

## Supplementary Information

### **An AIE-based Monofunctional Pt(II) Complex for Photodynamic Therapy through Synergism of Necroptosis-Ferroptosis**

Xiaoxue Zheng<sup>#a</sup>, Minglun Liu<sup>#a</sup>, Yanping Wu<sup>a</sup>, Yuncong Chen<sup>\*a,b</sup>, Weijiang He<sup>\*a</sup>, Zijian Guo<sup>\*a,b</sup>

<sup>a</sup> State Key Laboratory of Coordination Chemistry, School of Chemistry and Chemical Engineering, Chemistry and Biomedicine Innovation Center (ChemBIC), Nanjing University, Nanjing 210023, China

<sup>b</sup> Nanchuang (Jiangsu) Institute of Chemistry and Health, Nanjing 210000, China

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## Supplementary Methods

**Materials and instrument.** All chemicals for compounds synthesis were commercially available (Macklin, Bidepharm, Shandong Boyuan Pharmaceutical Co) and used directly without further purification. The silica gel (300-400 mesh) was used for column chromatography. High-resolution mass spectra (HR-MS) were recorded on an Agilent 6540Q-TOF HPLC-MS spectrometer. The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were determined on a Bruker DRX-400/-500 MHz spectrometer. The purity was detected using Thermo ultimate 3000 UHPLC. The dynamic light scattering (DLS) data were characterized by Zetasizer Nano ZS-90. The transmission electron microscopy (TEM) images were taken using a JEOL-JEM 2100 F transmission electron microscope (Japan). UV-visible absorption spectra were measured on a PerkinElmer Lambda 35 spectrophotometer. Emission spectra were measured on a FluoroMax-4 (Horiba). Fluorescence confocal imaging was carried out on a laser scanning confocal imaging system (Olympus TH4-200) consisting of ZEISS Laser Scanning Microscope (LSM 710) and a 20-mW output 488 nm argon-ion laser. ICP-MS was measured by PerkinElmer PQEXCell. The images of photocytotoxicity in MCSs were taken by OLYMPUS IX71 inverted microscope (external power TH4-200; fluorescent source X-120Q).

**Cell lines.** The human cervical cancer cell line HeLa were purchased from Stem Cell Bank, Chinese Academy of Science (Shanghai, China) and cultured in Dulbecco's Modified Eagle Medium (DMEM, KeyGEN) supplemented with 10% fetal bovine serum (FBS, HyClone) and 1% antibiotics, respectively, with 21%  $\text{O}_2$  and 5%  $\text{CO}_2$  at 37 °C. Before an experiment, the cells were passaged at least three times.

**The purity of TTC-Pt.** TTC-Pt was dissolved in acetonitrile solution containing 0.5% DMSO at 100  $\mu\text{M}$ . HPLC analyses were performed on a thermos ultimate3000 equipped with an ACE Excel 5 reverse phase column. The sample were injected directly into the instrument and eluted by a linear gradient (eluent A, acetonitrile; eluent B,  $\text{H}_2\text{O}$ ). Among them, 0-2 mins was 95% eluent B, 2-35 mins 95-5%, and 35-45 mins 5%. The detection wavelength is 254 nm.

**UV-vis and fluorescence spectroscopic study.** The test methods are consistent with the previously reported<sup>1</sup>. The stock solutions of TTC and TTC-Pt (1 mM) were prepared with DMSO and stored at 4 °C in a refrigerator. The absorption and fluorescence spectra of TTC (10  $\mu\text{M}$ ) and TTC-Pt (10  $\mu\text{M}$ ) were determined in water containing 1% DMSO or in pure DMSO solution at room temperature.

**The stability of TTC-Pt and TTC.** The stock solutions of TTC and TTC-Pt (1 mM) were prepared with DMSO and stored at 4 °C in a refrigerator. The absorption of TTC (10  $\mu\text{M}$ ) and TTC-Pt (10  $\mu\text{M}$ ) were determined in water containing 1% DMSO at room temperature for 10 h in the dark.

**Singlet oxygen detection in solution.** The test methods are consistent with the previously reported<sup>1</sup>. ABDA (9,10-Anthracenediyl-bis(methylene)-dimalonic acid) was used as a singlet oxygen indicator, and its decomposition rate determined the singlet oxygen yield. With RB (Rose Bengal, 10  $\mu\text{M}$ ) or  $\text{Ru}(\text{bpy})_3\text{Cl}_2$  (10  $\mu\text{M}$ ) as the

internal reference, the absorbance of the solution mixed with ABDA (83  $\mu\text{M}$ ) and AIEgens (10  $\mu\text{M}$ ) in DMSO/water (1:99, v/v) at 398 nm was measured over time during exposed to 450 nm laser irradiation (25 mW  $\text{cm}^{-2}$ ).

**Type I ROS evaluation.** The test methods are consistent with the previously reported<sup>1</sup>. Hydroxyl radical probe (hydroxyphenyl fluorescein, HPF, 5 $\mu\text{M}$ ,  $\lambda_{\text{ex}} = 490$  nm,  $\lambda_{\text{em}} = 515$  nm) and hydroxyl radical probe (dihydrorhodamine 123, DHR123, 5 $\mu\text{M}$ ,  $\lambda_{\text{ex}} = 488$  nm,  $\lambda_{\text{em}} = 525$  nm) were used to test whether TTC-Pt and TTC (1  $\mu\text{M}$ ) have the ability to produce type I ROS under 450 nm laser irradiation (25 mW  $\text{cm}^{-2}$ ).

**Cellular uptake.** For TTC-Pt and TTC, the AIEgens (10  $\mu\text{M}$ ) were incubated with HeLa cells respectively and the fluorescence imaging was performed using a confocal microscope with 63 $\times$  objective lens ( $\lambda_{\text{ex}} = 488$  nm,  $\lambda_{\text{em}} = 600\text{-}700$  nm). For TTC-Pt and CDDP, after the compounds were incubated with HeLa cells respectively, the cells were collected and washed three times with 1 $\times$ PBS. Then the cells were digested at 95  $^{\circ}\text{C}$  with nitric acid (100  $\mu\text{L}$ ) for two hours and a half,  $\text{H}_2\text{O}_2$  (50  $\mu\text{L}$ ) for half hour and concentrated HCl (100  $\mu\text{L}$ ) in sequence until total volume was less than 20  $\mu\text{L}$ . Followed, water was added to dilute the solution to 1 mL, and the Pt levels were measured by ICP-MS. Pt levels was normalized by intracellular protein concentration.

**Cellular uptake pathways.** The test methods are consistent with the previously reported<sup>1, 2</sup>. The cell internalization pathways of the AIEgens were investigated by different methods and blockers, including metabolic inhibitors (2-deoxy-Dglucose, 50 mM; and oligomycin, 5  $\mu\text{M}$ , at 37  $^{\circ}\text{C}$ ), endocytic inhibitor ( $\text{NH}_4\text{Cl}$ , 50 mM, at 37  $^{\circ}\text{C}$ ), ion channel inhibitor (TEA, 1 mM, at 37  $^{\circ}\text{C}$ ) and transmembrane protein inhibition (none any blockers at 4  $^{\circ}\text{C}$ ). As First, the HeLa cells were preincubated in DMEM for 1 h at 37  $^{\circ}\text{C}$  with different pathway inhibitors or without any inhibitors at 4  $^{\circ}\text{C}$ , then the medium was replaced with the inhibitors and different AIEgens (10  $\mu\text{M}$ ) including TTC-Pt and TTC at 37  $^{\circ}\text{C}$  or 4  $^{\circ}\text{C}$ . After 3 h, fluorescence imaging of AIEgens was performed using a confocal microscope with 63 $\times$  objective lens ( $\lambda_{\text{ex}} = 488$  nm,  $\lambda_{\text{em}} = 600\text{-}700$  nm).

**Colocalization of TTC-Pt and TTC with MitoTracker<sup>TM</sup> Blue in HeLa cells.** HeLa cells were co-incubated with TTC-Pt or TTC (1  $\mu\text{M}$ ) at 37  $^{\circ}\text{C}$  for 5 h and then washed three times with 1 $\times$ PBS. Subsequently, the cells were incubated with MitoTracker<sup>TM</sup> Blue in DMEM without FBS for 1 h at 37  $^{\circ}\text{C}$  and washed three times with 1 $\times$ PBS. Finally, the cells were imaged by CLSM immediately. And the imaging band path for TTC-Pt and TTC was 600-700 nm, and the related excitation wavelength was 488 nm, as well as MitoTracker<sup>TM</sup> Blue was 410-480 nm, respectively, 405 nm.

**Colocalization of TTC-Pt and TTC with LysoTracker<sup>TM</sup> Deep Red in HeLa cells.** HeLa cells were co-incubated with TTC-Pt or TTC (1  $\mu\text{M}$ ) at 37  $^{\circ}\text{C}$  for 5 h and then washed three times with 1 $\times$ PBS. Subsequently, the cells were incubated with LysoTracker<sup>TM</sup> Deep Red in DMEM without FBS for 40 min at 37  $^{\circ}\text{C}$  and washed three times with 1 $\times$ PBS. Finally, the cells were imaged by CLSM immediately. And the imaging band path for TTC-Pt and TTC was 600-700 nm, and the related

excitation wavelength was 488 nm, as well as LysoTracker™ Deep Red was 650-680 nm, respectively, 633 nm.

**Colocalization of TTC-Pt and TTC with ER-Tracker™ Blue in HeLa cells.** HeLa cells were co-incubated with TTC-Pt or TTC (1  $\mu$ M) at 37 °C for 5 h and then washed three times with 1 $\times$ PBS. Subsequently, the cells were incubated with ER-Tracker™ Blue in DMEM without FBS for 40 min at 37 °C and washed three times with 1 $\times$ PBS. Finally, the cells were imaged by CLSM immediately. And the imaging band path for TTC-Pt and TTC was 600-700 nm, and the related excitation wavelength was 488 nm, as well as ER-Tracker™ Blue was 455-520 nm, respectively, 405 nm.

**Colocalization of TTC-Pt with LD-Tracker™ Green in HeLa cells.** HeLa cells were co-incubated with TTC-Pt (1  $\mu$ M) at 37 °C for 5 h and then washed three times with 1 $\times$ PBS. Subsequently, the cells were incubated with LD-Tracker™ Green in DMEM without FBS for 40 min at 37 °C and washed three times with 1 $\times$ PBS. Finally, the cells were imaged by CLSM immediately. And the imaging band path for TTC-Pt was 600-700 nm, and the related excitation wavelength was 488 nm, as well as LD-Tracker™ Green was 495-535 nm, respectively, 488 nm.

**Colocalization of TTC with LD-Tracker™ Deep Red in HeLa cells.** HeLa cells were co-incubated with TTC (1  $\mu$ M) at 37 °C for 5 h and then washed three times with 1 $\times$ PBS. Subsequently, the cells were incubated with LD-Tracker™ Deep Red in DMEM without FBS for 30 min at 37 °C and washed three times with 1 $\times$ PBS. Finally, the cells were imaged by CLSM immediately. And the imaging band path for TTC was 600-700 nm, and the related excitation wavelength was 488 nm, as well as LD-Tracker™ Deep Red was 650-690 nm, respectively, 633 nm.

**Intracellular ROS detection.** The test methods are consistent with the previously reported<sup>1</sup>. 2',7'-Dichlorodihydrofluorescein diacetate (DCFH-DA) was used as ROS indicator to determine the capability of TTC-Pt, TTC and CDDP to produce intracellular ROS. The HeLa cancer cells were cultured in the special confocal chambers and incubated with various drugs including TTC (0.5  $\mu$ M), TTC-Pt (0.5  $\mu$ M) or CDDP (0.5  $\mu$ M) for 5 h at 37 °C. Subsequently, the cells were washed three times with 1 $\times$ PBS, followed by addition of 2',7'-Dichlorodihydrofluorescein diacetate (DCFH-DA, 10  $\mu$ M) in DMEM without FBS and incubation for half an hour. The above-mentioned process was performed in dark. Then, the cells were exposed to 450 nm laser irradiation (100 mW cm<sup>-2</sup>) for 1 min, and fluorescently imaged by CLSM ( $\lambda_{ex}$  = 488 nm,  $\lambda_{em}$  = 500-550 nm) after three times washes with PBS.

**Cytotoxicity Study.** The test methods are consistent with the previously reported<sup>1</sup>. First, 100  $\mu$ L microliters of HeLa cell suspension was added into each well of 96-well plate with a density of 4 $\times$ 10<sup>4</sup> cells mL<sup>-1</sup>. When the cell density reached 80%, fresh culture medium (the concentration of DMSO lower than 0.5%) containing a series of concentrations of compounds (TTC, TTC-Pt, CDDP and TTC+CDDP) were added, respectively, and incubated with the HeLa cells for 5 h at 37 °C in dark. Subsequently, the cells were illuminated at 450 nm (100 mW cm<sup>-2</sup>) for 2 min and then continued to incubated in the dark for 24 h. Therewith, the wells were replaced with freshly

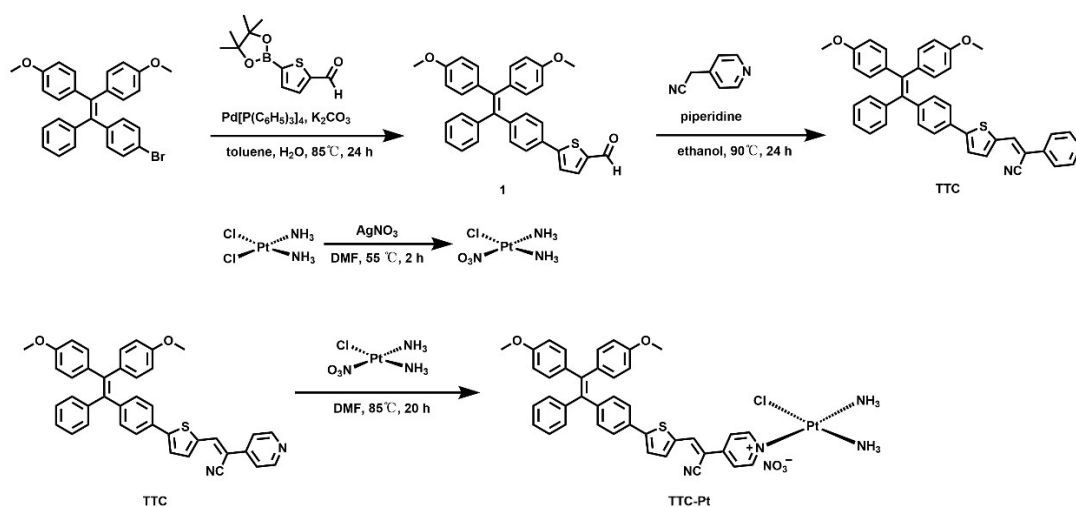
prepared 3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyl tetrazolium bromide (MTT, 2.5 mg mL<sup>-1</sup> in PBS) solution and the cells were incubated for 4 h. After that, the solution in each well was carefully removed and 150  $\mu$ L of DMSO was added to each well to dissolve the formazan. The plate was gently shaken for 10 min at room temperature and then the absorbance of MTT at 570 nm was monitored by the microplate reader (Thermo Scientific Varioskan Flash) in order to determine the cell viability. Cell viability rates (%) and IC<sub>50</sub> values were calculated on the data of three parallel tests.

**Lipid peroxidation detection in cells treated by TTC-Pt.** The test methods are consistent with the previously reported<sup>2</sup>. C11-BODIPY<sup>581/591</sup> acted as a lipid peroxidation probe to monitor the degree of intracellular lipid peroxidation. The HeLa cells were incubated with TTC-Pt (1  $\mu$ M) at 37 °C for 5 h and then washed three times with 1 $\times$ PBS. Subsequently, the cells were incubated with C11-BODIPY<sup>581/591</sup> (10  $\mu$ M) for 30 min in DMEM without FBS. After that, cells were illuminated with a laser of 450 nm (100 mW cm<sup>-2</sup>). Then the cells were imaged by CLSM ( $\lambda_{\text{ex}}$  = 488 nm,  $\lambda_{\text{em}}$  = 500-550 nm).

**Western Blot Analysis for HeLa cells treated by AIEgens and CDDP.** HeLa cells were seeded in 6 cm petri dish and incubated with TTC-Pt, TTC and CDDP (1  $\mu$ M) respectively. After 5 h of incubation in the dark, the cells of light groups were illuminated with a laser of 450 nm (100 mW cm<sup>-2</sup>) for 5 min. Then, after 5 h, cells were extracted with a cell scraper and washed three times with 1 $\times$ TBS. The cells were lysed by cell lysates containing protease inhibitors and phosphatase inhibitors on ice for 30 mins. After centrifugation at 4 °C, 12000 r for 20 mins, the upper protein extract concentration was measured by Bradford assay and uniformly quantified. The equal amounts of protein were added to each lane of SDS-PAGE gel for electrophoresis, and then transferred to the polyvinylidene fluoride (PVDF) membranes. After the protein was sealed with 5% BSA solution, the membranes were applied with the corresponding monoantibody ( $\beta$ -Actin: 4970T, Cell Signaling; FSP1: 20886-1-AP, proteintech; GPX4: ab125066, abcam) and kept at 4°C overnight. The next day, incubate with the second antibodies (ab205718, abcam) at room temperature for 1 h and expose.

**3D multicellular spheroids (MCSs) culture and related study.** The test methods are consistent with the previously reported<sup>2</sup>. First, 100  $\mu$ L microliters of HeLa cell suspension was added into each well of ultra-low attachment microplate 96-well round bottom (Corning) with a density of 2 $\times$ 10<sup>4</sup> cells mL<sup>-1</sup>. After 24 h, the HeLa MCSs were divided to different groups and cultured in media containing TTC, TTC-Pt, CDDP and TTC+CDDP (5  $\mu$ M) respectively. The medium containing the corresponding drug was changed every two days, and the cells was illuminated with a laser of 450 nm (100 mW cm<sup>-2</sup>, 2 min) after the change of 24 h. The MCSs were photographed with an inverted fluorescence microscope before each medium change.

## Synthesis and Characterization Parts



**Scheme S1.** Syntheses routes of TTC and TTC-Pt.

### Syntheses of compound 1.

The test methods are consistent with the previously reported<sup>1</sup>. Under N<sub>2</sub> atmosphere, 5-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-2-thiophenecarboxaldehyde (1.01 g, 4.08 mmol), 1,1'-[2-(4-Bromophenyl)-2-phenylethenylidene]bis[4-methoxybenzene] (0.40 g, 0.85 mmol), potassium carbonate (6.21 g) and Tetrakis(triphenylphosphine)palladium (0.200 g, 0.165 mmol) were added to a solution with toluene (90 mL) and water (18 mL), and then the mixture was heated to 85 °C for over 24 hours. After the reaction was completed, the mixture was cooled to room temperature and the solvent was removed. The resulting residue was extracted with dichloromethane and the crude product was purified by column chromatography using petroleum ether and DCM (1/1, R<sub>f</sub> = 0.3) as the eluent to give a brown solid (0.18 g, 42.2%). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 9.86 (s, 1H), 7.70 (d, J = 4.0 Hz, 1H), 7.42 (d, J = 8.4 Hz, 2H), 7.34 (d, J = 4.0 Hz, 1H), 7.12 (t, J = 7.4 Hz, 3H), 7.07 (d, J = 8.4 Hz, 2H), 7.04 (dd, J = 7.7, 1.6 Hz, 2H), 6.98 (d, J = 8.7 Hz, 2H), 6.94 (d, J = 8.7 Hz, 2H), 6.70-6.62 (m, 4H), 3.74 (d, J = 4.6 Hz, 6H).

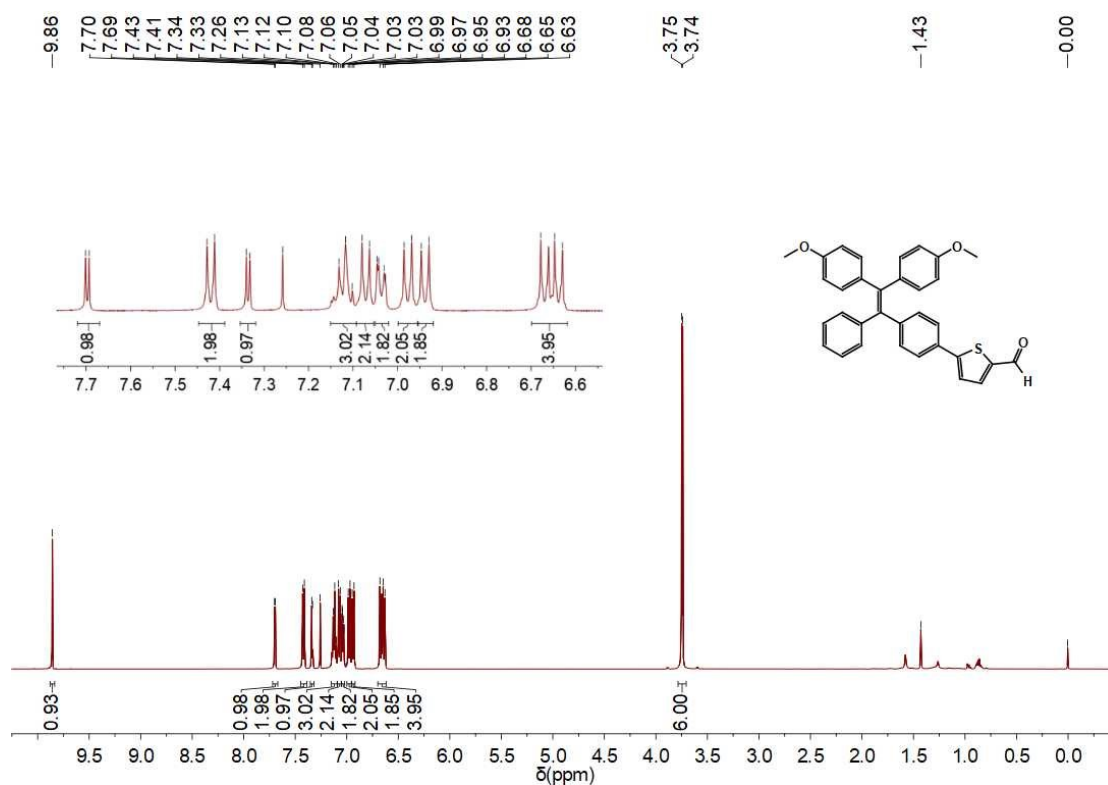
### Syntheses of compound TTC.

The test methods are consistent with the previously reported<sup>1</sup>. Compound 1 (180 mg, 0.358 mmol) and 2-(pyridin-4-yl)acetonitrile (126.9 mg, 1.07 mmol) were added to a 100 mL flask under N<sub>2</sub> atmosphere, then 50 mL ethanol and 0.3 mL piperidine were added. The mixture was heated to 85 °C and stirred for 24 hours. After the reaction was completed, the mixture was cooled to room temperature and the solvent was removed. Further separation using column to separate the crude products with DCM (R<sub>f</sub> = 0.4) as the eluent to give a red solid (99.8 mg, 46.2%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.67 (s, 2H), 7.85 (s, 1H), 7.66 (d, J = 4.0 Hz, 1H), 7.60 (d, J = 4.1 Hz, 2H), 7.44 (d, J = 8.5 Hz, 2H), 7.34 (d, J = 4.0 Hz, 1H), 7.17-7.10 (m, 3H), 7.10-7.02 (m, 4H), 7.01-6.92 (m, 4H), 6.71-6.61 (m, 4H), 3.75 (d, J = 6.7 Hz, 6H).

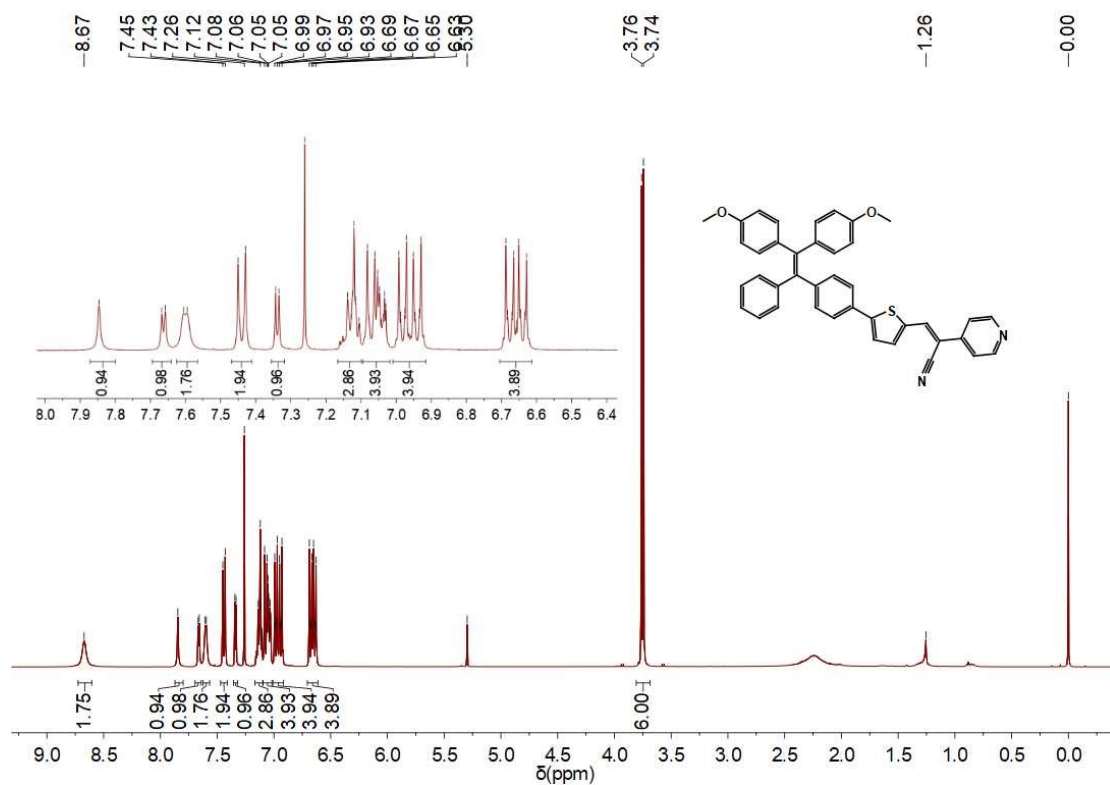
### Syntheses of compound TTC-Pt.

The test methods are consistent with the previously reported<sup>3</sup>. A mixture of cisplatin (30.00 mg, 0.1 mmol), AgNO<sub>3</sub> (13.60 mg, 0.08 mmol) and anhydrous DMF (2 mL)

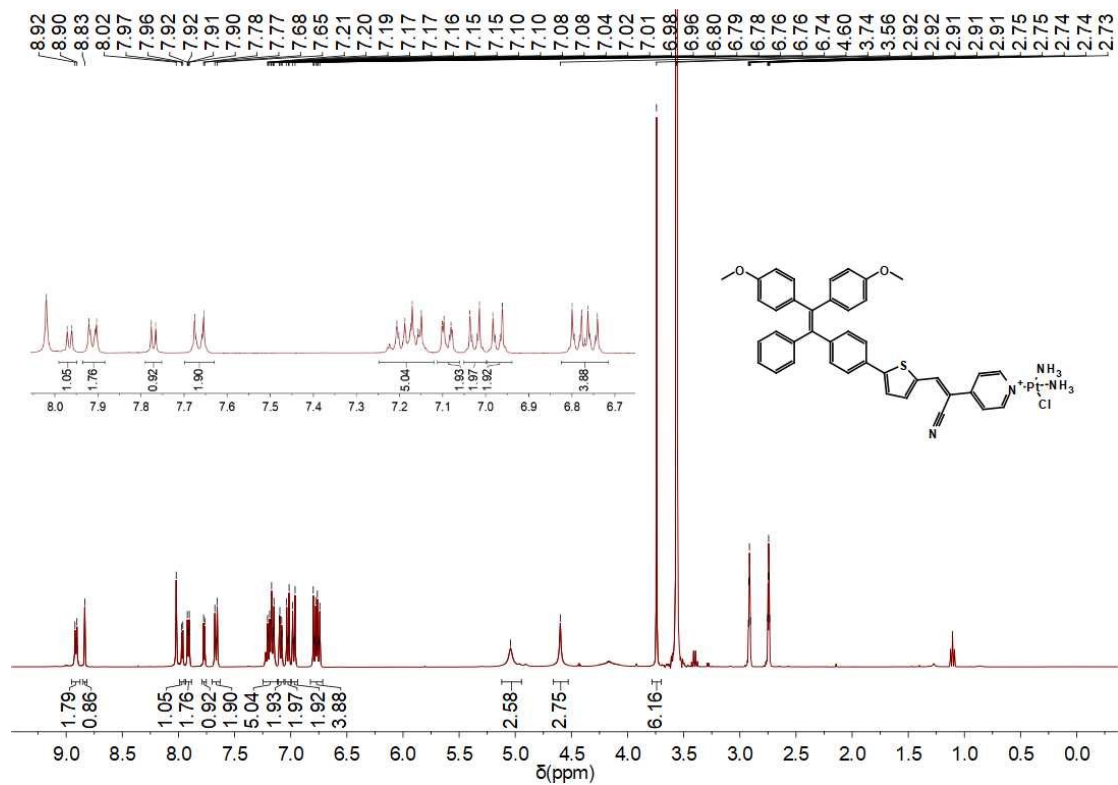
was added into a 10 mL flask. The mixture was heated to 55 °C for 2 h. After the reaction was completed, the mixture was centrifuged and washed with anhydrous DMF to collect the clear upper solution which contained cisplatin-NO<sub>3</sub> compound. The collected cisplatin-NO<sub>3</sub> solution and TTC (48.2 mg, 0.08 mmol) was added into a 10 mL flask under N<sub>2</sub> atmosphere. The mixture was heated to 55 °C and reacted for 20 h. When the reaction was finished, the solvent was removed by rotary evaporator. The residue was dissolved in methanol and filtered to collect the clear solution. The solution was added in ethyl ether dropwise for precipitation to yield the product TTC-Pt as orange solid (15 mg, yield 20.2 %). <sup>1</sup>H NMR (400 MHz, DMF-*d*<sub>7</sub>) δ 8.91 (d, J = 7.0 Hz, 2H), 8.83 (s, 1H), 7.97 (d, J = 4.2 Hz, 1H), 7.91 (dd, J = 5.6, 1.5 Hz, 2H), 7.77 (d, J = 4.0 Hz, 1H), 7.70 – 7.63 (m, 2H), 7.18 (ddd, J = 8.5, 5.3, 1.8 Hz, 5H), 7.11–7.06 (m, 2H), 7.05–7.00 (m, 2H), 7.00–6.94 (m, 2H), 6.82–6.72 (m, 4H), 5.04 (s, 3H), 4.60 (s, 3H), 3.74 (s, 6H). <sup>13</sup>C NMR (101 MHz, DMF-*d*<sub>7</sub>) δ 162.60, 158.84, 158.70, 153.61, 152.01, 146.15, 144.22, 144.17, 141.66, 140.96, 139.81, 138.69, 136.45, 136.18, 136.16, 132.67, 132.61, 132.40, 131.39, 130.79, 128.22, 126.75, 125.82, 125.24, 121.70, 117.11, 113.58, 113.41, 101.88, 55.06, 55.04. HR-MS (ESI, positive mode, m/z): calcd for C<sub>40</sub>H<sub>36</sub>ClN<sub>4</sub>O<sub>2</sub>PtS<sup>+</sup> [M]<sup>+</sup>: 866.1890, found: 866.1873.



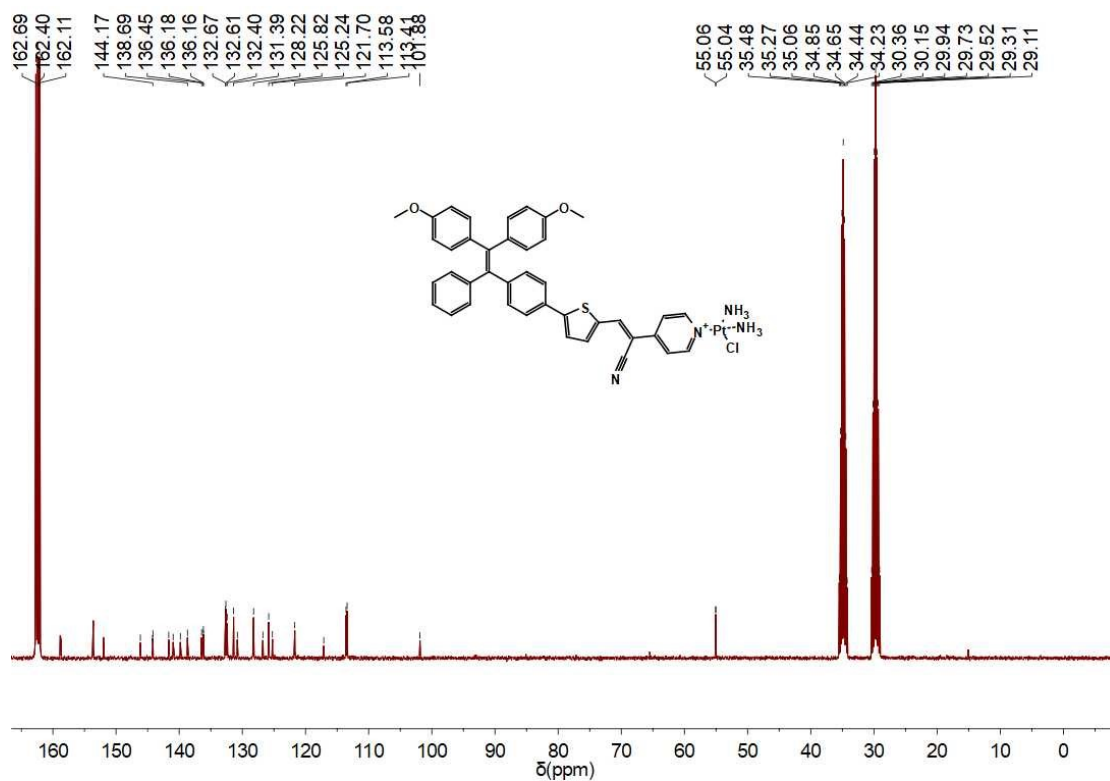
**Supplementary Figure 1.** <sup>1</sup>H NMR spectra of compound 1 in CDCl<sub>3</sub>.



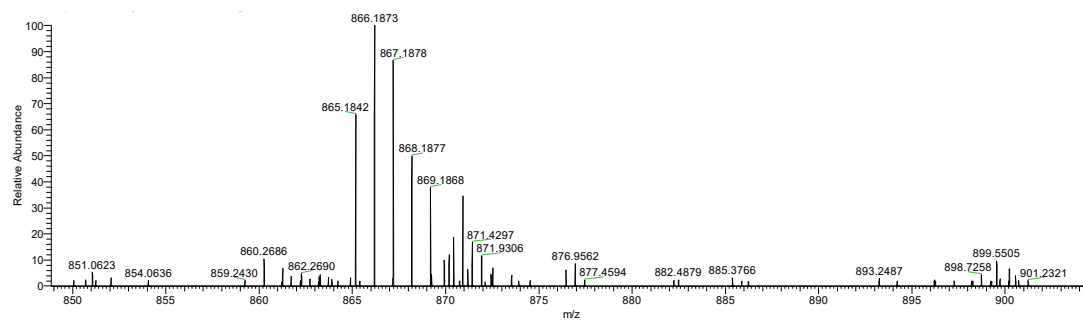
**Supplementary Figure 2. <sup>1</sup>H NMR spectra of compound TTC in CDCl<sub>3</sub>.**



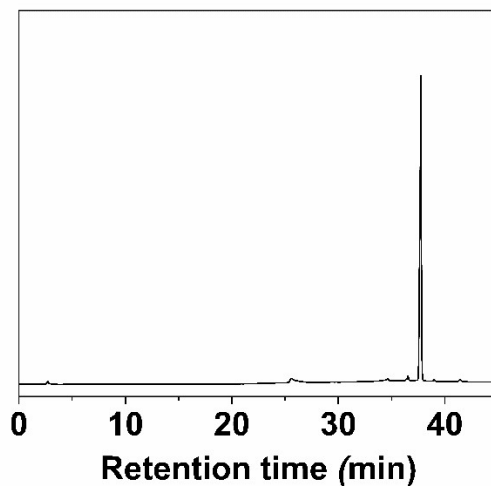




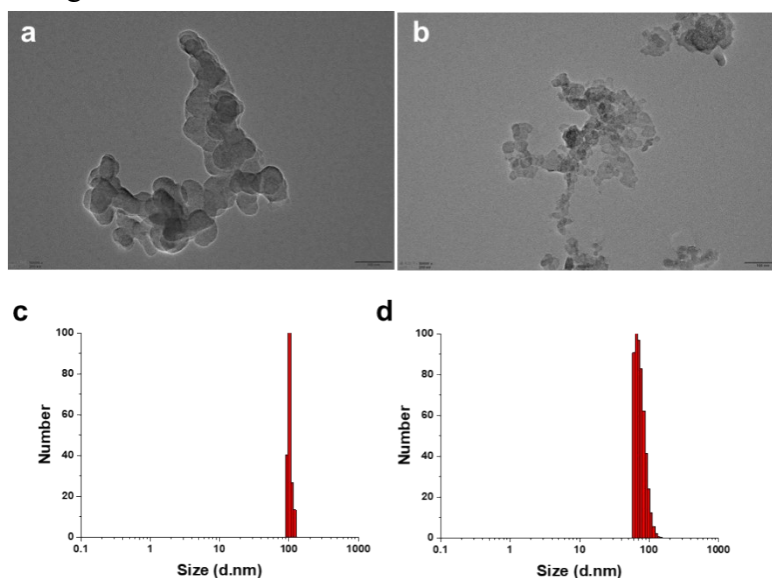
Supplementary Figure 3.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of compound TTC-Pt in  $\text{DMF-}d_7$ .



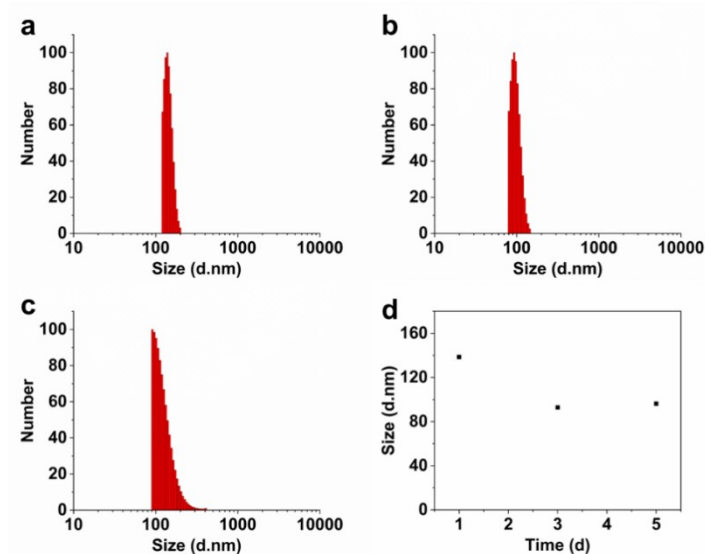
Supplementary Figure 4. HR-ESI-MS spectrum of compound TTC-Pt.



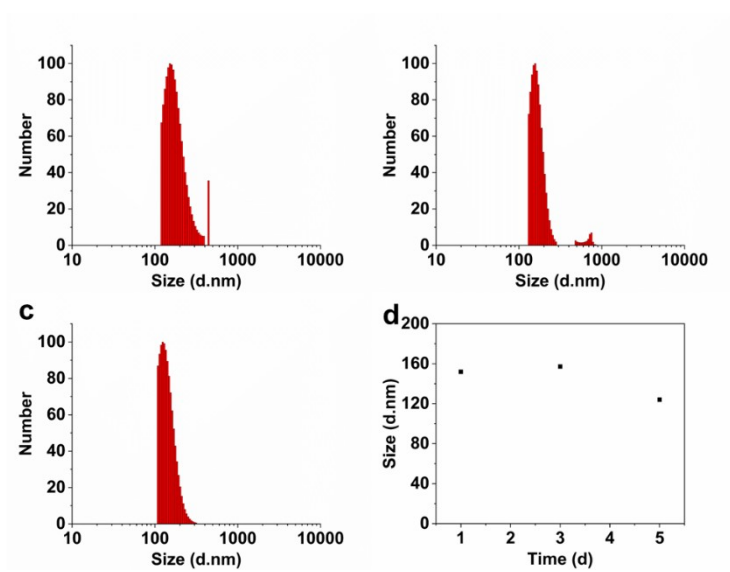
**Supplementary Figure 5.** HPLC chromatogram of compound **TTC-Pt**. The detection wavelength is 254 nm.



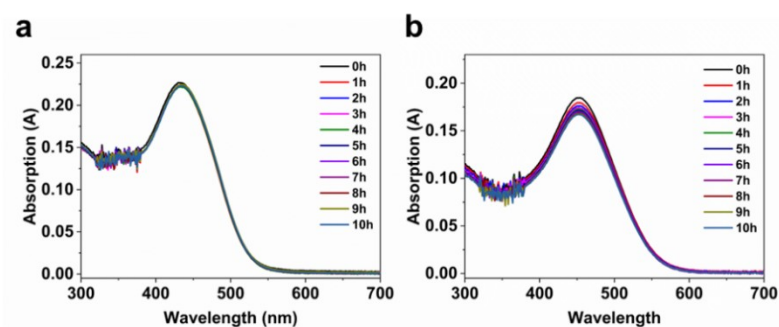
**Supplementary Figure 6.** The transmission electron microscopy (TEM) and dynamic laser scattering (DLS) data of the AIEgens. The TEM images of the AIEgens including (a) TTC-Pt and (b) TTC in aqueous solution. Scale bar: 100 nm. The DLS data of the AIEgens including (c) TTC-Pt and (d) TTC in aqueous solution.



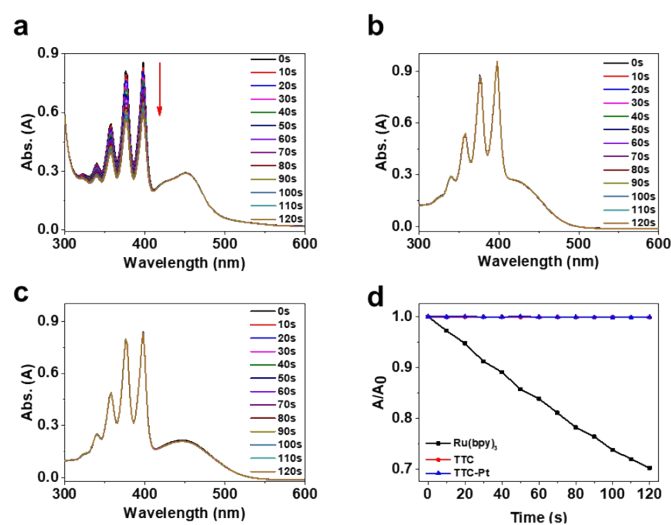
**Supplementary Figure 7.** The stability of the formed nano-aggregates in water with **TTC**. DLS images of the TTC-Pt in water, respectively, in the (a) first, (b) third and (c) fifth days. (d) The size of TTC-Pt in different days.



**Supplementary Figure 8. The stability of the formed nano-aggregates in water with TTC-Pt.** DLS images of the TTC-Pt in water, respectively, in the (a) first, (b) third and (c) fifth days. (d) The size of TTC-Pt in different days.

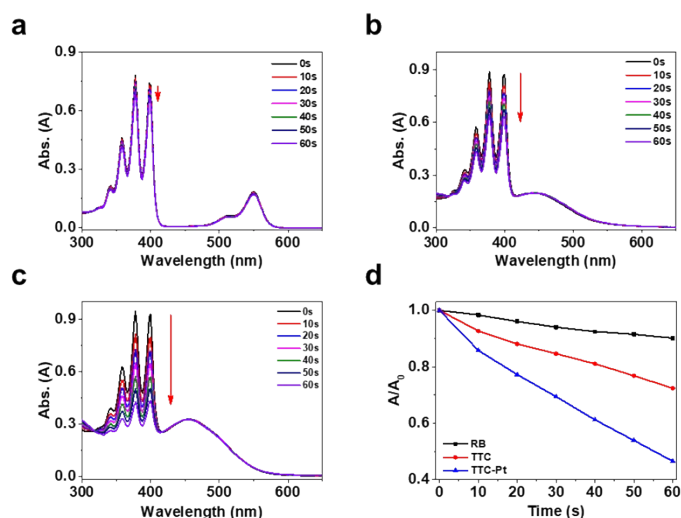


**Supplementary Figure 9. The stability of AIEgens.** The absorbance of the AIEgens including (a) TTC and (b) TTC-Pt (10  $\mu\text{M}$ ) in aqueous solution in the dark for 10 h.

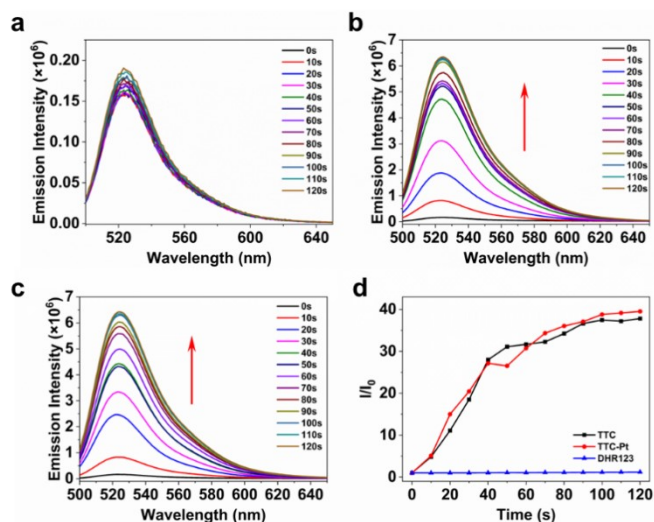


**Supplementary Figure 10. ROS generation capacity of the AIEgens in the presence of ABDA under light irradiation in acetonitrile.** Changes of UV-vis spectra of ABDA (83  $\mu\text{M}$ ) in the presence of (a)  $\text{Ru}(\text{bpy})_3\text{Cl}_2$  (10  $\mu\text{M}$ ) and different

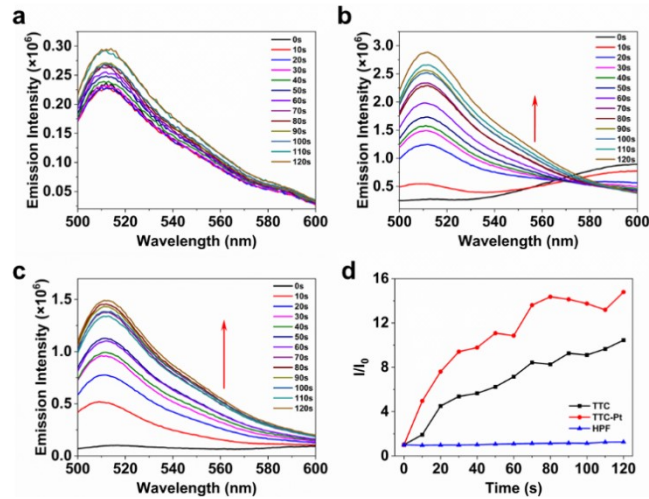
AIEgens (10  $\mu\text{M}$ ) including (b) TTC, (c) TTC-Pt under different durations of light irradiation (25  $\text{mW cm}^{-2}$ ) in acetonitrile. (d) The decomposition rates of ABDA in different AIEgens under light irradiation ( $A_0$  and  $A$  represent the absorption of ABDA before and after laser irradiation, respectively).



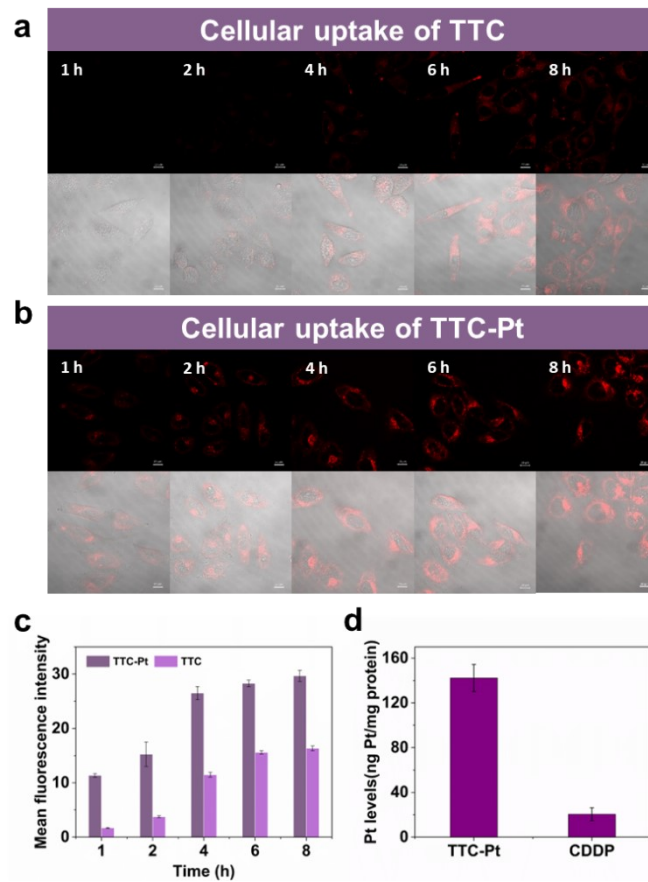
**Supplementary Figure 11. ROS generation capacity of the AIEgens in the presence of ABDA under light irradiation in water.** Changes of UV-vis spectra of ABDA (83  $\mu\text{M}$ ) in the presence of (a) RB (10  $\mu\text{M}$ ) and different AIEgens (10  $\mu\text{M}$ ) including (b) TTC, (c)TTC-Pt under different durations of light irradiation (25  $\text{mW cm}^{-2}$ ) in water. (d) The decomposition rates of ABDA in different AIEgens under light irradiation ( $A_0$  and  $A$  represent the absorption of ABDA before and after laser irradiation, respectively).



**Supplementary Figure 12.  $\text{O}_2^{\cdot-}$  generation capacity of the AIEgens in the presence of DHR123 under light irradiation in water.** Changes of fluorescence spectra of DHR123 (5  $\mu\text{M}$ ) with only DHR123 or in the presence of different AIEgens (1  $\mu\text{M}$ ) including (b) TTC and (c) TTC-Pt under different durations of light irradiation (25  $\text{mW cm}^{-2}$ ) in the water. (d) The decomposition rates of DHR123 in different conditions under light irradiation ( $I_0$  and  $I$  represent the fluorescence intensity of DHR123 before and after laser irradiation, respectively).

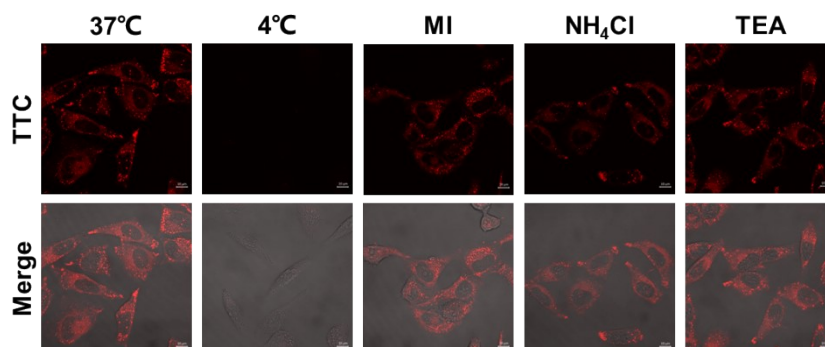


**Supplementary Figure 13. •OH generation capacity of the AIEgens in the presence of HPF under light irradiation in water.** Changes of fluorescence spectra of HPF (5  $\mu\text{M}$ ) with only HPF or in the presence of different AIEgens (1  $\mu\text{M}$ ) including (b) TTC and (c) TTC-Pt under different durations of light irradiation (25  $\text{mW cm}^{-2}$ ) in the water. (d) The decomposition rates of HPF in different conditions under light irradiation ( $I_0$  and  $I$  represent the fluorescence intensity of HPF before and after laser irradiation, respectively).

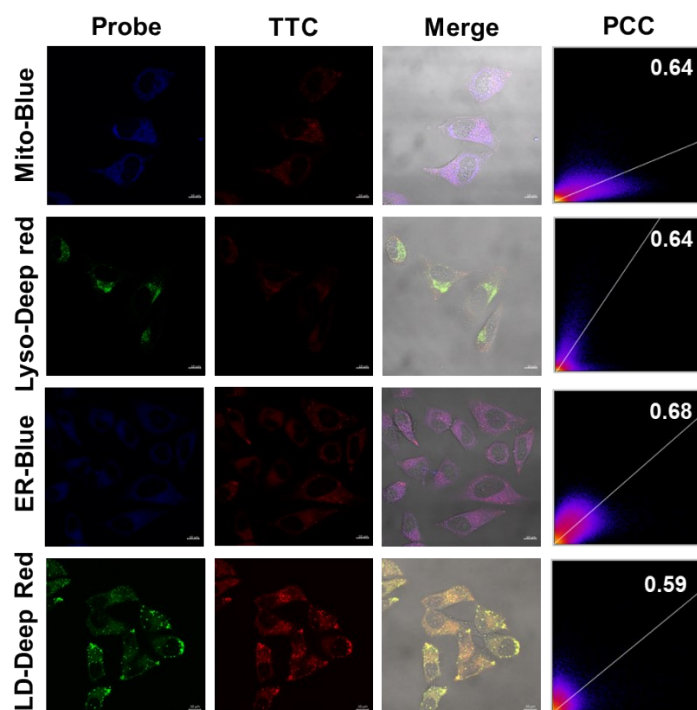


**Supplementary Figure 14. CLSM images of the cell uptake with different**

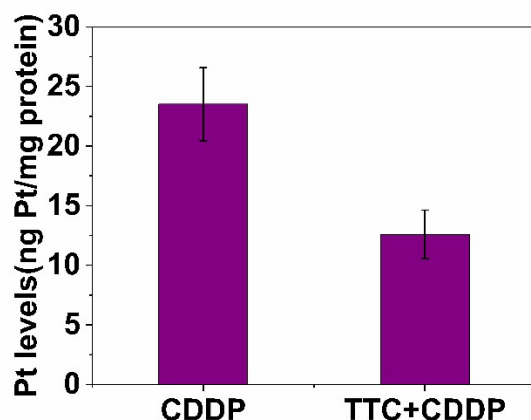
**AI-Egens (10  $\mu\text{M}$ ) at 37  $^{\circ}\text{C}$ .** CLSM images of different AI-Egens including (a) TTC and (b) TTC-Pt in HeLa cells after co-incubation with cells for different times from 1 h to 8 h, (c) fluorescence intensity change stained cells in (a) and (b). Excitation: 488 nm; Scanning channel: 600-700 nm; Scale bar: 10  $\mu\text{m}$ . (d) Pt accumulation in HeLa cells after incubated with TTC-Pt or CDDP (10  $\mu\text{M}$ ) for 8 h.



**Supplementary Figure 15. Different inhibitors were used to analyze the uptake pathway of TTC.** CLSM images of HeLa cells incubated with TTC (1  $\mu\text{M}$ ) in different conditions. The cells were pre-incubated by different inhibitors for 1h or at different temperature. Excitation: 488 nm; Scanning channel: 600-700 nm; Scale bar: 10  $\mu\text{m}$ . MI: metabolic inhibitors; 2-deoxy-D-glucose/oligomycin; TEA: triethylamine.



**Supplementary Figure 16. Subcellular co-localization of TTC.** CLSM of HeLa cells stained with different commercial probes (Mito-Blue: 410-480 nm,  $\lambda_{\text{ex}}$ , 405 nm; Lyso-Deep Red: 650-680 nm,  $\lambda_{\text{ex}}$ , 633 nm; ER-Blue: 455-520 nm,  $\lambda_{\text{ex}}$ , 405 nm; LD-Deep Red: 650-690 nm,  $\lambda_{\text{ex}}$ , 633 nm) and TTC (1  $\mu\text{M}$ , 600-700 nm,  $\lambda_{\text{ex}}$ , 488 nm). Scale bar: 10  $\mu\text{m}$ .



**Supplementary Figure 17. Pt levels in HeLa cells.** Pt accumulation in HeLa cells after cubulated with CDDP or TTC+CDDP (10  $\mu$ M) for 12 h.

**Supplementary Table 1.** Optical properties and lipophilicity of AIEgens including TTC and TTC-Pt.

AIEgens	$\lambda_{\text{abs}}^{\text{a}}$ (nm)	$\lambda_{\text{em}}^{\text{b}}$ (nm)	$\Phi_{\text{f}}/\%$	
			water	ACN
<b>TTC-Pt</b>	<b>451</b>	<b>650</b>	<b>2.90</b>	<b>0.6</b>
<b>TTC</b>	<b>434</b>	<b>603</b>	<b>11.45</b>	<b>0.46</b>

(a) Absorption maximum in water (containing 1% DMSO). (b) Emission maximum in water (containing 1% DMSO). (c) Fluorescence quantum yield of AIEgens in water and acetonitrile.

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