.7, 116.8, 116.4; MS (EI), m/z: $[\text{M}^+]$, calcd. for $\text{C}_{18}\text{H}_9\text{ClF}_3\text{NNaO}_2\text{S}$, 417.9888; found, 417.9887.

PTZO-Br: 2-Bromo-10H-phenothiazine (2.78 g, 10 mmol), 3,4,5-trifluoriodobenzene (2.58 g, 10 mol), potassium *tert*-butoxide (1.68 g, 15 mmol), palladium acetate (0.11 g, 0.5 mmol) and tri-*tert*-butylphosphine solution (0.5 mL, 0.25 mmol) were dissolved in toluene (100 mL) in a Schlenk tube. The resultant mixture was refluxed for 12 hours under nitrogen, then extracted with water. The combined organic extracts were dried over anhydrous Na_2SO_4 and concentrated by rotary evaporation. The crude product was purified by column chromatography on silica gel using petroleum ether as eluent. Then the collected product was dissolved in dichloromethane (90 mL), acetic acid (45 mL) and H_2O_2 (2 mL). After reacting for another 24 hours at 60 °C, the reaction mixture was extracted with dichloromethane and further purified by column chromatography using petroleum dichloromethane/petroleum ether (2:1 v/v) as eluent to afford a white solid in a yield of 62%. $^1\text{H-NMR}$ (600 MHz, CDCl_3): δ 8.18-8.16 (t, H), 8.04-8.02 (d, H), 7.51-7.43 (m, 2H), 7.36-7.33 (t, H), 7.16-7.13 (t, 2H), 6.79 (s, H), 6.65-6.63 (d, H); $^{13}\text{C-NMR}$ (150 MHz, CDCl_3): δ 141.1, 140.0, 133.4, 128.0, 126.2, 125.4, 123.8, 123.6, 123.5, 122.3, 119.4, 117.0, 116.0, 115.8, 115.7, 100.0; MS (EI), m/z: $[\text{M}^+]$, calcd. for $\text{C}_{18}\text{H}_9\text{BrF}_3\text{NNaO}_2\text{S}$, 461.9392; found, 461.9382.

Preparation of single crystal

The single crystal of PTZO-Cl was cultured by slow solvent evaporation of the saturated solution of PTZO-Cl in dichloromethane/methyl alcohol. The single crystal of PTZO-Cl (RTP-inactive) was cultured by slow solvent evaporation of the saturated solution of PTZO-Cl in dichloromethane/petroleum ether.

2. Figures and Tables

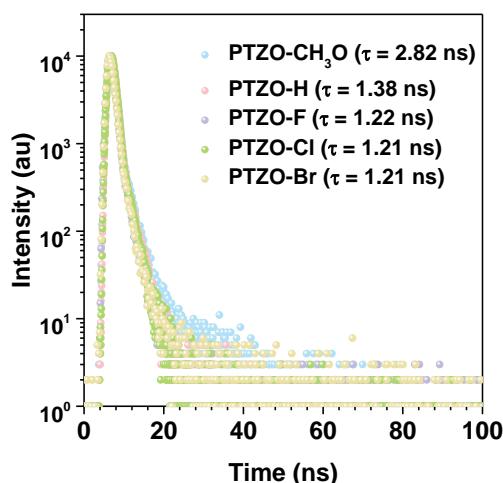


Figure S1. Fluorescence decay curves of phenothiazine 5,5-dioxide derivatives in crystal state at room temperature.

Table S1. The PL quantum yields of PTZO-CH₃O, PTZO-Br, PTZO-H, PTZO-F and PTZO-Cl and PTZO-Cl (RTP-inactive) in crystal state.

Samples	Quantum yield (Φ , %)
	Φ (%) (@ 300 nm)
PTZ-CH ₃ O	2.55%
PTZ-H	1.71%
PTZ-F	1.10%
PTZ-Cl	1.79%
PTZ-Br	0.22%
PTZ-Cl-RTP inactive	0.94%

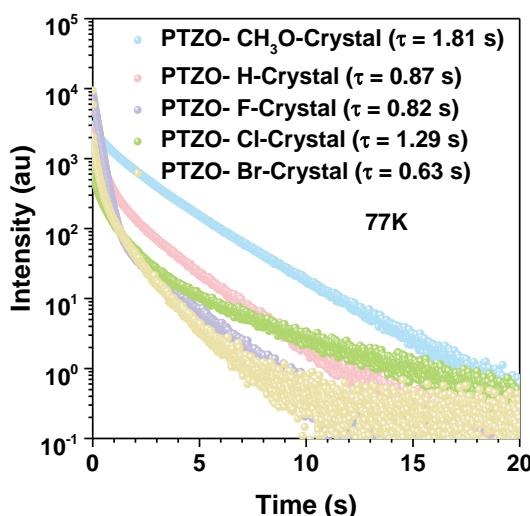


Figure S2. Time-resolved PL-decay curves for their low temperature phosphorescence in crystal state.

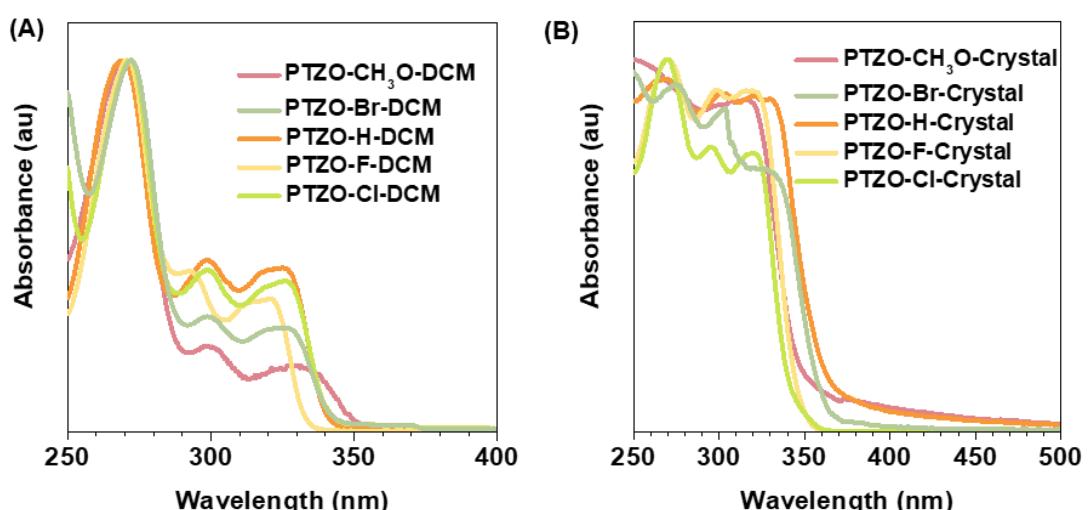


Figure S3. (A) The UV-visible spectra of the dilute dichloromethane solutions of PTZO-CH₃O, PTZO-Br, PTZO-H, PTZO-F and PTZO-Cl at room temperature (Concentration: 10⁻⁵ M). (B) The UV-visible spectra of the crystals of PTZO-CH₃O, PTZO-Br, PTZO-H, PTZO-F and PTZO-Cl at room temperature.

room temperature.

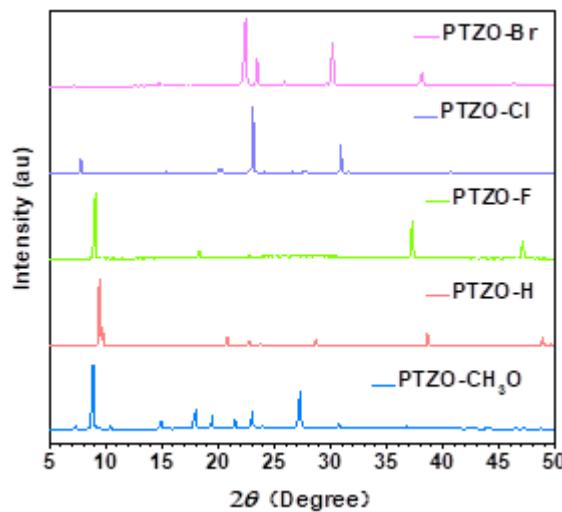


Figure S4. The PXRD patterns of the crystals of PTZO-CH₃O, PTZO-Br, PTZO-H, PTZO-F and PTZO-Cl at room temperature.

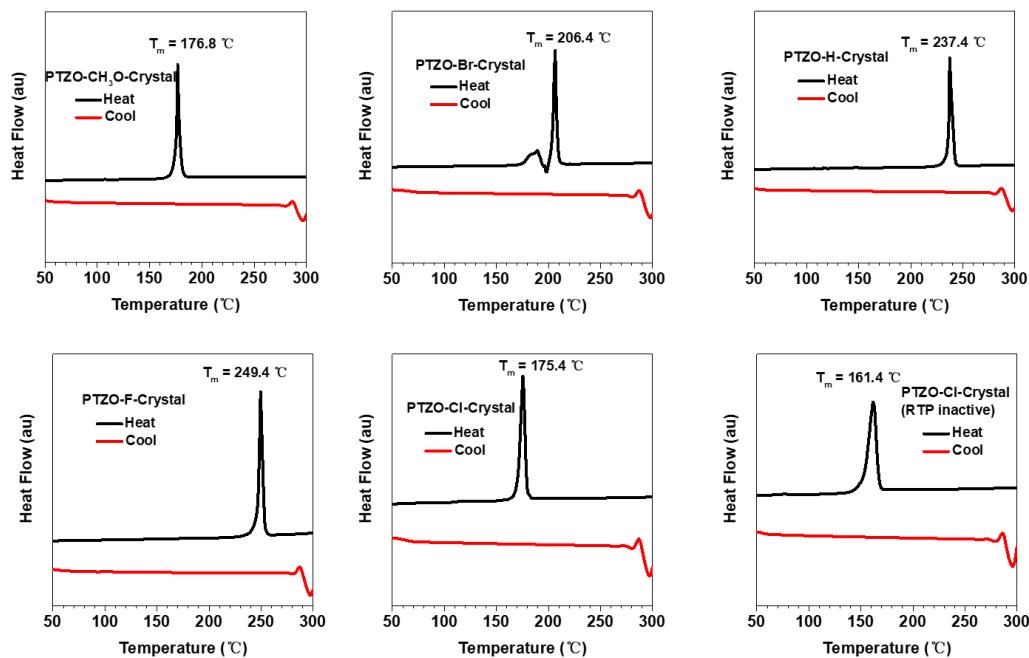


Figure S5. The DSC curves of PTZO-CH₃O, PTZO-Br, PTZO-H, PTZO-F and PTZO-Cl and PTZO-Cl (RTP-inactive) in crystal state.

Table S2. Single crystal data of PTZO-CH₃O, PTZO-Br, PTZO-H, PTZO-F and PTZO-Cl and

PTZO-Cl (RTP-inactive).

Name	PTZO-CH ₃ O	PTZO-Br	PTZO-H	PTZO-F	PTZO-Cl	PTZO-Cl (RTP inactive)
Formula	C ₁₉ H ₁₂ F ₃ NO ₃ S	C ₁₈ H ₉ BrF ₃ NO ₂ S	C ₁₈ H ₁₀ F ₃ NO ₂ S	C ₁₈ H ₉ F ₄ NO ₂ S	C ₁₈ H ₉ ClF ₃ NO ₂ S	C ₁₈ H ₉ ClF ₃ NO ₂ S
Wavelength (Å)	1.54184	1.54184	1.54184	0.71073	1.54184	1.54184
Space Group	P 1 21/c 1	R -3	P b c a	P b c a	P b c a	P 1 21/c 1
Cell Lengths (Å)	a=11.89904(16) b=19.4573(3) c=9.08595(14)	a=23.3444(4) b=23.3444(4) c=16.0383(3)	a=10.6605(2) b=15.5563(2) c=18.6230(3)	a=10.5098(6) b=15.5106(7) c=19.2985(11)	a=10.2364(2) b=15.4778(3) c=21.0591(4)	a=12.2251(3) b=17.7371(4) c=7.86265(19)
Cell Angles (°)	alpha=90 beta=102.1856(15) gamma=90	alpha=90 beta=90 gamma=120	alpha=90 beta=90 gamma=90	alpha=90 beta=90 gamma=90	alpha=90 beta=90 gamma=90	alpha=90 beta=92.328(2) gamma=90
Cell Volume	2056.21(5)	7569.3(2)	3088.40(9)	3145.9(3)	3336.54(10)	1703.50(8)
Z	4	18	7	8	8	4
Density (g/cm ³)	1.538	1.738	1.554	1.602	1.576	1.543
F(000)	968.0	3930.8	1472.0	1536.0	1600.0	800.0
h _{max} , k _{max} , l _{max}	14,23,10	23,24,19	12,18,22	15,23,28	12,18,25	14,21,9
CCDC Number	2132708	2132716	2132713	2132715	2132714	2132717

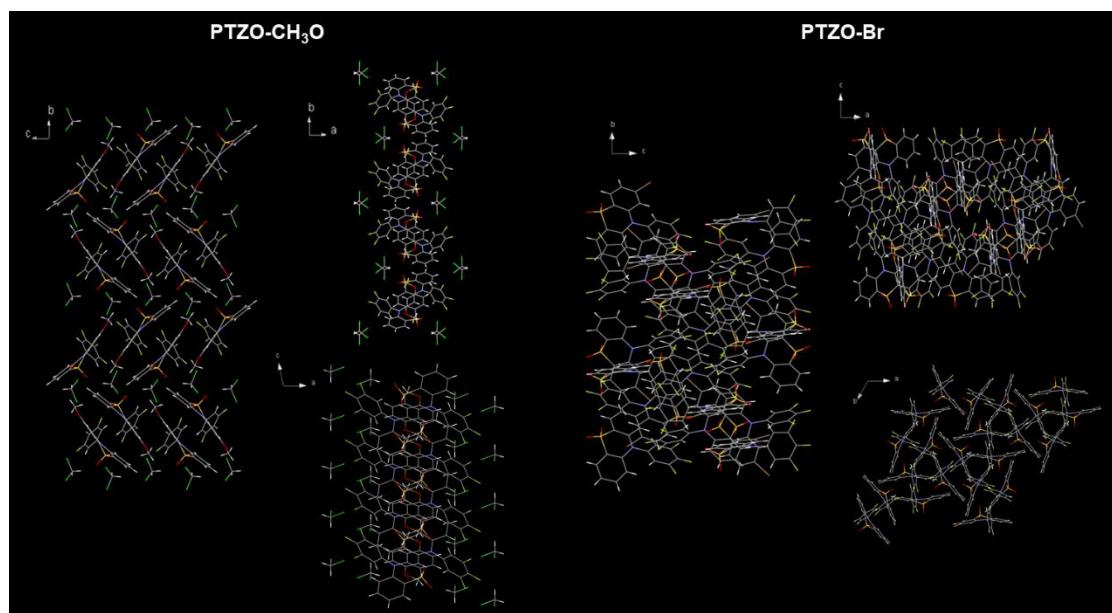


Figure S6. Molecular packing of PTZO-CH₃O and PTZO-Br crystals observed from a, b and c directions.

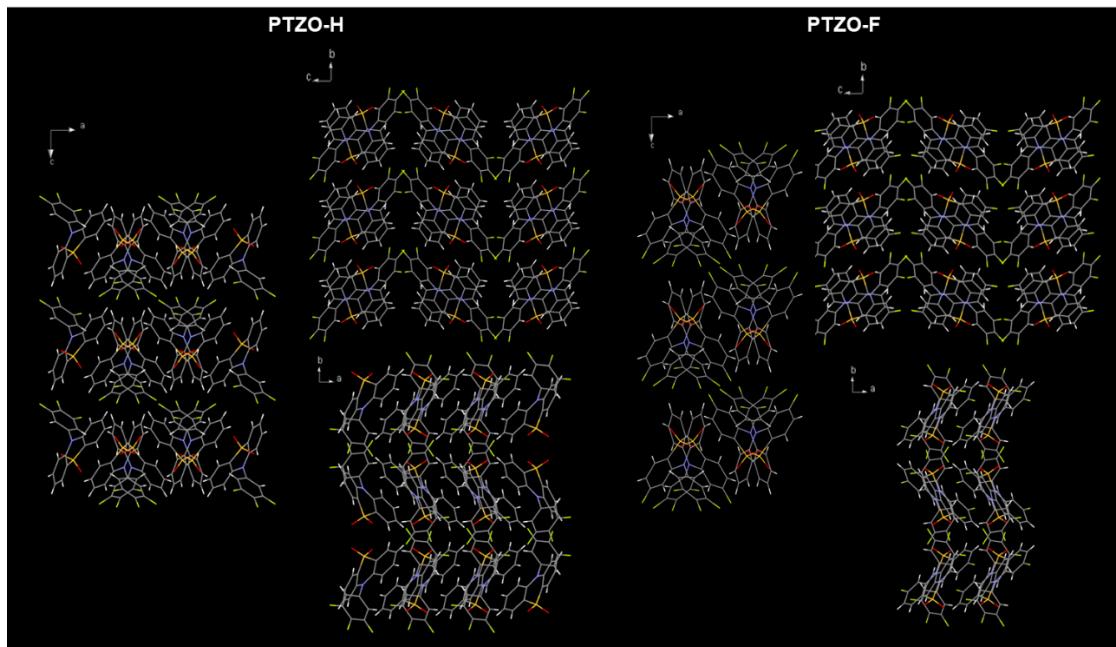


Figure S7. Molecular packing of PTZO-H and PTZO-F crystals observed from a, b and c directions.

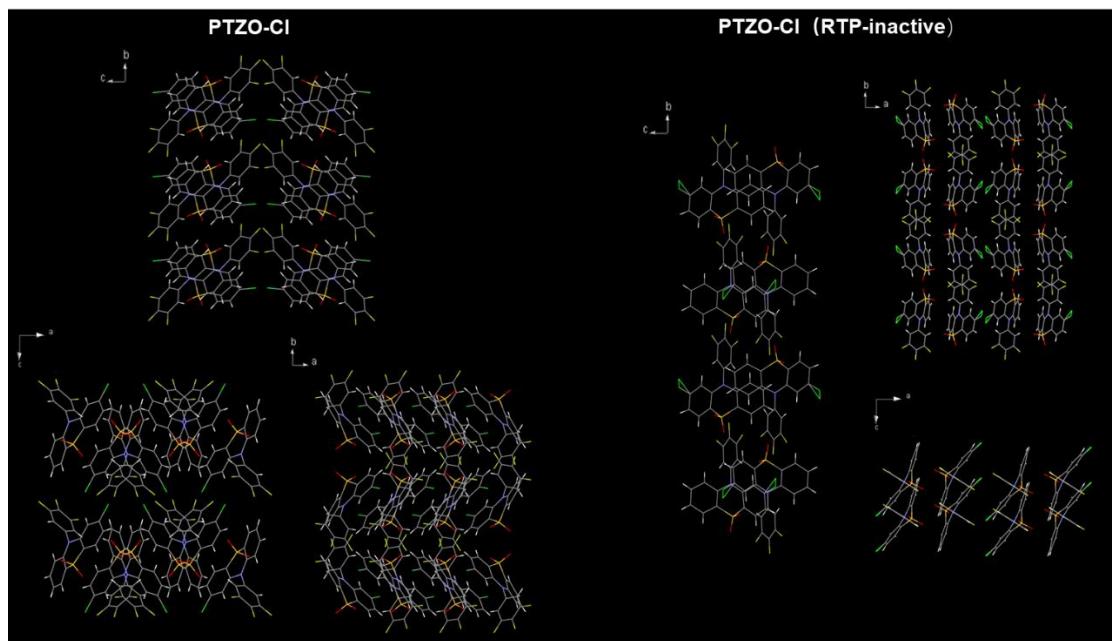


Figure S8. Molecular packing of PTZO-Cl and PTZO-Cl (RTP-inactive) crystals observed from a, b and c directions.

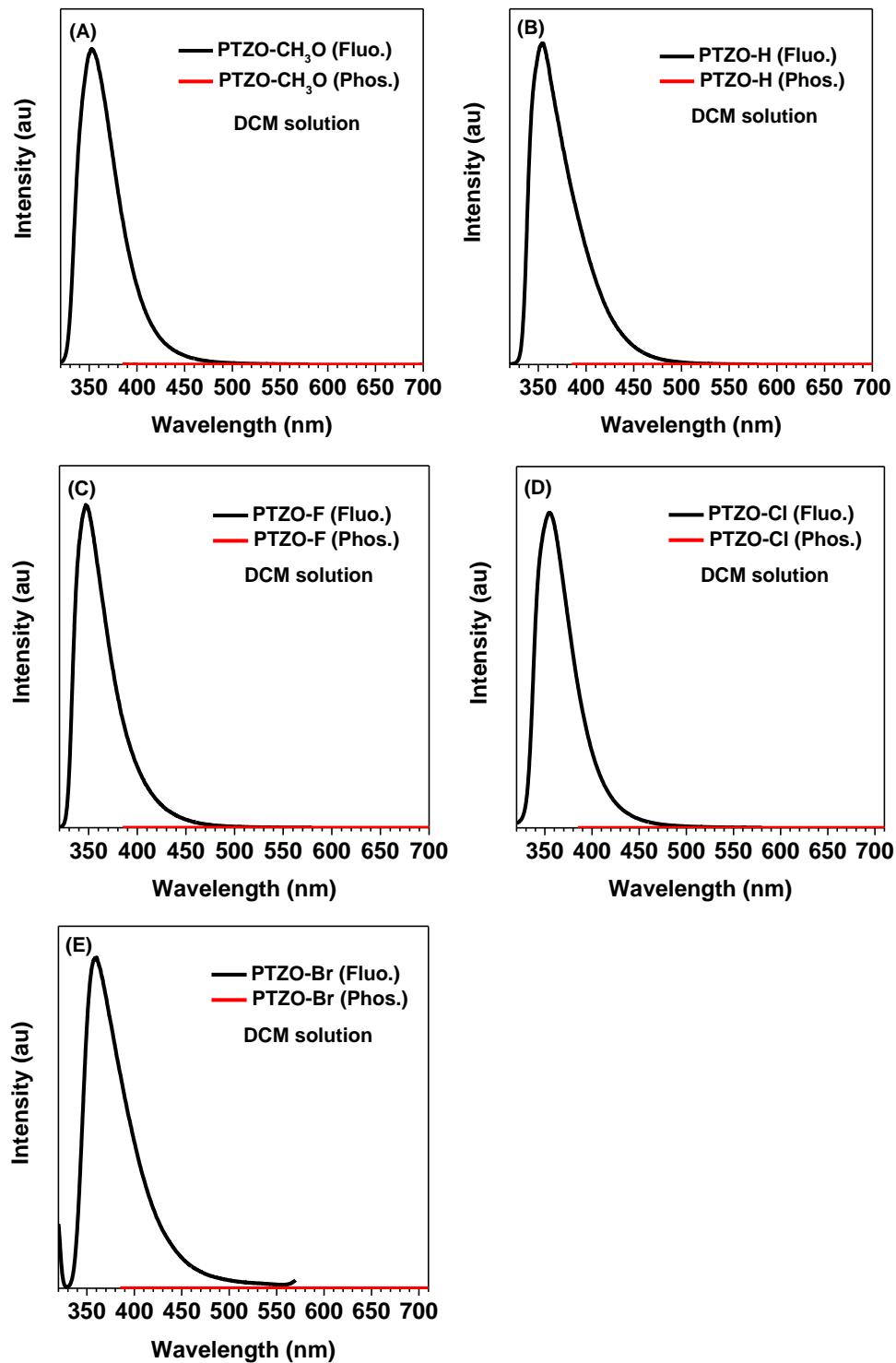


Figure S9. (A) The fluorescence and phosphorescence spectra of PTZO-CH₃O in dichloromethane solution. (B) The fluorescence and phosphorescence spectra of PTZO-H in dichloromethane solution. (C) The fluorescence and phosphorescence spectra of PTZO-F in dichloromethane solution. (D) The fluorescence and phosphorescence spectra of PTZO-Cl in dichloromethane solution. (E) The fluorescence and phosphorescence spectra of PTZO-Br in dichloromethane solution.

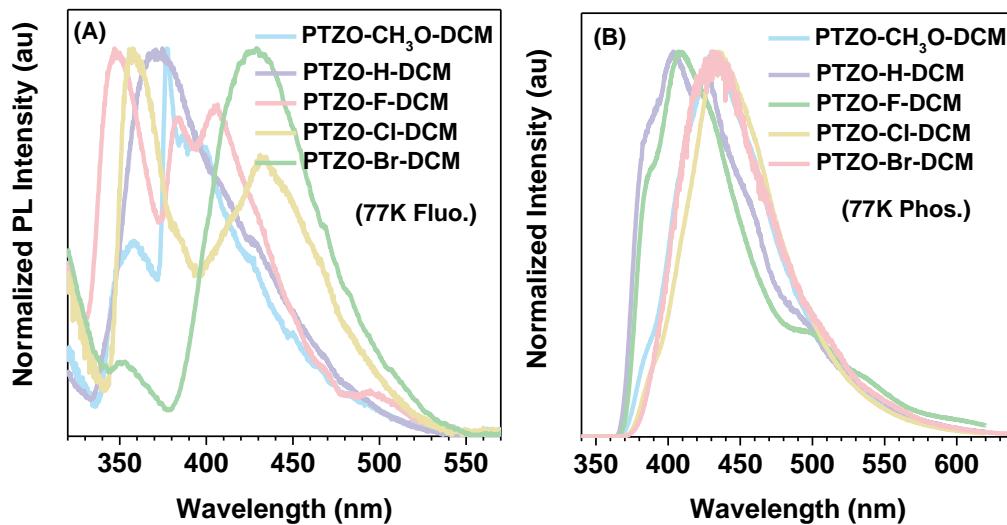


Figure S10. (A) The normalized low temperature (77 K) fluorescence spectra in dichloromethane solution. (B) The normalized low temperature (77 K) phosphorescence spectra in dichloromethane solution.

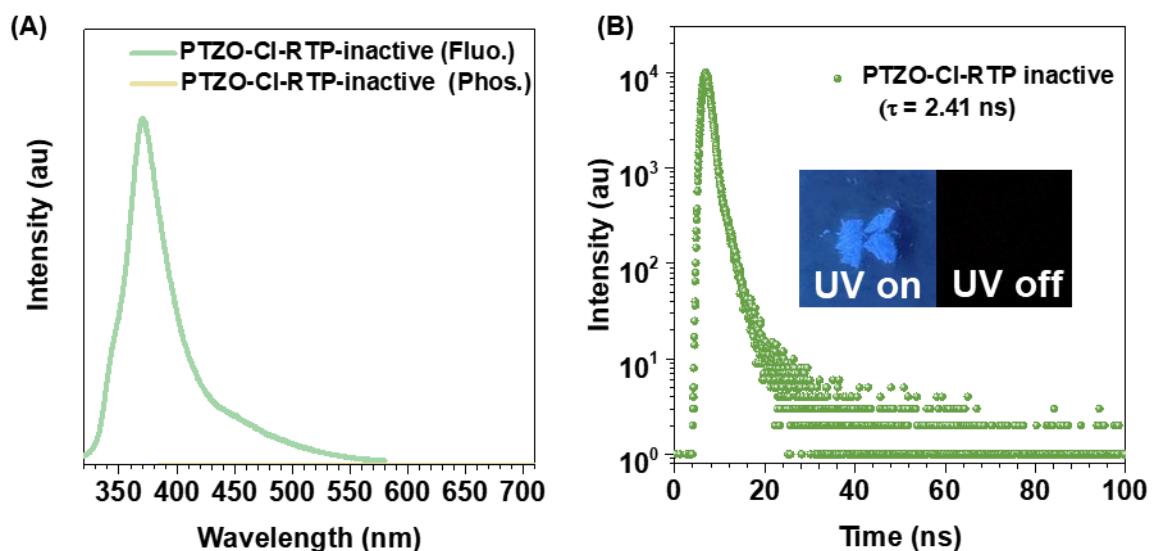


Figure S11. (A) Fluorescence and phosphorescence spectra of PTZO-Cl (RTP-inactive) crystal. (B) Fluorescence decay of PTZO-Cl (RTP-inactive) crystal at room temperature; insets: photos taken before and after irradiation (365 nm) under ambient conditions.

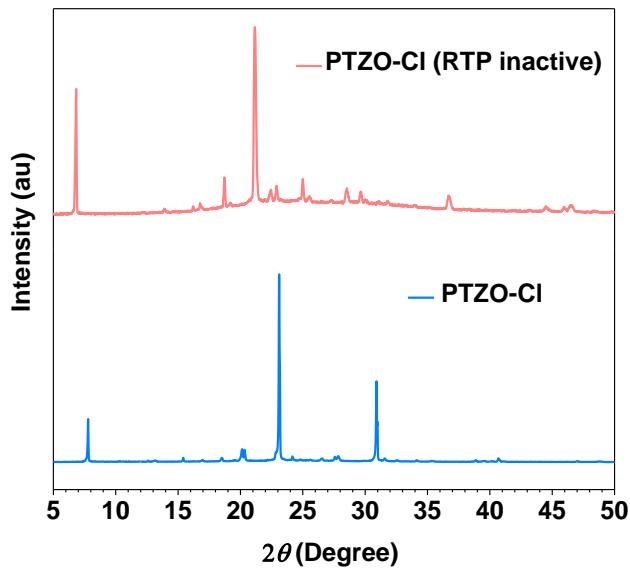


Figure S12. The PXRD patterns of the crystals of PTZO-Cl and PTZO-Cl (RTP inactive) at room temperature.

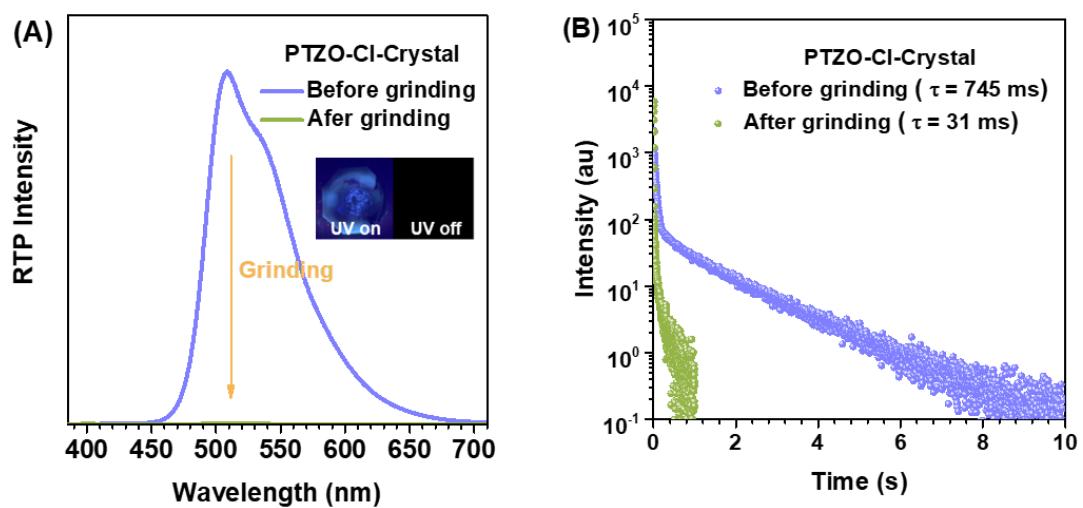


Figure S13. (A) The changed PL spectra of single crystal PTZO-Cl crystal before and after grinding; (B) The changed PL decay curves of single crystal PTZO-Cl crystal before and after grinding.

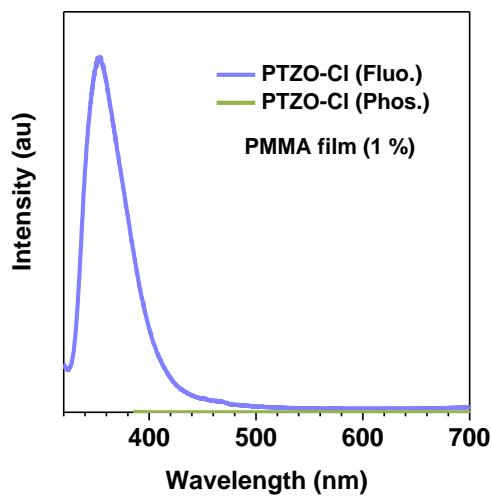


Figure S14. The fluorescence and phosphorescence spectra of PTZO-Cl doped in PMMA film.

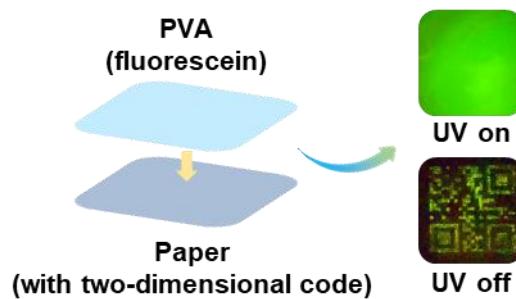


Figure S15. The anti-counterfeiting of two-dimensional code.

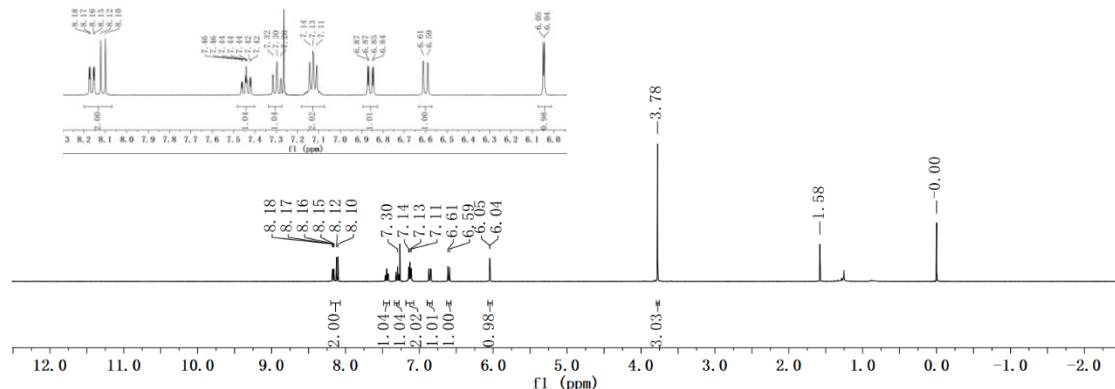


Figure S16. ^1H NMR spectrum of PTZO-CH₃O in CDCl₃.

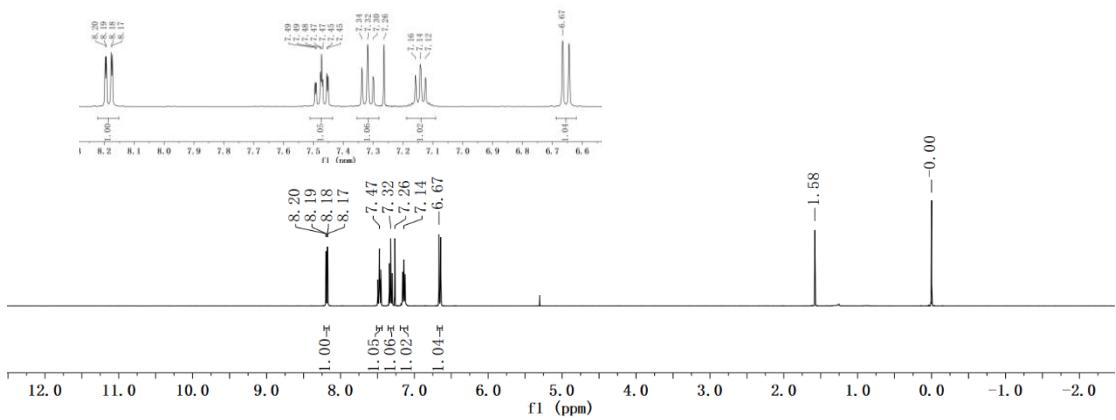


Figure S17. ^1H NMR spectrum of PTZO-H in CDCl_3 .

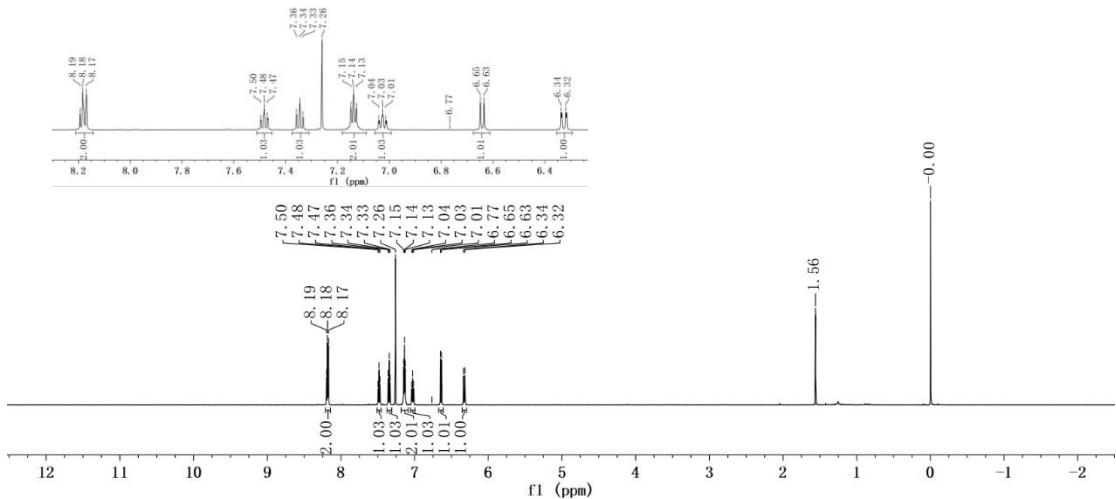


Figure S18. ^1H NMR spectrum of PTZO-F in CDCl_3 .

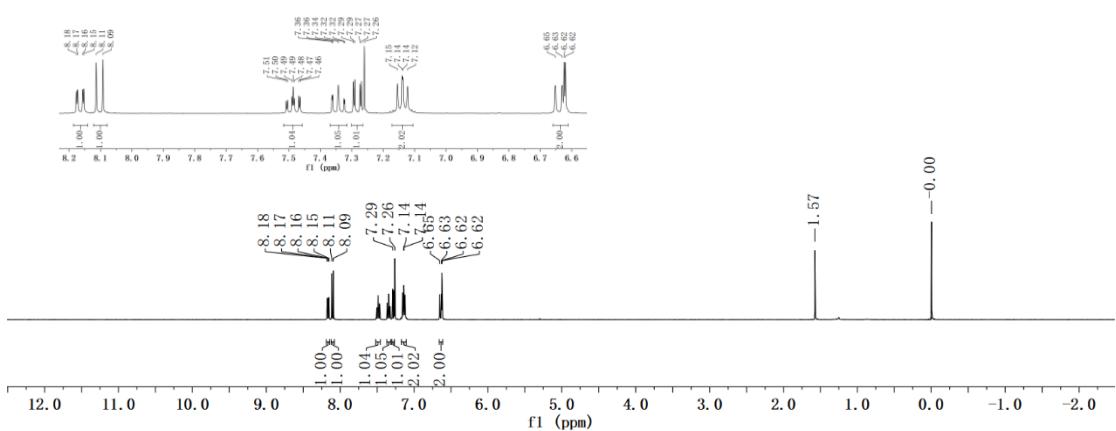


Figure S19. ^1H NMR spectrum of PTZO-Cl in CDCl_3 .

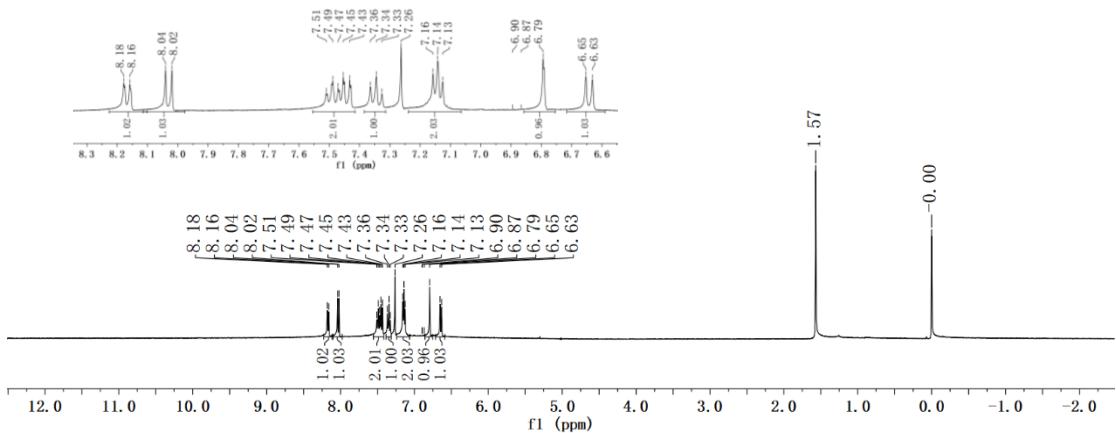


Figure S20. ^1H NMR spectrum of PTZO-Br in CDCl_3 .

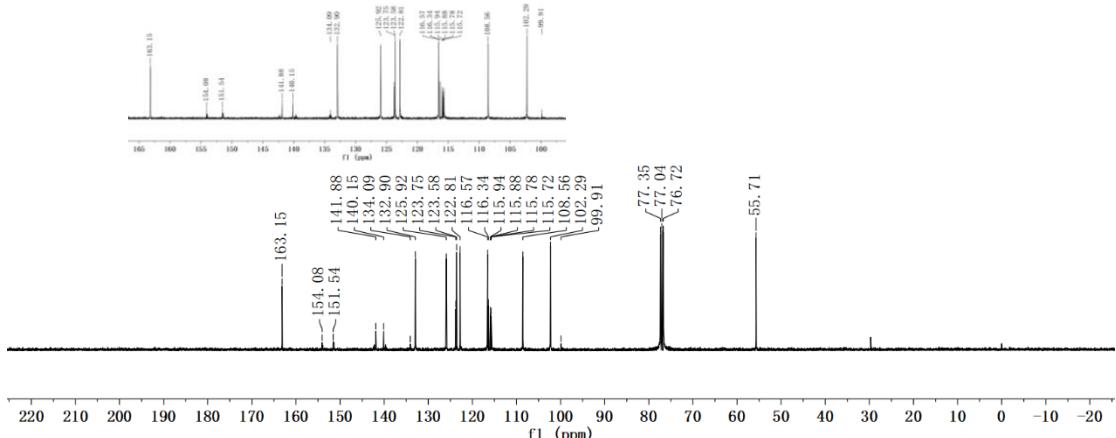


Figure S21. ^{13}C NMR spectrum of PTZO-CH₃O in CDCl₃.

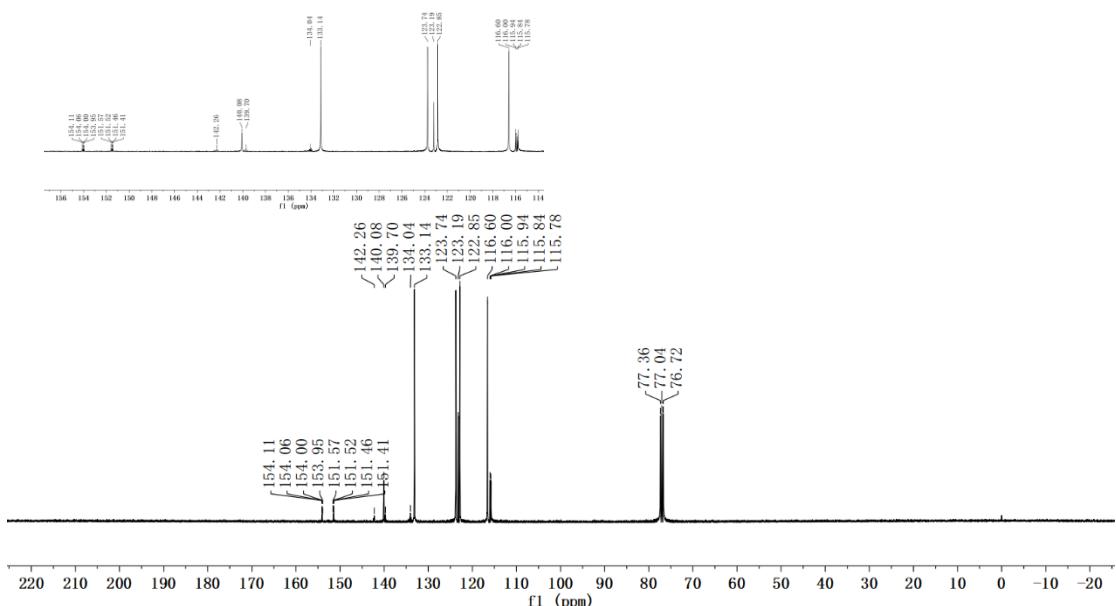


Figure S22. ^{13}C NMR spectrum of PTZO-H in CDCl_3 .

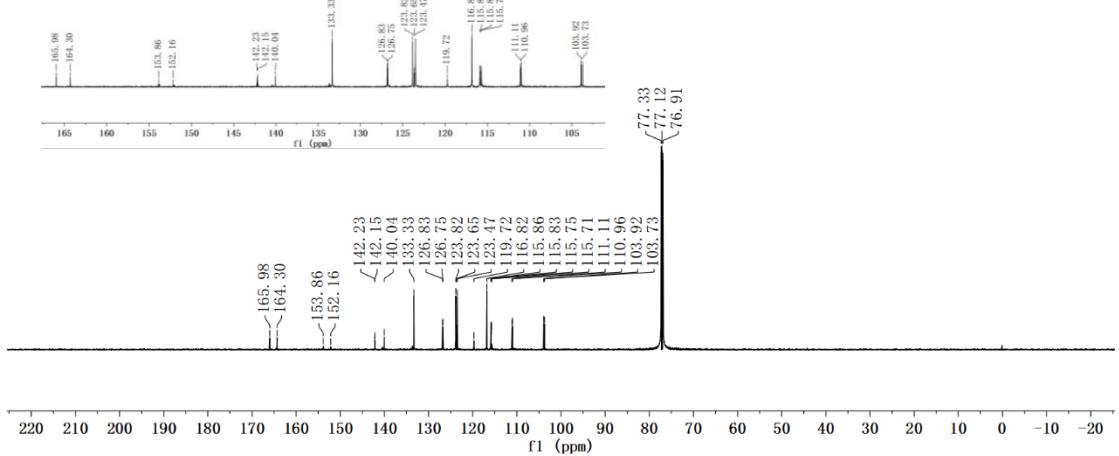


Figure S23. ^{13}C NMR spectrum of PTZO-F in CDCl_3

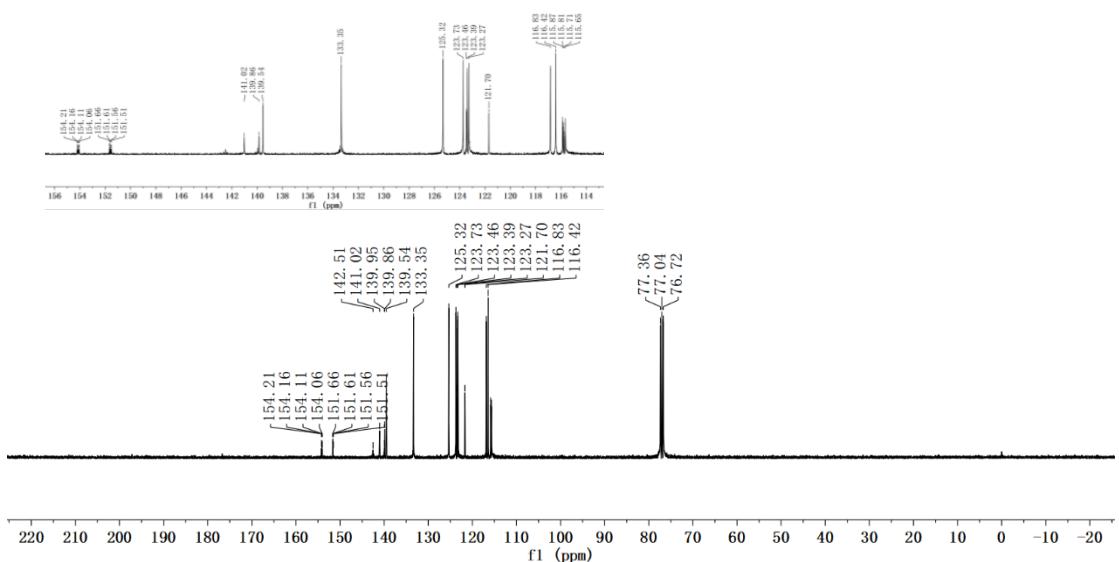


Figure S24. ^{13}C NMR spectrum of PTZO-Cl in CDCl_3 .

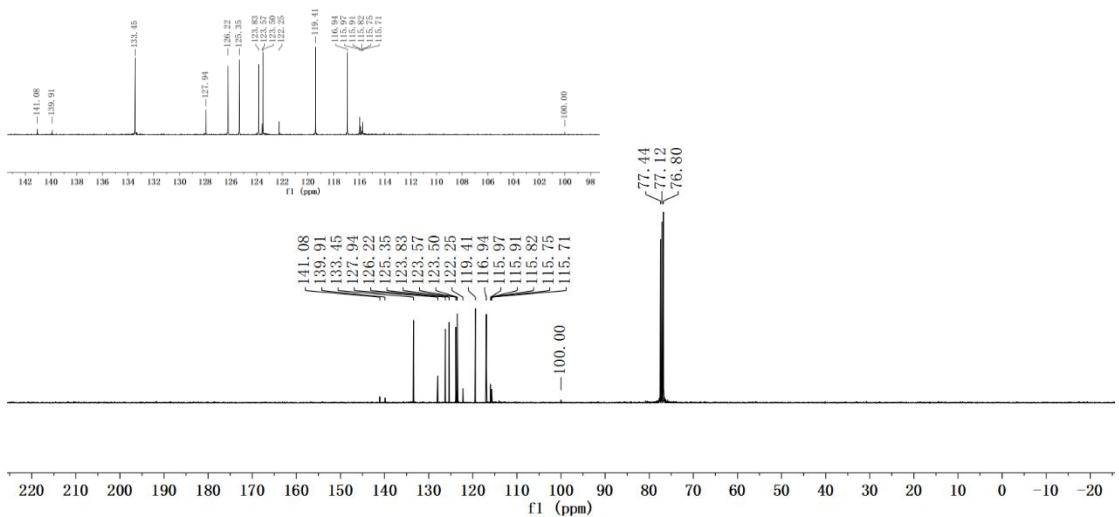


Figure S25. ^{13}C NMR spectrum of PTZO-Br in CDCl_3 .

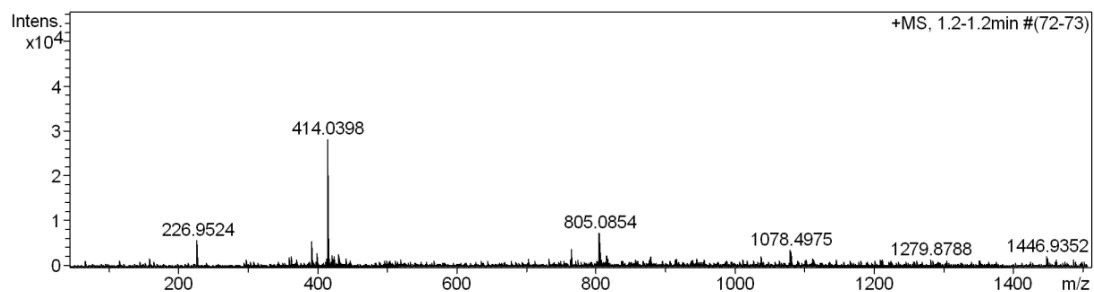


Figure S26. HRMS (FTMS-ESI) spectrum of PTZO- CH_3O .

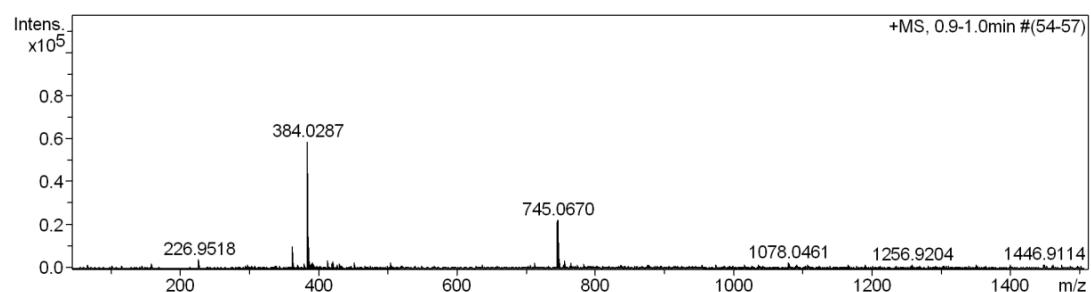


Figure S27. HRMS (FTMS-ESI) spectrum of PTZO-H.

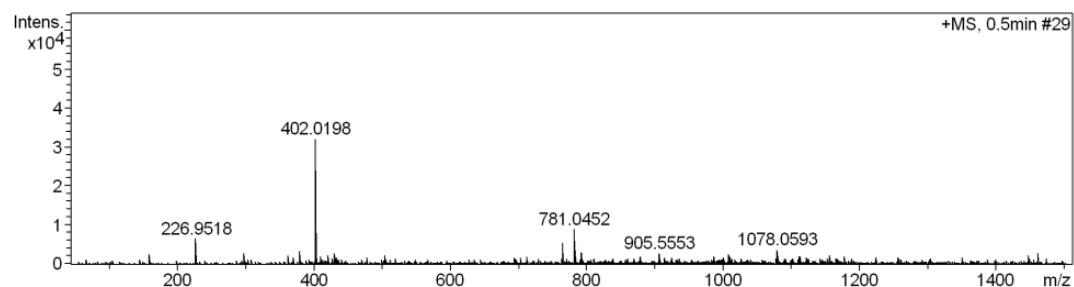


Figure S28. HRMS (FTMS-ESI) spectrum of PTZO-F.

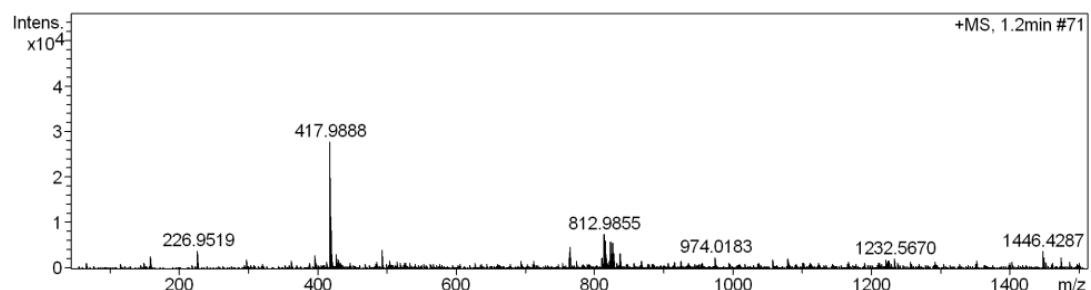


Figure S29. HRMS (FTMS-ESI) spectrum of PTZO-Cl.

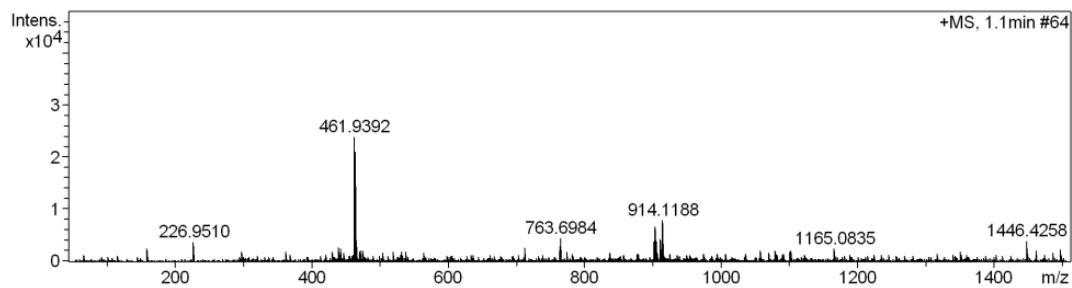


Figure S30. HRMS (FTMS-ESI) spectrum of PTZO-Br.

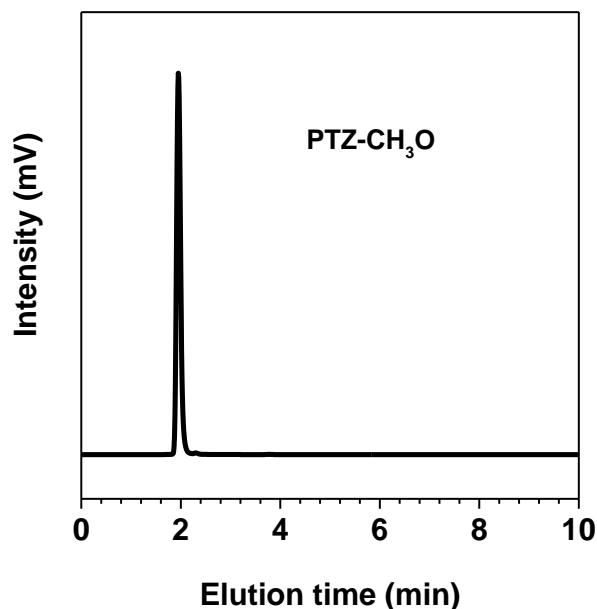


Figure S31. High performance liquid chromatogram (HPLC) spectrum of PTZO-CH₃O.

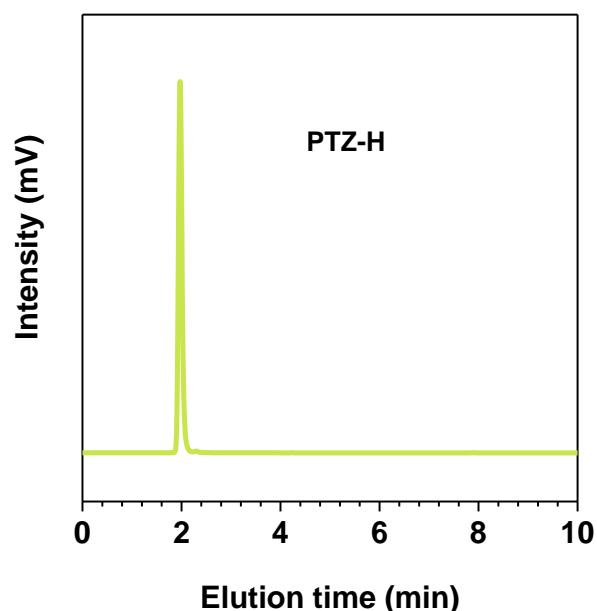


Figure S32. High performance liquid chromatogram (HPLC) spectrum of PTZO-H.

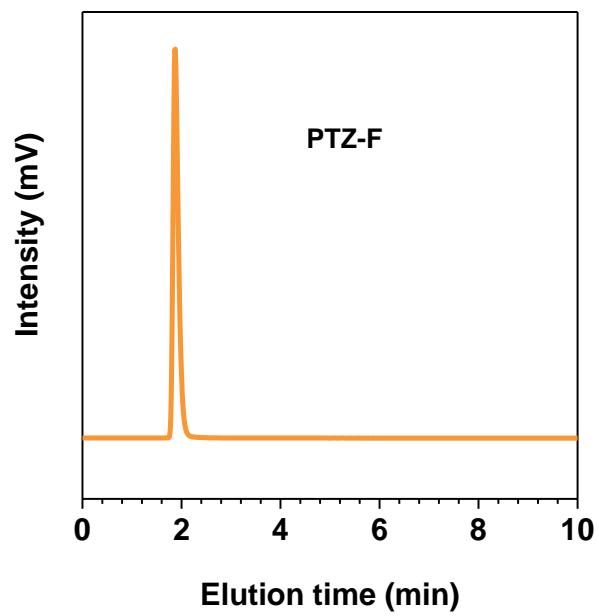


Figure S33. High performance liquid chromatogram (HPLC) spectrum of PTZO-F.

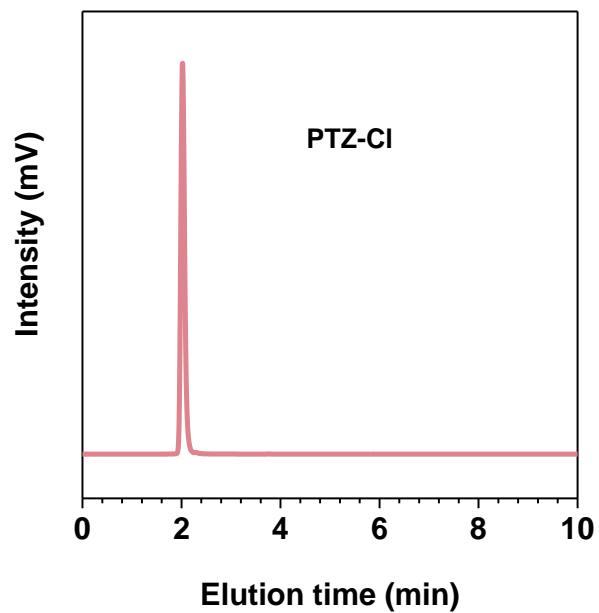


Figure S34. High performance liquid chromatogram (HPLC) spectrum of PTZO-Cl.

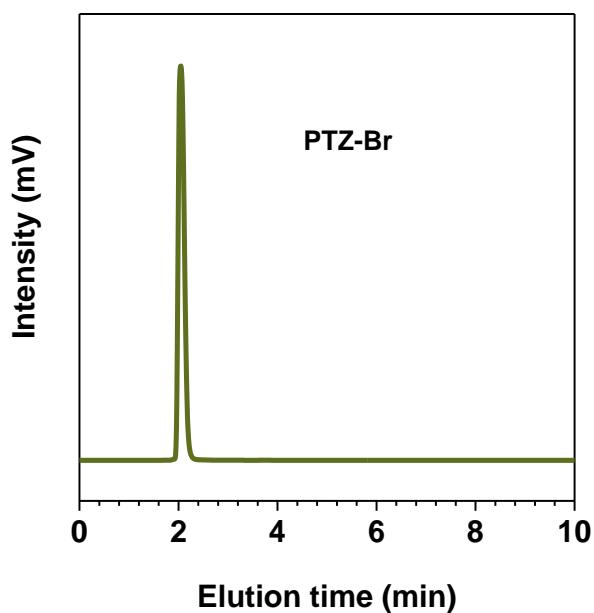


Figure S35. High performance liquid chromatogram (HPLC) spectrum of PTZO-Br.

3. Supplementary References

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