A Benzothiazole - Based Fluorescent Probe for Hypochlorite - Highly Sensitive Detection and Live - Cell Imaging Research

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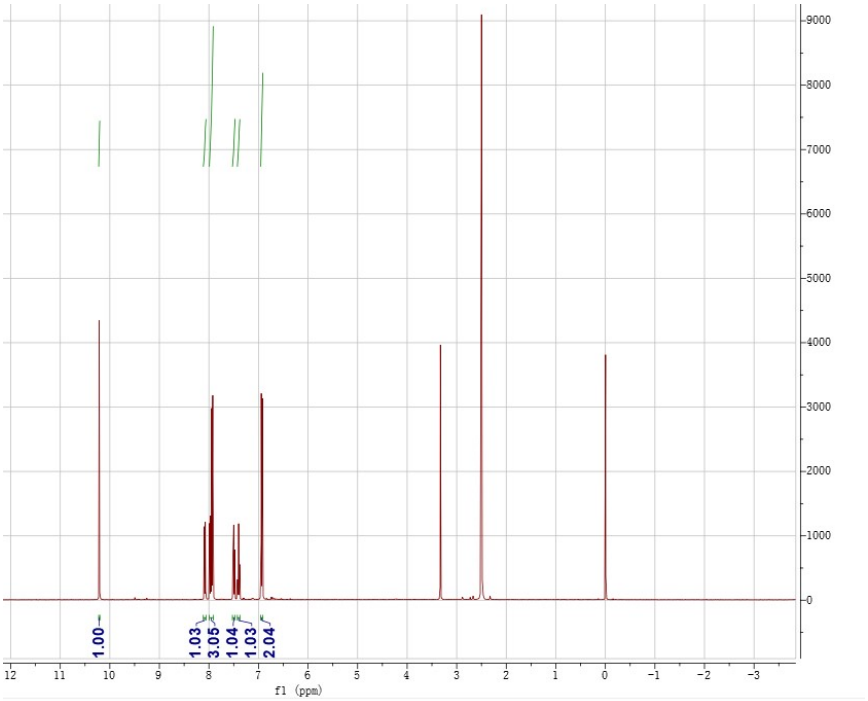
1、Synthesis of 2-1

In a 100 mL three-necked flask,o-aminothiophenol (20 mmol, 2.20 mL),p-hydroxybenzal dehyde (20 mmol, 2.4424 g), sodium metabisulfite ($Na_2S_2O_5$, 40 mmol, 7.5636 g) and N, N-dimethylformamide (DMF, 30 mL) were added sequentially. The mixture was stirred at 120 °C for 3 h, and the reaction progress was monitored by thin-layer chromatography (TLC) until completion. After the reaction, the mixture was allowed to stand and cooled t o room temperature, then poured into ice water, resulting in a large amount of milky white precipitate. The precipitate was collected by vacuum filtration, washed with ice wa ter for 3 times, and dried under vacuum to obtain Compound 2-1 as a white powder w ith a yield of 72.50% (3.2921 g). ^{1}H NMR (400 MHz,DMSO-d_6) δ 10.21(s,1H),8.10-8.06(m,1 H),7.99-7.91(m,3H),7.50(ddd,J=8.3,7.2,1.3Hz,1H),7.40(ddd,J=8.3,7.2,1.2Hz,1H),6.95 6.91(m,2H).^{13}C NMR (101 MHz, DMSO-d_6): δ 167.45, 160.5, 153.73, 134.10, 129.04, 126.42, 124.89, 1 24.04, 122.30, 122.11, 116.09, 116.08.

2、Synthesis of 2-2

Hexamethylenetetramine (HMTA, 15 mmol, 2.1030 g) was added to a 100 mL three-necked flask, followed by the slow addition of trifluoroacetic acid (25 mL). The mixture was heated to 80 °C and reacted for 1 h. Then, Compound 2-1 (3 mmol, 0.6811 g) was added into the flask, and the reaction was continued for another 12 h. The reaction pr ogress was monitored by thin-layer chromatography (TLC) until completion. After cooling to room temperature, the reaction solution was poured into ice water, and a yellow pre cipitate formed immediately. The precipitate was collected by vacuum filtration, washed with water three times, and dried under vacuum. The crude product was purified by col umn chromatography using petroleum ether/ethyl acetate (15:1,V/V) as the eluent, afford ing Compound 2-2 as a yellow solid with a yield of 48.33% (0.3698 g). ^{1}H NMR (400MH z,DMSO-d_6)δ11.44(s,1H),10.37(s,1H),8.33(d,J=2.4Hz,1H),8.23(dd,J=8.7,2.5Hz,1H),8.15-8.12(m,1H),8 .04(dt,J=8.1,1.0Hz,1H),7.54(ddd,J=8.3,7.2,1.3Hz,1H),7.45(ddd,J=8.3,7.2,1.2Hz,1H),7.20(d,=8.7Hz,1 H). ^{13}C NMR (101 MHz, DMSO-d_6): δ 190.39, 166.25, 162.98, 153.55, 134.55, 129.02, 127.5 0, 126.62, 125.28, 124.42, 122.58, 122.27, 118.46, 116.07.

3、(A)



(B)

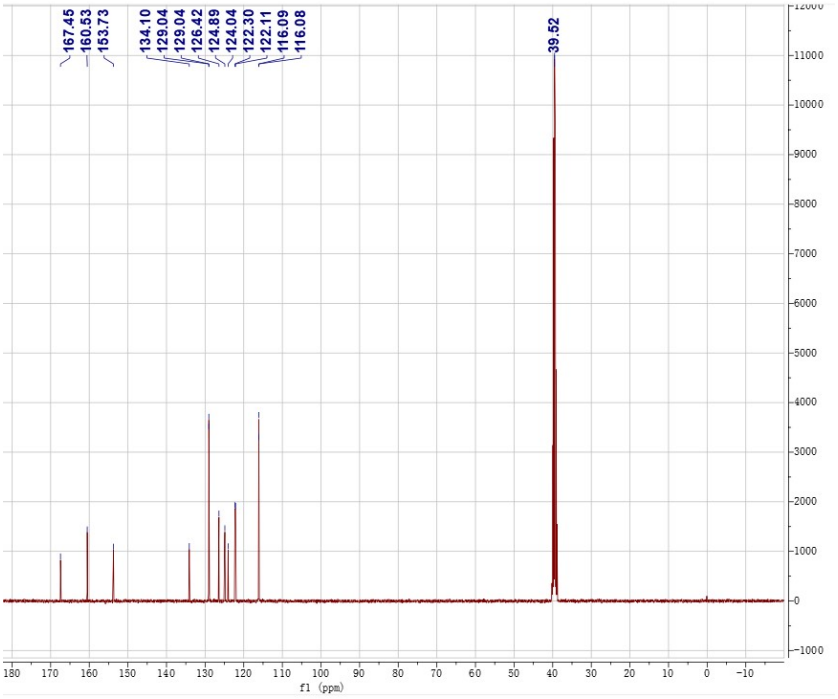
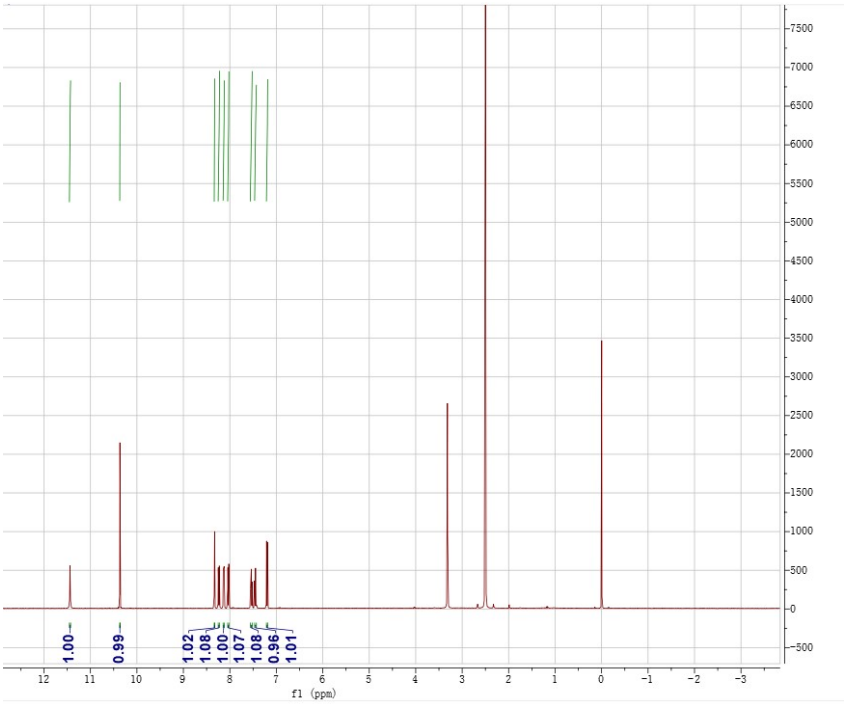


Fig. S1. ^{1}H NMR (A) and ^{13}C NMR (B) of 2-1

4、(A)



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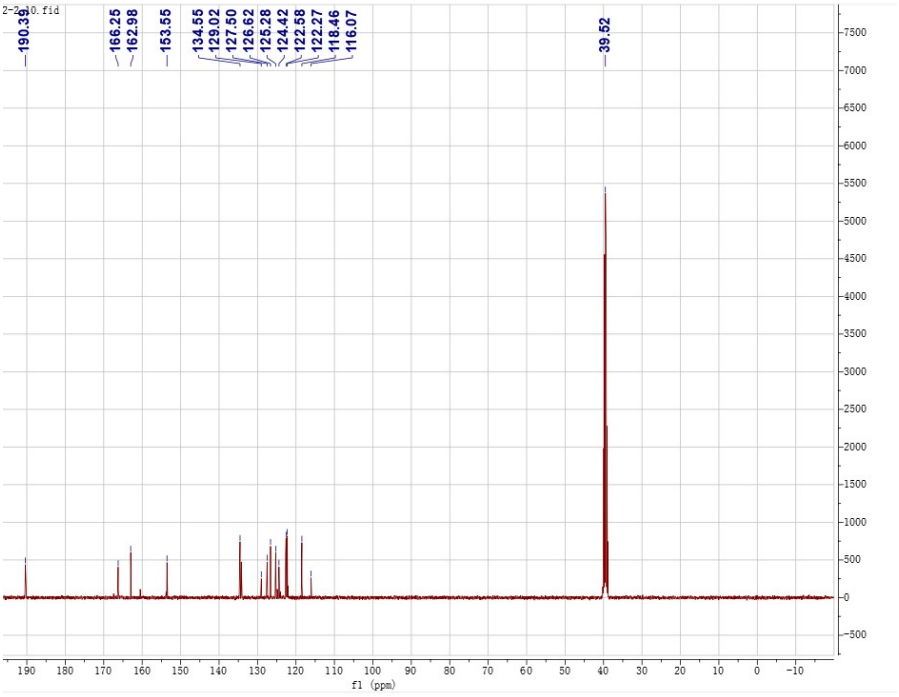
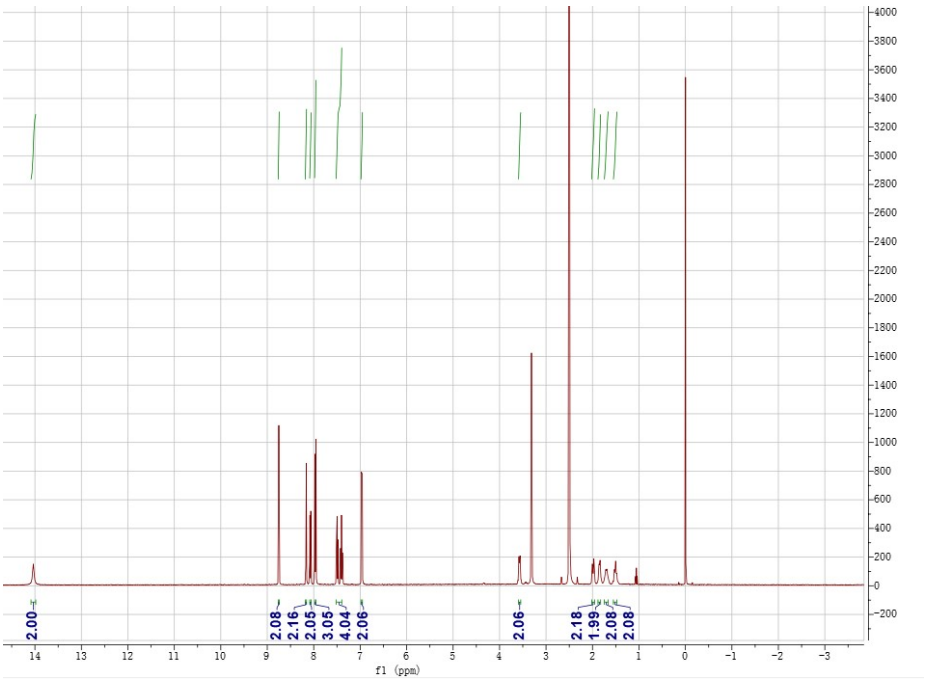


Fig. S2. ^{1}H NMR (A) and ^{13}C NMR (B) of 2-2

5、(A)



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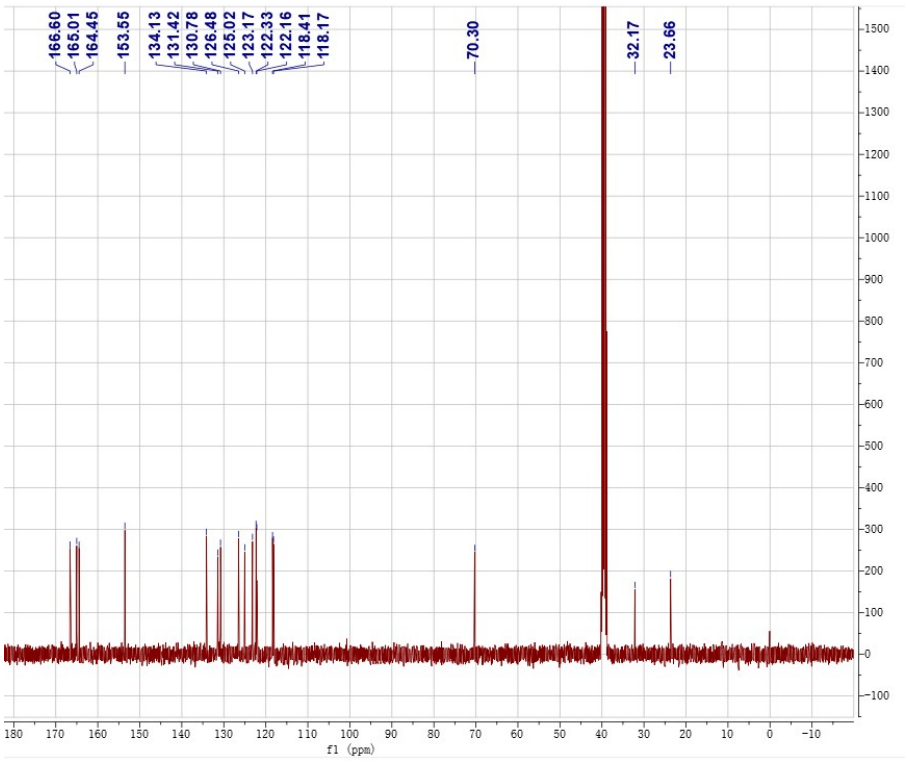

Fig. S3. ^{1}H NMR (A) and ^{13}C NMR (B) of the probe BZDC

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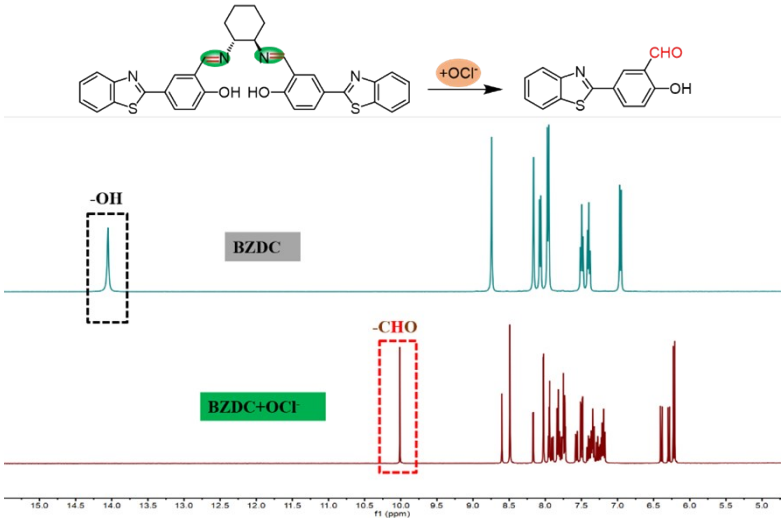

Fig. S4. Comparison of partial ^{1}H NMR of probe BZDC in DMSO-d6 before and after addition of excess OCl^-.

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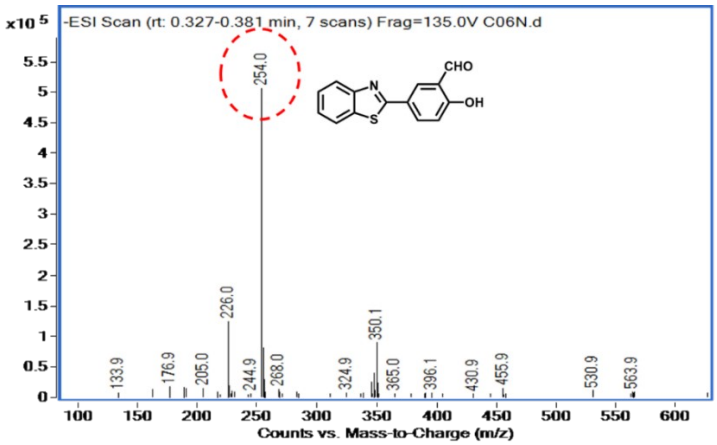

Fig. S5. Mass spectrum data of the product after the response between probe BZDC and OCl^-.

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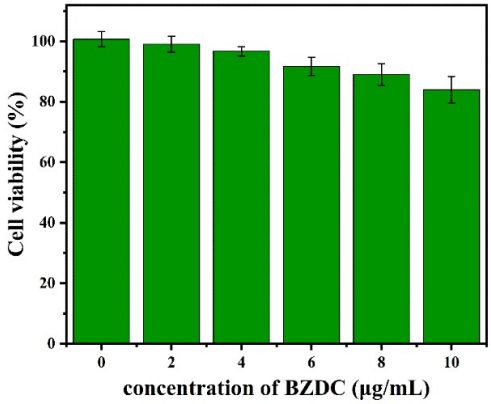

Fig. S6. Survival rates of Hela cells cultured with different concentrations of probe solution measured by MTT method.