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Supporting Information for:

Photoactuators based on the dynamic molecular crystals of naphthalene acrylic acids driven by sterospecific [2+2] cycloaddition reactions

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Measurements and characterizations

^{1}H NMR and ^{13}C NMR spectra were recorded with a Bruker-AvanceIII 400 MHz and 101 MHz spectrometer using DMSO-$d_6$and $CDCl_3$ as solvents and tetramethylsilane (TMS) as the internal standard. The samples for irradiation time-dependent ^{1}H NMR measurements were obtained via the irradiation of the crystals of **1FNaAA**, **1ClNaAA**, **1BrNaAA**, **1INaAA** and **6BrNaAA** by 365 nm (3 W) light for different times, followed by dissolving in DMSO-d_6. FT-IR spectra were obtained with a Nicolet-360 FT-IR spectrometer by the incorporation of samples into KBr disks. High-resolution mass analyses were measured on Bruker micro TOF-QII apparatus. UV-vis absorption spectra were measured by a Shimadzu UV-1601PC spectrophotometer. Fluorescence emission spectra were taken on a Shimadzu RF-5301 Luminescence Spectrometer. The samples of **1FNaAA**, **1ClNaAA**, **1BrNaAA**, **1INaAA** and **6BrNaAA** for electronic spectra measurements were prepared by grinding the crystals into microcrystals, which were smeared on the quartz slice and irradiated by 365 nm (3 W) light for different times. To reveal the geometrical structures, frontier molecular orbitals and energy band gaps of the target compounds, density functional theory (DFT) calculations were performed by using the Gaussian 09W program with the B3LYP/3-21G(d) basis set for **1FNaAA**, **1ClNaAA**, **1BrNaAA**, **1INaAA** and **6BrNaAA**.

The single crystals of **1FNaAA**,**D-1FNaAA**, **1ClNaAA**,**D-1ClNaAA**, **1BrNaAA**, **1INaAA** and **6BrNaAA**were selected for X-ray diffraction analysis on a Rigaku RA XIS-RA PID diffractometer using graphite-monochromated MoK α radiation (λ =

0.71073 Å), and the crystals were kept at 99.97 K, 100.0 K, 100.02 K, 100.0 K, 293.76 K, 150.0 K and 293.76 K during data collection, respectively. The single crystals of **1FNaAA**, **1ClNaAA** and **1BrNaAA** were obtained in CH_2Cl_2, methanol and acetone, respectively, under petroleum ether steam by vapor diffusion method. The single crystals of **1INaAA** and **6BrNaAA** were obtained in THF under petroleum ether steam by vapor diffusion method. The single crystals of β-type **D-1FNaAA** and **D-1ClNaAA** were obtained in acetone and THF, respectively, under petroleum ether steam by vapor diffusion method. In addition, the β-type **D-1FNaAA** and **D-1ClNaAA** were isolated from the microcrystals of **1FNaAA** and **1ClNaAA**, which were firstly irradiated by a hand-held 365 nm lamp (3 W) for 30 min, via column chromatography (silica gel) using ethylacetate/petroleum ether (v/v = 1/4) containing 3% acetic acid as the eluent. It should be noted that the absorption at 365 nm for the naphthalene acrylic acids in THF were quite low, but the microcrystals gave broad peaks with tails covering 365 nm. Indeed, the photochemical reactions could take place in microcrystals under 365 nm light. Therefore, the hand-held 365 nm lamp (3 W) was used as light source for [2+2] cycloaddition reactions and photomechanical studies.

The photomechanical effects were observed under an optical microscope at 298 K under ambient conditions. The crystals were put on the glass slices and then irradiated by a hand-held 365 nm lamp (3 W) for different times. The crystals of **1FNaAA** and **1ClNaAA**were obtained by slow evaporation from the solutions in CH_2Cl_2. The crystals of **1BrNaAA** and **6BrNaAA**were obtained by slow evaporation from the

solutions in CH_2Cl_2/petroleum ether (v/v = 1/4).

THF was dried over sodium and benzophenone. DMF was dried over phosphorus pentoxide. CH_2Cl_2 was dried over calcium hydride. The other chemicals and reagents were used as received without further purification.

Syntheses

1-Fluoro-2-naphthaldehyde (1)

The solution of *n*-butyllithium (8.5 mL, 13.6 mmol, 1.6 M in hexane) in cyclohexane was added to a solution of 1-fluoronaphthalene (2.0 g, 13.6 mmol) in dry THF (40 mL) at -78 °C. The mixture was stirred at -78 °C for 2 h, and then dry DMF (2.2 mL, 27.2 mmol) was added. After stirring for 5 min at -78 °C, the mixture was quenched with water and the crude product was extracted with ethyl acetate (20 mL × 3). The combined organic phase was dried over anhydrous Na_2SO_4. After removal of the solvent, the crude product was purified by column chromatography (silica gel, petroleum ether/ethyl acetate, v/v = 20/1) to afford **1** (1.8 g) as a yellow solid. Yield 75%. ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ 10.49 (s, 1H), 8.31-8.19 (m, 1H), 8.10 (d, J = 8.1 Hz, 1H), 7.94-7.86 (m, 1H), 7.86-7.72 (m, 3H) (Figure S23).

1-Chloro-3,4-dihydronaphthalene-2-carbaldehyde (2)

A flame-dried flask containing DMF (5.3 mL, 68.4 mmol) was cooled to 0 °C and $POCl_3$ (5 mL, 54.7 mmol) was added dropwise under stirring. The mixture was warmed to room temperature and stirred for 15 min. Then, it was cooled to 0 °C and 3,4-dihydronaphthalen-1-(2H)-one (4.6 mL, 34.2 mmol) was added dropwise. After that, the mixture was warmed to room temperature and stirred for another 1 h. The

mixture was poured into ice water (100 mL) and the saturated aqueous $NaHCO_3$ was added to adjust pH to ca. 6.0. The mixture was extracted with ethyl acetate (30 mL × 3) and the combined organic phase was dried over anhydrous Na_2SO_4. After removal of the solvent, the crude product was purified by column chromatography (silica gel, petroleum ether/ethyl acetate, v/v = 20/1) to afford **2** (5.9 g) as a slightly yellow oil. Yield 91%. ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ 10.28 (s, 1H), 7.82 (dd, J = 7.7, 1.1 Hz, 1H), 7.46 (td, J = 7.4, 1.4 Hz, 1H), 7.44-7.37 (m, 1H), 7.34 (dd, J = 7.3, 0.8 Hz, 1H), 2.88-2.78 (m, 2H), 2.58-2.52 (m, 2H) (Figure S24).

1-Chloro-2-naphthaldehyde (3)

The mixture of compound **2** (3.0 g, 15.6 mmol) and DDQ (2,3-dichloro-5,6-dicyano-1,4-benzoquinone, 7.1 g, 31.2 mmol) in chlorobenzene (100 mL) was refluxed for 72 h. It was then poured into saturated aqueous solution of $NaHCO_3$ and extracted with ethyl acetate (20 mL × 3). The combined organic phase was dried over anhydrous Na_2SO_4. After removal of the solvent, the crude product was purified by column chromatography (silica gel, petroleum ether/ethyl acetate, v/v = 20/1) to afford **3** (1.9 g) as a pale yellow solid. Yield 63%. ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ 10.68 (s, 1H), 8.49 (dd, J = 5.8, 3.4 Hz, 1H), 8.16 (dd, J = 5.8, 3.6 Hz, 1H), 8.11 (d, J = 8.5 Hz, 1H), 7.92 (d, J = 8.6 Hz, 1H), 7.87 (dd, J = 6.3, 3.3 Hz, 2H) (Figure S25).

2-(1-Bromonaphthalen-2-yl)-1,3-dioxolane (4)

1-Bromo-2-naphthaldehyde(2.0 g, 8.5 mmol) was dissolved in toluene (60 mL), and 4-methylbenzenesulfonic acid monohydrate (17.4 mg, 0.091 mmol) as well as

ethylene glycol (2.4 mL, 43.5 mmol) were added. Then, the mixture was stirred at 125 °C for 18 h under nitrogen atmosphere. After that, the excess toluene was removed under vacuum. The crude product was purified by column chromatography (silica gel, petroleum ether/ethyl acetate, v/v = 20/1) to afford **4** (2.2 g) as a yellow liquid. Yield 93%. ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ 8.33 (d, J = 8.5 Hz, 1H), 8.06 (d, J = 8.6 Hz, 2H), 7.81-7.74 (m, 1H), 7.74-7.67 (m, 2H), 6.30 (s, 1H), 4.27-4.05 (m, 4H) (Figure S26).

1-Iodo-2-naphthaldehyde (5)

Compound **4** (2.2 g, 7.9 mmol) was dissolved in dry THF (20 mL) and the solution was cooled to -78 °C under nitrogen atmosphere. Then, *n*-butyllithium (6.5 mL, 15.8 mmol, 2.5 M in hexane) was added dropwise and stirred for 20 min. Iodine (3 g, 11.9 mmol) in THF (15 mL) was added at -78 °C. After that, the solution was allowed to warm up to room temperature and stirred for 14 h. The reaction was quenched by saturated aqueous solution of NH_4Cl and $NaHSO_3$. The mixture was extracted with ethyl acetate (20 mL × 3). The combined organic phase was dried over anhydrous Na_2SO_4. After removal of the solvent, the crude product of 2-(1-iodonaphthalen-2-yl)-1,3-dioxolane was not further purified. It was dissolved in MeOH/toluene (21 mL, v/v = 4/17), to which HCl (15 mL, 20%) was added. The mixture was stirred at room temperature for 18 h under nitrogen. The saturated aqueous $NaHCO_3$ was added to adjust pH to ca. 7.0. The mixture was extracted by ethyl acetate (20 mL × 3), and the combined organic phase was dried over anhydrous Na_2SO_4. After removal of the solvent, the crude product was purified by column

chromatography (silica gel, petroleum ether/ethyl acetate, v/v = 20/1) to afford **5** (1.2 g) as a yellow solid. Yield 55%. ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ 10.37 (d, J = 0.6 Hz, 1H), 8.47 (dd, J = 6.3, 3.4 Hz, 1H), 8.15-8.02 (m, 2H), 7.91-7.78 (m, 3H) (Figure S27).

Methyl 6-bromo-2-naphthoate (6)

6-Bromo-2-naphthoic acid (5.0 g, 19.9 mmol) was suspended in methanol (100 mL), and concentrated sulphuric acid (2 mL) was added. After the mixture was refluxed for 12 h, it was cooled and the precipitate was collected by filtration. The crude product was purified by column chromatography (silica gel, dichloromethane/petroleum ether, v/v = 1/1) to afford **6** (4.9 g) as a white solid. Yield 93%. ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.56 (s, 1H), 8.07 (dd, J = 8.6, 1.5 Hz, 1H), 8.03 (s, 1H), 7.79 (t, J = 9.4 Hz, 2H), 7.61 (dd, J = 8.7, 1.8 Hz, 1H), 3.98 (s, 3H) (Figure S28). ^{13}C NMR (101 MHz, $CDCl_3$, ppm) δ 166.88, 136.40, 130.91, 130.85, 130.18, 129.92, 127.85, 127.24, 126.36, 122.63, 52.35 (Figure S29).

(6-Bromonaphthalen-2-yl)methanol (7)

To a solution of diisobutylaluminium hydride (DIBALH) (1.0 M in heptane, 99.6 mL, 99.6 mmol) at -78 °C under nitrogen atmosphere a solution of compound **6** (8.8 g, 33.2 mmol) in anhydrous THF (40 mL) was added dropwise. The mixture was allowed to warm up to room temperature and stirred for 14 h. The reaction was quenched by methanol (20 mL), followed by adding saturated solution of sodium potassium tartrate, and the mixture was stirred at room temperature for another 2 h. After extracted by ethyl acetate (20 mL × 3), the combined organic phase was washed

with saturated aqueous solution of NH_4Cl and brine, dried over anhydrous Na_2SO_4. After removal of the solvent, the crude product was purified by column chromatography (silica gel, petroleum ether/ethyl acetate, v/v = 1/1) to afford **7** (7.7 g) as a white solid. Yield 98%. ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.03 (s, 1H), 7.81 (s, 1H), 7.78 (d, J = 8.5 Hz, 1H), 7.73 (d, J = 8.7 Hz, 1H), 7.59 (dd, J = 8.7, 1.9 Hz, 1H), 7.53 (dd, J = 8.5, 1.3 Hz, 1H), 4.88 (s, 2H) (Figure S30). ^{13}C NMR (101 MHz, $CDCl_3$, ppm) δ 138.85, 133.99, 131.80, 129.79, 129.60, 129.54, 127.43, 126.18, 125.30, 119.84, 65.27 (Figure S31).

6-Bromo-2-naphthaldehyde (8)

A solution of compound **7** (2.1 g, 8.7 mmol) in dry CH_2Cl_2 was added to a suspension of pyridinium chromate (2.1 g, 9.7 mmol) in dry CH_2Cl_2 (30 mL). The mixture was refluxed for 5 h, and then CH_2Cl_2 was removed under vacuum. The crude product was purified by column chromatography (silica gel, petroleum ether/ethyl acetate, v/v = 3/1) to afford **8** (1.9 g) as a white solid. Yield 90%. ^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 10.21 (s, 1H), 8.37 (s, 1H), 8.14 (s, 1H), 8.04 (d, J = 8.5 Hz, 1H), 7.93 (dt, J = 13.7, 6.8 Hz, 2H), 7.73 (dd, J = 5.3, 3.3 Hz, 1H) (Figure S32). ^{13}C NMR (101 MHz, $CDCl_3$, ppm) δ 191.82, 137.31, 134.37, 134.06, 131.10, 130.99, 130.68, 130.29, 128.19, 124.07, 123.62 (Figure S33).

(*E*)-3-(1-Fluoronaphthalen-2-yl)acrylic acid (1FNaAA)

Compound **1** (1.0 g, 5.7 mmol), malonic acid (1.7 g, 16.1 mmol) and piperidine (1.0 mL) were dissolved in pyridine (60 mL), and the mixture was stirred at 90 °C for 8 h. Then, the mixture was poured into water (200 mL) and dilute HCl was added to

adjust the pH to around 1.0. The white solid **1FNaAA** (0.9 g) was collected by filtration and recrystallized in ethanol. Yield 75%. m.p. 161.0-162.8 °C. ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ 12.63 (s, 1H), 8.13 (dd, J = 5.9, 3.7 Hz, 1H), 8.04 (dd, J = 4.8, 2.4 Hz, 1H), 7.94 (dd, J = 17.6, 12.3 Hz, 2H), 7.83 (d, J = 8.7 Hz, 1H), 7.73-7.65 (m, 2H), 6.76 (d, J = 16.1 Hz, 1H) (Figure S34). ^{13}C NMR (101 MHz, DMSO-d_6, ppm) δ 167.32, 157.54, 154.97, 135.07, 135.02, 134.88, 134.83, 128.34, 127.79, 127.76, 127.47, 124.41, 124.37, 123.76, 123.73, 122.69, 122.52, 121.79, 121.75, 120.68, 120.62, 116.53, 116.43 (Figure S35). FT-IR (KBr, cm^{-1}): 3451, 2989, 1700, 1620, 1420, 1381, 1277, 1075, 982, 869, 750, 559. HRMS (ESI): m/z calcd. for $C_{13}H_9FO_2$ $[M–H]^-$ 215.0541; found 215.0502 (Figure S36).

(*E*)-3-(1-Chloronaphthalen-2-yl)acrylic acid (1ClNaAA)

By following the synthetic procedure for **1FNaAA**, **1ClNaAA** was prepared from compound **3** (1.0 g, 5.2 mmol) and malonic acid (1.5 g, 14.7 mmol) in pyridine (60 mL) in the presence of piperidine (1.0 mL). The crude product was purified by crystallization in ethanol to afford white solid **1ClNaAA** (1.2 g). Yield 98%. m.p. 157.5-160.0 °C. ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ 12.70 (s, 1H), 8.30 (d, J = 8.3 Hz, 1H), 8.17 (d, J = 15.9 Hz, 1H), 8.07-8.00 (m, 2H), 7.97 (d, J = 8.7 Hz, 1H), 7.79-7.61 (m, 2H), 6.76 (d, J = 15.9 Hz, 1H) (Figure S38). ^{13}C NMR (101 MHz, DMSO-d_6, ppm) δ 167.25, 139.09, 134.52, 131.60, 130.21, 129.39, 128.43, 128.30, 128.07, 127.71, 124.58, 123.97, 123.04 (Figure S39). FT-IR (KBr, cm^{-1}): 3415, 2985, 1695, 1618, 1419, 1338, 1281, 1233, 979, 809, 749, 695. HRMS (ESI): m/z calcd. for $C_{13}H_9ClO_2$ $[M–H]^-$ 231.0218; found 231.0206 (Figure S40).

(*E*)-3-(1-Bromonaphthalen-2-yl)acrylic acid (1BrNaAA)

By following the synthetic procedure for **1FNaAA**, **1BrNaAA** was prepared from 1-bromo-2-naphthaldehyde (0.56 g, 2.4 mmol) and malonic acid (0.7 g, 6.7 mmol) in pyridine (50 mL) in the presence of piperidine (0.5 mL). The crude product was purified by crystallization in ethanol to afford white solid **1BrNaAA** (0.57 g). Yield 86%. m.p. 176.0-178.0 °C. ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ 12.71 (s, 1H), 8.31 (d, J = 8.3 Hz, 1H), 8.20 (d, J = 15.9 Hz, 1H), 8.08-7.93 (m, 3H), 7.81-7.61 (m, 2H), 6.74 (d, J = 15.8 Hz, 1H) (Figure S42). ^{13}C NMR (101 MHz, DMSO-d_6, ppm) δ 167.19, 142.27, 134.59, 131.67, 128.53, 128.47, 128.40, 128.03, 127.41, 125.52, 124.39, 123.20 (Figure S43). FT-IR (KBr, cm^{-1}): 3392, 2984, 1693, 1620, 1418, 1331, 1276, 1228, 978, 810, 749, 692. HRMS (ESI): m/z calcd. for $C_{13}H_9BrO_2$ $[M–H]^-$ 274.9713; found 274.9717 (Figure S44).

(*E*)-3-(1-Iodonaphthalen-2-yl)acrylic acid (1INaAA)

By following the synthetic procedure for **1FNaAA**, **1INaAA** was prepared from compound **5** (1.2 g, 4.3 mmol) and malonic acid (1.3 g, 11.9 mmol) in pyridine (80 mL) in the presence of piperidine (1.0 mL). The crude product was purified by crystallization in ethanol to afford white solid **1INaAA** (0.8 g). Yield 57%. mp: 178.2-180.5 °C. ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ 12.70 (s, 1H), 8.25 (d, J = 8.3 Hz, 1H), 8.15 (d, J = 15.7 Hz, 1H), 7.98 (t, J = 7.5 Hz, 2H), 7.91 (d, J = 8.6 Hz, 1H), 7.68 (ddd, J = 21.2, 13.9, 6.9 Hz, 2H), 6.63 (d, J = 15.7 Hz, 1H) (Figure S45). ^{13}C NMR (101 MHz, DMSO-d_6, ppm) δ168.06, 151.24, 136.03, 134.49, 134.05, 132.85, 129.19, 128.71, 128.57, 127.89, 124.44, 123.20, 108.68, 99.49 (Figure S46). FT-IR

(KBr, cm^{-1}): 3415, 2985, 1695, 1618, 1419, 1338, 1281, 1233, 979, 809, 749, 695. HRMS (ESI): m/z calcd. for $C_{13}H_9IO_2$ $[M–H]^-$ 322.9574; found 322.9553 (Figure S47).

(*E*)-3-(6-Bromonaphthalen-2-yl)acrylic acid (6BrNaAA)

By following the synthetic procedure for **1FNaAA**, **6BrNaAA** was prepared from compound **8** (0.5 g, 2.2 mmol) and malonic acid (0.6 g, 6.2 mmol) in pyridine (40 mL) in the presence of piperidine (0.5 mL). The crude product was purified by crystallization in ethanol to afford white solid **6BrNaAA** (0.5 g). Yield 88%. m.p. 174.0-176.8 °C. ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ 12.54 (s, 1H), 8.35-8.11 (m, 2H), 7.94 (dd, J = 10.1, 4.8 Hz, 3H), 7.82-7.60 (m, 2H), 6.71 (d, J = 16.0 Hz, 1H) (Figure S48). ^{13}C NMR (101 MHz, DMSO-d_6, ppm) δ 167.52, 143.37, 134.73, 132.48, 131.41, 130.57, 129.71, 129.63, 129.38, 127.71, 125.24, 120.44, 120.24 (Figure S49). FT-IR (KBr, cm^{-1}): 3365，2919, 2849, 2596, 1702, 1620, 1424, 1316, 1286, 1219,1154, 1123, 1058, 973, 857, 810, 687, 626, 545, 471, 417. HRMS (ESI): m/z calcd. for $C_{13}H_9BrO_2$ $[M–H]^-$ 274.9713; found 274.9711 (Figure S50).

Table S1 Photophysical data of **1FNaAA**, **1ClNaAA**, **1BrNaAA**, **1INaAA** and **6BrNaAA** in THF.

Compound	Absorption [a] (nm) (ε / $\times 10^4$ $M^{-1}cm^{-1}$)	Emission [b] (nm)
1FNaAA	266 (5.9), 297 (3.7), 308 (3.4)	382, 368(shoulder)
1ClNaAA	263 (2.2), 301 (1.3), 312 (1.1)	387
1BrNaAA	263 (1.5), 302 (1.8), 312 (0.7)	403
1INaAA	267 (6.2), 307 (2.7), 319 (2.7)	392
6BrNaAA	271 (4.8), 303 (2.9), 315 (3.0)	376, 359(shoulder)

[a] Measured in THF (1.0×10^{-5} M); [b] Excited at 320 nm.

Table S2 Main electronic transitions calculated with TD-DFT.

Compound	λ_{abs}[a] (nm)	λ_{abs}[b] (nm)	f[c]	Transition (%)[d]
1FNaAA	353	336.83	0.1823	H→L (88.0)
				H→L+1 (2.5)
	308	305.50	0.0005	H−2→L (92.5)
				H−2→L+1 (2.9)
	297	294.32	0.2830	H−1→L (60.1)
				H→L (10.3)
1ClNaAA	361	336.54	0.1193	H→L (88.4)
				H→L+1 (2.1)
	312	304.28	0.0863	H−2→L (60.2)
				H−2→L+1 (2.9)
				H−1→L (26.4)
	301	295.54	0.2508	H−1→L (42.5)
				H→L (7.8)
1BrNaAA	347	340.76	0.1286	H→L (91.4)
	312	301.41	0.1298	H−2→L (37.9)
				H−2→L+1 (2.3)
				H−1→L (43.4)
				H→L (2.6)
	302	294.66	0.1274	H−2→L (46.7)
				H→L+1 (14.9)
1INaAA	362	344.05	0.1464	H→L (90.0)
				H→L+1 (2.2)
	319	300.87	0.1589	H−4→L (30.7)
				H−1→L (47.8)
				H→L (3.1)
	307	292.86	0.0427	H−4→L (28.4)
				H−3→L (11.6)
				H−2→L (7.8)
				H→L+1 (28.2)
6BrNaAA	351	339.90	0.3627	H→L (92.5)
				H−1→L (4.5)
	315	310.01	0.2387	H−1→L (69.0)
				H→L+1 (22.9)
	303	305.52	0.0005	H−2→L (92.1)
				H−2→L+1 (3.7)

[a]Experimental absorption in THF; [b]Compound transition wavelength *in vacuo*; [c]Compound oscillator strength *in vacuo*; [d]H represents HOMO, L represents LUMO.

Table S3 Single crystal data of **1FNaAA**,**D-1FNaAA**, **1ClNaAA** and **D-1ClNaAA**.

	1FNaAA	**D-1FNaAA**	**1ClNaAA**	**D-1ClNaAA**
Formula	$C_{13}H_9FO_2$	$C_{29}H_{24}F_2O_5$	$C_{13}H_9ClO_2$	$C_{30}H_{26}Cl_2O_5$
Formula weight	216.20	490.48	232.65	537.41
Space group	P21/c	P21/n	$P\bar{1}$	P21/c
Crystal system	Monoclinic	Monoclinic	Triclinic	Monoclinic
a (Å)	6.7599(8)	7.4331(7)	3.7709(7)	13.8413(5)
b (Å)	3.7695(4)	21.337(2)	7.9900(14)	6.6231(2)
c (Å)	38.685(4)	15.4508(17)	17.205(3)	27.4197(9)
α (deg)	90	90	91.451(7)	90
β (deg)	93.899(4)	98.686(5)	90.871(7)	91.7890(10)
γ (deg)	90	90	91.865(7)	90
V (Å^3)	983.46(18)	2422.4(4)	517.87(16)	2512.40(14)
Z	4	4	2	4
D_{calc}(g/cm^3)	1.460	1.345	1.492	1.421
μ (mm^{-1})	0.110	0.101	0.347	0.299
Final R indices	R_1 = 0.0558	R_1 = 0.0562	R_1 = 0.1117	R_1 = 0.0292
[I>2sigma(I)]	wR_2 = 0.1367	wR_2 = 0.0934	wR_2 =0.2969	wR_2 = 0.0724
R indices(all data)	R_1 = 0.0674	R_1 = 0.1494	R_1 = 0.1782	R_1 = 0.0335
	wR_2 = 0.1436	wR_2 = 0.1275	wR_2 = 0.3717	wR_2 = 0.0766
GoF	1.131	1.019	1.135	1.067
CCDC	1948533	1955133	1948658	1955134

Table S4 Single crystal data of **1BrNaAA**,**1INaAA** and **6BrNaAA**.

	1BrNaAA	**1INaAA**	**6BrNaAA**
Formula	$C_{13}H_9BrO_2$	$C_{13}H_9IO_2$	$C_{13}H_9BrO_2$
Formula weight	277.11	324.10	277.11
Space group	$P\bar{1}$	$P\bar{1}$	$P\bar{1}$
Crystal system	Triclinic	Triclinic	Triclinic
a (Å)	3.8948(4)	4.8142(2)	3.9738(2)
b (Å)	8.1149(8)	8.2211(4)	6.2790(4)
c (Å)	17.1274(16)	14.3363(7)	21.6685(13)
α (deg)	90.194(3)	102.141(2)	97.582(2)
β (deg)	91.725(4)	91.410(2)	93.911(2)
γ (deg)	92.396(4)	93.280(2)	92.391(2)
V (Å^3)	540.60(9)	553.41(4)	533.98(5)
Z	2	2	2
D_{calc}(g/cm^3)	1.702	1.945	1.723
μ (mm^{-1})	3.781	2.873	3.828
Final R indices	R_1 = 0.0264	R_1 = 0.0302	R_1 = 0.0383
[I>2sigma(I)]	wR_2 = 0.0609	wR_2 = 0.0601	wR_2 = 0.0996
R indices(all data)	R_1 = 0.0325	R_1 = 0.0408	R_1 = 0.0405
	wR_2 = 0.0633	wR_2 = 0.0633	wR_2 = 0.1009
GoF	1.050	1.086	1.092
CCDC	1948592	1948594	1948602

Table S5 Geometric parameters of **1FNaAA**, **1ClNaAA**, **1BrNaAA**,**1INaAA** and **6BrNaAA** in single crystals (parameter definitions are given in **Chart S1**).

	d (Å)	θ_1 (deg)	θ_2 (deg)	θ_3 (deg)
1FNaAA	3.769	0	103.73	69.39, 70.25
1ClNaAA	3.771	0	112.94	68.35, 87.98
1BrNaAA	3.895	0	112.17	67.69, 85.68
1INaAA	4.814	0	121.07	58.00, 52.82
6BrNaAA	3.974	0	96.35	64.04, 62.78
Ideal	<4.2	0	90	90

Table S6 Calculated attachment energies (E_{att}) for different crystalline planes in the single crystal of **1ClNaAA**.

hkl	Multiplicity	d_{hkl} / Å	Eatt (total) / kcal·mol^{-1}	Total facet area/ %
(001)	2	17.197	-6.662	57.541
(010)	2	7.983	-13.072	29.925
(100)	2	3.768	-31.389	8.069
(10-1)	2	3.693	-31.218	4.167
(11-1)	2	3.318	-33.015	0.217
(111)	2	3.287	-33.408	0.081

Table S7 Calculated attachment energies (E_{att}) for different crystalline planes in the single crystal of **6BrNaAA**.

hkl	Multiplicity	d_{hkl} / Å	Eatt (total) / kcal·mol^{-1}	Total facet area/ %
(001)	2	21.419	-4.669	67.063
(010)	2	6.216	-16.088	29.925
(01-1)	2	6.198	-16.134	9.311
(100)	2	3.960	-25.257	7.188
(10-1)	2	3.946	-25.341	4.836
(110)	2	3.420	-29.241	0.936
(1-11)	2	3.382	-29.565	0.045

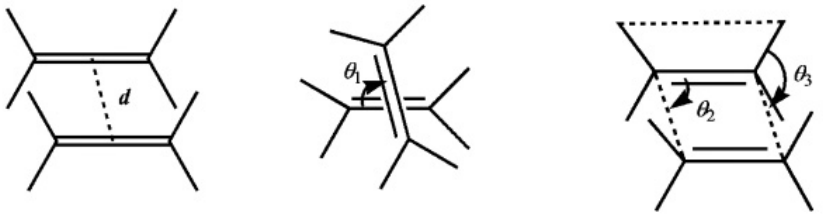


Chart S1 Geometric parameters for [2+2] topochemical dimerization. The angle θ_1 corresponds to the rotational angle of one double bond with respect to the other, while θ_2 corresponds to the obtuse angle of the parallelogram formed by the double bond carbons, whereas θ_3 measures the angle between the alkene substituents and cyclobutane planes, d is the distance between the two reactive olefin centers and the reaction double bonds of the adjacent molecules satisfy the parallel arrangement. The ideal values for θ_1, θ_2, and θ_3 for the best overlap of π-orbitals are 0°, 90° and 90°and d = 3.5-4.2 Å, respectively.[1]

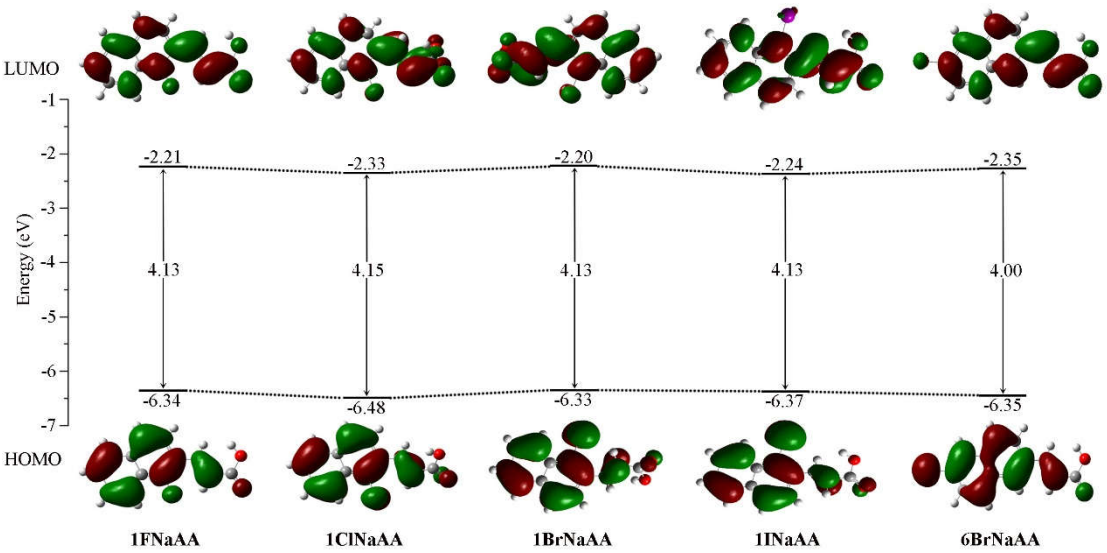


Figure S1 The frontier molecular orbital plots and energy levels for the HOMOs and LUMOs of **1FNaAA**, **1ClNaAA**, **1BrNaAA**, **1INaAA** and **6BrNaAA**.

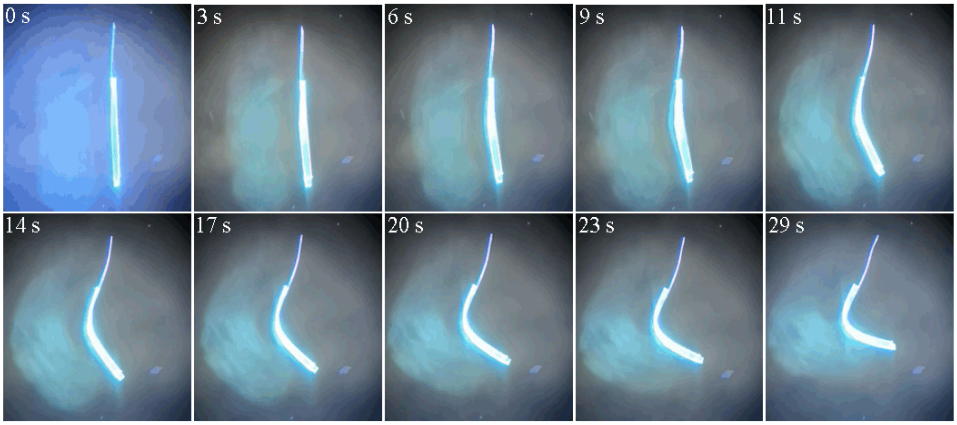


Figure S2 Optical microscopy photos of the bending of the slice-like crystal (700 μm × 110 μm × 15 μm) attached by a needle-like crystal (360 μm × 33 μm × 15 μm) of **1ClNaAA**upon irradiation with 365 nm light (3W) for different times (the irradiation direction: from left to right).

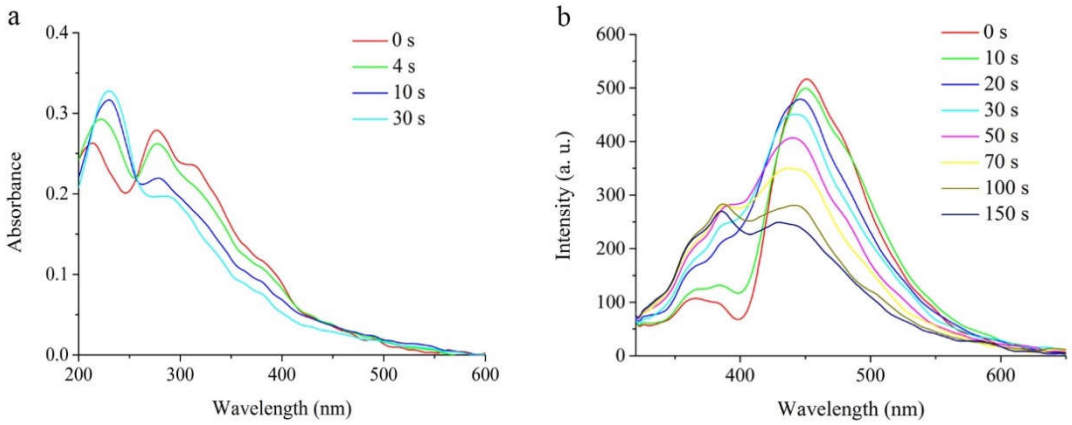


Figure S3 UV-vis absorption (a) and fluorescence emission (b; λ_{ex} = 320 nm) spectra of **1ClNaAA** in the microcrystals before and after irradiation by 365 nm light (3 W) for different times.

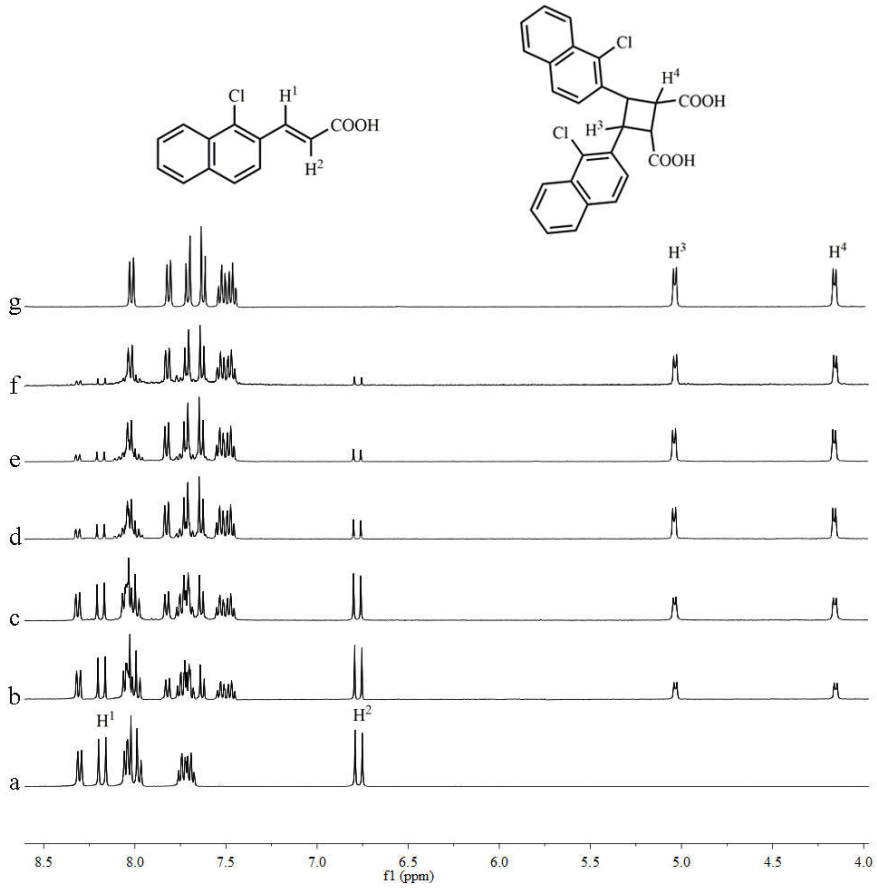


Figure S4 ^{1}H NMR spectra of the samples before (a) and after irradiation of the microcrystals **1ClNaAA** by 365 nm light (3 W) for 30 s (b), 1 min (c), 3 min (d), 5 min (e) and 10 min (f), followed by dissolving in DMSO-d_6;(g) ^{1}H NMR spectrum of **D-1ClNaAA** in DMSO-d_6.

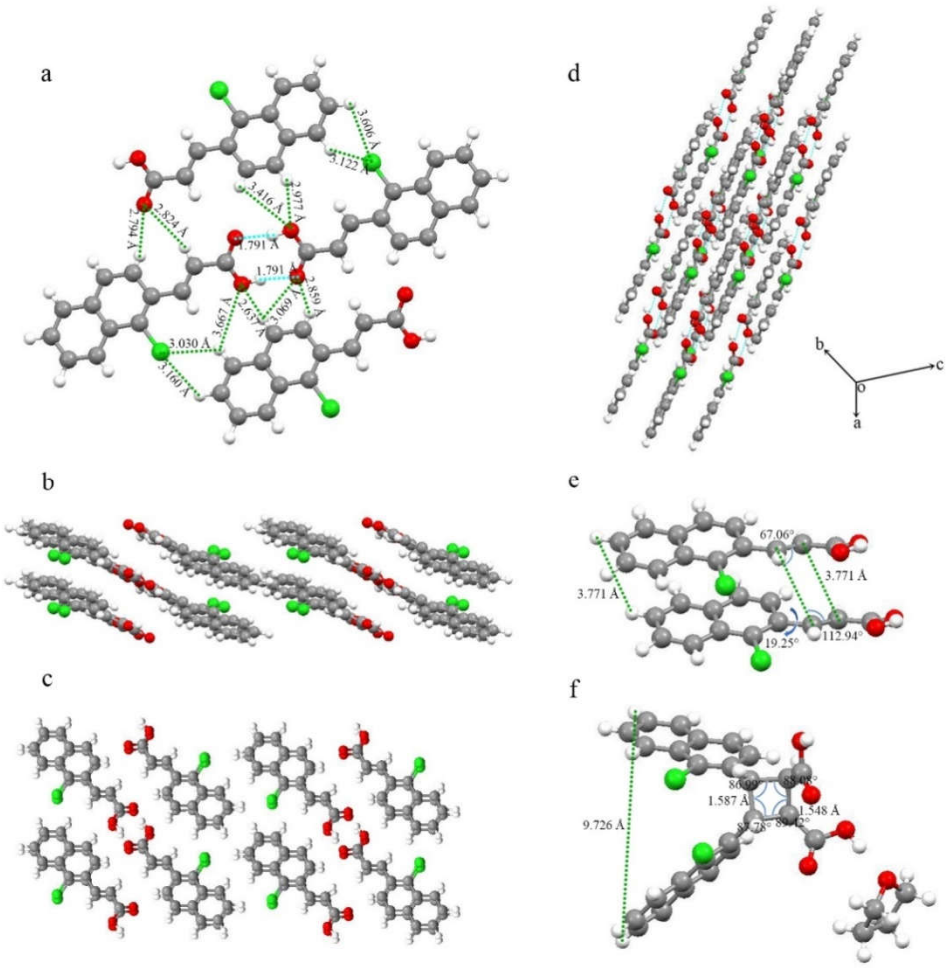


Figure S5 (a) The H-bonds in the single crystal of **1ClNaAA**; the single crystal structure of viewed along the b-axis (b) and a-axis (c); (d) the packing of H-bonded dimer in single crystal; (e) the dihedral angle between the plane of the double bond and naphthalene and distance between two adjacent parallel molecules; (f) the molecular configuration of β-type **D-1ClNaAA** in the single crystal.

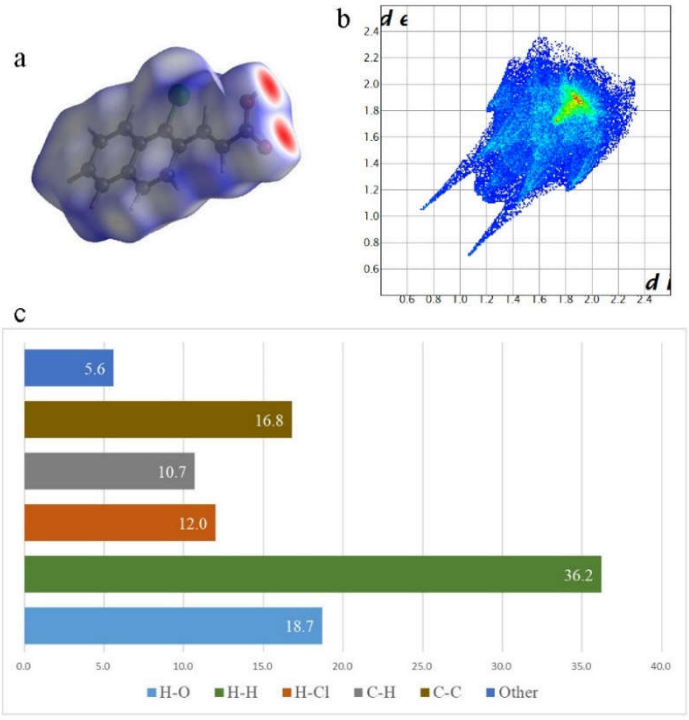


Figure S6 (a) Hirshfeld surface mapped with d_{norm} of **1ClNaAA**; (b) the two-dimensional fingerprint plots in crystal stacking for **1ClNaAA**; (c) the percentage of individual atomic contacts contribution to Hirshfeld surface. The surfaces are shown as transparent to allow visualization of the orientation and conformation of the functional groups in the molecules.

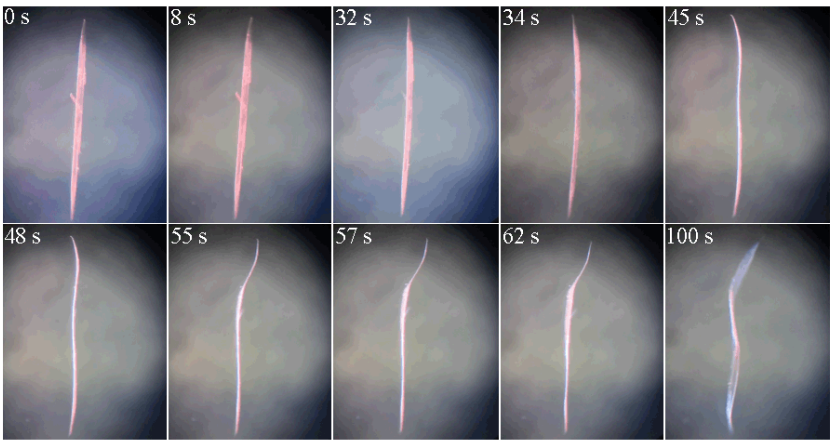


Figure S7 Optical microscopy photos of the bending, rotating and coiling of the slice-like crystal of **1BrNaAA** (700 μm × 110 μm × 15 μm) upon irradiation with 365 nm light (3W) for different times (the irradiation direction: from left to right).

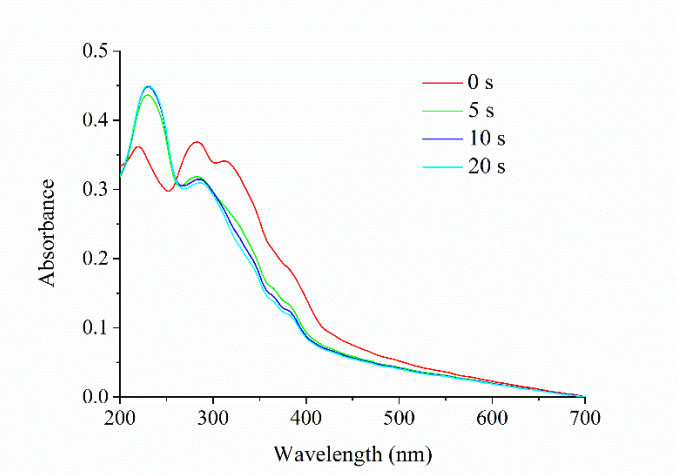


Figure S8 UV-vis absorption spectra of **1BrNaAA** in the microcrystals before and after irradiation by 365 nm light (3 W) for different times.

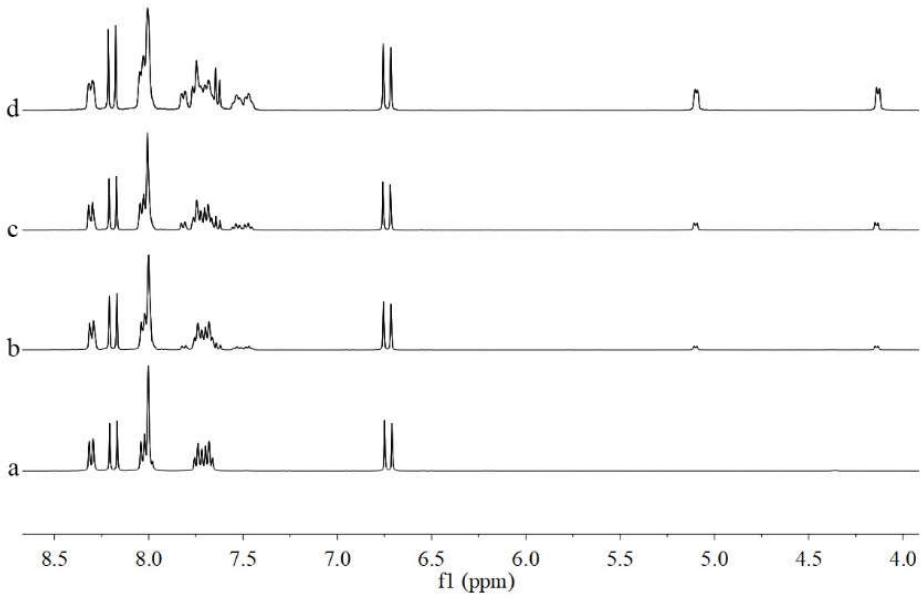


Figure S9 ^{1}H NMR spectra of the samples before (a) and after irradiation of the microcrystals **1BrNaAA** by 365 nm light (3 W) for 1 min (b), 5 min (c) and 10 min (d), followed by dissolving in DMSO-d_6.

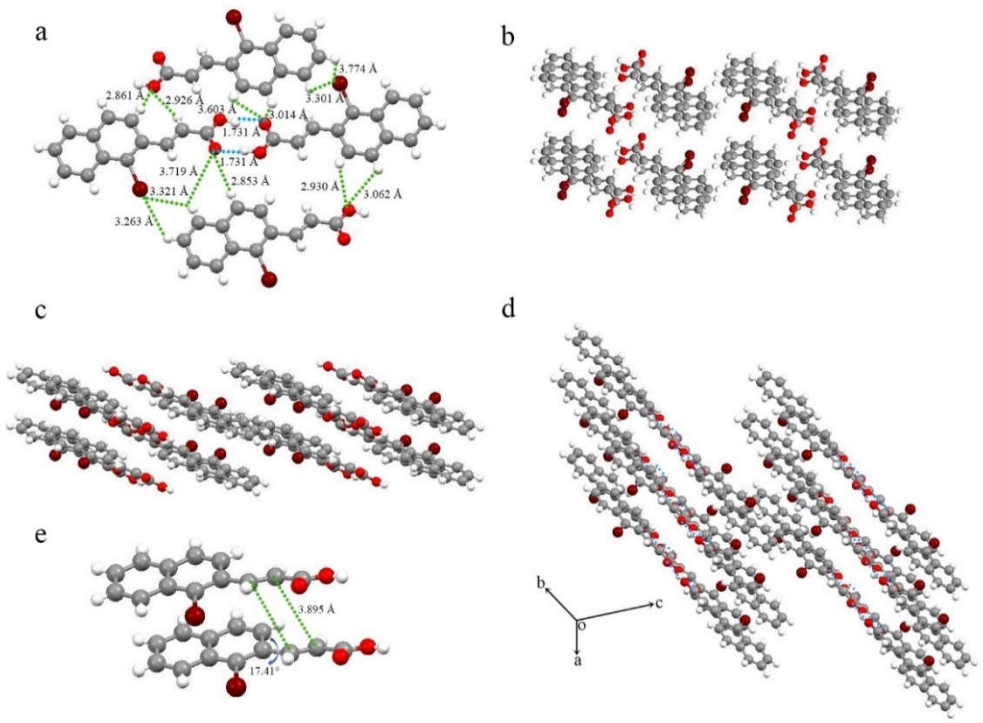


Figure S10 (a) The H-bonds in the single crystal of **1BrNaAA**; the single crystal structure of viewed along the a-axis (b) and b-axis (c); (d) the packing of H-bonded dimer in single crystal; (e) the dihedral angle between the plane of the double bond and naphthalene and distance between two adjacent parallel molecules.

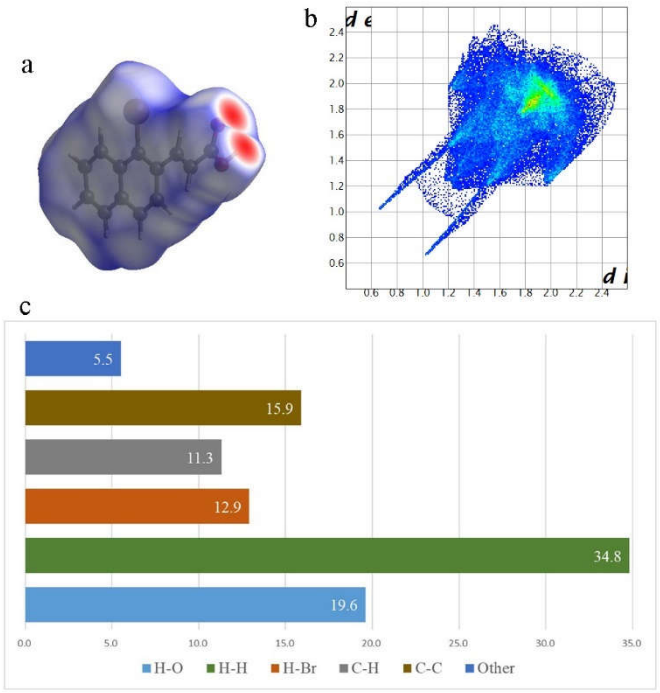


Figure S11 (a) Hirshfeld surface mapped with d_{norm} of **1BrNaAA**; (b) the two-dimensional fingerprint plots in crystal stacking for **1BrNaAA**; (c) the percentage of individual atomic contacts contribution to Hirshfeld surface. The surfaces are shown as transparent to allow visualization of the orientation and conformation of the functional groups in the molecules.

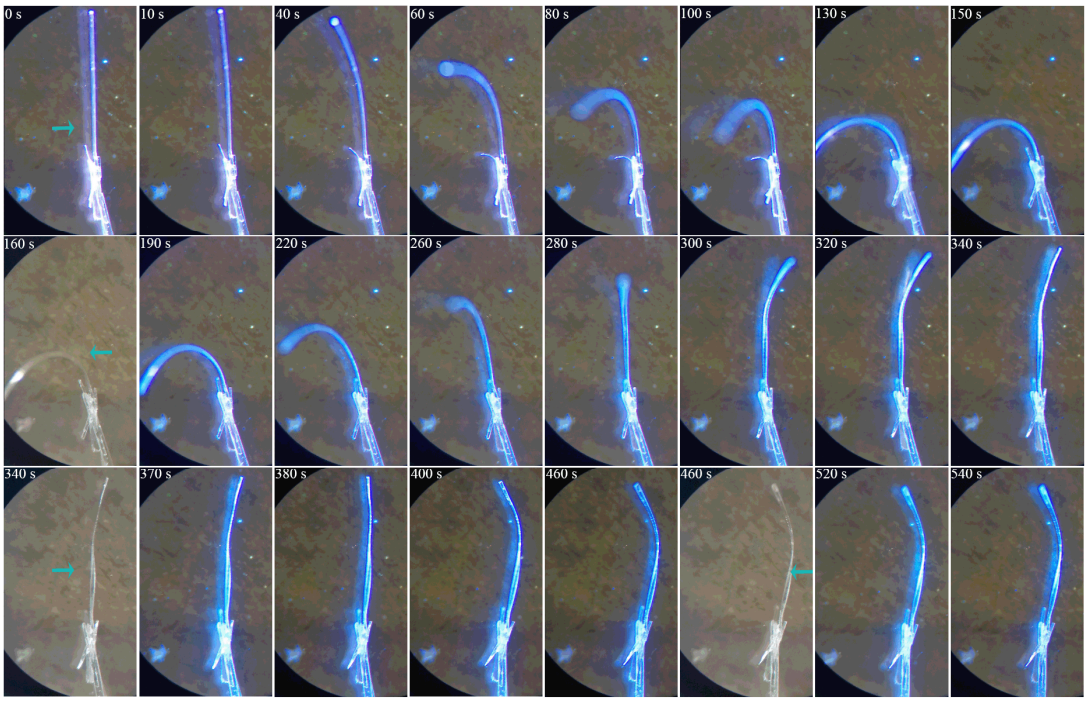


Figure S12 Optical microscopy photos of the bending and twisting of the slice-like crystals of **6BrNaAA** (2400 μm × 80 μm × 40 μm) upon irradiation with 365 nm light (3W) for different times(the arrows indicate the irradiation direction).

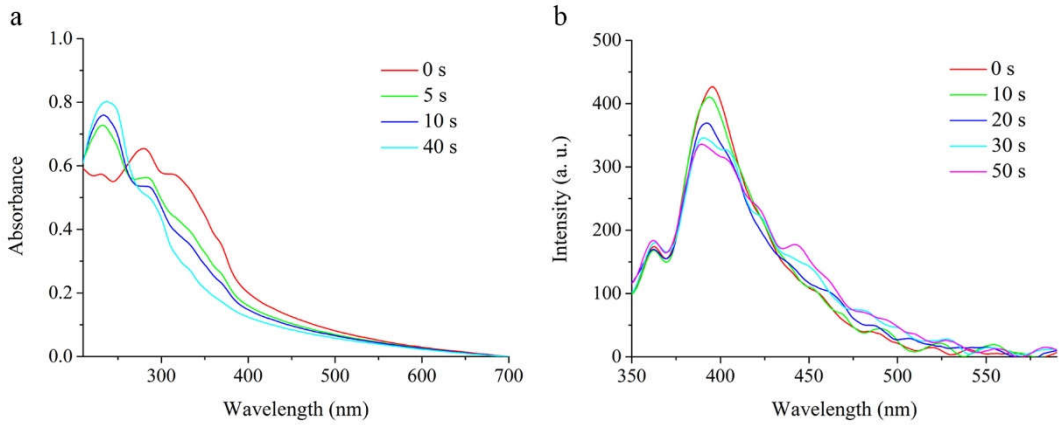


Figure S13 UV-vis absorption (a) and fluorescence emission (b; λ_{ex} = 320 nm) spectra of **6BrNaAA** in the microcrystals before and after irradiation by 365 nm light (3 W) for different times.

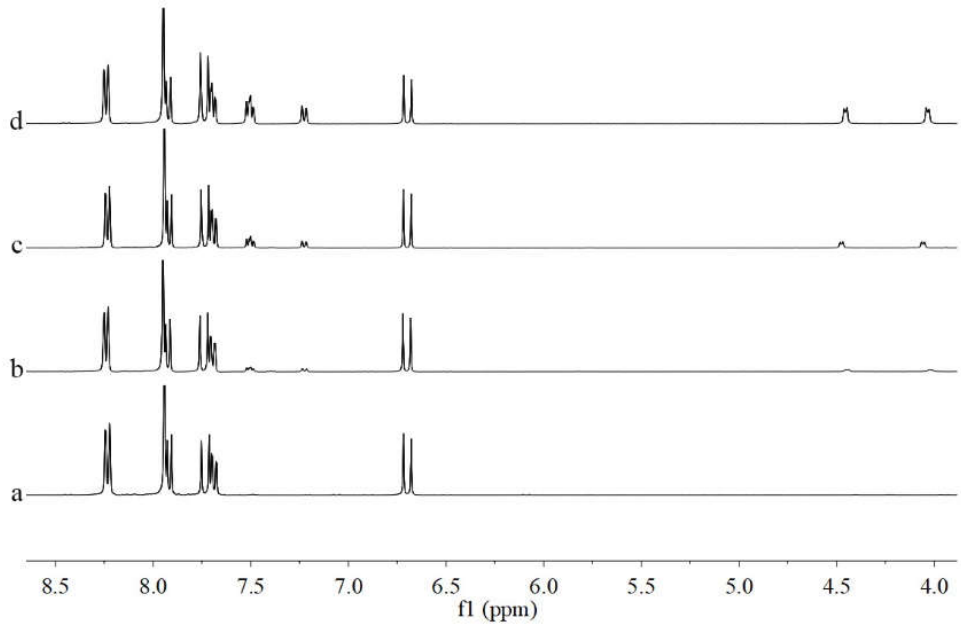


Figure S14 ^{1}H NMR spectra of the samples before (a) and after irradiation of the microcrystals **6BrNaAA** by 365 nm light (3 W) for 1 min (b), 5 min (c) and 10 min (d), followed by dissolving in DMSO-d_6.

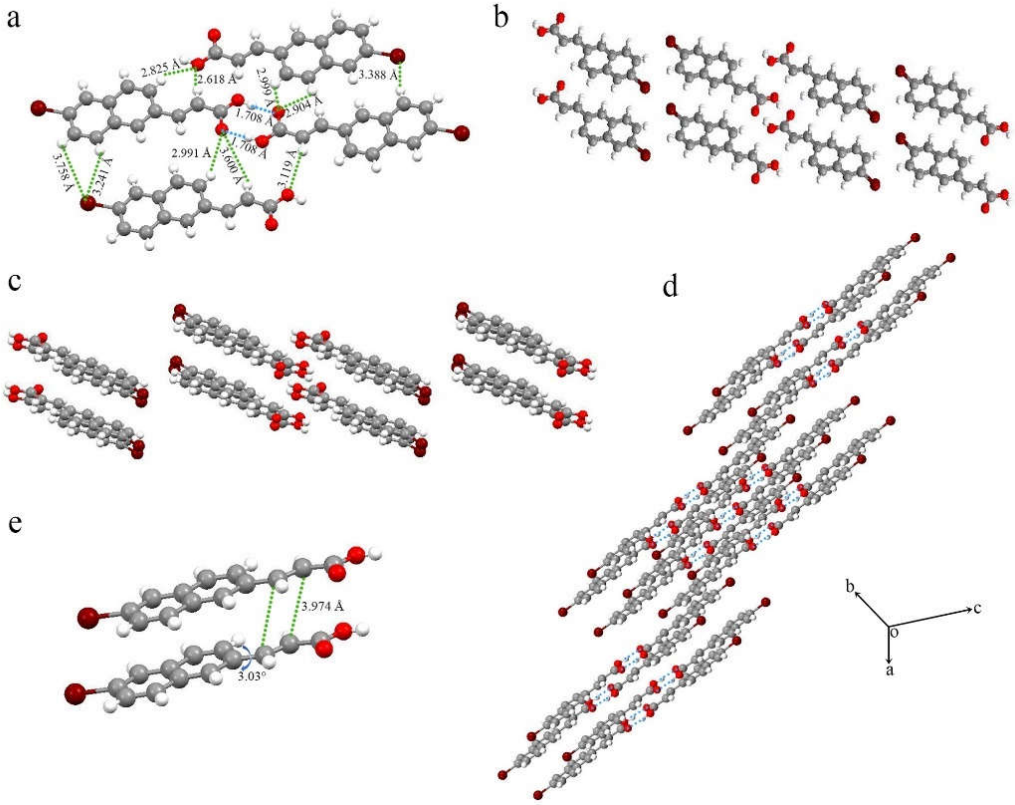


Figure S15 (a) The H-bonds in the single crystal of **6BrNaAA**; the single crystal structure of viewed along the a-axis (b) and b-axis (c); (d) the packing of H-bonded dimer in single crystal; (e) the dihedral angle between the plane of the double bond and naphthalene and distance between two adjacent parallel molecules.

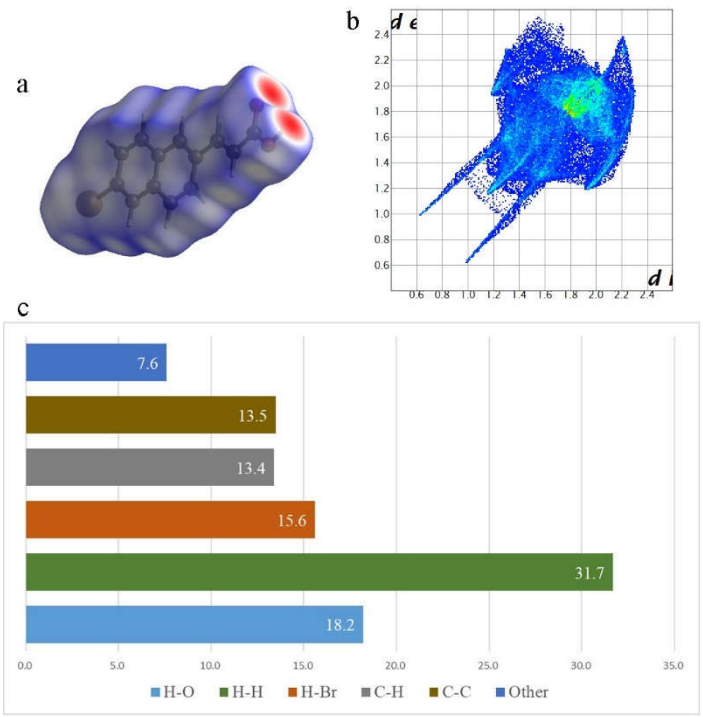


Figure S16 (a) Hirshfeld surface mapped with d_{norm} of **6BrNaAA**; (b) the two-dimensional fingerprint plots in crystal stacking for **6BrNaAA**; (c) the percentage of individual atomic contacts contribution to Hirshfeld surface. The surfaces are shown as transparent to allow visualization of the orientation and conformation of the functional groups in the molecules.

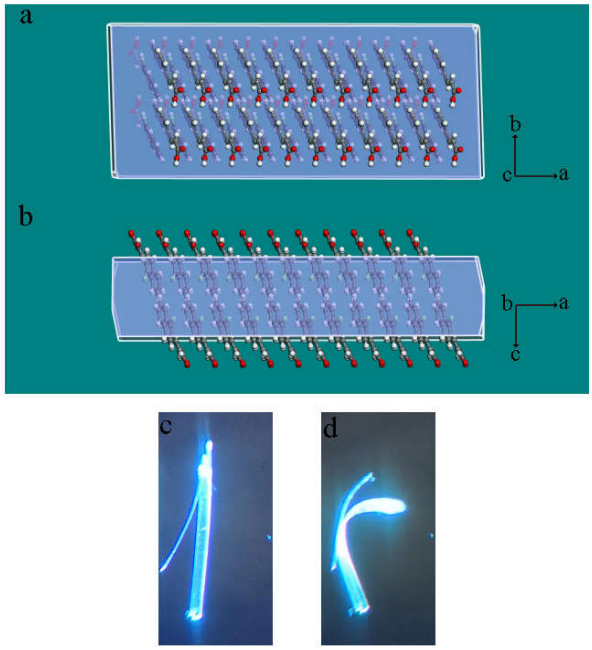


Figure S17 Calculated growth morphology of **1ClNaAA**in single crystal (a, b) and microscopic images showing the widest face (c) and the bending of the widest face of **1ClNaAA**crystal(d).

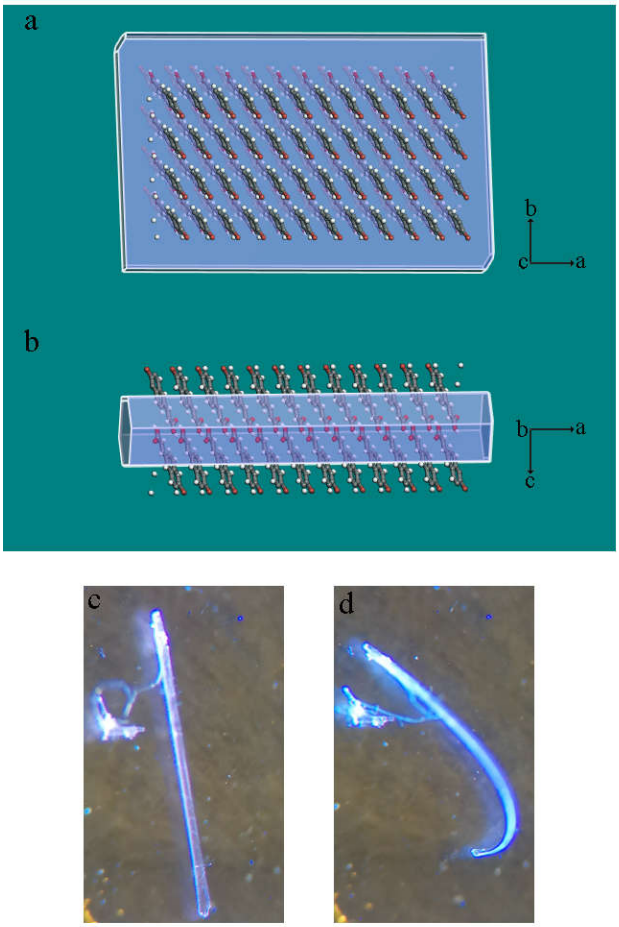


Figure S18 Calculated growth morphology of **6BrNaAA**in single crystal (a, b) and microscopic images showing the widest face (c) and the bending of the widest face of **6BrNaAA**crystal(d).

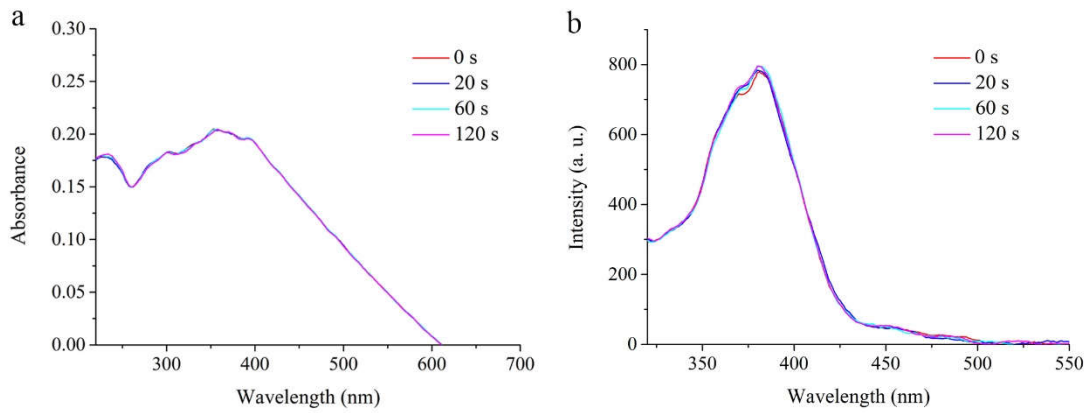


Figure S19 UV-vis absorption (a) and fluorescence emission (b; λ_{ex} = 320 nm) spectra of **1INaAA** in the microcrystals before and after irradiation by 365 nm light (3 W) for different times.

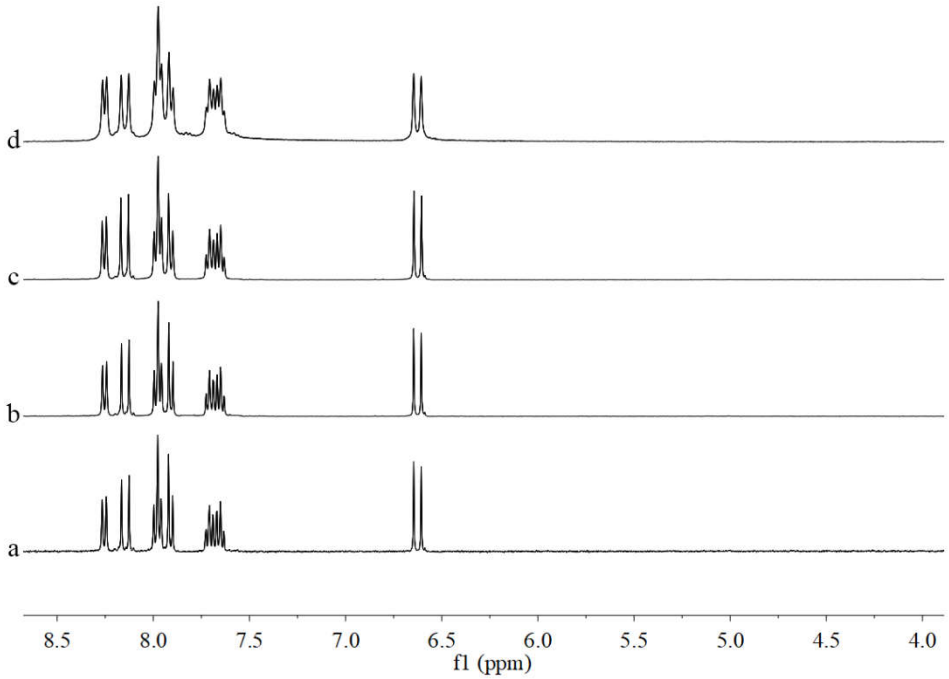


Figure S20 ^{1}H NMR spectra of the samples before (a) and after irradiation of the microcrystals **1INaAA** by 365 nm light (3 W) for 5 min (b), 10 min (c) and 1 h (d), followed by dissolving in DMSO-d_6.

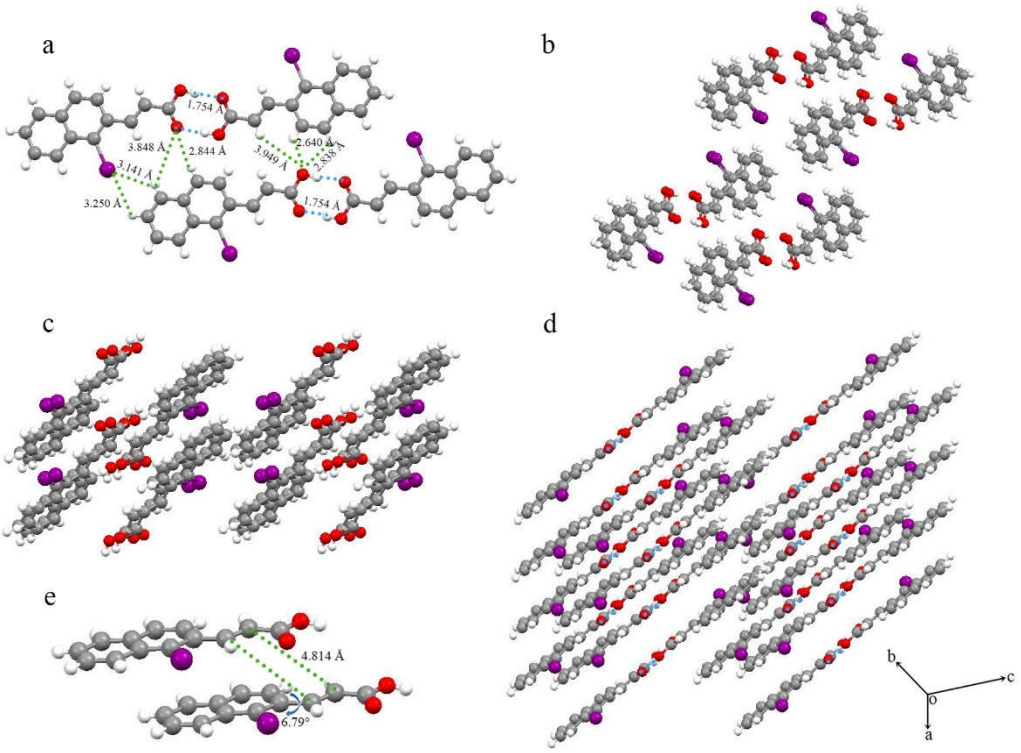


Figure S21 (a) The H-bonds in the single crystal of **1INaAA**; the single crystal structure of viewed along the a-axis (b) and b-axis (c); (d) the packing of H-bonded dimer in single crystal; (e) the dihedral angle between the plane of the double bond and naphthalene and distance between two adjacent parallel molecules.

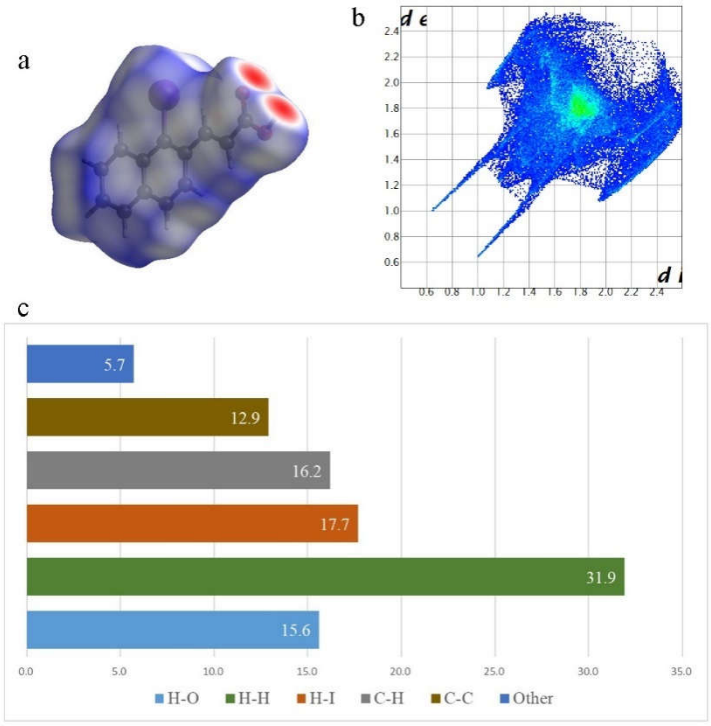


Figure S22 (a) Hirshfeld surface mapped with d_{norm} of **1INaAA**; (b) the two-dimensional fingerprint plots in crystal stacking for **1INaAA**; (c) the percentage of individual atomic contacts contribution to Hirshfeld surface. The surfaces are shown as transparent to allow visualization of the orientation and conformation of the functional groups in the molecules.

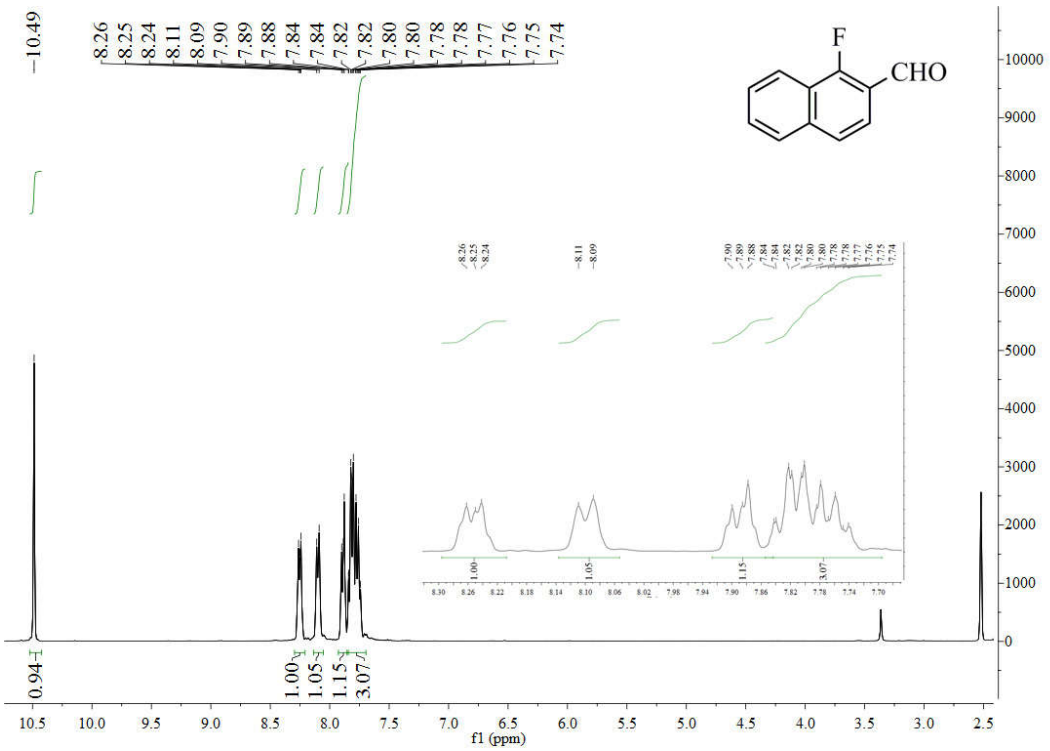


Figure S23 ^{1}H NMR (400 MHz) spectrum of compound **1** in DMSO-d_6.

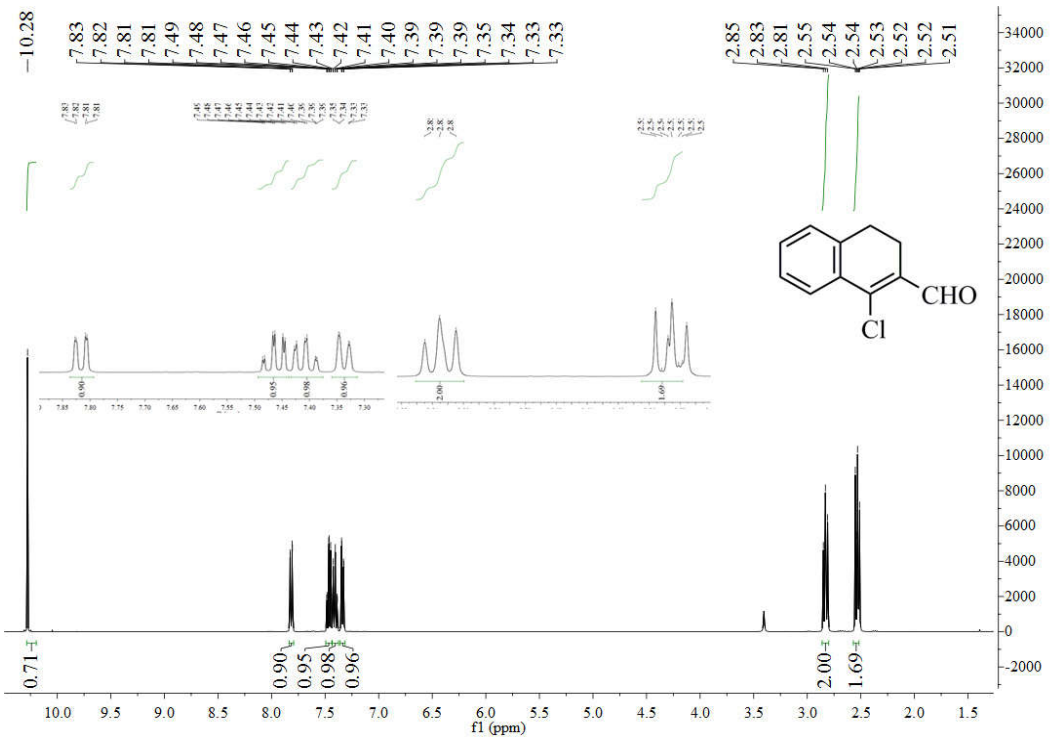


Figure S24 ^{1}H NMR (400 MHz) spectrum of compound **2** in DMSO-d_6.

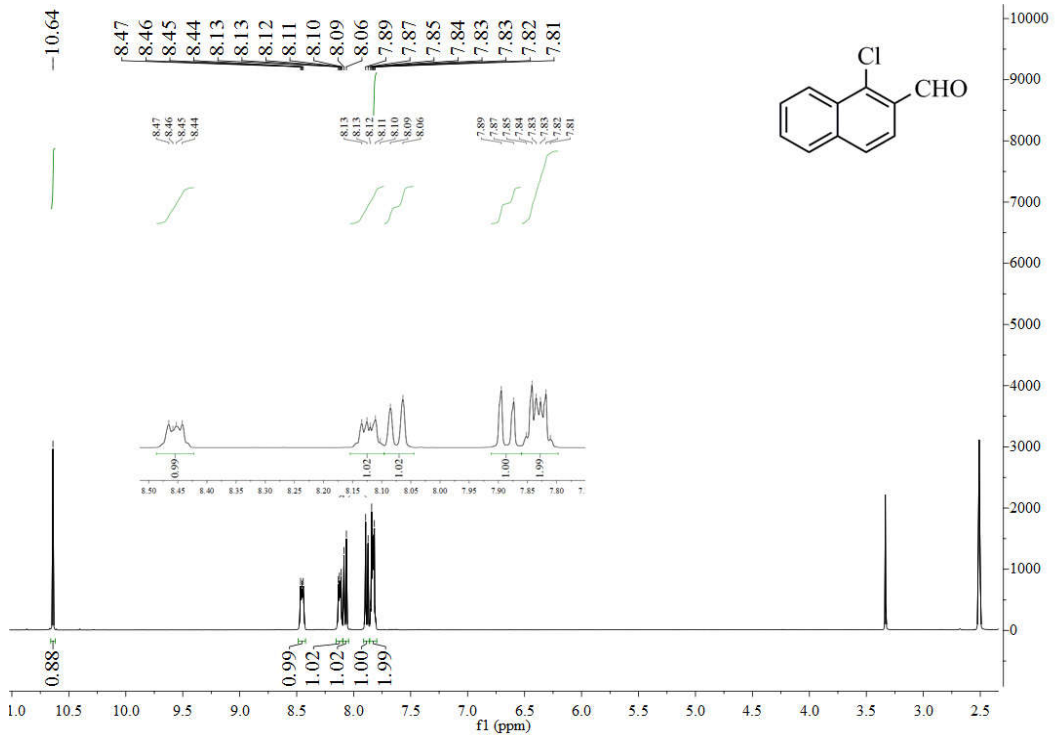


Figure S25 ^{1}H NMR (400 MHz) spectrum of compound **3** in DMSO-d_6.

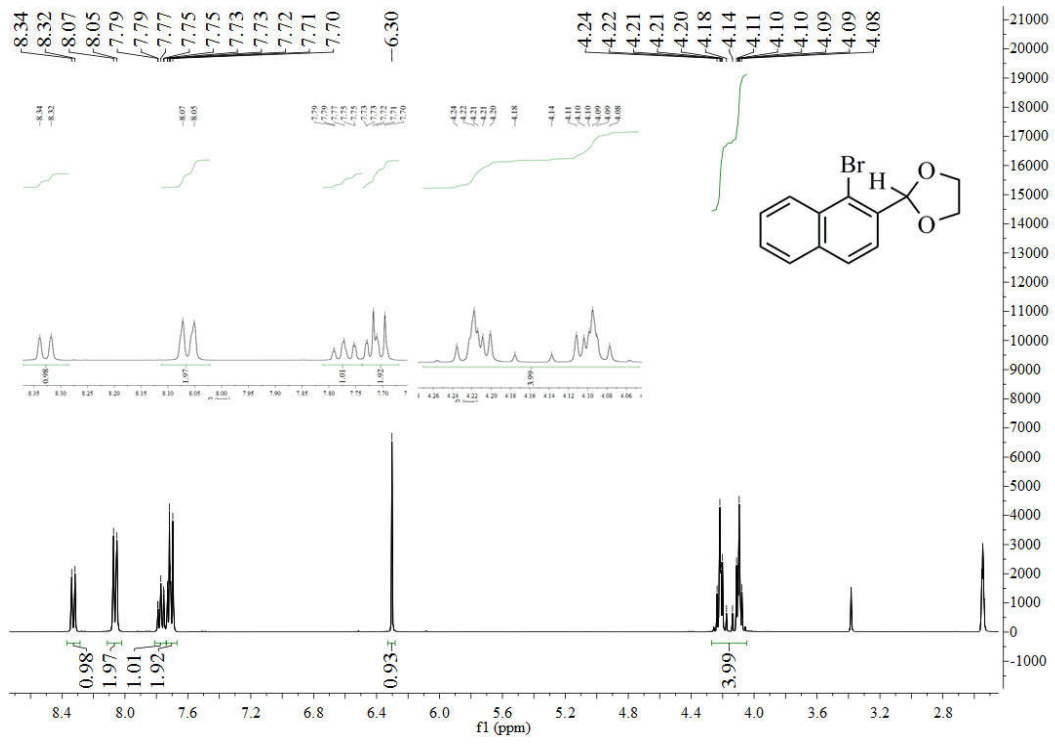


Figure S26 ^{1}H NMR (400 MHz) spectrum of compound **4** in DMSO-d_6.

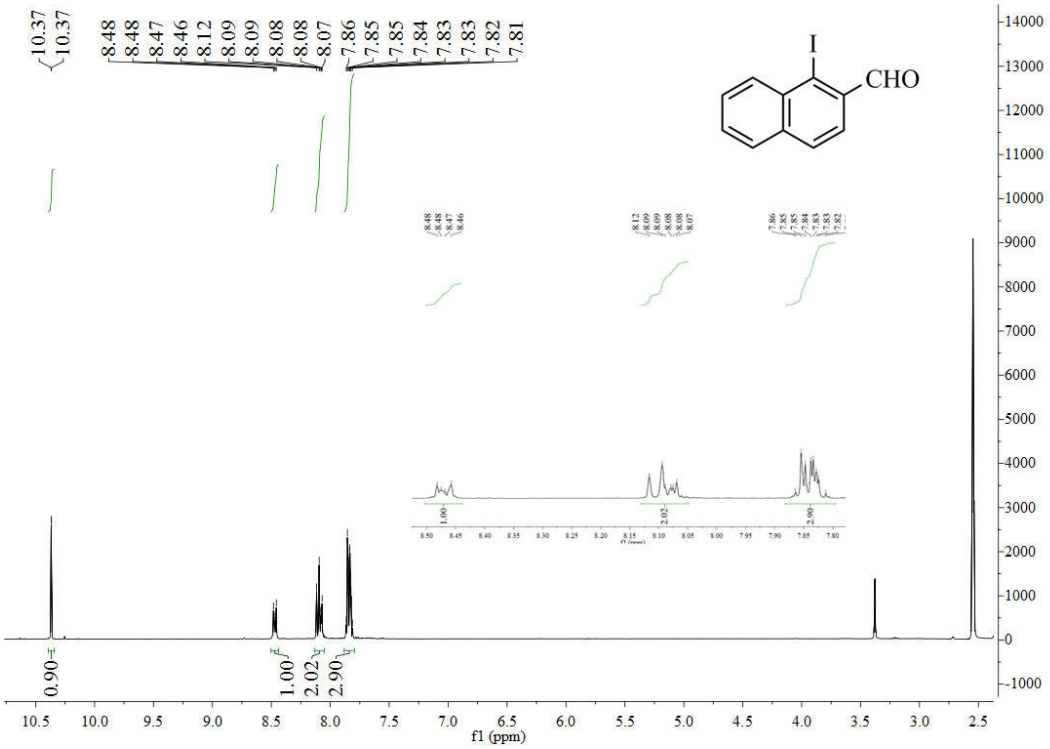


Figure S27 ^{1}H NMR (400 MHz) spectrum of compound **5** in DMSO-d_6.

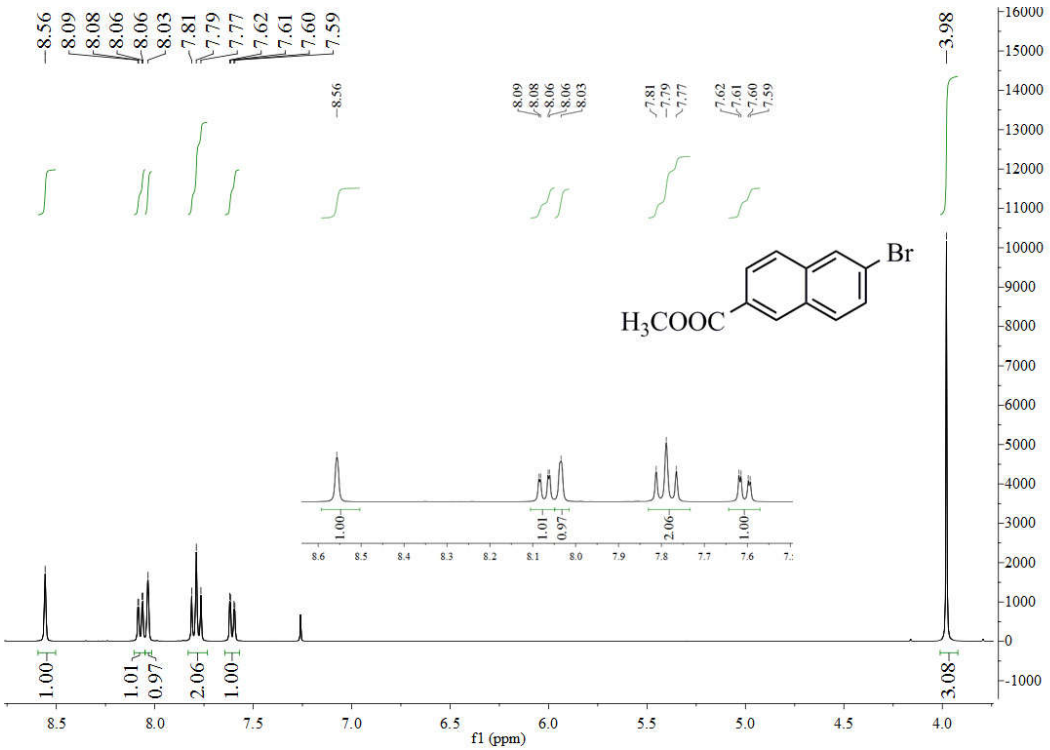


Figure S28 ^{1}H NMR (400 MHz) spectrum of compound **6** in $CDCl_3$.

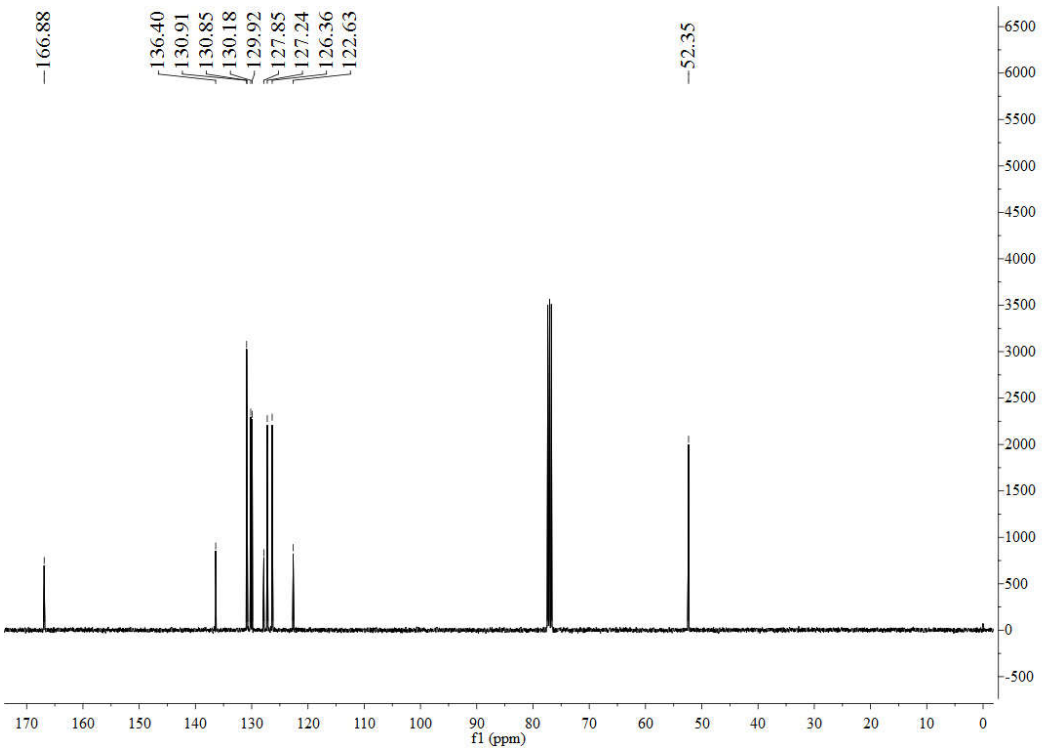


Figure S29 ^{13}C NMR (101 MHz) spectrum of compound **6** in $CDCl_3$.

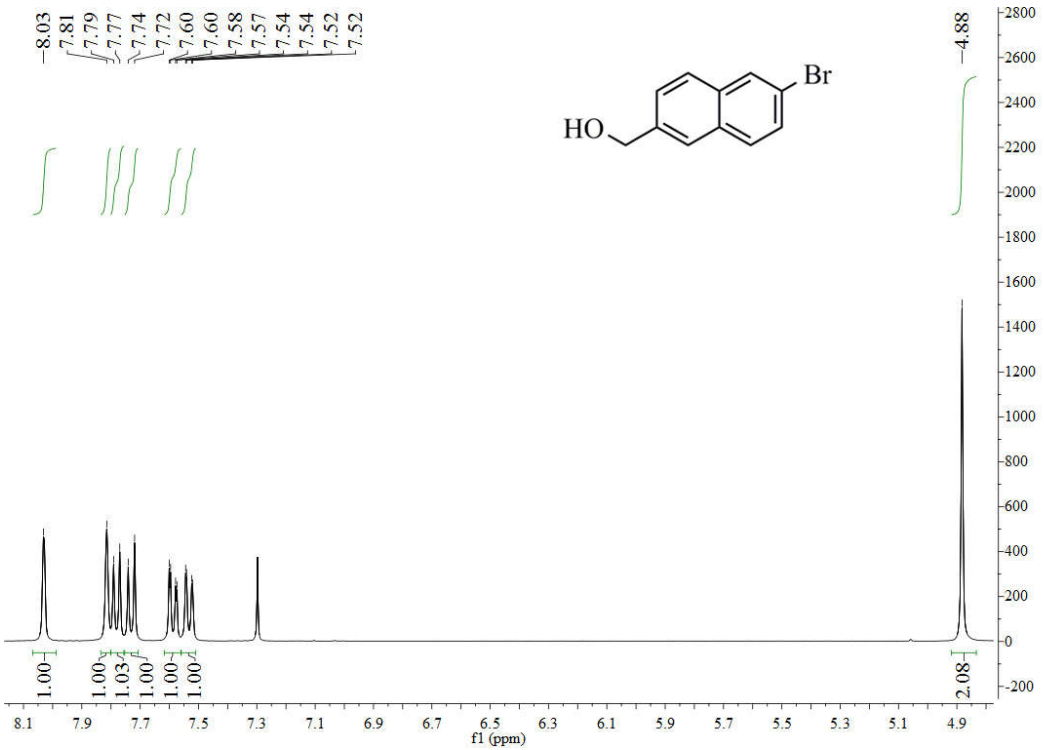


Figure S30 ^{1}H NMR (400 MHz) spectrum of compound **7** in $CDCl_3$.

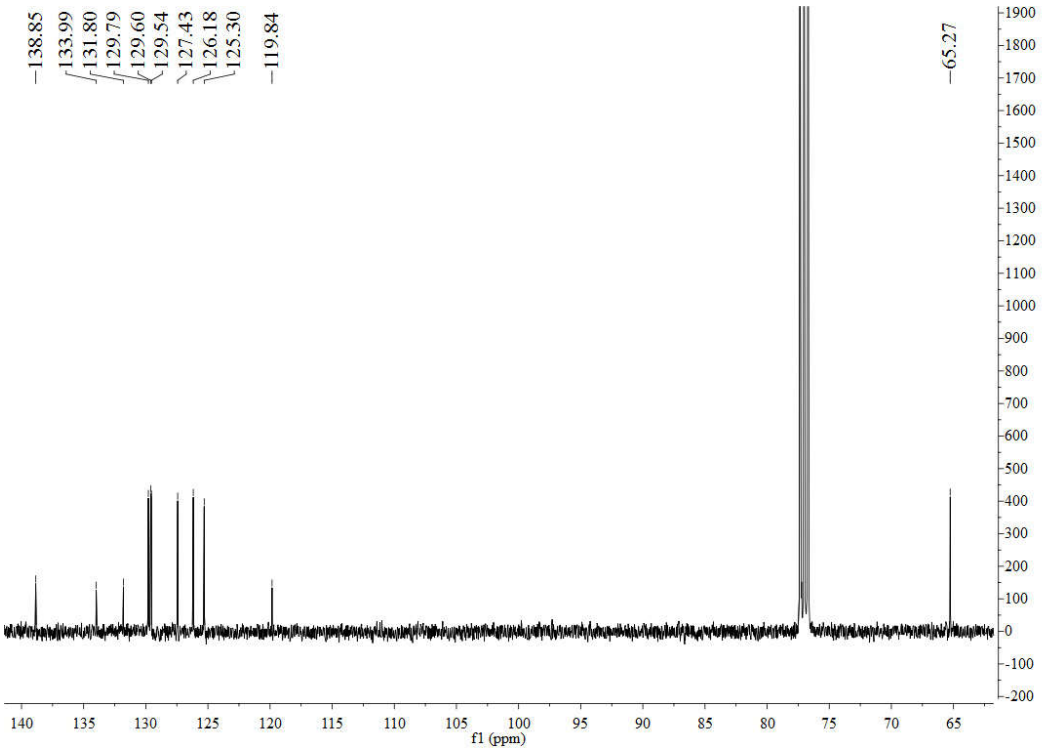


Figure S31 ^{13}C NMR (101 MHz) spectrum of compound **7** in $CDCl_3$.

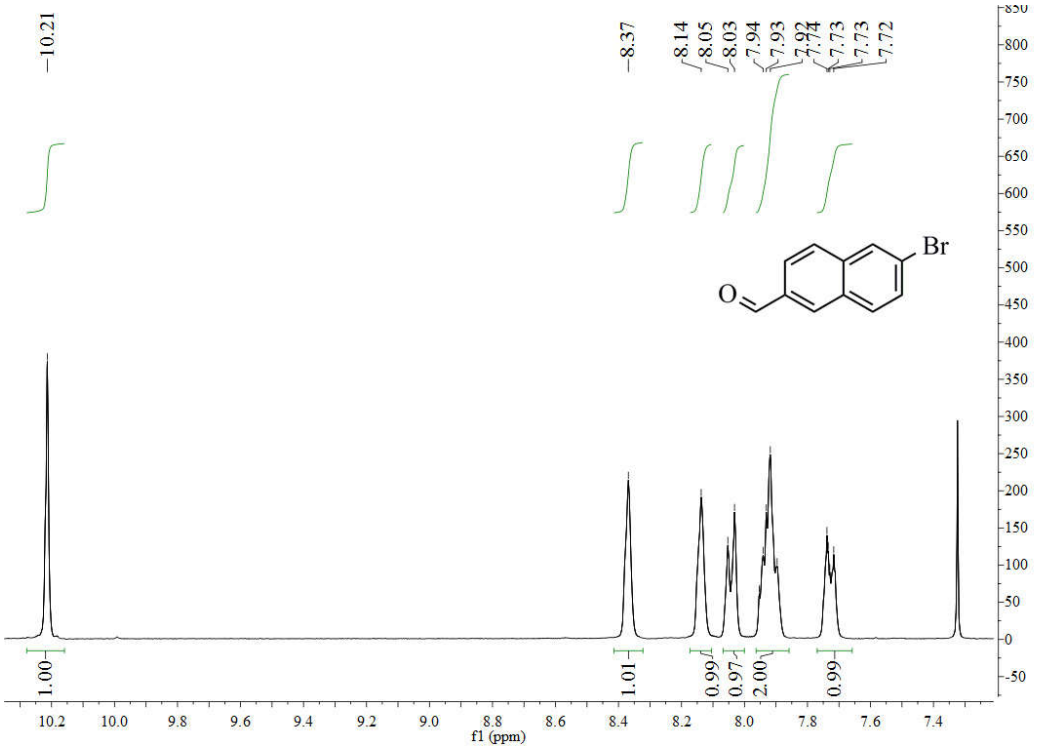


Figure S32 ^{1}H NMR (400 MHz) spectrum of compound **8** in $CDCl_3$.

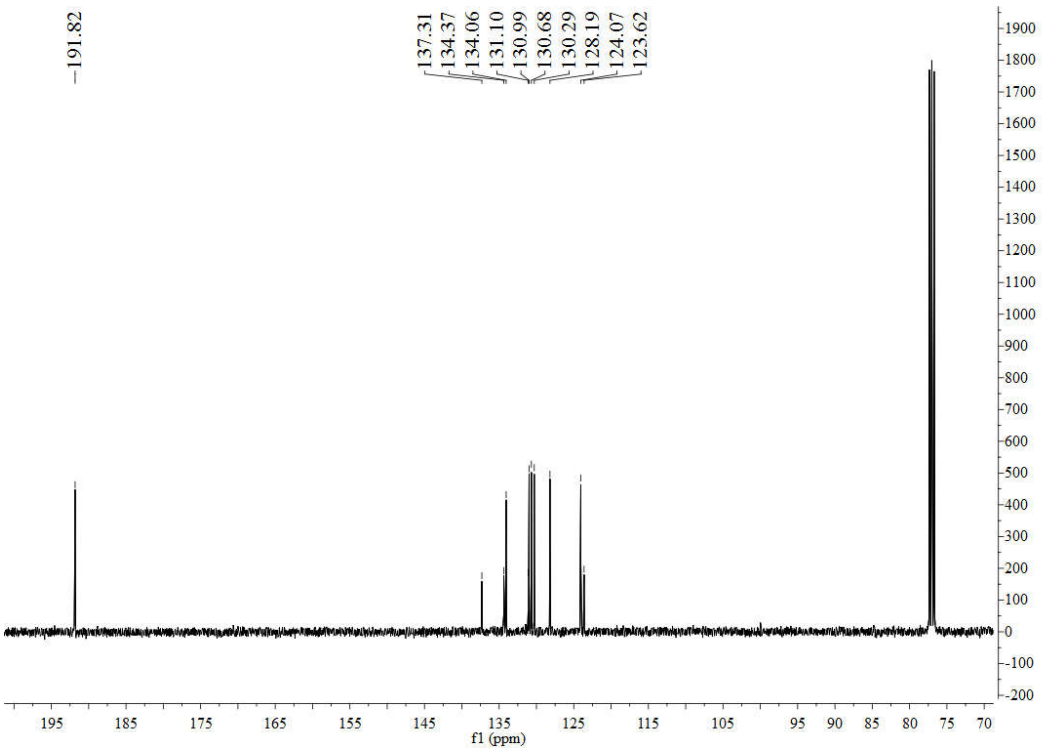


Figure S33 ^{13}C NMR (101 MHz) spectrum of compound **8** in $CDCl_3$.

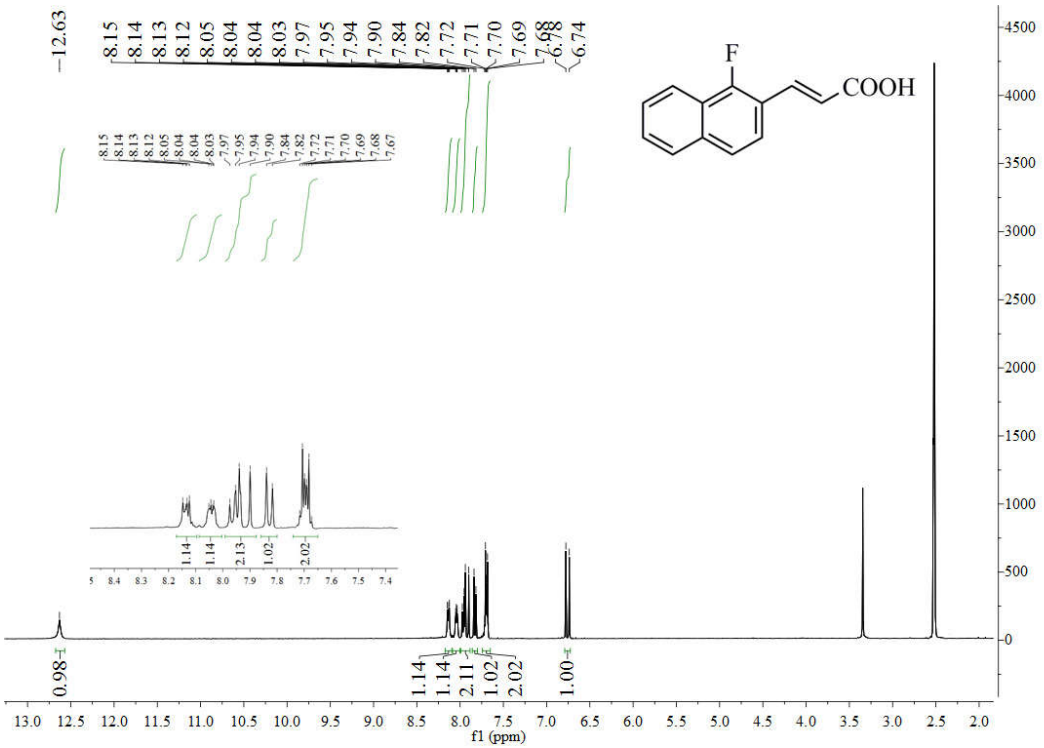


Figure S34 ^{1}H NMR (400 MHz) spectrum of **1FNaAA** in DMSO-d_6.

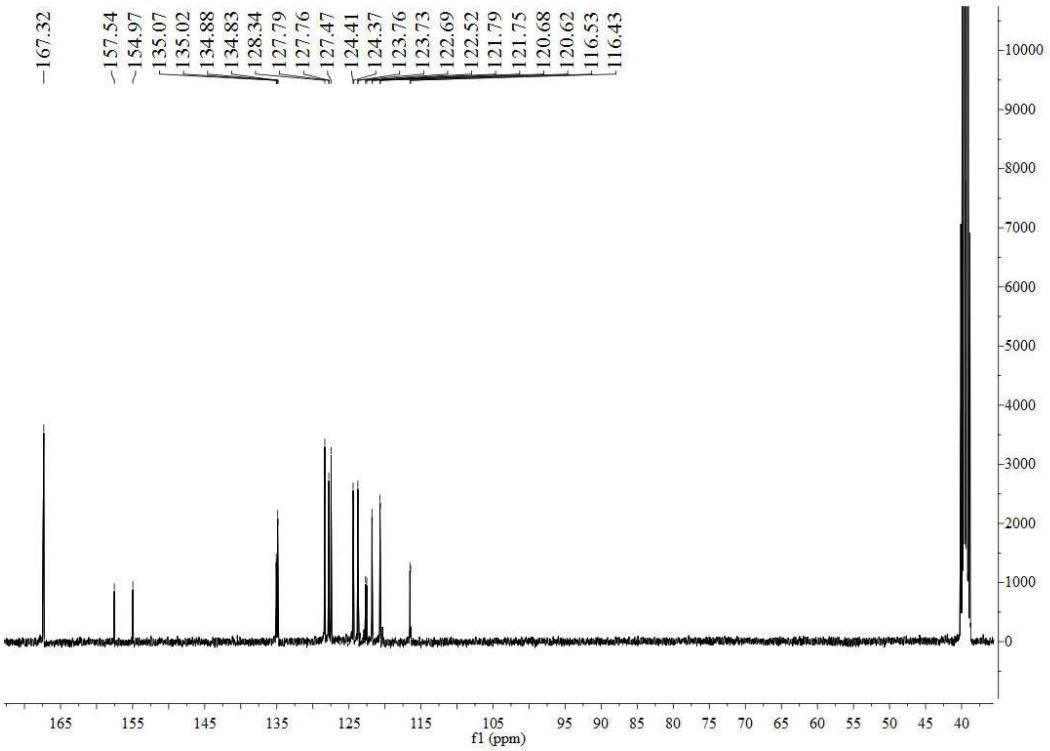
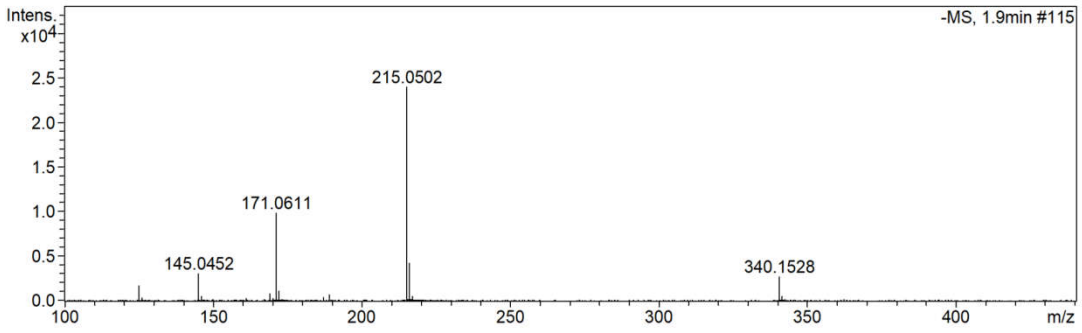


Figure S35 ^{13}C NMR (101 MHz) spectrum of **1FNaAA** in DMSO-d_6.



Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e¡¥ Conf	mSigma	Std I	Std Mean m/z	Std I VarNorm	Std m/z Diff	Std Comb Dev
215.0502	1	C 13 H 8 F O 2	215.0514	5.5	5.3	9.5	ok	even	20.58	0.0399	0.0012	0.0162	0.0004	0.8427

Figure S36 The HRMS of **1FNaAA**.

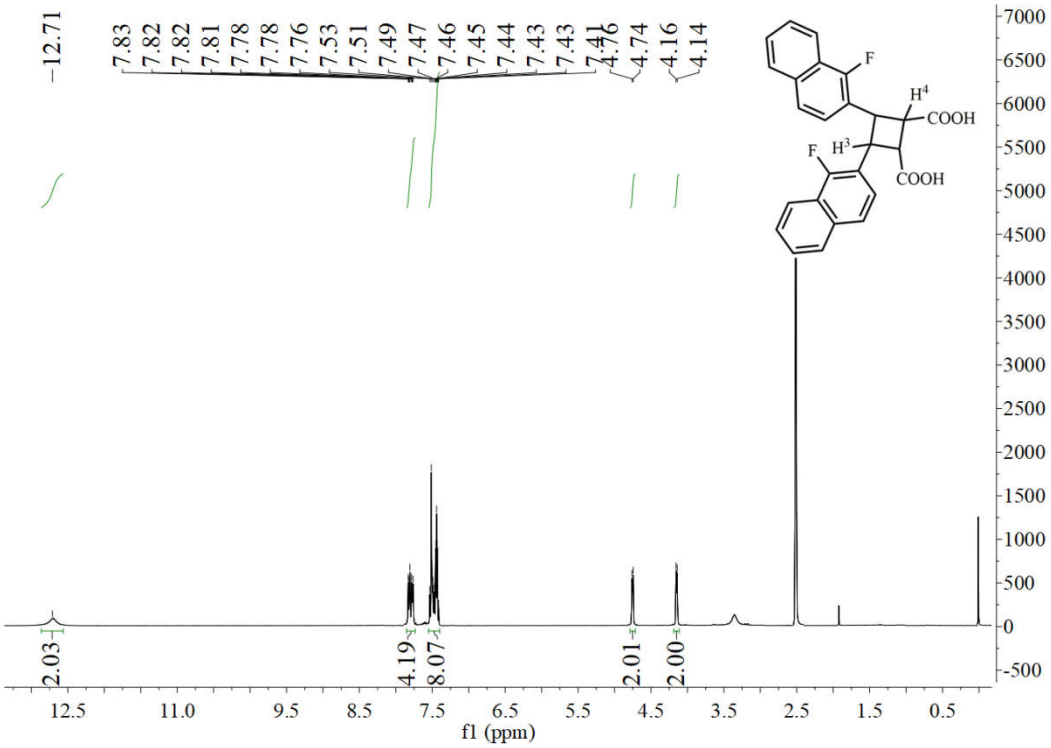


Figure S37 ^{1}H NMR (400 MHz) spectrum of **D-1FNaAA** in DMSO-d_6.

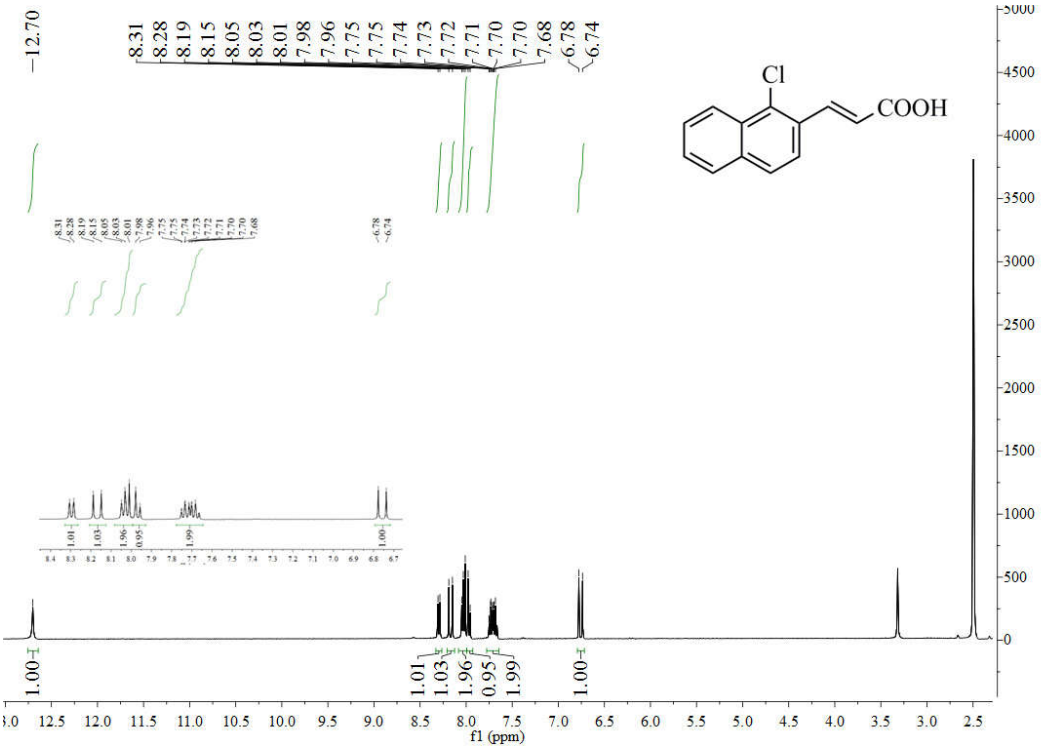


Figure S38 ^{1}H NMR (400 MHz) spectrum of **1ClNaAA** in DMSO-d_6.

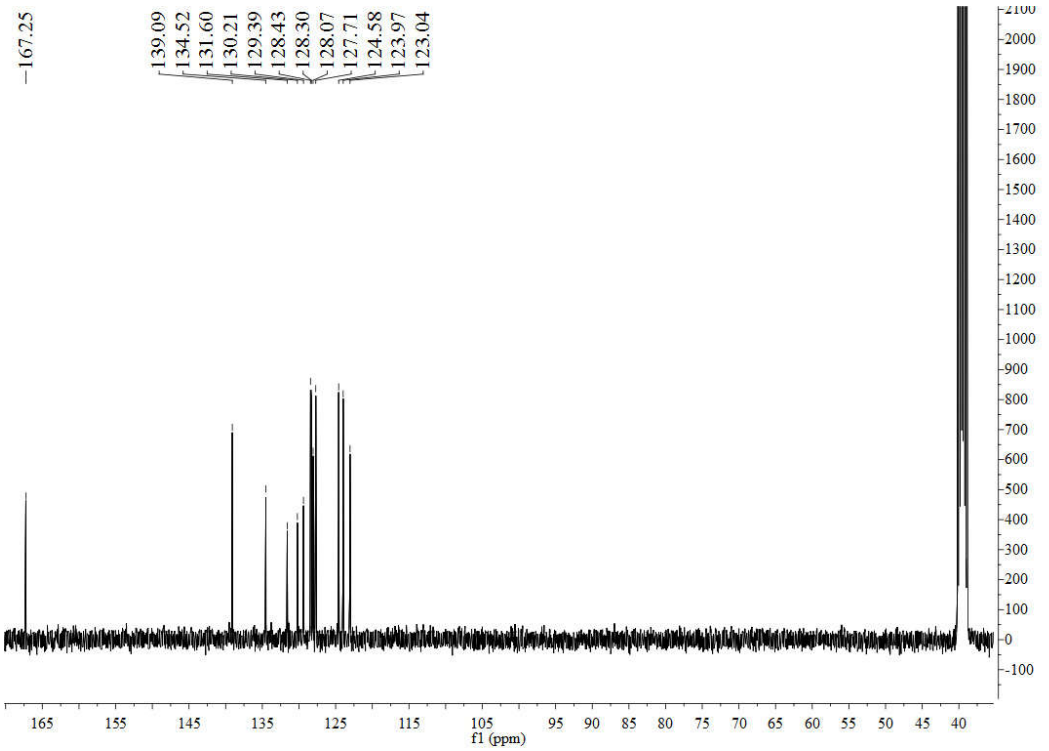
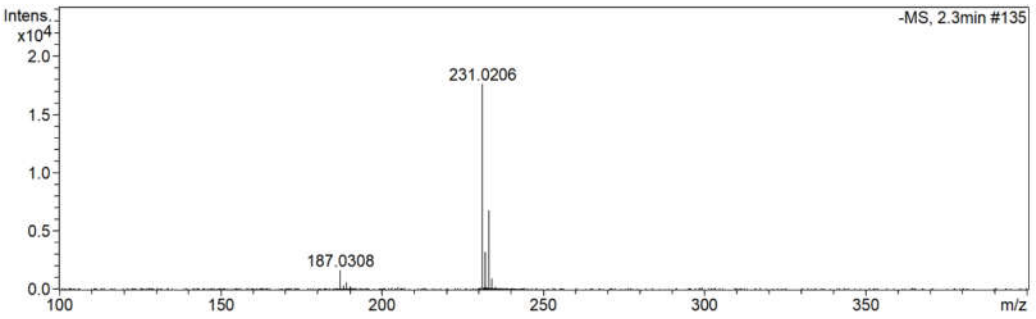


Figure S39 ^{13}C NMR (101 MHz) spectrum of **1ClNaAA** in DMSO-d_6.



Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma	Std I	Std Mean m/z	Std I VarNorm	Std m/z Diff	Std Comb Dev
231.0206	1	C 13 H 8 Cl O 2	231.0218	5.1	4.1	9.5	ok	even	32.16	0.0471	0.0010	0.0175	0.0006	0.8427

Figure S40 The HRMS of **1ClNaAA**.

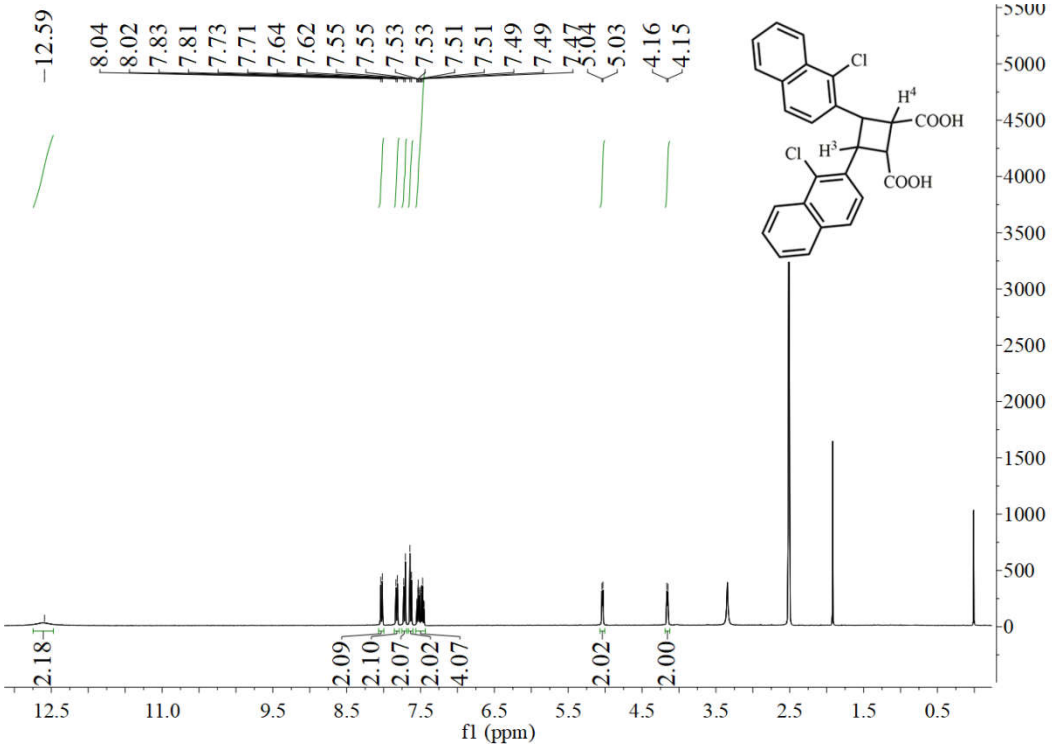


Figure S41 ^{1}H NMR (400 MHz) spectrum of **D-1ClNaAA** in DMSO-d_6.

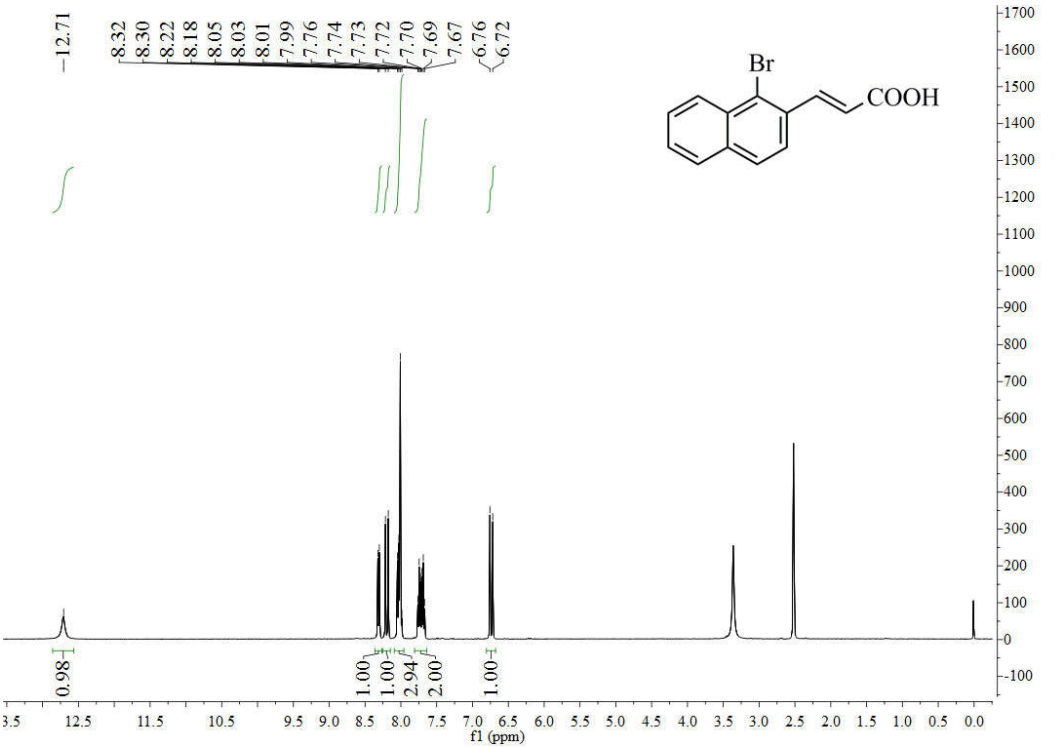


Figure S42 ^{1}H NMR (400 MHz) spectrum of **1BrNaAA** in DMSO-d_6.

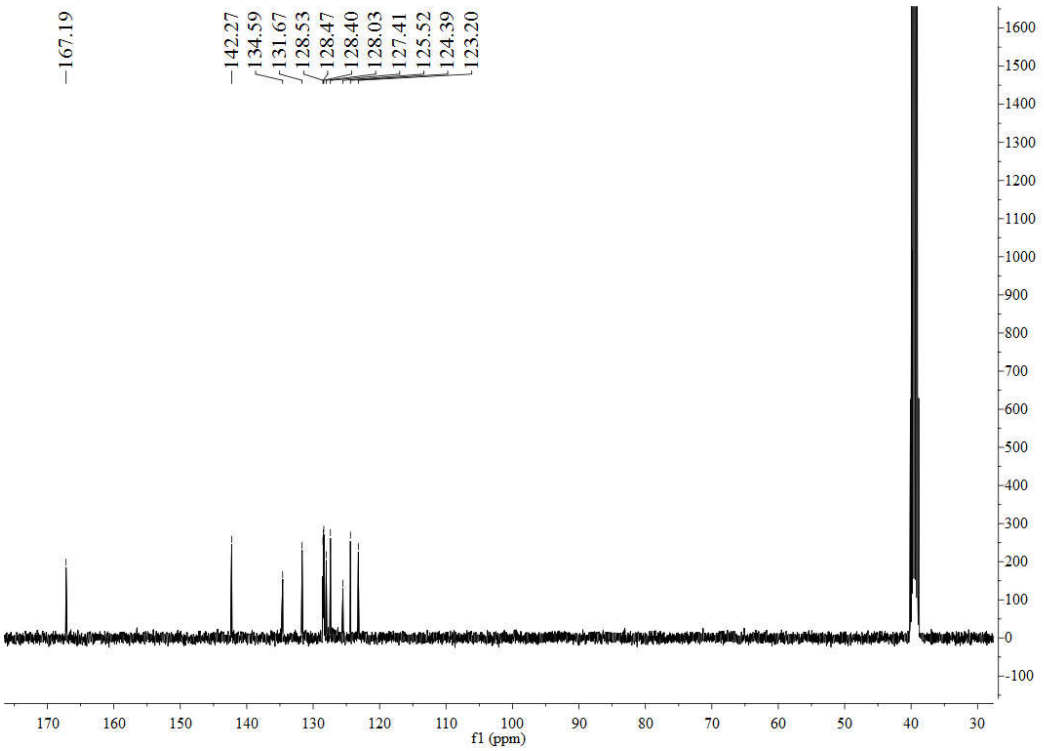
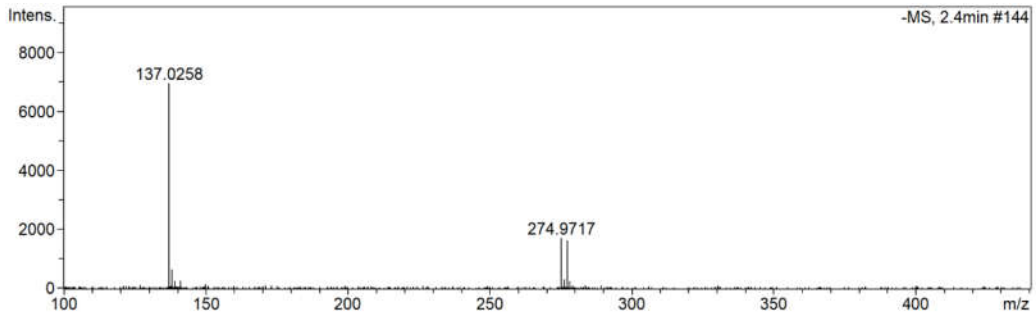


Figure S43 ^{13}C NMR (101 MHz) spectrum of **1BrNaAA** in DMSO-d_6.



Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e¡¥ Conf	mSigma	Std I	Std Mean m/z	Std I VarNorm	Std m/z Diff	Std Comb Dev
274.9717	1	C 13 H 8 Br O 2	274.9713	-1.3	3.0	9.5	ok	even	29.73	0.0324	0.0015	0.0143	0.0023	0.8427

Figure S44 The HRMS of **1BrNaAA**.

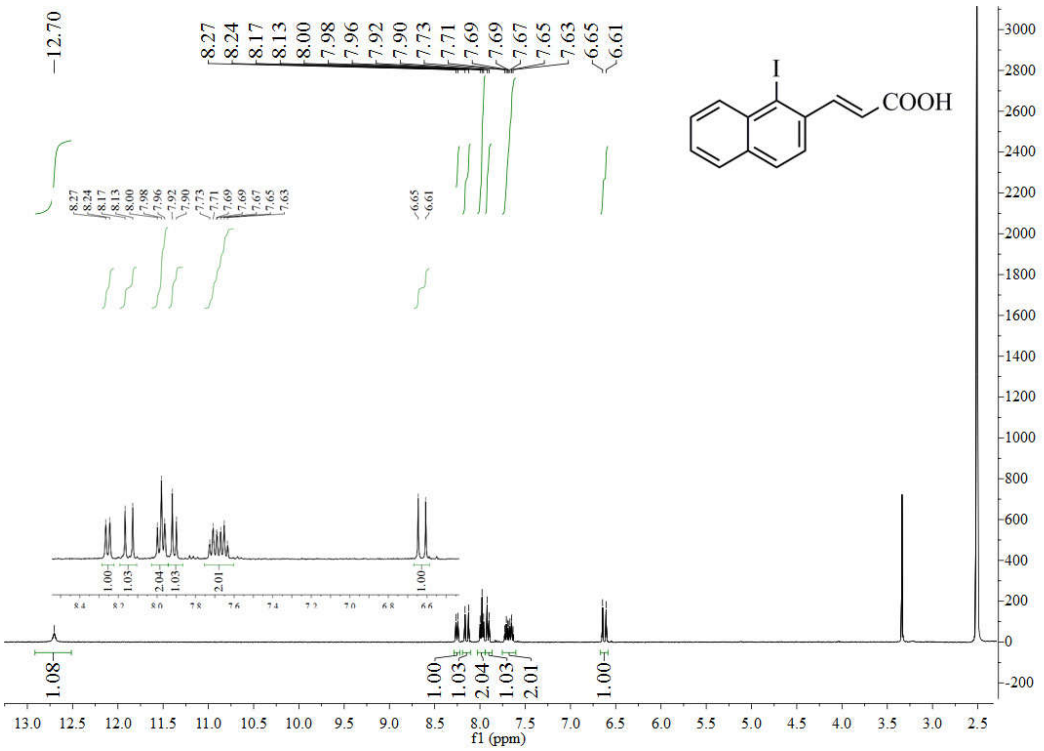


Figure S45 ^{1}H NMR (400 MHz) spectrum of **1INaAA** in DMSO-d_6.

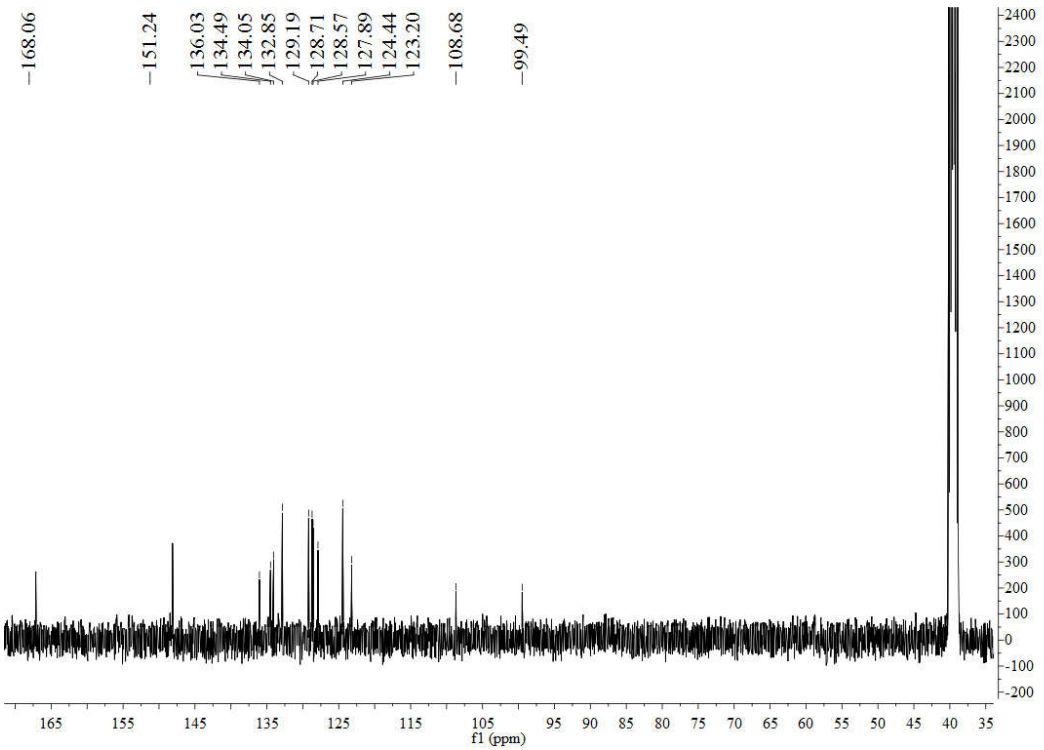
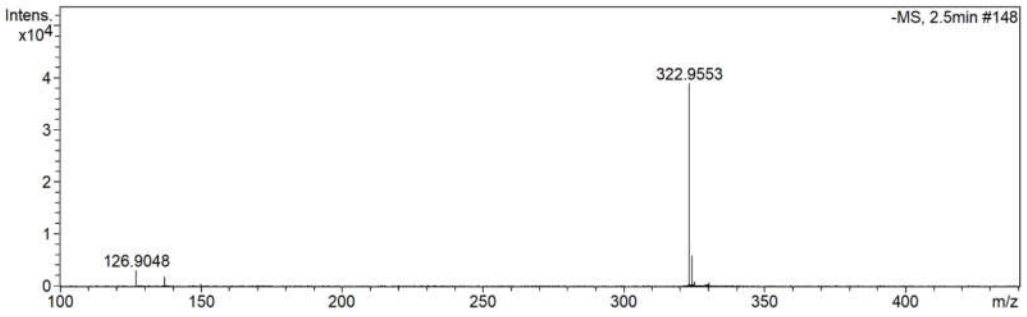


Figure S46 ^{13}C NMR (101 MHz) spectrum of **1INaAA** in DMSO-d_6.



Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma	Std I	Std Mean m/z	Std I VarNorm	Std m/z Diff	Std Comb Dev
322.9553	1	C 13 H 8 I O 2	322.9574	6.7	6.8	9.5	ok	even	5.74	0.0125	0.0023	0.0056	0.0003	0.8427

Figure S47 The HRMS of **1INaAA**.

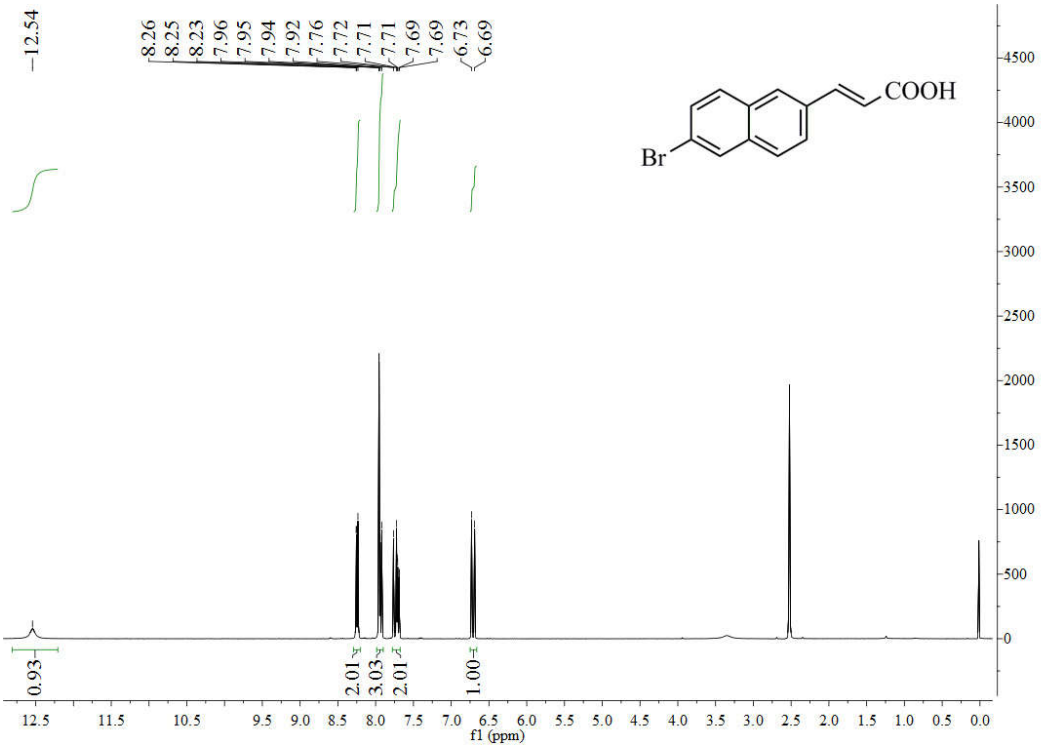


Figure S48 ^{1}H NMR (400 MHz) spectrum of **6BrNaAA** in DMSO-d_6.

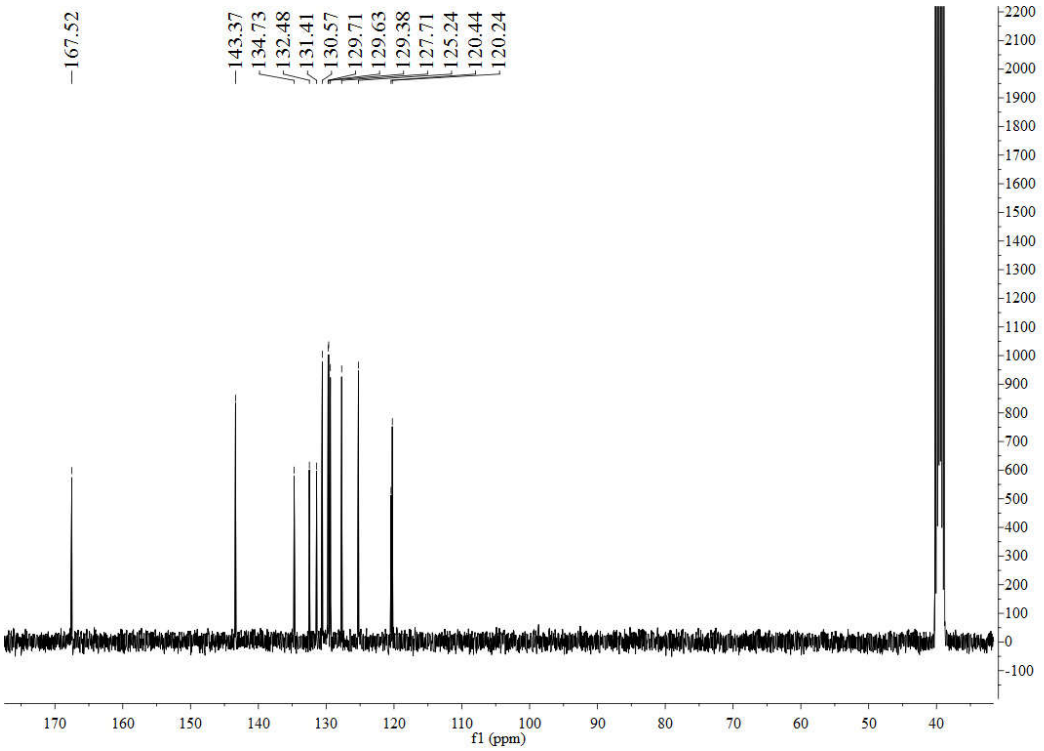
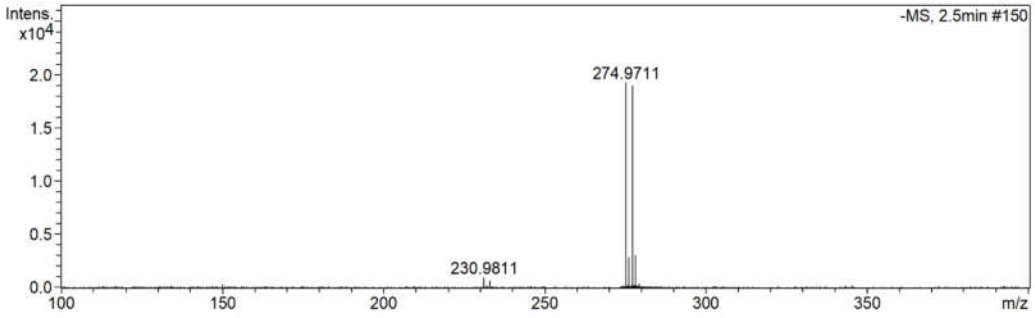


Figure S49 ^{13}C NMR (101 MHz) spectrum of **6BrNaAA** in DMSO-d_6.



Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e¡¥ Conf	mSigma	Std I	Std Mean m/z	Std I VarNorm	Std m/z Diff	Std Comb Dev
274.9711	1	C 13 H 8 Br O 2	274.9713	0.7	1.0	9.5	ok	even	10.15	0.0122	0.0003	0.0045	0.0003	0.8427

Figure S50 The HRMS of **6BrNaAA**.

Reference

1. K. Biradha and R. Santra, *Chem. Soc. Rev.*, 2013, **42**, 950.

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 1FNaAA

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: 1FNaAA

Bond precision: C-C = 0.0021 A Wavelength=0.71073

Cell: a=6.7599(8) b=3.7695(4) c=38.685(4)
alpha=90 beta=93.899(4) gamma=90
Temperature: 100 K

	Calculated	Reported
Volume	983.47(19)	983.46(18)
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C26 H18 F2 O4	C13 H9 F O2
Sum formula	C26 H18 F2 O4	C13 H9 F O2
Mr	432.40	216.20
Dx,g cm-3	1.460	1.460
Z	2	4
Mu (mm-1)	0.110	0.110
F000	448.0	448.0
F000’	448.27	
h,k,lmax	9,5,53	9,5,53
Nref	2766	2755
Tmin,Tmax		
Tmin’		

Correction method= Not given

Data completeness= 0.996 Theta(max)= 29.596

R(reflections)= 0.0558(2339) wR2(reflections)= 0.1436(2755)

S = 1.131 Npar= 181

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

```
PLAT053_ALERT_1_C Minimum Crystal Dimension Missing (or Error) ...     Please Check
PLAT054_ALERT_1_C Medium  Crystal Dimension Missing (or Error) ...     Please Check
PLAT055_ALERT_1_C Maximum Crystal Dimension Missing (or Error) ...     Please Check
PLAT222_ALERT_3_C Non-Solv.  Resd 1  H   Uiso(max)/Uiso(min) Range       8.0 Ratio
PLAT303_ALERT_2_C Full Occupancy Atom H003      with # Connections      2.00 Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......     6.133 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600         2 Report
```

Alert level G

```
PLAT042_ALERT_1_G Calc. and Reported MoietyFormula Strings  Differ     Please Check
PLAT045_ALERT_1_G Calculated and Reported Z Differ by a Factor ...      0.50 Check
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels ..........        25 Note
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min).        2 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600        7 Note
PLAT955_ALERT_1_G Reported (CIF) and Actual (FCF) Lmax Differ by .        1 Units
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.       13 Info
```

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0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
7 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
7 **ALERT level G** = General information/check it is not something unexpected

6 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
2 ALERT type 2 Indicator that the structure model may be wrong or deficient
4 ALERT type 3 Indicator that the structure quality may be low
2 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

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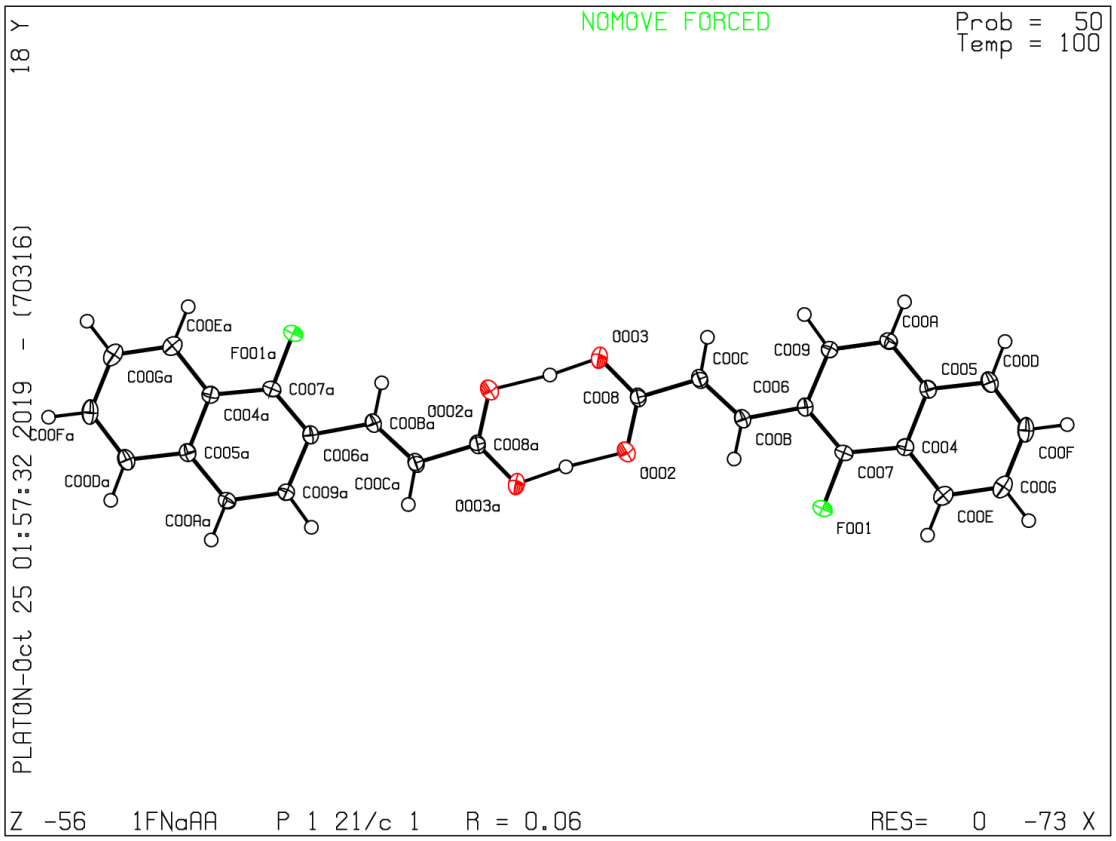
PLATON version of 07/08/2019; check.def file version of 30/07/2019

Datablock 1FNaAA - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 1ClNaAA

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: 1ClNaAA

```
Bond precision:  C-C = 0.0070 A          Wavelength=0.71073

Cell:          a=3.7709(7)       b=7.9900(14)     c=17.205(3)
               alpha=91.451(7)   beta=90.871(7)   gamma=91.865(7)
Temperature:   100 K
```

	Calculated	Reported
Volume	517.87(16)	517.87(16)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C13 H9 Cl O2	C13 H9 Cl O2
Sum formula	C13 H9 Cl O2	C13 H9 Cl O2
Mr	232.65	232.65
Dx,g cm-3	1.492	1.492
Z	2	2
Mu (mm-1)	0.347	0.347
F000	240.0	240.0
F000’	240.40	
h,k,lmax	5,11,25	5,11,24
Nref	3432	3229
Tmin,Tmax	0.951,0.963	
Tmin’	0.949	

```
Correction method= Not given

Data completeness= 0.941          Theta(max)= 31.388

R(reflections)= 0.1117( 1859)     wR2(reflections)= 0.3717( 3229)

S = 1.134                 Npar= 178
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level B

PLAT084_ALERT_3_B High wR2 Value (i.e. > 0.25) ..... 0.37 Report

---

Alert level C

PLAT029_ALERT_3_C _diffrn_measured_fraction_theta_full value Low . 0.979 Why?
PLAT082_ALERT_2_C High R1 Value ..... 0.11 Report
PLAT245_ALERT_2_C U(iso) H2 Smaller than U(eq) C2 by 0.013 Ang**2
PLAT245_ALERT_2_C U(iso) H11 Smaller than U(eq) C11 by 0.013 Ang**2
PLAT250_ALERT_2_C Large U3/U1 Ratio for Average U(i,j) Tensor .... 2.1 Note
PLAT340_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.007 Ang.
PLAT351_ALERT_3_C Long C-H (X0.96,N1.08A) C10 - H10 . 1.11 Ang.
PLAT905_ALERT_3_C Negative K value in the Analysis of Variance ... -0.282 Report
PLAT910_ALERT_3_C Missing # of FCF Reflection(s) Below Theta(Min). 5 Note
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 34 Report

---

Alert level G

PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..... 1 Report
PLAT072_ALERT_2_G SHELXL First Parameter in WGHT Unusually Large 0.20 Report
PLAT154_ALERT_1_G The s.u.’s on the Cell Angles are Equal ..(Note) 0.007 Degree
PLAT169_ALERT_4_G The CIF-Embedded .res File Contains AFIX 1 Recds 1 Report
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 161 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 3 Info
PLAT992_ALERT_5_G Repd & Actual _reflns_number_gt Values Differ by 1 Check

---

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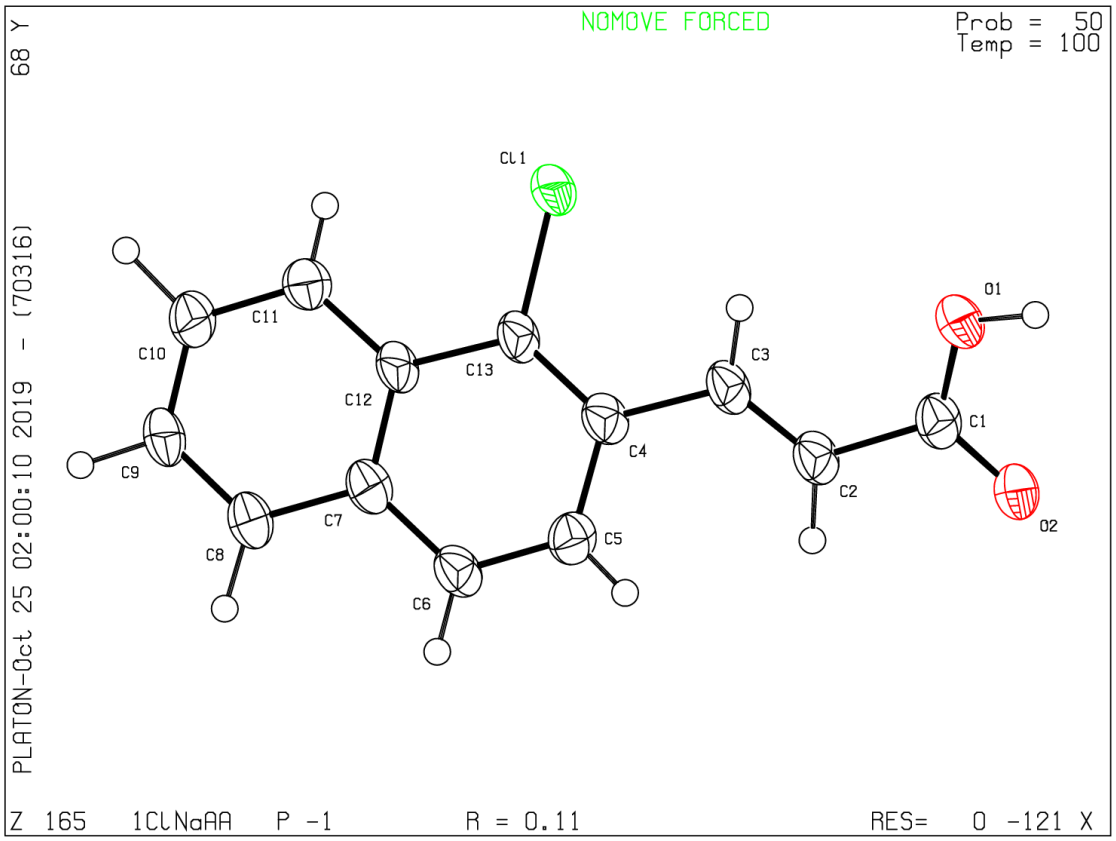
PLATON version of 07/08/2019; check.def file version of 30/07/2019

Datablock 1ClNaAA - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 1BrNaAA

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: 1BrNaAA

Bond precision: C-C = 0.0033 A Wavelength=0.71073

Cell: a=3.8948(4) b=8.1149(8) c=17.1274(16)
alpha=90.194(3) beta=91.725(4) gamma=92.396(4)
Temperature: 294 K

	Calculated	Reported
Volume	540.60(9)	540.60(9)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C13 H9 Br O2	C13 H9 Br O2
Sum formula	C13 H9 Br O2	C13 H9 Br O2
Mr	277.10	277.11
Dx,g cm-3	1.702	1.702
Z	2	2
Mu (mm-1)	3.781	3.781
F000	276.0	276.0
F000’	275.56	
h,k,lmax	4,10,21	4,10,21
Nref	2174	2164
Tmin,Tmax	0.573,0.635	
Tmin’	0.562	

Correction method= Not given

Data completeness= 0.995 Theta(max)= 26.245

R(reflections)= 0.0264(1929) wR2(reflections)= 0.0633(2164)

S = 1.050 Npar= 177

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

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PLAT057_ALERT_3_C Correction for Absorption Required   RT(exp) ...       1.11 Do !
PLAT218_ALERT_3_C Constrained U(ij) Components(s) for H1         .          6 Check
```

Alert level G

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PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..............          1 Report
PLAT164_ALERT_4_G Nr. of Refined C-H H-Atoms in Heavy-Atom Struct.          8 Note
PLAT169_ALERT_4_G The CIF-Embedded .res File Contains AFIX 1 Recds          1 Report
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min).          3 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600          8 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          5 Info
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   2 ALERT level C = Check. Ensure it is not caused by an omission or oversight
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```

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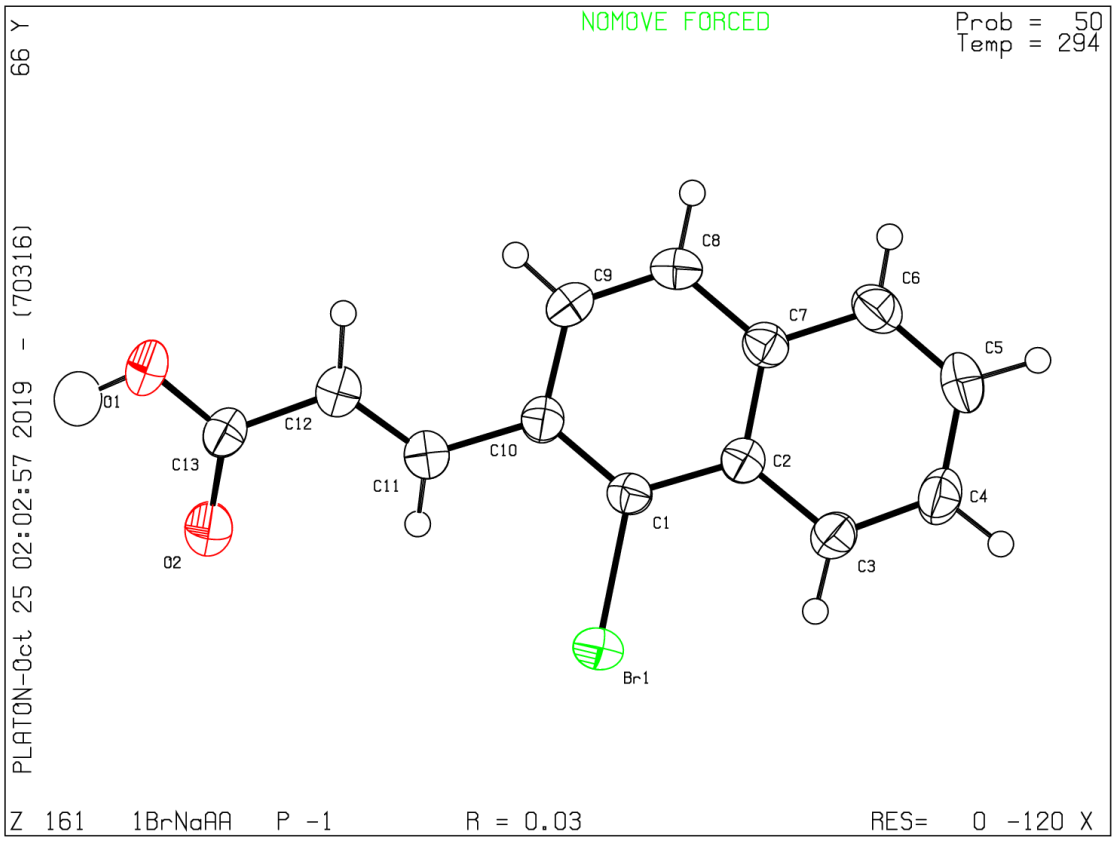
PLATON version of 07/08/2019; check.def file version of 30/07/2019

Datablock 1BrNaAA - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 1INaAA

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: 1INaAA

Bond precision: C-C = 0.0042 A Wavelength=0.71073

Cell: a=4.8142(2) b=8.2211(4) c=14.3363(7)
alpha=102.141(2) beta=91.410(2) gamma=93.280(2)
Temperature: 150 K

	Calculated	Reported
Volume	553.41(4)	553.41(4)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C13 H9 I O2	C13 H9 I O2
Sum formula	C13 H9 I O2	C13 H9 I O2
Mr	324.10	324.10
Dx,g cm-3	1.945	1.945
Z	2	2
Mu (mm-1)	2.873	2.873
F000	312.0	312.0
F000’	311.18	
h,k,lmax	6,11,19	6,11,19
Nref	2982	2953
Tmin,Tmax		
Tmin’		

Correction method= Not given

Data completeness= 0.990 Theta(max)= 29.104

R(reflections)= 0.0302(2537) wR2(reflections)= 0.0633(2953)

S = 1.086 Npar= 149

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

```
PLAT053_ALERT_1_C Minimum Crystal Dimension Missing (or Error) ...     Please Check
PLAT054_ALERT_1_C Medium  Crystal Dimension Missing (or Error) ...     Please Check
PLAT055_ALERT_1_C Maximum Crystal Dimension Missing (or Error) ...     Please Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600         10 Report
```

Alert level G

```
PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note)      0.002 Degree
PLAT432_ALERT_2_G Short Inter X...Y Contact  C9       ..C9              3.20 Ang.
                                              2-x,1-y,1-z  =      2_766 Check
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min).          3 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600         16 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          8 Info
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   4 ALERT level C = Check. Ensure it is not caused by an omission or oversight
   5 ALERT level G = General information/check it is not something unexpected

   4 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   2 ALERT type 2 Indicator that the structure model may be wrong or deficient
   2 ALERT type 3 Indicator that the structure quality may be low
   1 ALERT type 4 Improvement, methodology, query or suggestion
   0 ALERT type 5 Informative message, check
```

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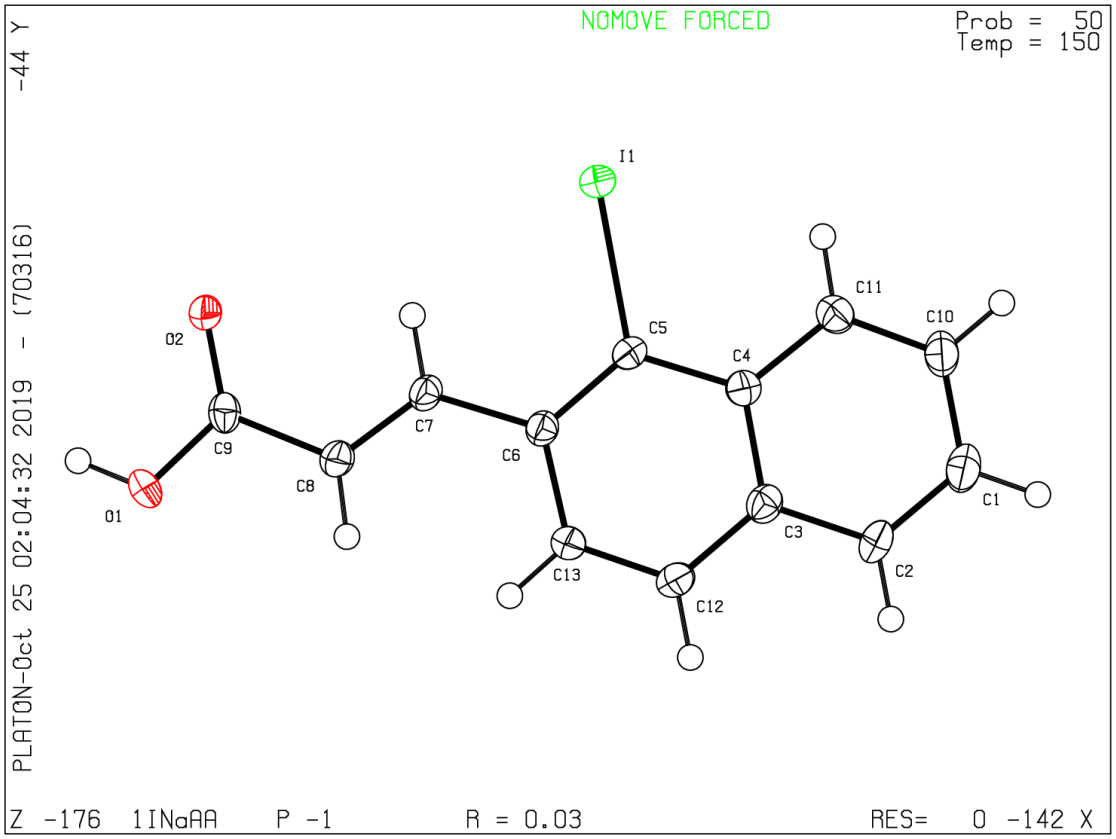
PLATON version of 07/08/2019; check.def file version of 30/07/2019

Datablock 1INaAA - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 6BrNaAA

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 6BrNaAA

Bond precision: C-C = 0.0041 A Wavelength=0.71073

Cell: a=3.9738(2) b=6.2790(4) c=21.6685(13)
alpha=97.582(2) beta=93.911(2) gamma=92.391(2)
Temperature: 294 K

	Calculated	Reported
Volume	533.98(5)	533.98(5)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C13 H9 Br O2	C13 H9 Br O2
Sum formula	C13 H9 Br O2	C13 H9 Br O2
Mr	277.10	277.11
Dx,g cm-3	1.723	1.723
Z	2	2
Mu (mm-1)	3.828	3.828
F000	276.0	276.0
F000'	275.56	
h,k,lmax	4,7,26	4,7,26
Nref	2083	2076
Tmin,Tmax		
Tmin'		

Correction method= Not given

Data completeness= 0.997 Theta(max)= 25.994

R(reflections)= 0.0383(1950) wR2(reflections)= 0.1009(2076)

S = 1.092 Npar= 181

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

```
PLAT053_ALERT_1_C Minimum Crystal Dimension Missing (or Error) ...     Please Check
PLAT054_ALERT_1_C Medium  Crystal Dimension Missing (or Error) ...     Please Check
PLAT055_ALERT_1_C Maximum Crystal Dimension Missing (or Error) ...     Please Check
PLAT094_ALERT_2_C Ratio of Maximum / Minimum Residual Density ....       3.23 Report
PLAT222_ALERT_3_C Non-Solv.  Resd 1  H   Uiso(max)/Uiso(min) Range        4.2 Ratio
PLAT790_ALERT_4_C Centre of Gravity not Within Unit Cell: Resd.  #          1 Note
              C13 H9 Br O2
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600          4 Report
```

Alert level G

```
PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note)      0.002 Degree
PLAT164_ALERT_4_G Nr. of Refined C-H H-Atoms in Heavy-Atom Struct.          8 Note
PLAT434_ALERT_2_G Short Inter HL..HL Contact Br1      ..Br1             3.54 Ang.
                                                 1-x,-y,1-z  =      2_656 Check
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min).          2 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600          2 Note
PLAT913_ALERT_3_G Missing # of Very Strong Reflections in FCF ....          3 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          6 Info
```

---

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
7 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
7 **ALERT level G** = General information/check it is not something unexpected

4 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
3 ALERT type 2 Indicator that the structure model may be wrong or deficient
4 ALERT type 3 Indicator that the structure quality may be low
3 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check

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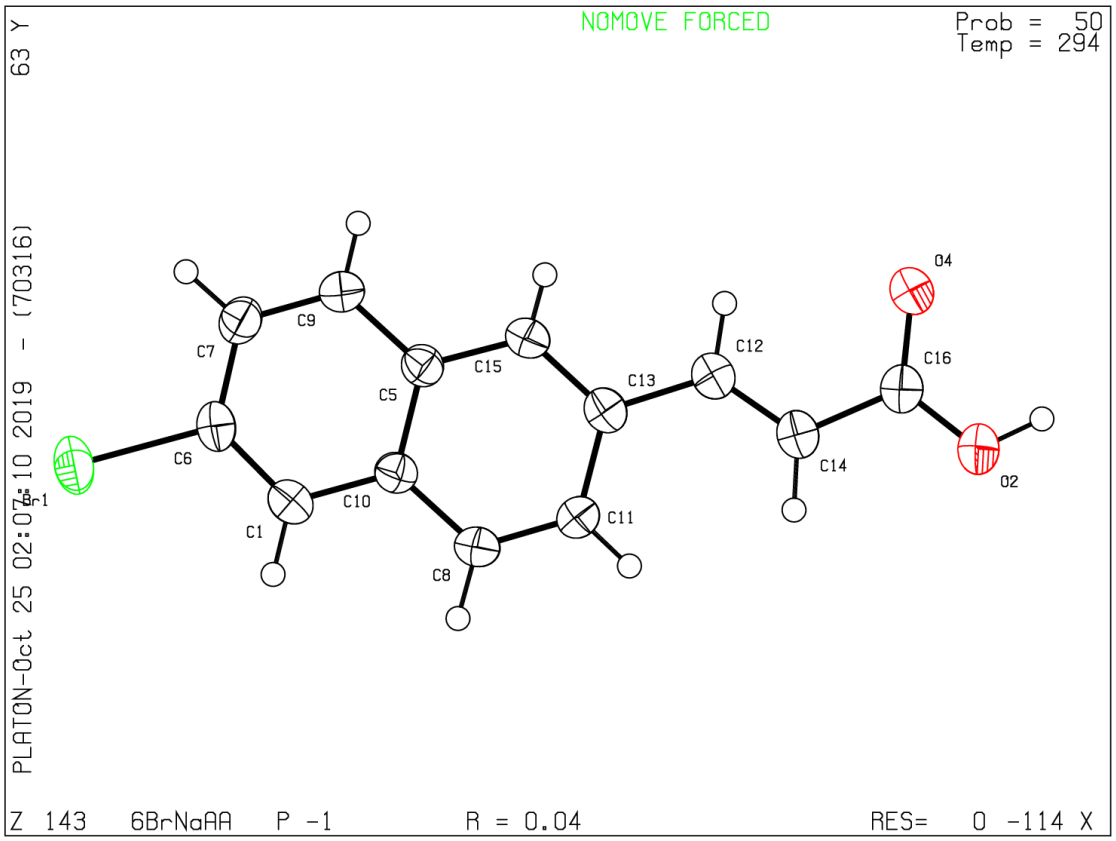
PLATON version of 07/08/2019; check.def file version of 30/07/2019

Datablock 6BrNaAA - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) D-1FNaAA

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: D-1FNaAA

Bond precision: C-C = 0.0033 A Wavelength=0.71073

Cell: a=7.4331(7) b=21.337(2) c=15.4508(17)
alpha=90 beta=98.686(5) gamma=90
Temperature: 100 K

	Calculated	Reported
Volume	2422.4(4)	2422.4(4)
Space group	P 21/n	P 1 21/n 1
Hall group	-P 2yn	-P 2yn
Moiety formula	C52 H36 F4 O8, 2(C3 H6 O)	C26 H18 F2 O4, C3 H6 O
Sum formula	C58 H48 F4 O10	C29 H24 F2 O5
Mr	980.96	490.48
Dx,g cm-3	1.345	1.345
Z	2	4
Mu (mm-1)	0.101	0.101
F000	1024.0	1024.0
F000’	1024.62	
h,k,lmax	10,29,21	10,29,21
Nref	6753	6668
Tmin,Tmax		
Tmin’		

Correction method= Not given

Data completeness= 0.987 Theta(max)= 29.488

R(reflections)= 0.0562(3525) wR2(reflections)= 0.1275(6668)

S = 1.019 Npar= 421

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

```
RINTA01_ALERT_3_C  The value of Rint is greater than 0.12
           Rint given   0.175
PLAT053_ALERT_1_C Minimum Crystal Dimension Missing (or Error) ...     Please Check
PLAT054_ALERT_1_C Medium  Crystal Dimension Missing (or Error) ...     Please Check
PLAT055_ALERT_1_C Maximum Crystal Dimension Missing (or Error) ...     Please Check
PLAT222_ALERT_3_C Non-Solv.  Resd 1  H   Uiso(max)/Uiso(min) Range       10.0 Ratio
PLAT245_ALERT_2_C U(iso) H00W      Smaller than U(eq) C00W      by      0.011 Ang**2
PLAT303_ALERT_2_C Full Occupancy Atom H003      with # Connections       2.00 Check
PLAT355_ALERT_3_C Long   O-H (X0.82,N0.98A)  O004     - H004     .      1.07 Ang.
PLAT391_ALERT_3_C Deviating Methyl C010      H-C-H Bond Angle ....        117 Degree
PLAT391_ALERT_3_C Deviating Methyl C010      H-C-H Bond Angle ....        101 Degree
PLAT905_ALERT_3_C Negative K value in the Analysis of Variance ...    -2.764 Report
PLAT910_ALERT_3_C Missing # of FCF Reflection(s) Below Theta(Min).          5 Note
```

Alert level G

```
PLAT020_ALERT_3_G The Value of Rint is Greater Than 0.12 .........      0.175 Report
PLAT042_ALERT_1_G Calc. and Reported MoietyFormula Strings  Differ     Please Check
PLAT045_ALERT_1_G Calculated and Reported Z Differ by a Factor ...       0.50 Check
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels ..........         60 Note
PLAT793_ALERT_4_G Model has Chirality at C00C        (Centro SPGR)          R Verify
PLAT793_ALERT_4_G Model has Chirality at C00G        (Centro SPGR)          S Verify
PLAT793_ALERT_4_G Model has Chirality at C00I        (Centro SPGR)          S Verify
PLAT793_ALERT_4_G Model has Chirality at C00J        (Centro SPGR)          R Verify
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600         80 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          6 Info
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
  12 ALERT level C = Check. Ensure it is not caused by an omission or oversight
  10 ALERT level G = General information/check it is not something unexpected

   5 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   3 ALERT type 2 Indicator that the structure model may be wrong or deficient
   8 ALERT type 3 Indicator that the structure quality may be low
   6 ALERT type 4 Improvement, methodology, query or suggestion
   0 ALERT type 5 Informative message, check
```

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

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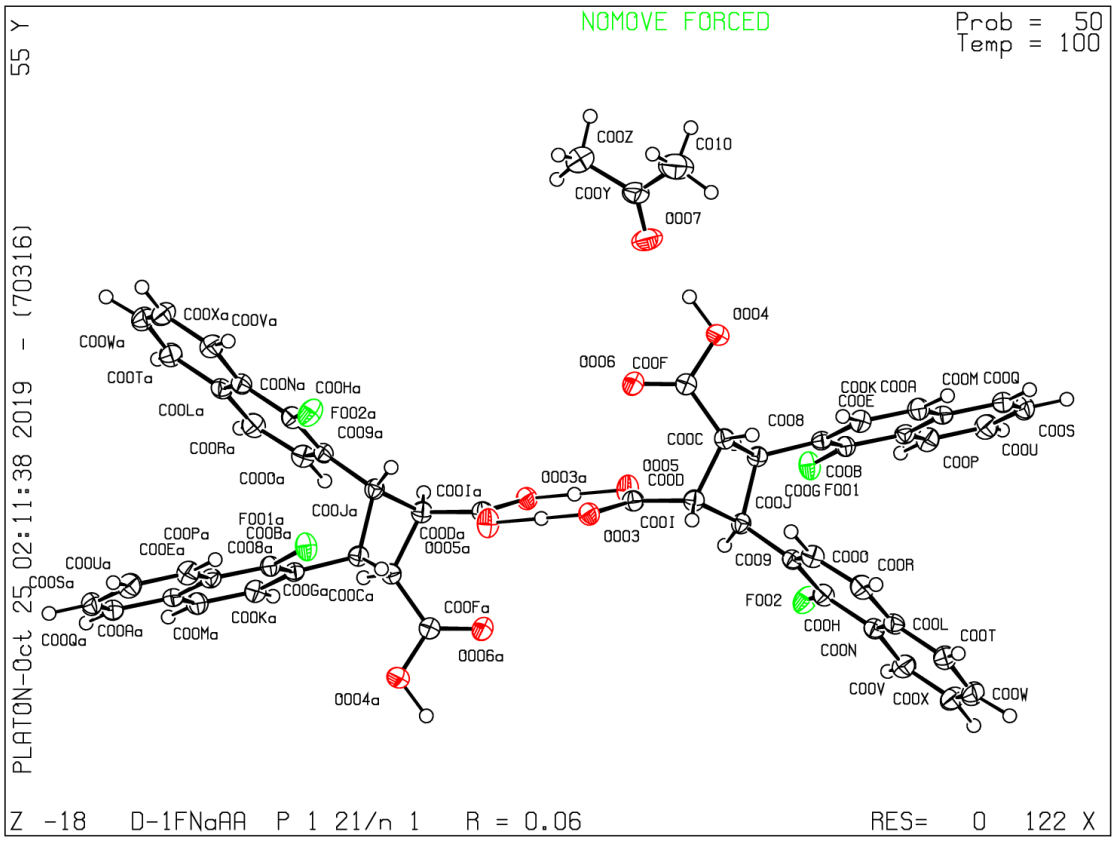
PLATON version of 07/08/2019; check.def file version of 30/07/2019

Datablock D-1FNaAA - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) D-1ClNaAA

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: D-1ClNaAA

Bond precision: C-C = 0.0017 A Wavelength=0.71073

Cell: a=13.8413(5) b=6.6231(2) c=27.4197(9)
alpha=90 beta=91.789(1) gamma=90
Temperature: 100 K

	Calculated	Reported
Volume	2512.40(14)	2512.40(14)
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C26 H18 Cl2 O4, C4 H8 O	C26 H18 Cl2 O4, C4 H8 O
Sum formula	C30 H26 Cl2 O5	C30 H26 Cl2 O5
Mr	537.41	537.41
Dx,g cm-3	1.421	1.421
Z	4	4
Mu (mm-1)	0.299	0.299
F000	1120.0	1120.0
F000’	1121.69	
h,k,lmax	18,8,35	18,8,35
Nref	5869	5819
Tmin,Tmax		
Tmin’		

Correction method= Not given

Data completeness= 0.991 Theta(max)= 27.666

R(reflections)= 0.0292(5293) wR2(reflections)= 0.0766(5819)

S = 1.067 Npar= 438

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

```
CRYSC01_ALERT_1_C The word below has not been recognised as a standard
           identifier.
           dull
CRYSC01_ALERT_1_C The word below has not been recognised as a standard
           identifier.
           whiteish
PLAT053_ALERT_1_C Minimum Crystal Dimension Missing (or Error) ...     Please Check
PLAT054_ALERT_1_C Medium  Crystal Dimension Missing (or Error) ...     Please Check
PLAT055_ALERT_1_C Maximum Crystal Dimension Missing (or Error) ...     Please Check
```

Alert level G

```
PLAT398_ALERT_2_G Deviating  C-O-C    Angle From 120 for O004          105.0 Degree
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels .....            63 Note
PLAT793_ALERT_4_G Model has Chirality at C00D       (Centro SPGR)         S Verify
PLAT793_ALERT_4_G Model has Chirality at C00J       (Centro SPGR)         R Verify
PLAT793_ALERT_4_G Model has Chirality at C00K       (Centro SPGR)         R Verify
PLAT793_ALERT_4_G Model has Chirality at C00N       (Centro SPGR)         S Verify
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min).        4 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600       41 Note
PLAT953_ALERT_1_G Reported (CIF) and Actual (FCF) Hmax Differ by .        1 Units
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.       21 Info
```

```
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```

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PLATON version of 07/08/2019; check.def file version of 30/07/2019

Datablock D-1ClNaAA - ellipsoid plot