

## Supplementary Information

### **Immobilized Ni on TMEDA@ $\beta$ SiO<sub>2</sub>@ $\alpha$ SiO<sub>2</sub>@Fe<sub>3</sub>O<sub>4</sub>: as a novel magnetic nanocatalyst for preparation of pyrido[2,3-*d*:6,5-*d'*]dipyrimidines**

Yasir Qasim Almajidi<sup>1</sup>, Mohd Ubaidullah<sup>2\*</sup>, Bidhan Pandit<sup>3</sup>, A. K. Kareem<sup>4</sup>, Rosario Mireya Romero-Parra<sup>5</sup>, Adizov Bobirjon<sup>6</sup>, Wesam R. Kadhum<sup>7</sup>, Amran M. AL-Erjan<sup>8</sup>, Munther Abosaooda<sup>9</sup>, Aisha Kamal Mahmoud<sup>10</sup>

<sup>1</sup> *Department of Pharmacy (pharmaceutics), Baghdad College of Medical Sciences, Baghdad, Iraq*

<sup>2</sup> *Department of Chemistry, College of Science, King Saud University, P.O. Box 2455, Riyadh 11451, Saudi Arabia*

<sup>3</sup> *Department of Materials Science and Engineering and Chemical Engineering, Universidad Carlos III de Madrid, Avenida de la Universidad 30, 28911 Leganés, Madrid, Spain*

<sup>4</sup> *Department of Biomedical Engineering, Al-Mustaqbal University College, Babylon, Iraq*

<sup>5</sup> *Department of General Studies, Universidad Continental, Lima, Peru*

<sup>6</sup> *Chief Researcher of the Institute of General and Inorganic Chemistry, Academy of Sciences of the Republic of Uzbekistan, 100071, Uzbekistan, Tashkent, Mirzo Ulugbek, Mirzo Ulugbek avenue 77A*

<sup>7</sup> *Department of Pharmacy, Kut University College, Kut 52001, Wasit, Iraq*

<sup>8</sup> *Department of Anesthesia, College of Health & Medical Technology, Al-Ayen University, Thi-Qar, Iraq*

<sup>9</sup> *College of pharmacy, the Islamic University, 54001 Najaf, Iraq*

<sup>10</sup> *Al-Nisour University College, Baghdad, Iraq*

\*Corresponding author. Mohd Ubaidullah: mtayyab@ksu.edu.sa; mohdubaidullah2023@gmail.com

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## Materials and Apparatuses

The chemical compounds, reagents, and solvents were supplied from Fluka, Sinopharm, and Chemical Companies. For recording the FT-IR spectra, a Thermo device (model Perkin–Elmer BX- II) was used (KBr plates). The  $^1\text{H}$  NMR (500 MHz) and  $^{13}\text{C}$  NMR (125.7 MHz) spectra were recorded in  $\text{DMSO-}d_6$  solvent using a Bruker Avance DPX FT-NMR spectrometer. The spectrometer 5975CVL MSD model triple-axis detector was applied for running mass spectra. Melting points were measured by Büchi B-545 apparatus in open capillary tubes. Thin layer chromatography (TLC) was used to check the progress of the reaction. A Panalytical X'Pert PRO MPD apparatus was utilized to record XRD patterns with  $\text{Cu K}\alpha$  radiation ( $\lambda=1.5408 \text{ \AA}$ ) from  $2\theta=10^\circ$ - $80^\circ$ . Apparatuses model TESCAN MIRA3 LMH and Philips CM200 were used for recording FE-SEM and TEM images of the nanocatalyst. Nitrogen adsorption–desorption isotherms were recorded on a BELSORP-mini II surface area analyzer. A Vibrating Sample Magnetometer (VSM, ADE-DMS 1660, MA, USA) was used to observe the magnetic property of the catalyst. A Varian inductively coupled plasma atomic emission spectrometer (ICP-OES) (model: VISTA-MPX, made in Australia) was used to determine the amount of nickel in the catalytic system. TGA was recorded using Perkin Elmerpyris apparatus, with temperature increase rate of  $10 \text{ }^\circ\text{C}\cdot\text{min}^{-1}$  in nitrogen atmosphere at 25-900  $^\circ\text{C}$ .

## Spectroscopic and physical data of pyrido-dipyrimidines

**Product 1a:** 5-Phenyl-2,8-dithioxo-2,3,5,8,9,10-hexahydropyrido[2,3-*d*:6,5-*d'*]dipyrimidine-4,6(1*H*,7*H*)-dione; cream powder, m.p.: 207–209 °C (208-210 °C) [38]; <sup>1</sup>H NMR (500 MHz; DMSO-*d*<sub>6</sub>): δ (ppm) 17.16 (br., 1H, NH), 11.54 (br., 3H, NH), 7.26 (br., 1H, NH), 7.17 (t, *J* = 7.6 Hz, 2H, H<sub>Ar</sub>), 7.06 (t, *J* = 7.2 Hz, 1H, H<sub>Ar</sub>), 7.03 (d, *J* = 7.8 Hz, 2H, H<sub>Ar</sub>), 5.97 (s, 1H, CH) (Fig. S1).

**Product 2a:** 5-(2-Nitrophenyl)-2,8-dithioxo-2,3,5,8,9,10-hexahydropyrido[2,3-*d*:6,5-*d'*]dipyrimidine-4,6(1*H*,7*H*)-dione; yellow powder, m.p.: 227-231 °C (228-230 °C) [38]; <sup>1</sup>H NMR (500 MHz; DMSO-*d*<sub>6</sub>): δ (ppm) 11.67 (br., 2H, NH), 10.80 (br., 1H, NH), 7.73 (br., 2H, NH), 7.57 (d, *J* = 7.9 Hz, 1H, H<sub>Ar</sub>), 7.44 (t, *J* = 7.8 Hz, 1H, H<sub>Ar</sub>), 7.23 (t, *J* = 7.6 Hz, 1H, H<sub>Ar</sub>), 7.11 (d, *J* = 8.0 Hz, 1H, H<sub>Ar</sub>), 5.92 (s, 1H, CH) (Fig. S2).

**Product 3a:** 5-(4-Nitrophenyl)-2,8-dithioxo-2,3,5,8,9,10-hexahydropyrido[2,3-*d*:6,5-*d'*]dipyrimidine-4,6(1*H*,7*H*)-dione; light brown powder, m.p.: 329-331 °C (329-331 °C) [38]; <sup>1</sup>H NMR (500 MHz; DMSO-*d*<sub>6</sub>): δ (ppm) 17.22 (br., 1H, NH), 11.52 (br., 3H, NH), 7.24 (br., 1H, NH), 8.05 (d, *J* = 8.5 Hz, 2H, H<sub>Ar</sub>), 7.24 (d, *J* = 8.5 Hz, 2H, H<sub>Ar</sub>), 7.00 (br., 1H, NH), 6.06 (s, 1H, CH) (Fig. S3).

**Product 4a:** 5-(*p*-tolyl)-2,8-dithioxo-2,3,5,8,9,10-hexahydropyrido[2,3-*d*:6,5-*d'*]dipyrimidine-4,6(1*H*,7*H*)-dione; white powder, m.p.: 315-317 °C (316-318 °C) [38]; <sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>): δ (ppm) 17.15 (br., 1H, NH), 11.51 (br., 3H, NH), 7.10 (br., 1H, NH), 6.96 (d, *J* = 7.6 Hz, 2H, H<sub>Ar</sub>), 6.87 (d, *J* = 7.8 Hz, 2H, H<sub>Ar</sub>), 5.90 (s, 1H, CH), 2.20 (s, 3H, CH<sub>3</sub>) (Fig. S3); <sup>13</sup>C NMR (125 MHz, DMSO-*d*<sub>6</sub>): δ (ppm) 173.2, 163.9, 163.0, 140.3, 134.0, 128.7, 126.9, 96.4, 30.5, 21.0 (Fig. S4).

**Product 5a:** 5-(2,4-dimethylphenyl)-2,8-dithioxo-2,3,5,8,9,10-hexahydropyrido[2,3-*d*:6,5-*d'*]dipyrimidine-4,6(1*H*,7*H*)-dione; yellow powder, m.p.: >300 °C; <sup>1</sup>H NMR (500 MHz,

DMSO-*d*<sub>6</sub>):  $\delta$  (ppm) 11.57 (br., 2H, NH), 10.87 (br., 1H, NH), 7.10 (br., 2H, NH), 6.89 (d,  $J = 7.9$  Hz, 1H, H<sub>Ar</sub>), 6.72 (d,  $J = 7.9$  Hz, 1H, H<sub>Ar</sub>), 6.72 (s, 1H, H<sub>Ar</sub>), 5.88 (s, 1H, CH), 2.27 (s, 3H, CH<sub>3</sub>), 2.16 (s, 3H, CH<sub>3</sub>) (Fig. S5); <sup>13</sup>C NMR (125 MHz, DMSO-*d*<sub>6</sub>):  $\delta$  (ppm) 174.0, 164.3, 163.3, 140.6, 136.7, 132.1, 130.5, 127.2, 125.8, 95.3, 32.9, 21.6, 16.2 (Fig. S6); IR (KBr): 3535 (N-H), 2972 (SP<sup>2</sup>-CH), 2912 (SP<sup>3</sup>-CH), 1666 (C=O), 1554 and 1435 (C=C), 1303 and 1136 (C=S) cm<sup>-1</sup> (Fig. S7); Mass:  $m/z$  385 [M<sup>+</sup>], 386 [M<sup>+</sup> + 1] (Fig. S8).

**Product 6a.** IR (KBr): 5-(4-hydroxyphenyl)-2,8-dithioxo-2,3,5,8,9,10-hexahydropyrido[2,3-*d*:6,5-*d'*]dipyrimidine-4,6(1*H*,7*H*)-dione; red powder, m.p.: >300 °C; <sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>):  $\delta$  (ppm) 13.20 (br., 1H, NH), 8.03 (br., 2H, NH), 7.84 (br., 2H, NH), 7.18 (s, 1H, OH), 7.13 (d,  $J = 10.5$  Hz, 2H, H<sub>Ar</sub>), 6.81 (d,  $J = 10.5$  Hz, 2H, H<sub>Ar</sub>), 5.80 (s, 1H, CH) (Fig. S9); <sup>13</sup>C NMR (125 MHz, DMSO-*d*<sub>6</sub>):  $\delta$  (ppm) 174.6, 164.1, 159.1, 157.8, 134.8, 128.3, 113.9, 86.4, 55.4 (Fig. S10); 3448 (NH), 2910 (SP<sup>3</sup>-CH), 1617 (C=O), 1545 and 1440 (C=C), 1201 (C-O), 1402, 1304 and 1140 (C=S) cm<sup>-1</sup> (Fig. S11); Mass:  $m/z$  373 [M<sup>+</sup>], 374 [M<sup>+</sup> + 1] (Fig. S12).

**Product 7a:** 5-(3-Methoxyphenyl)-2,8-dithioxo-2,3,5,8,9,10-hexahydropyrido[2,3-*d*:6,5-*d'*]dipyrimidine-4,6(1*H*,7*H*)-dione; orange powder, m.p.: 236-238 °C (237-239 °C) [38]; <sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>):  $\delta$  (ppm) 11.54 (br., 4H, NH), 7.27 (br., 1H, NH), 7.06 (t,  $J = 7.9$  Hz, 1H, H<sub>Ar</sub>), 6.63 (d,  $J = 8.1$  Hz, 1H, H<sub>Ar</sub>), 6.57 (d,  $J = 7.7$  Hz, 1H, H<sub>Ar</sub>), 6.49 (s, 1H, H<sub>Ar</sub>), 5.93 (s, 1H, CH), 3.63 (s, 3H, OCH<sub>3</sub>) (Fig. S13).

**Product 8a:** 5-(4-Methoxyphenyl)-2,8-dithioxo-2,3,5,8,9,10-hexahydropyrido[2,3-*d*:6,5-*d'*]dipyrimidine-4,6(1*H*,7*H*)-dione; orange powder, m.p.: 277-279 °C (277-279 °C) [38]; <sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>):  $\delta$  (ppm) 11.61 (br., 2H, NH) (Fig. S14), 11.47 (br., 2H, NH), 7.08 (br., 1H, NH), 6.88 (d,  $J = 8.4$  Hz, 2H, H<sub>Ar</sub>), 6.71 (d,  $J = 8.5$  Hz, 2H, H<sub>Ar</sub>), 5.90 (s, 1H, CH), 3.67 (s, 3H, CH<sub>3</sub>).

**Product 9a:** 5-(3,4-Methoxyphenyl)-2,8-dithioxo-2,3,5,8,9,10-hexahydropyrido[2,3-*d*:6,5-*d'*]dipyrimidine-4,6(1*H*,7*H*)-dione; orange powder, m.p.: >300 °C (>300 °C) [38]; <sup>1</sup>H NMR

(500 MHz, DMSO-*d*<sub>6</sub>): δ (ppm) 11.90 (br., 2H, NH), 10.76 (br., 1H, NH), 7.13 (br., 2H, NH), 6.93 (d, *J* = 8.1 Hz, 1H, H<sub>Ar</sub>), 6.80 (s, 1H, H<sub>Ar</sub>), 6.61 (d, *J* = 8.1 Hz, 1H, H<sub>Ar</sub>), 5.62 (s, 1H, CH), 3.72 (s, 3H, OCH<sub>3</sub>), 3.68 (s, 3H, OCH<sub>3</sub>) (Fig. S15).

**Product 10a:** 5-(4-Chlorophenyl)-2,8-dithioxo-2,3,5,8,9,10-hexahydropyrido[2,3-*d*:6,5-*d'*]dipyrimidine-4,6(1*H*,7*H*)-dione; yellow powder, m.p.: 255-257 °C (254-256 °C) [38]; <sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>): δ (ppm) 11.55 (br., 2H, NH), 10.53 (br., 1H, NH), 7.56 (br., 2H, NH), 7.19 (d, *J* = 8.0 Hz, 2H, H<sub>Ar</sub>), 6.98 (d, *J* = 8.0 Hz, 2H, H<sub>Ar</sub>), 5.93 (s, 1H, CH), 2.20 (Fig. S16).

**Product 11a:** 5-(4-Fluorophenyl)-2,8-dithioxo-2,3,5,8,9,10-hexahydropyrido[2,3-*d*:6,5-*d'*]dipyrimidine-4,6(1*H*,7*H*)-dione; white powder, m.p.: 228-230 °C (227-229 °C) [38]; <sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>): δ (ppm) 17.15 (br., 1H, NH), 11.54 (br., 3H, NH), 10.49 (br., 1H, NH), 6.96-7.00 (m, 4H, H<sub>Ar</sub>), 5.94 (s, 1H, CH) (Fig. S17).

**Product 12a:** 5-(2-Fluorophenyl)-2,8-dithioxo-2,3,5,8,9,10-hexahydropyrido[2,3-*d*:6,5-*d'*]dipyrimidine-4,6(1*H*,7*H*)-dione; white powder, m.p.: 239-241 °C (239-241 °C) [38]; <sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>): δ (ppm) 16.98 (br., 1H, NH), 11.49 (br., 2H, NH), 10.59 (br., 1H, NH), 7.55 (br., 1H, NH), 7.12 (d, *J* = 6.8 Hz, 2H, H<sub>Ar</sub>), 6.94-7.02 (m, 2H, H<sub>Ar</sub>), 6.03 (s, 1H, CH) (Fig. S18).

**Product 13a:** 5-(2-Bromophenyl)-2,8-dithioxo-2,3,5,8,9,10-hexahydropyrido[2,3-*d*:6,5-*d'*]dipyrimidine-4,6(1*H*,7*H*)-dione; white powder, m.p.: 254-256 °C (255-257 °C) [38]; <sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>): δ (ppm) 9.36 (br., 5H, NH), 7.36 (d, *J* = 7.5 Hz, 1H, H<sub>Ar</sub>), 7.12-7.16 (m, 2H, H<sub>Ar</sub>), 6.95-6.98 (m, 1H, H<sub>Ar</sub>), 5.63 (s, 1H, CH) (Fig. S19).

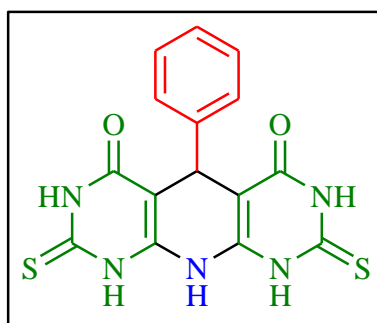
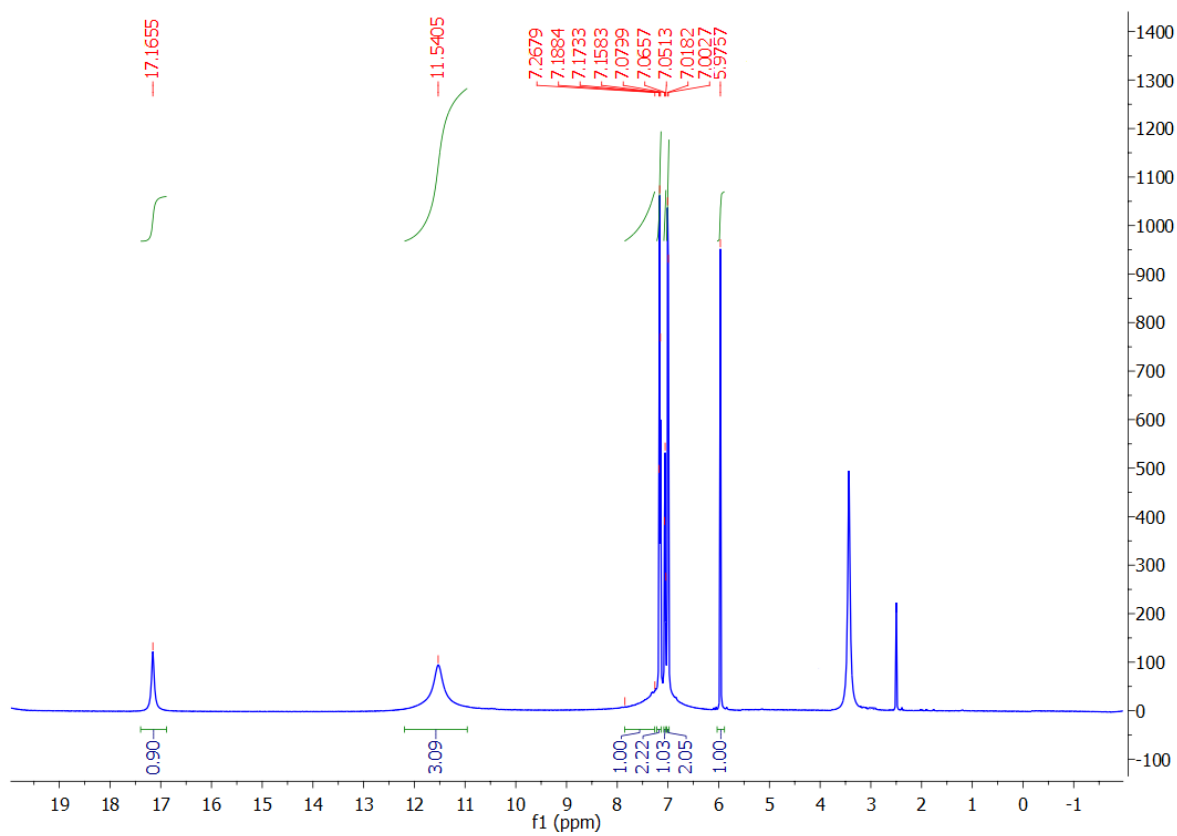
**Product 14a:** 5-(3-Bromophenyl)-2,8-dithioxo-2,3,5,8,9,10-hexahydropyrido[2,3-*d*:6,5-*d'*]dipyrimidine-4,6(1*H*,7*H*)-dione; white powder, m.p.: 248-250 °C (248-250 °C) [38]; <sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>): δ (ppm) 11.51 (s, 5H, NH), 7.20 (d, *J* = 7.8 Hz, 1H, H<sub>Ar</sub>), 7.09

(t,  $J = 7.8$  Hz, 1H, H<sub>Ar</sub>), 7.02 (s, 1H, H<sub>Ar</sub>), 6.93 (d,  $J = 7.8$  Hz, 1H, H<sub>Ar</sub>), 5.90 (s, 1H, CH) (Fig. S20).

**Product 15a:** 5,5'-(1,4-Phenylene)bis(2,8-dithioxo-2,3,7,8,9,10-hexahydropyrido[2,3-*d*:6,5-*d'*]dipyrimidine-4,6(1*H*,5*H*)-dione); red powder, m.p.: 298-300 °C (300 °C) [39]; <sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>): δ (ppm) 16.96 (br., 2H, NH), 11.48 (br., 3H, NH), 10.58 (br., 1H, NH), 7.12-8.18 (br., 4H, NH), 6.93-7.02 (m, 4H, H<sub>Ar</sub>), 6.06 (s, 1H, CH) (Fig. S21).

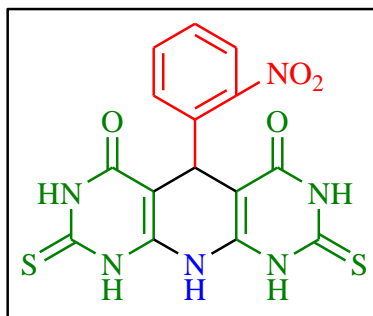
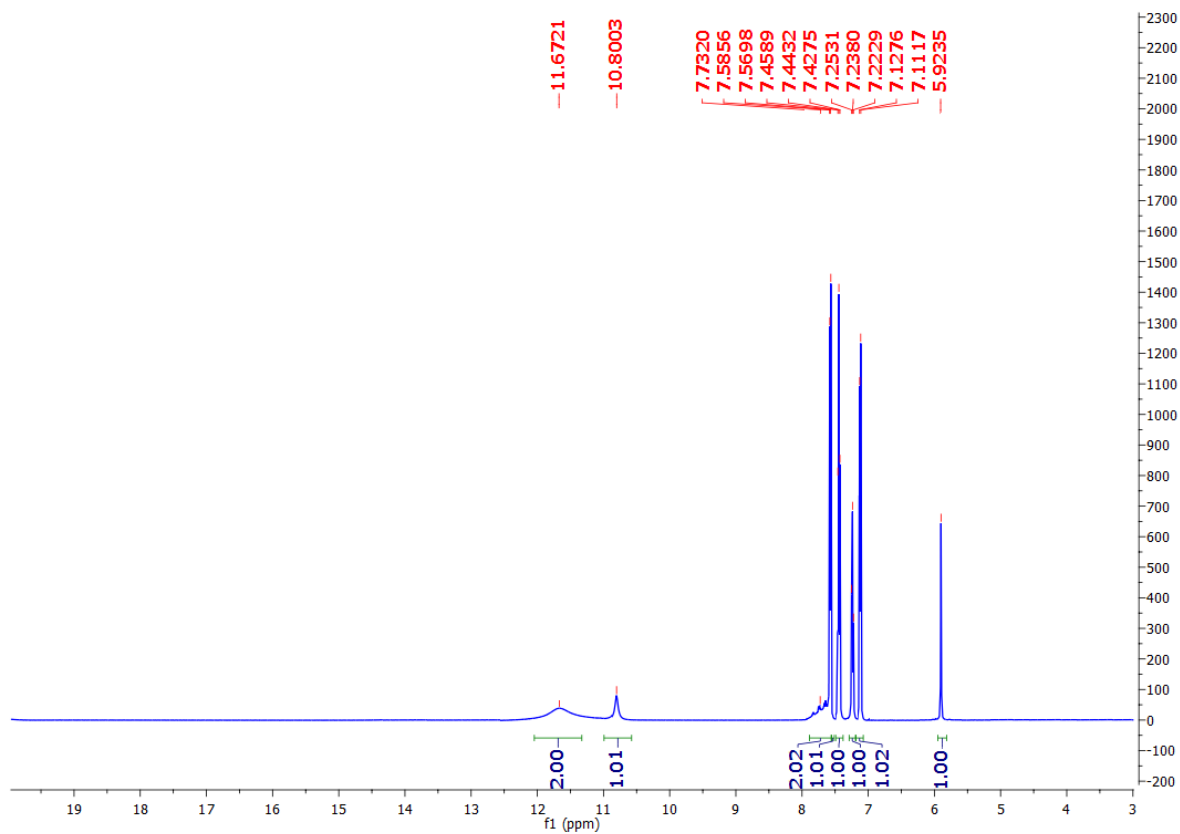
**Product 16a:** 5,5'-(1,3-Phenylene)bis(2,8-dithioxo-2,3,7,8,9,10-hexahydropyrido[2,3-*d*:6,5-*d'*]dipyrimidine-4,6(1*H*,5*H*)-dione); red powder, m.p.: >300 °C; <sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>): δ (ppm) 17.00 (br., 1H, NH), 11.55 (br., 4H, NH), 10.71 (br., 2H, NH), 7.08 (a triplet and a broad peak,  $J = 7.9$  Hz, 1H, H<sub>Ar</sub> and 3H, NH), 6.65 (dd,  $J = 8.0, 2.4$  Hz, H<sub>Ar</sub>), 6.58 (d,  $J = 8.0$  Hz, 1H, H<sub>Ar</sub>), 6.50 (s, 1H, H<sub>Ar</sub>), 5.92 (s, 2H, CH) (Fig. S22); <sup>13</sup>C NMR (125 MHz, DMSO-*d*<sub>6</sub>): δ (ppm) 174.7, 164.2, 163.2, 142.8, 131.9, 128.2, 118.7, 95.4, 33.1 (Fig. S23); IR (KBr): 3430 (NH), 2900 (SP<sup>3</sup>-CH), 1610 (C=O), 1534 and 1435 (C=C), 1400, 1302 and 1139 (C=S) (Fig. S24); Mass:  $m/z$  636 [M<sup>+</sup>], 638 [M<sup>+</sup> + 2] (Fig. S25).

## Original spectrums of pyrido-dipyrimidines

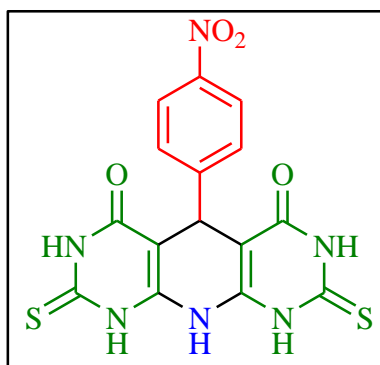
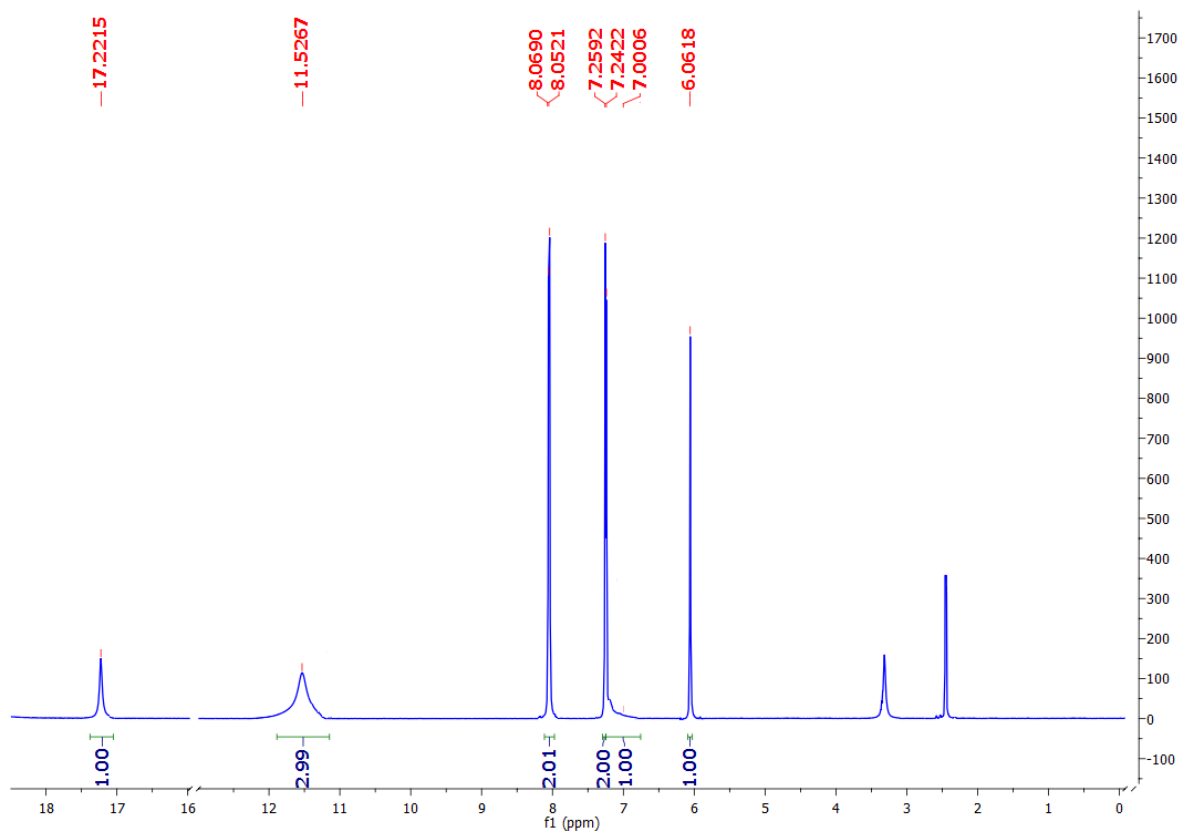


**Fig. S1** The structure and <sup>1</sup>H NMR spectrum of product **1a**

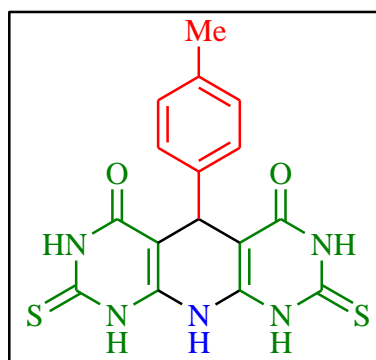
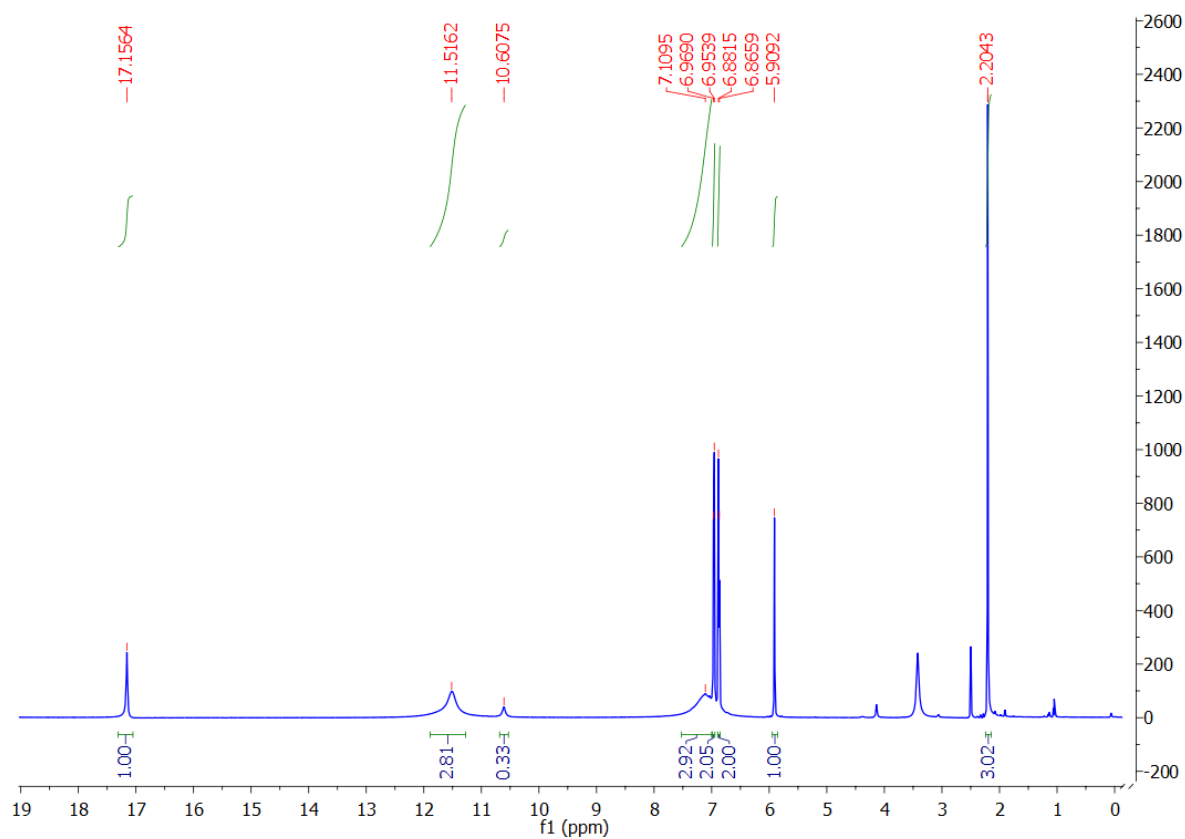




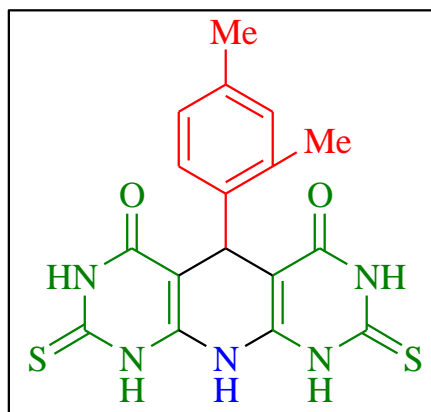
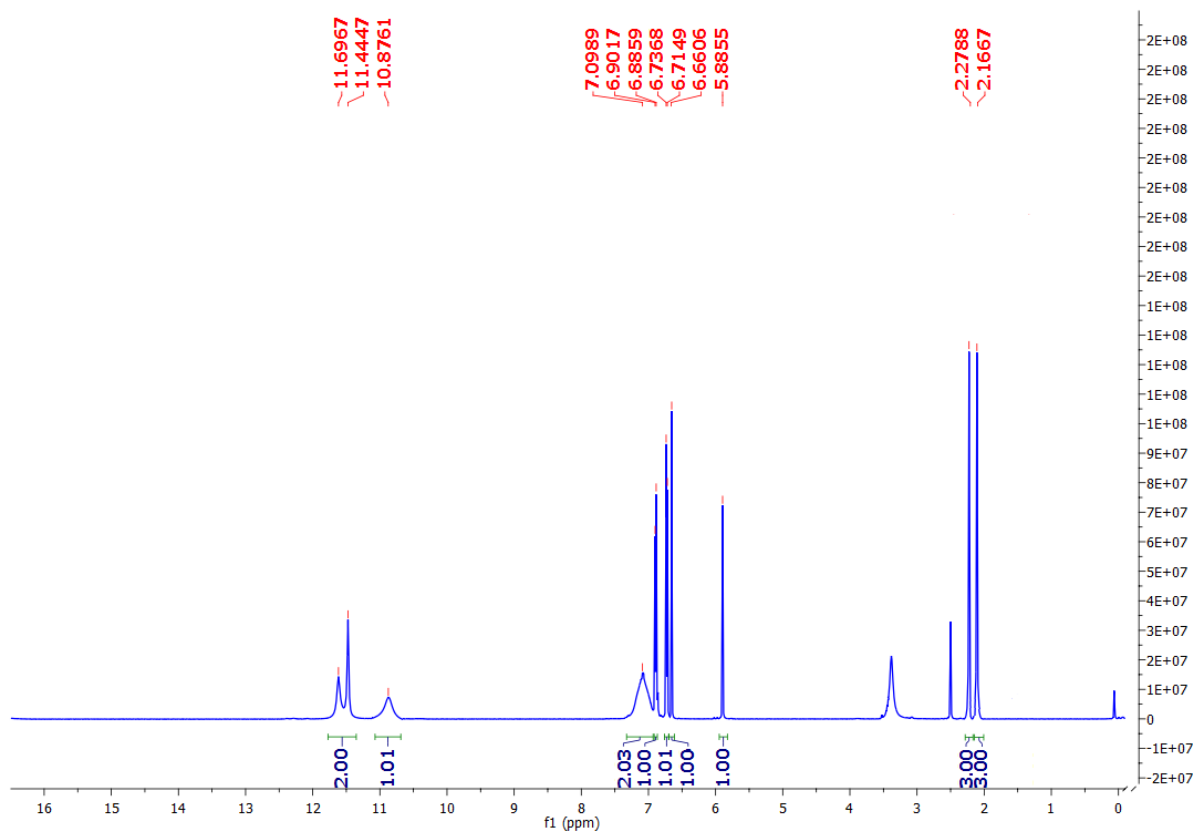
**Fig. S2** The structure and  $^1\text{H}$  NMR spectrum of product **2a**



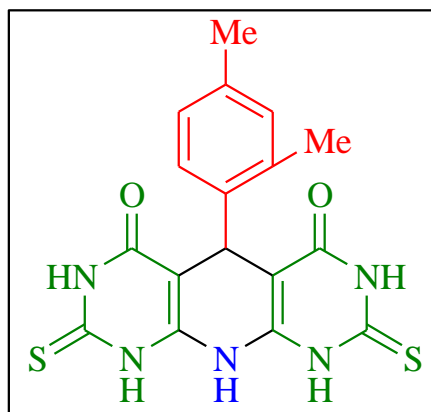
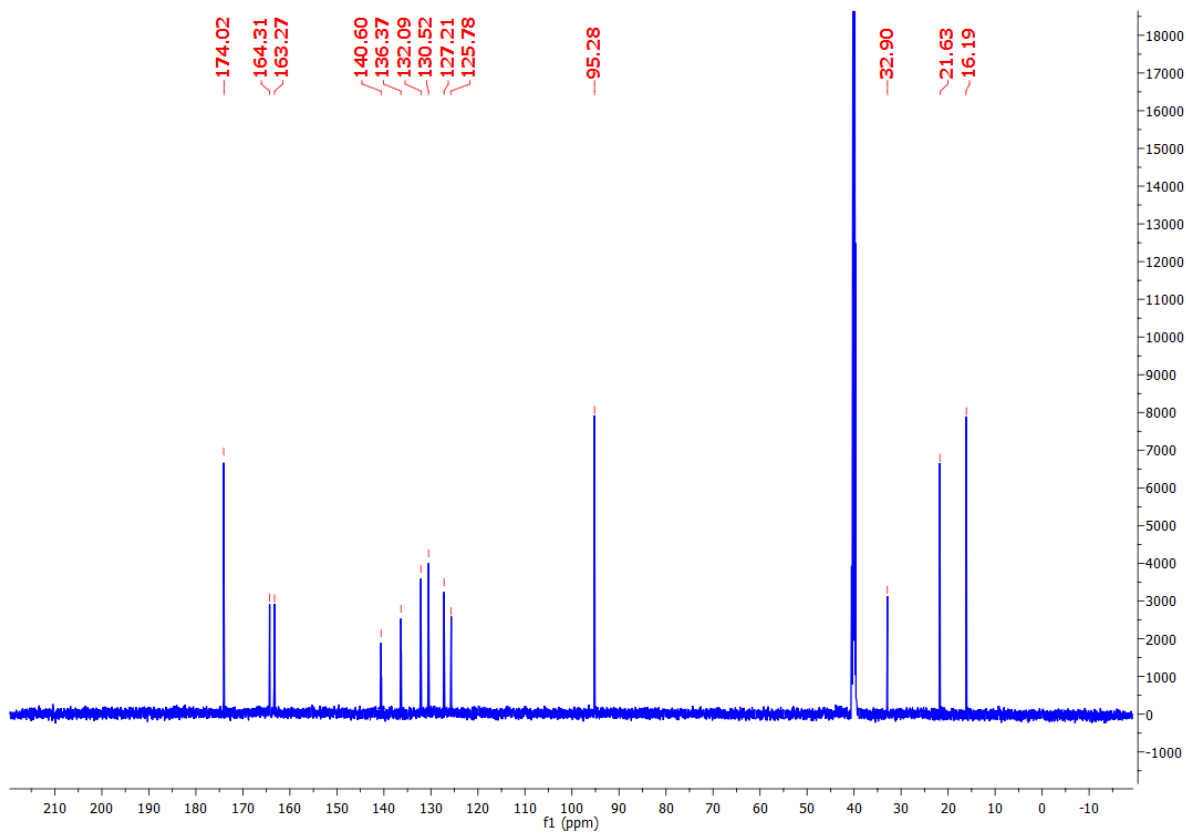
**Fig. S3** The structure and  $^1\text{H}$  NMR spectrum of product **3a**



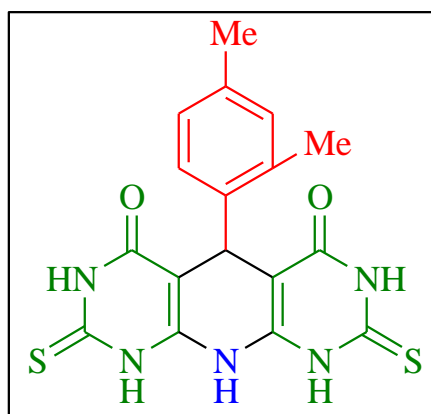
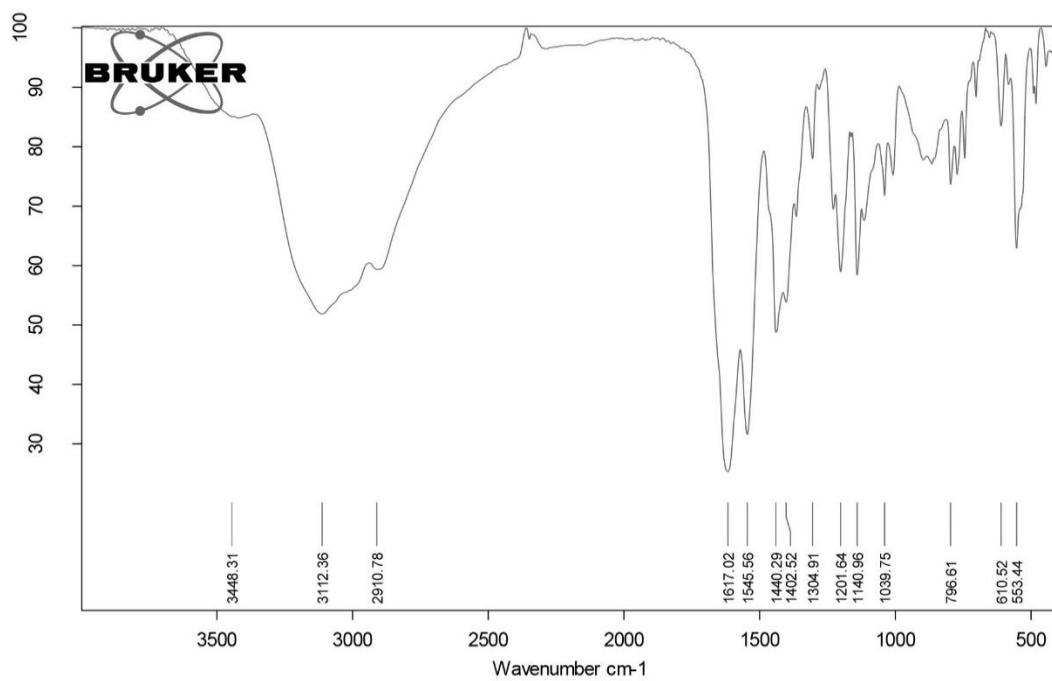
**Fig. S4** The structure and  $^1\text{H}$  NMR spectrum of product **4a**



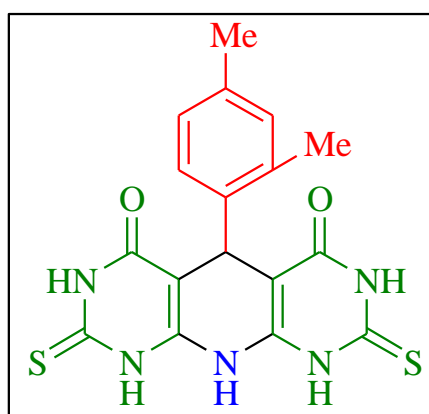
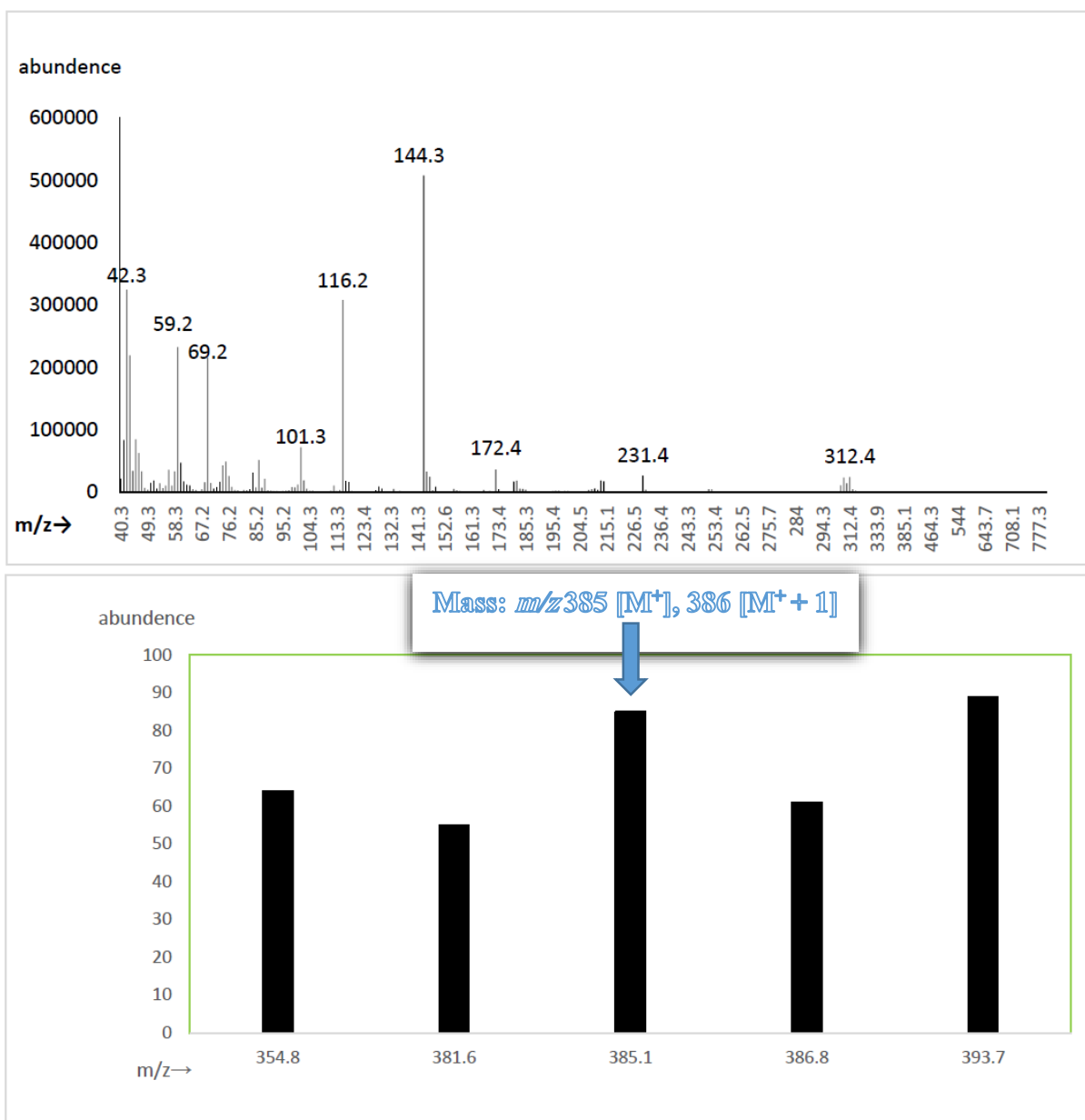
**Fig. S5** The structure and  $^1\text{H}$  NMR spectrum of product **5a**



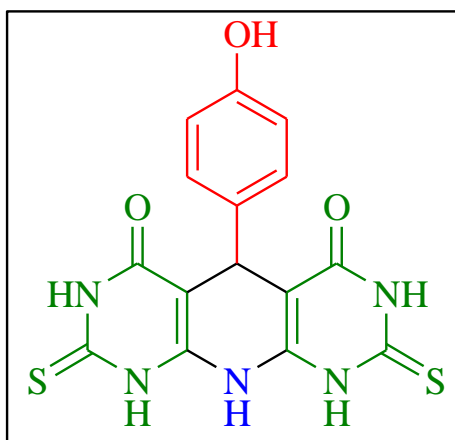
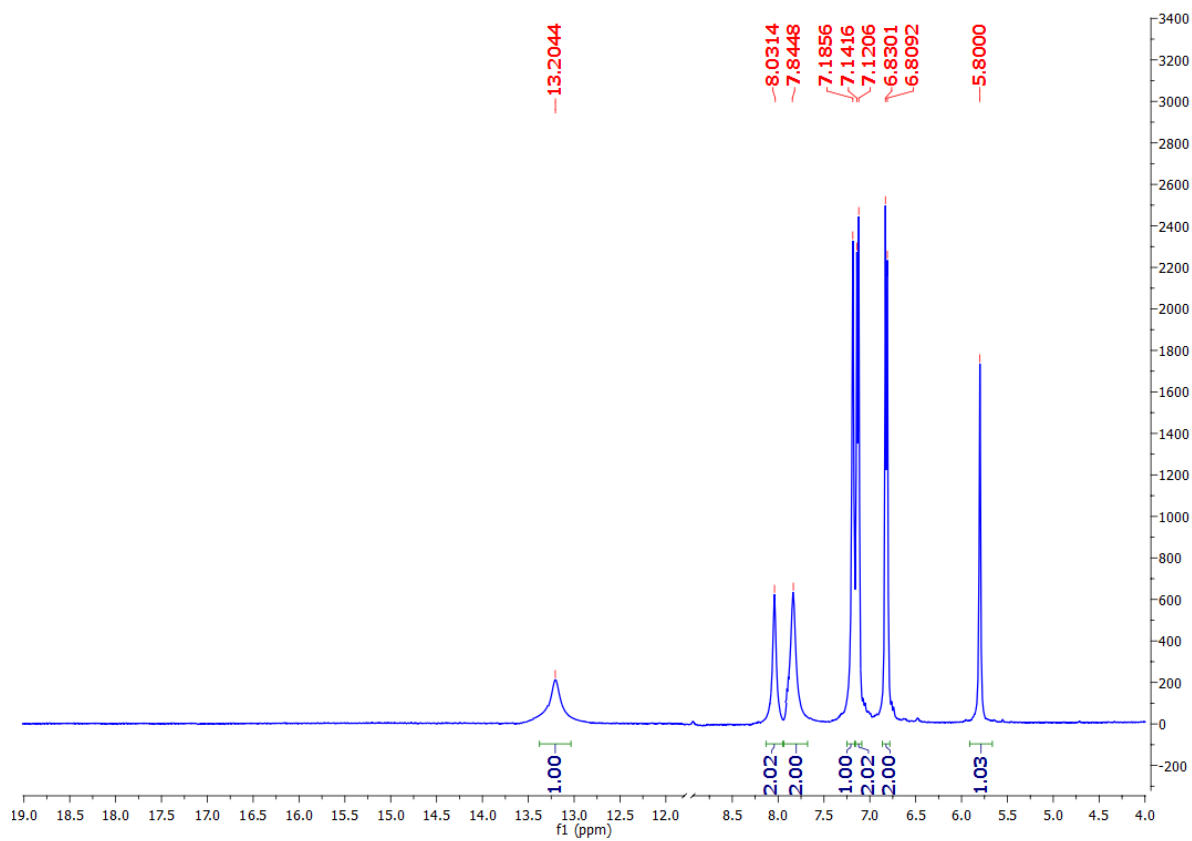
**Fig. S6** The structure and  $^{13}\text{C}$  NMR spectrum of product **5a**



**Fig. S7** The structure and FT-IR spectrum of product **5a**

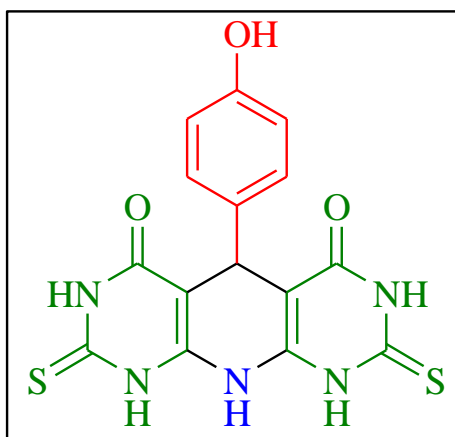
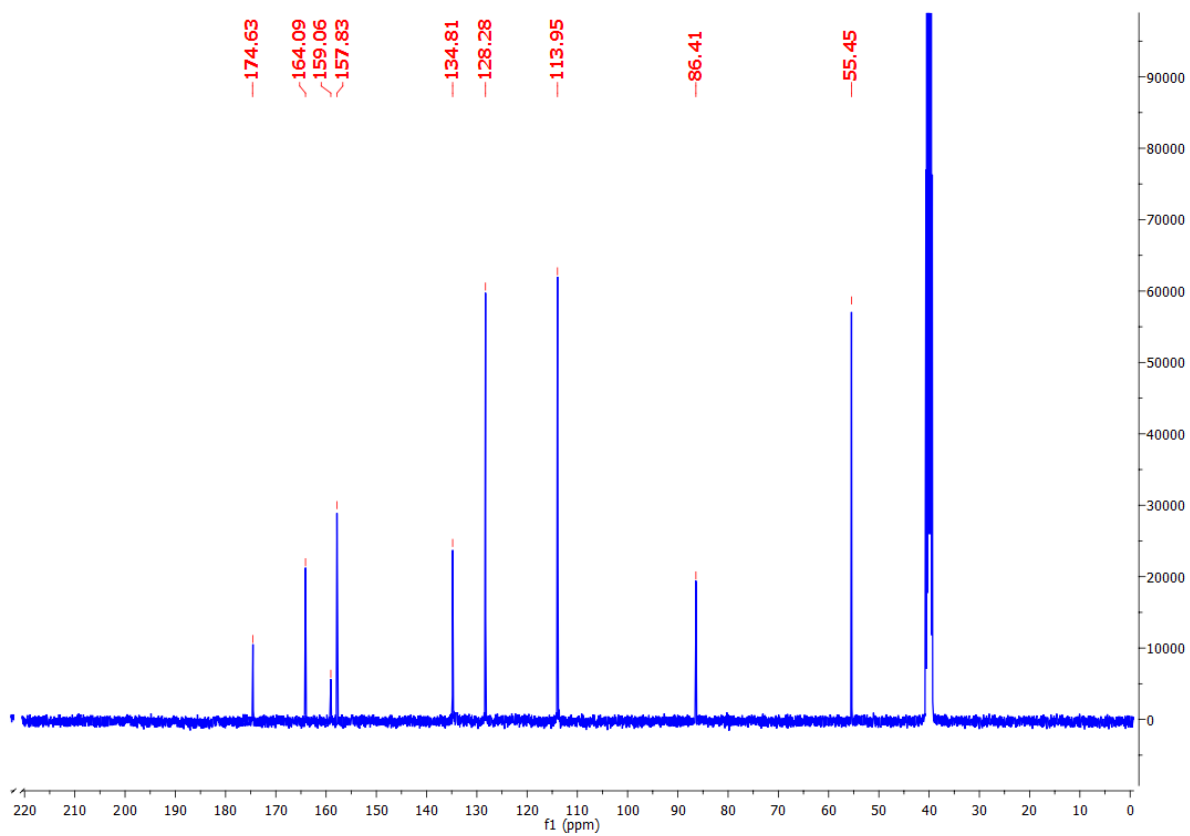


**Fig. S8** The structure and mass spectrum of product **5a**

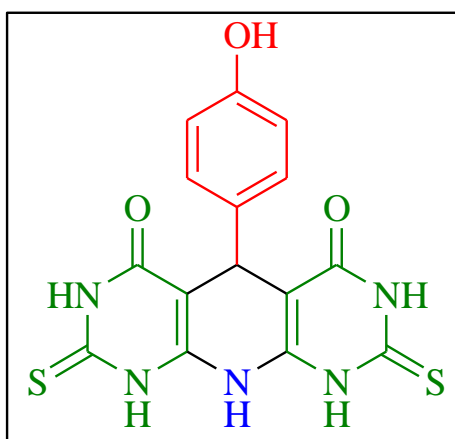
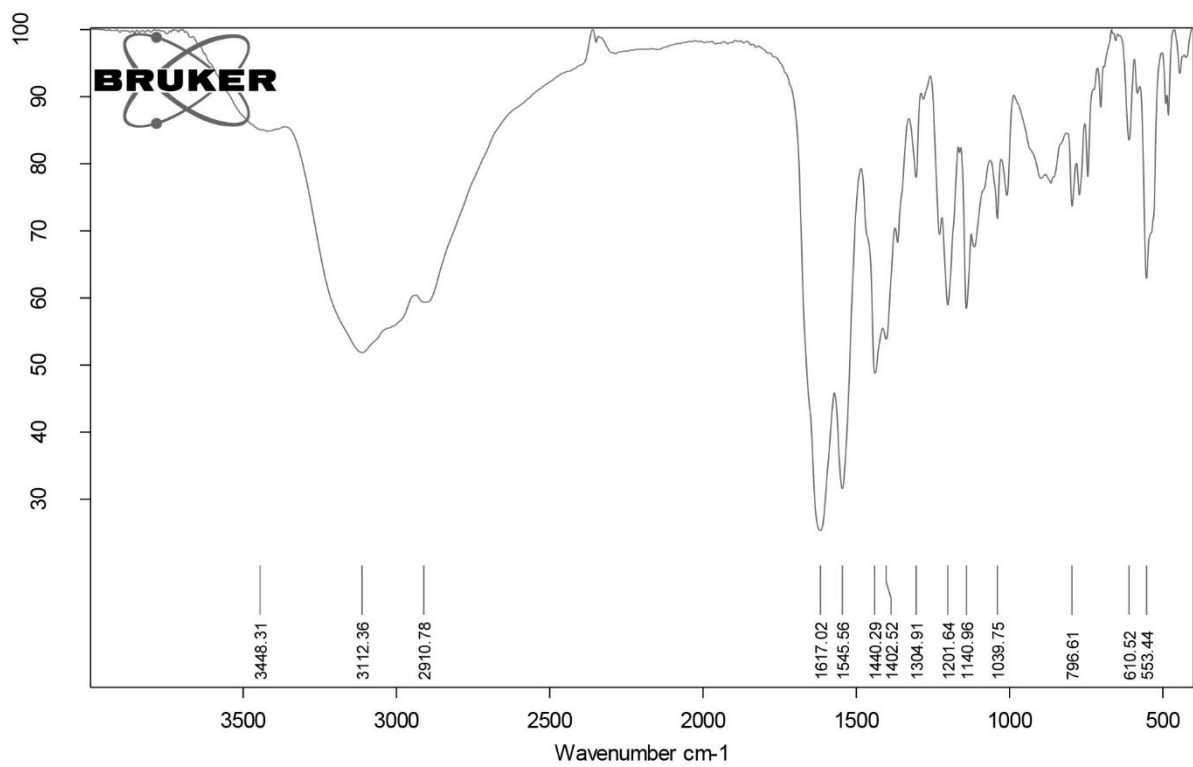


**Fig. S9** The structure and  $^1\text{H}$  NMR spectrum of product **6a**





**Fig. S10** The structure and  $^{13}\text{C}$  NMR spectrum of product **6a**



**Fig. S11** The structure and FT-IR spectrum of product **6a**

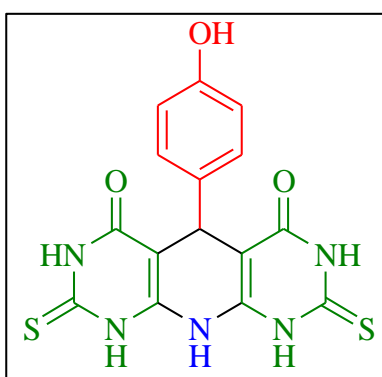
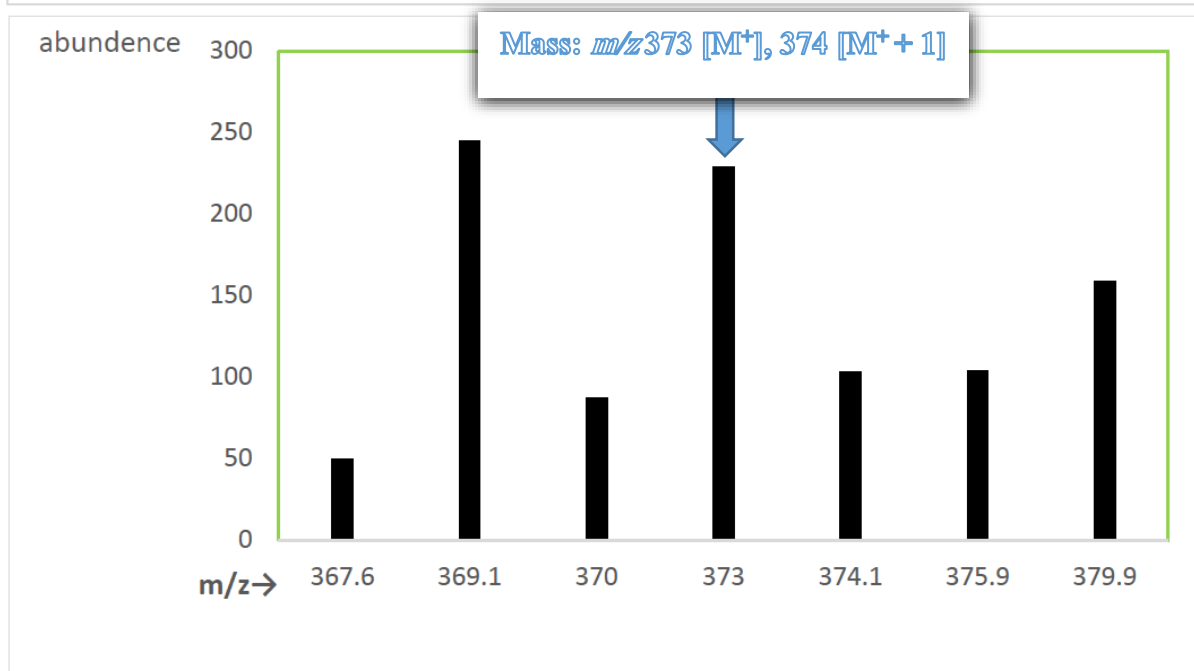
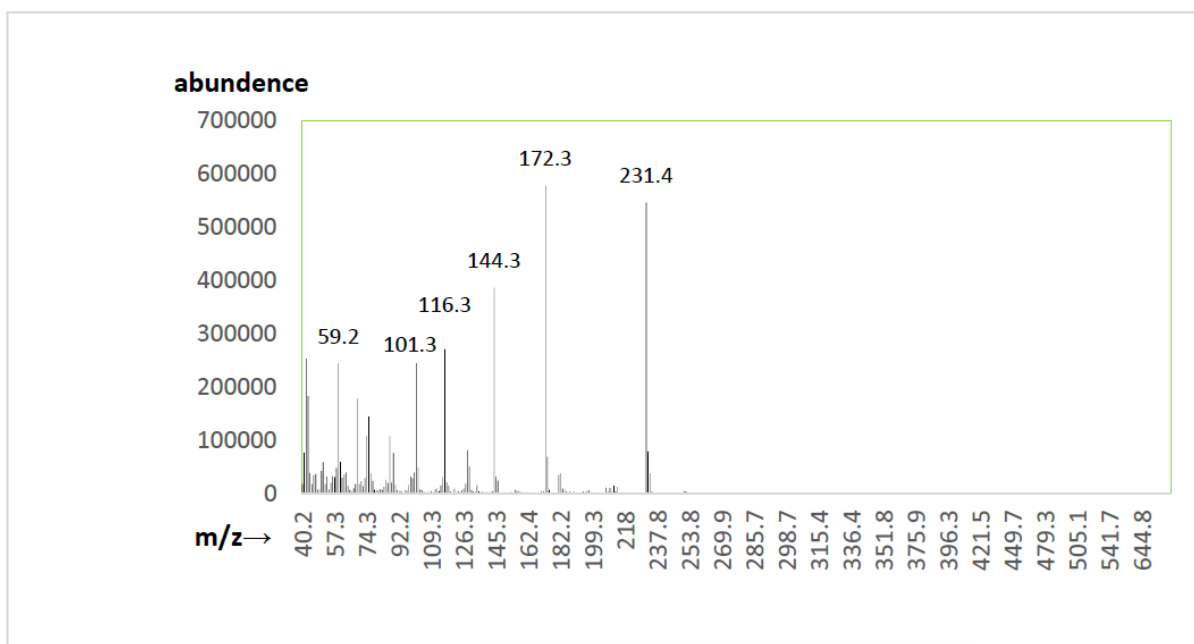
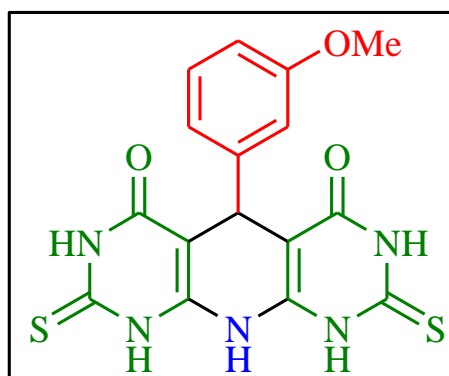
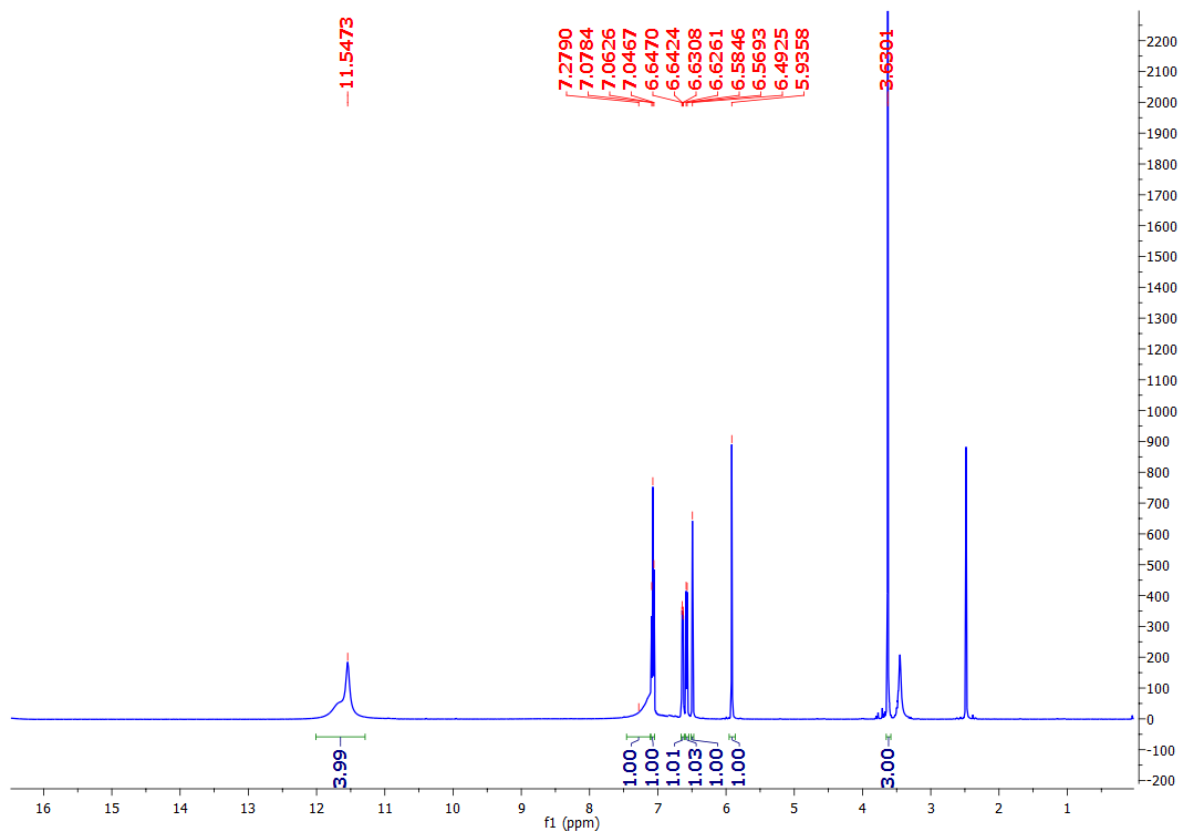
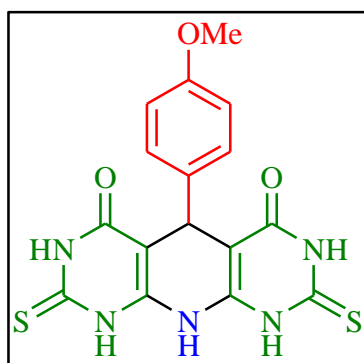
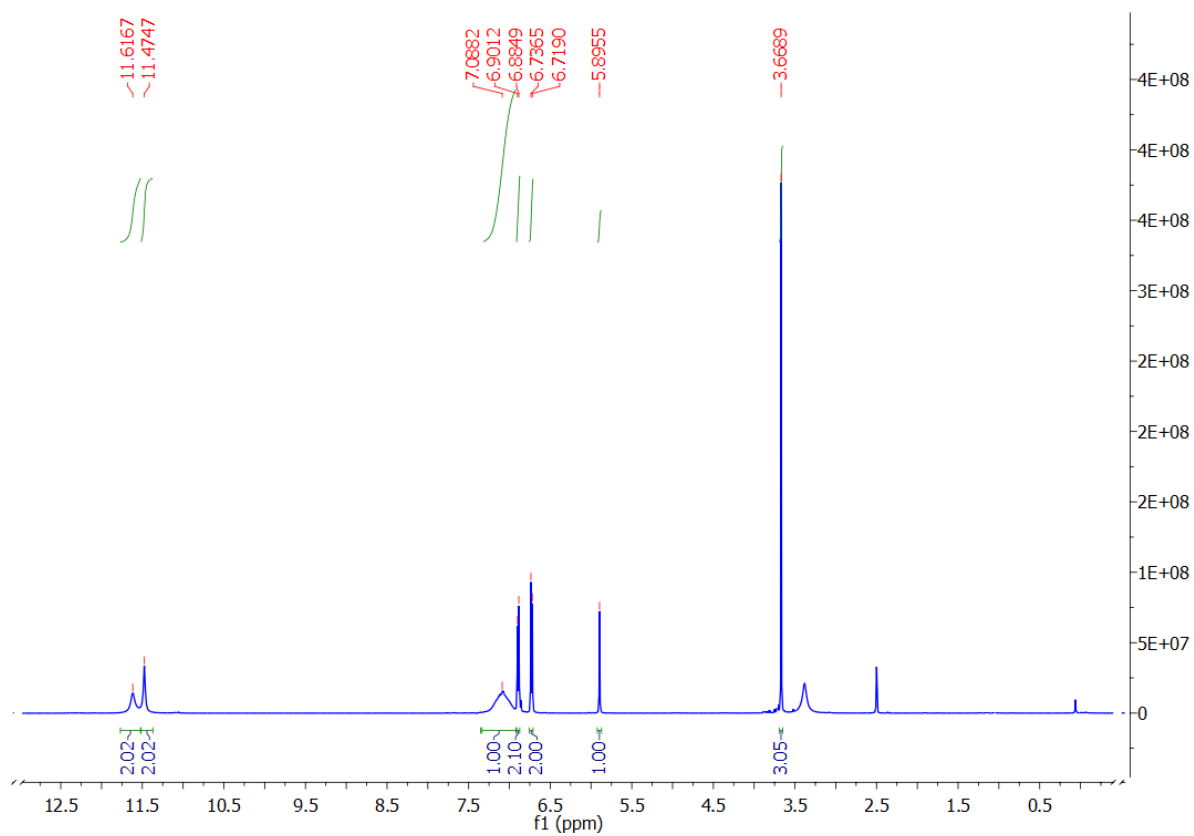


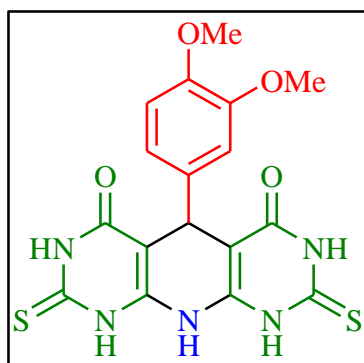
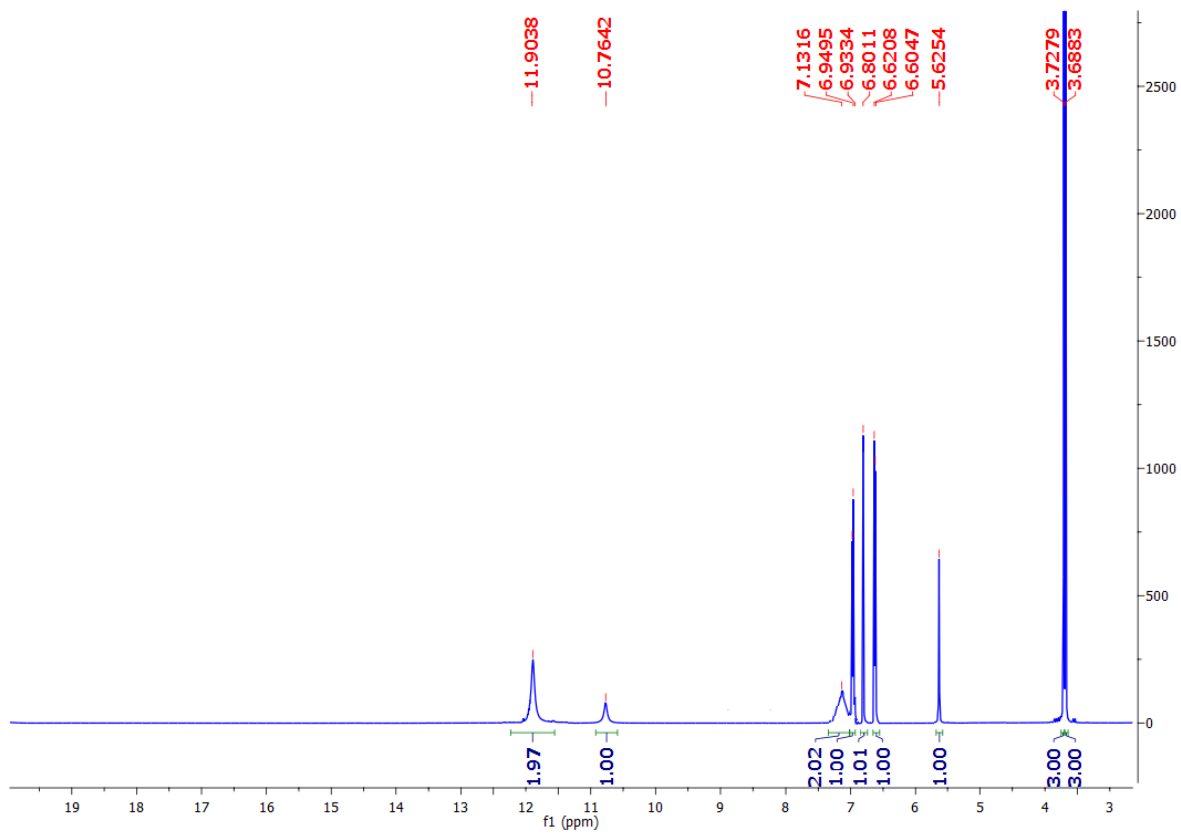
Fig. S12 The structure and mass spectrum of product 6a



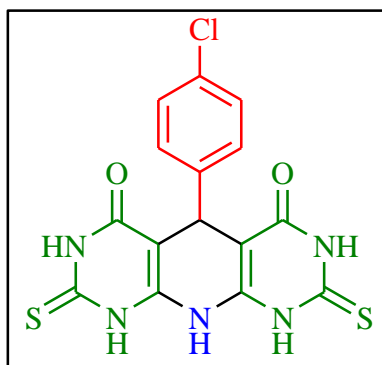
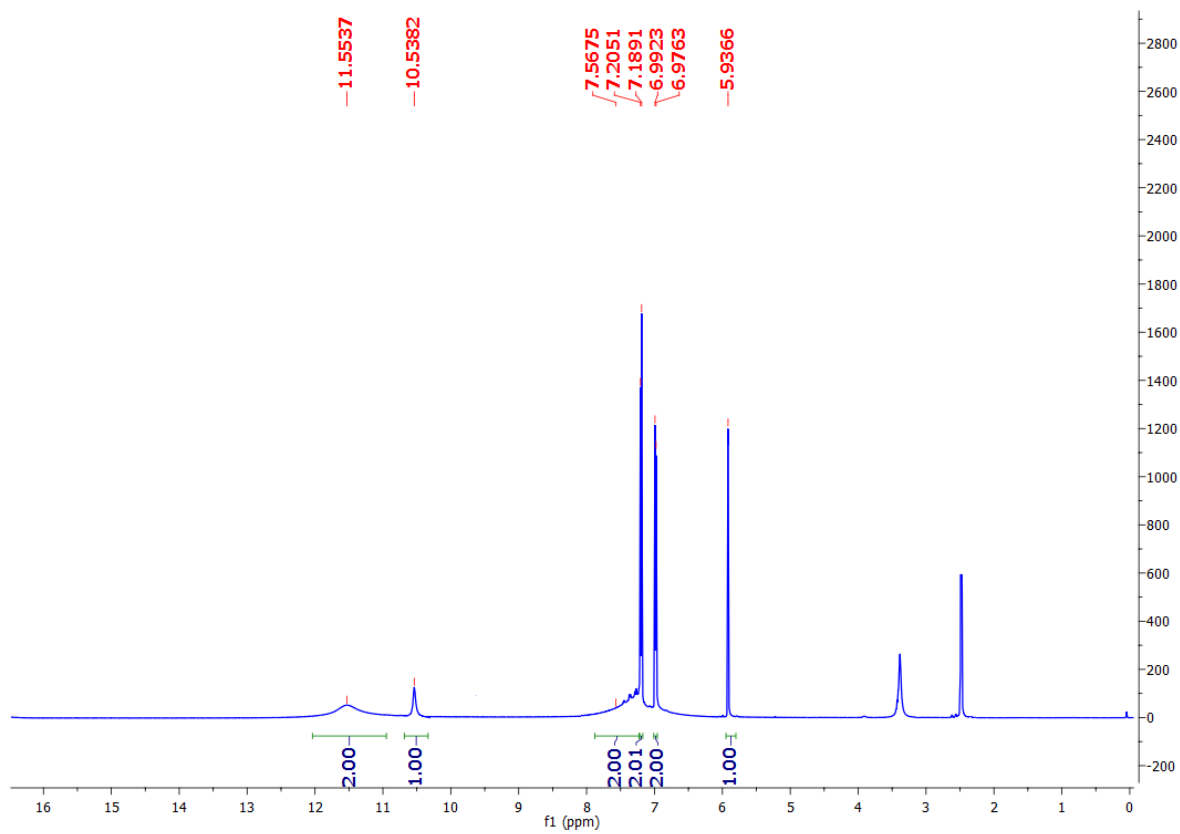
**Fig. S13** The structure and  $^1\text{H}$  NMR spectrum of product **7a**



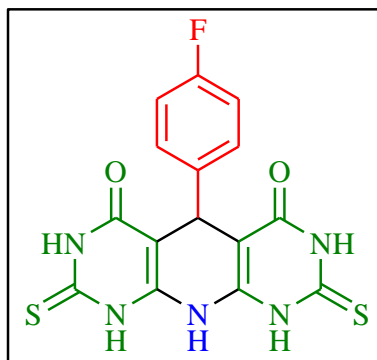
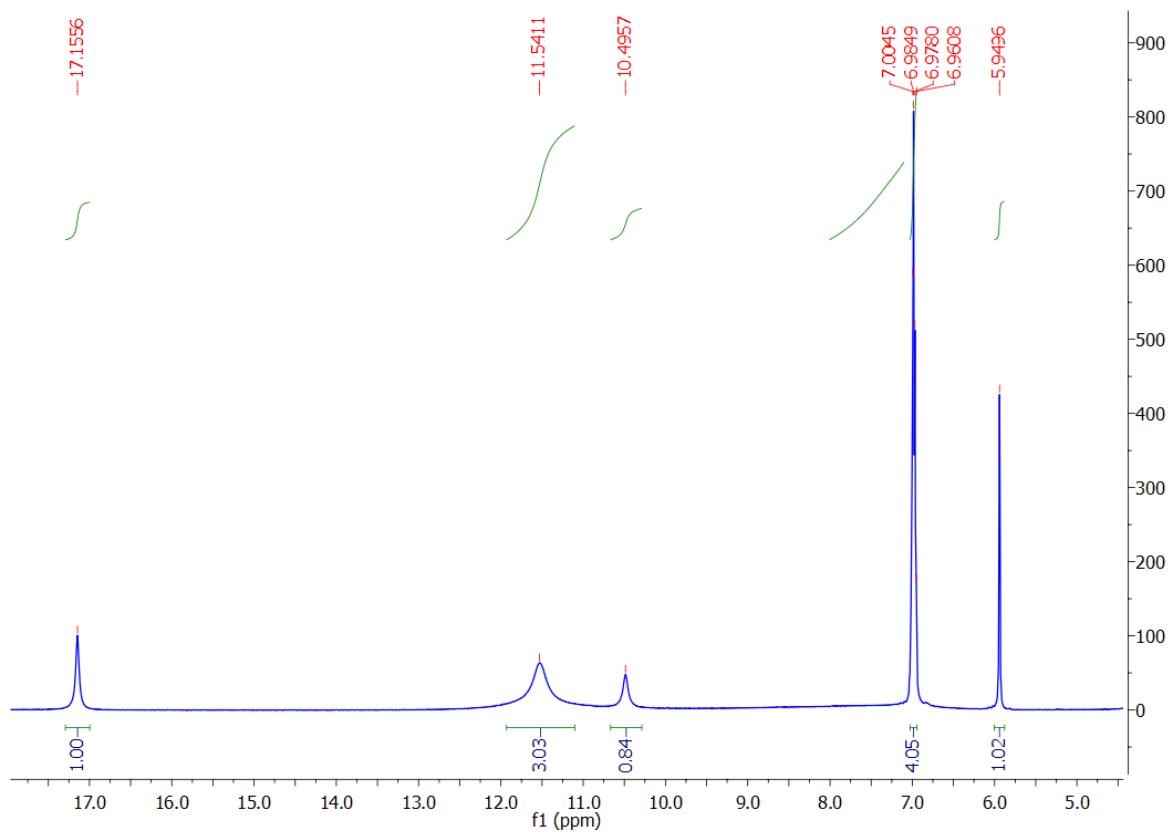
**Fig. S14** The structure and  $^1\text{H}$  NMR spectrum of product **8a**



**Fig. S15** The structure and  $^1\text{H}$  NMR spectrum of product **9a**

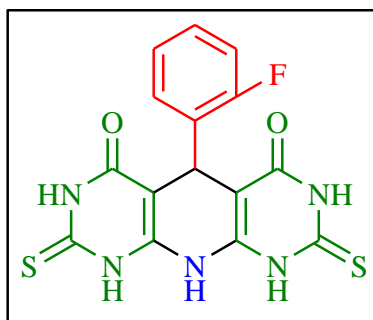
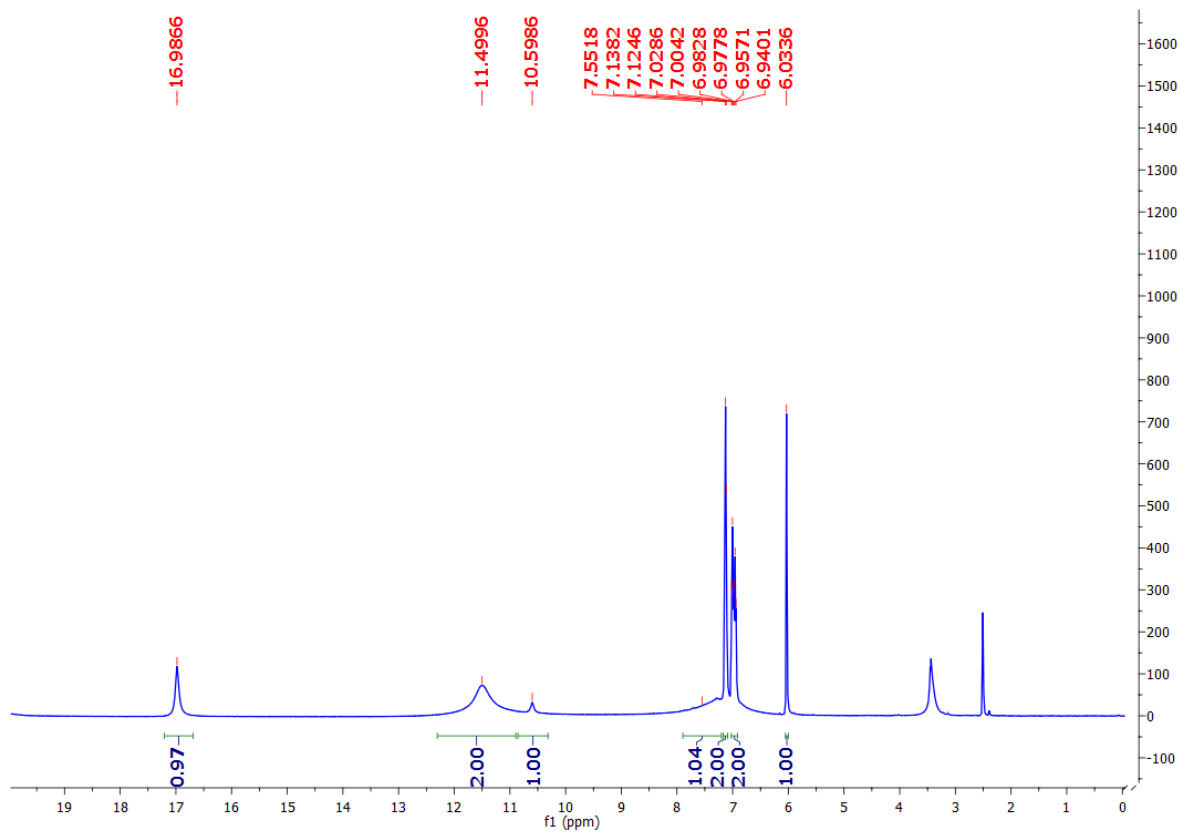


**Fig. S16** The structure and  $^1\text{H}$  NMR spectrum of product **10a**



**Fig. S17** The structure and  $^1\text{H}$  NMR spectrum of product **11a**





**Fig. S18** The structure and  $^1\text{H}$  NMR spectrum of product **12a**

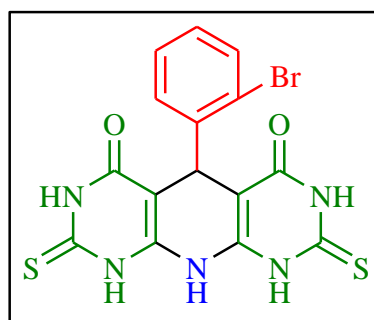
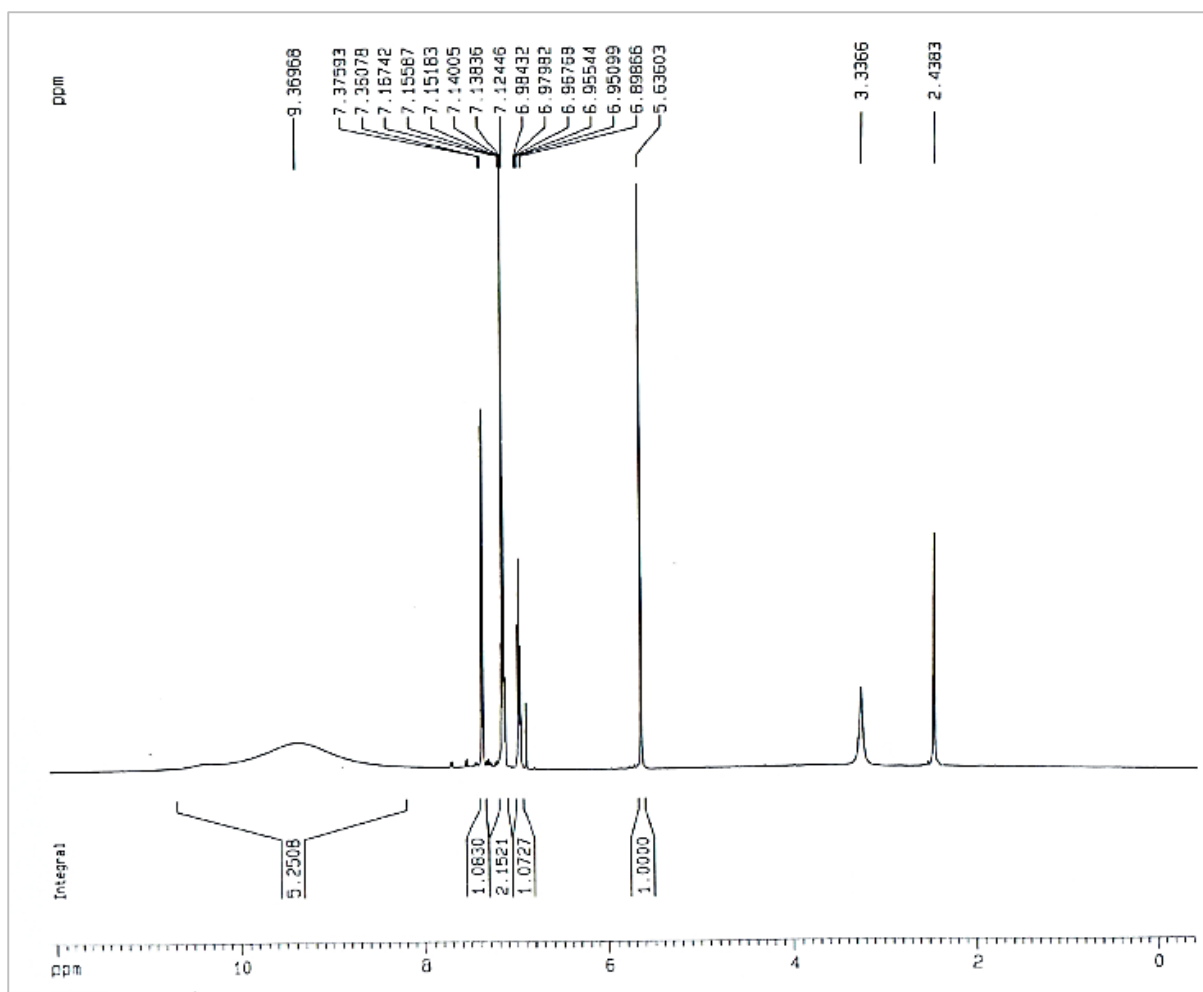
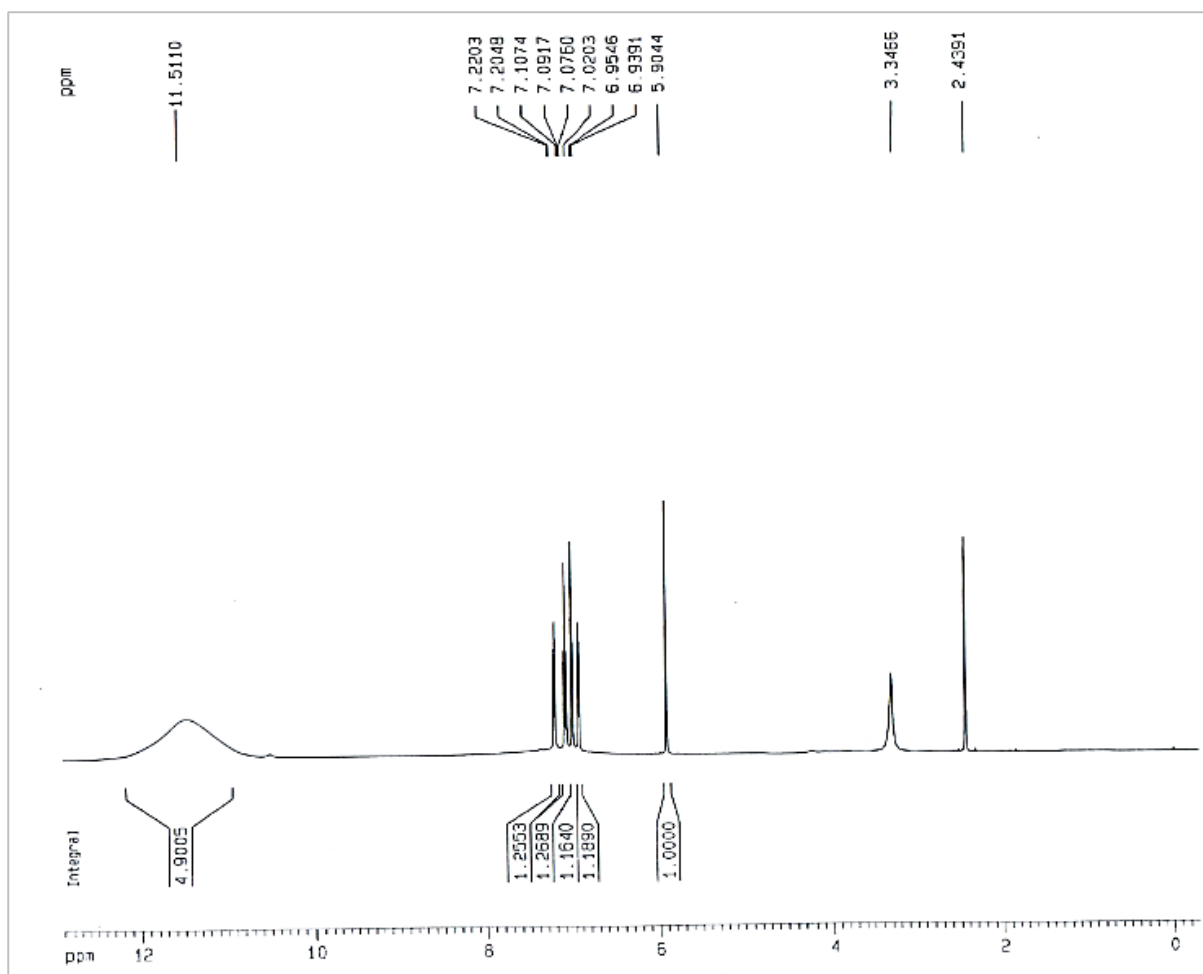
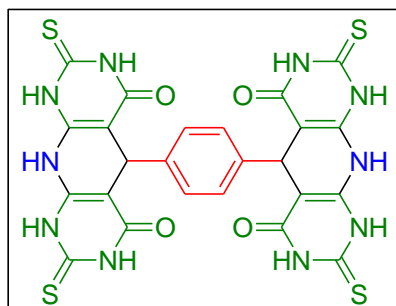
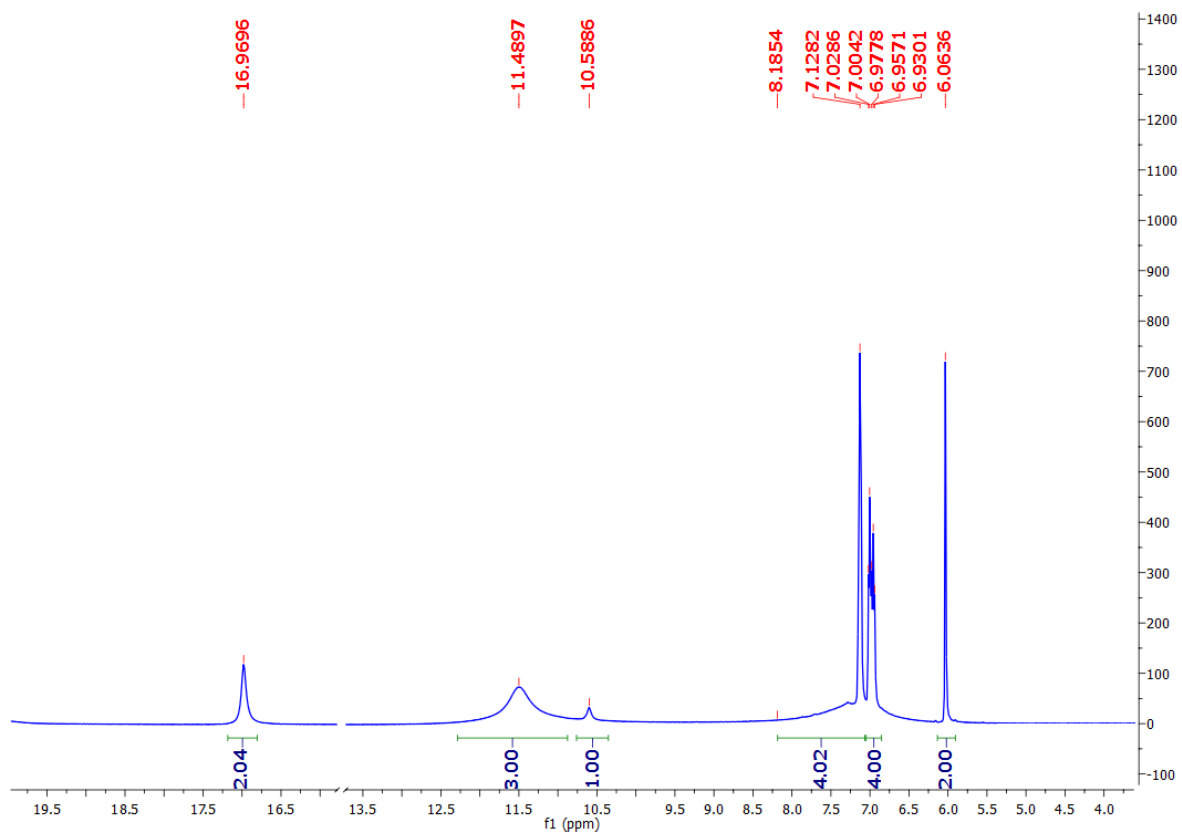


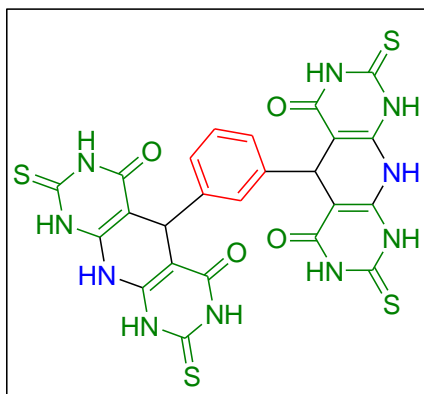
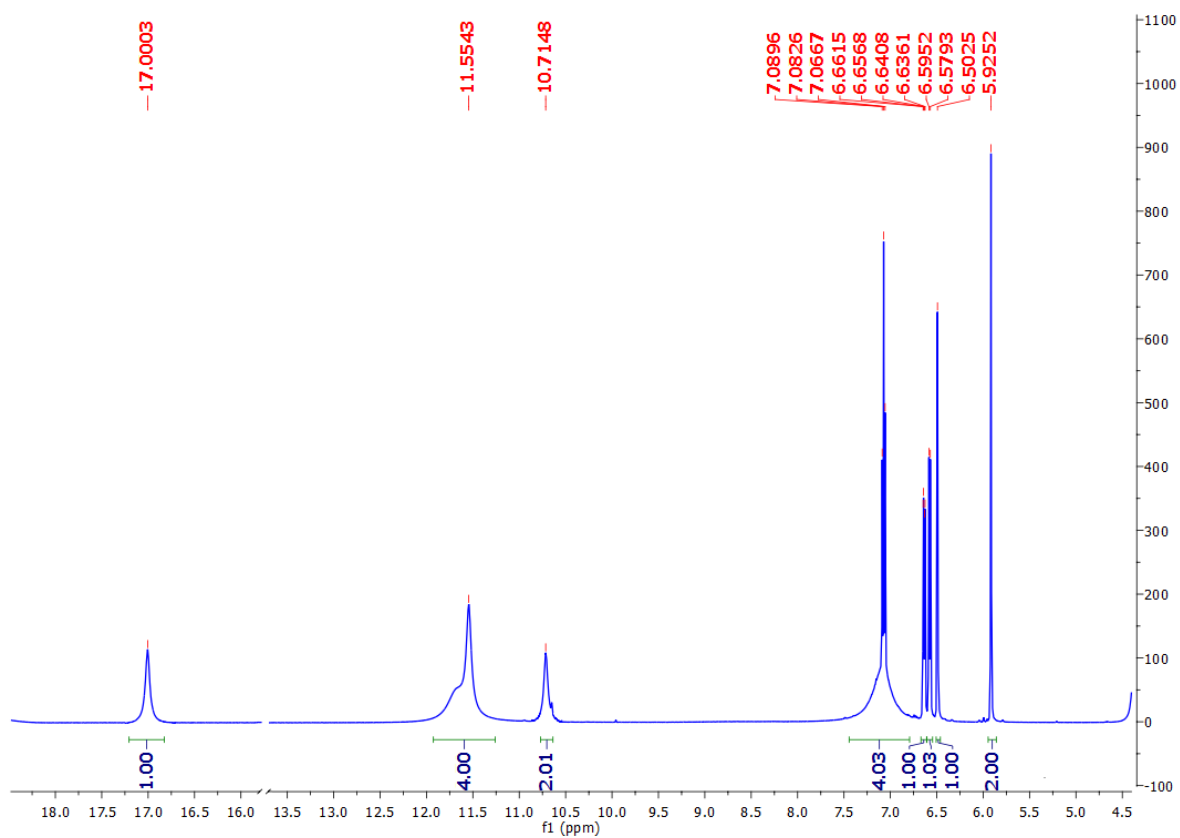
Fig. S19 The structure and  $^1\text{H}$  NMR spectrum of product 13a



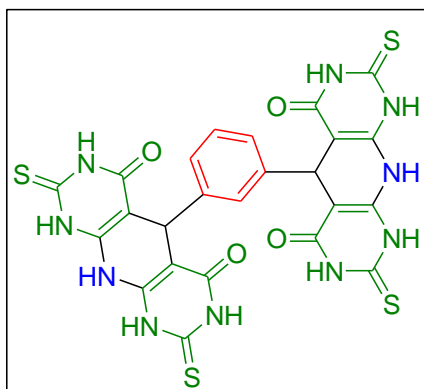
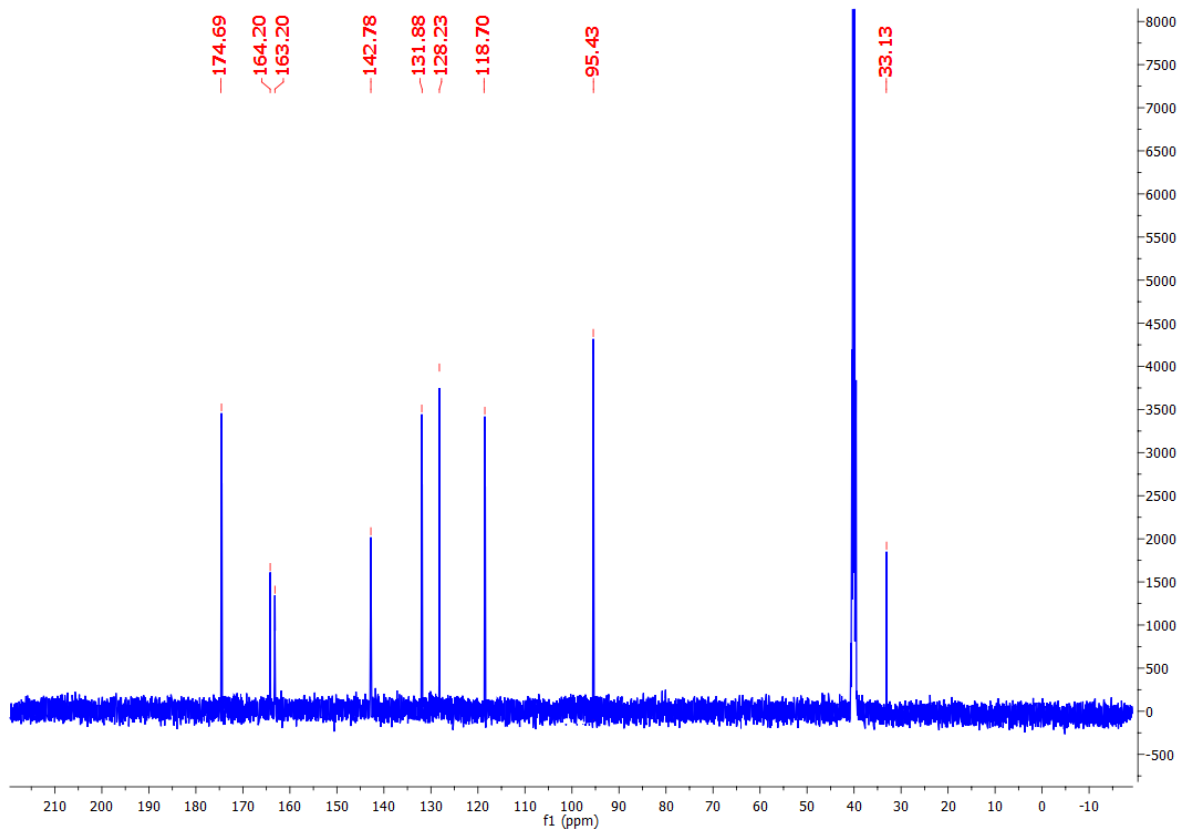
**Fig. S20** The structure and  $^1\text{H}$  NMR spectrum of product **14a**



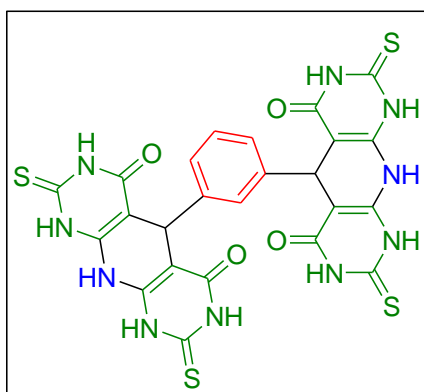
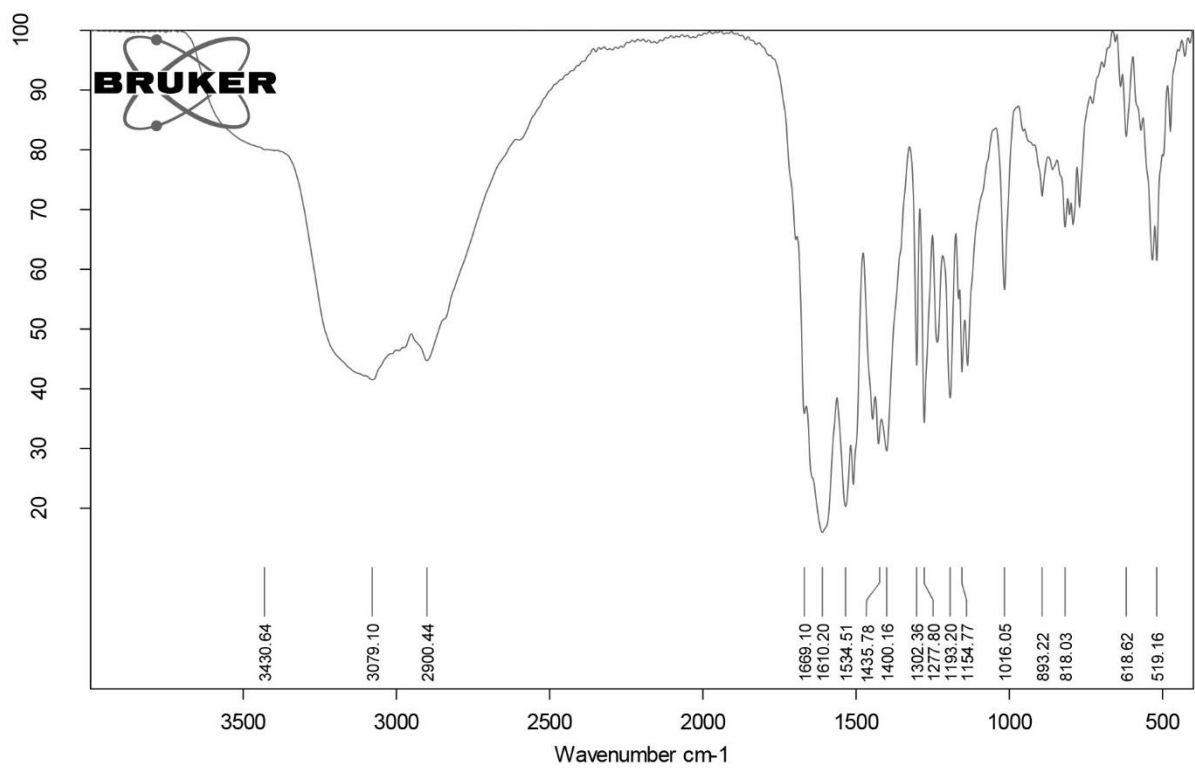
**Fig. S21** The structure and  $^1\text{H}$  NMR spectrum of product **15a**



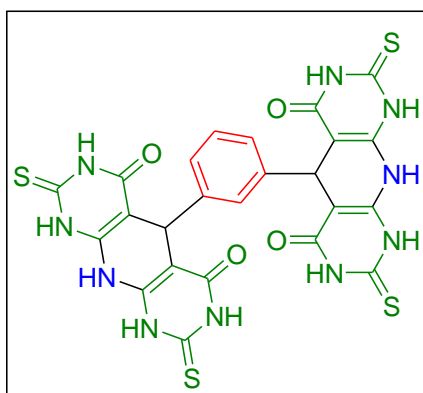
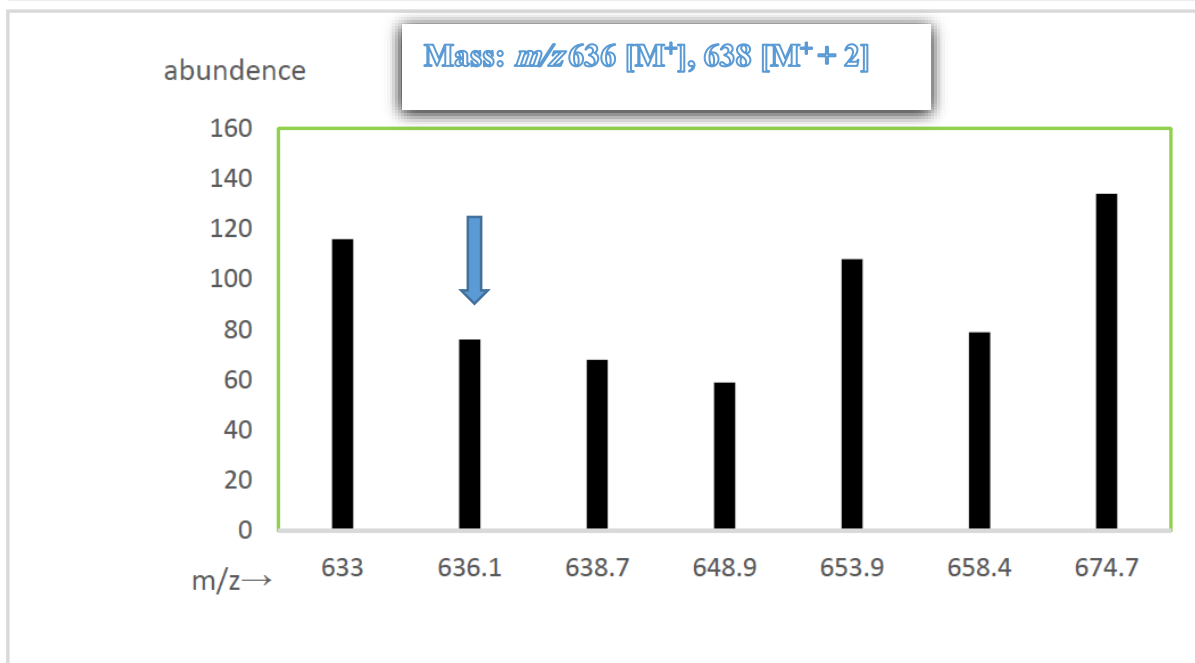
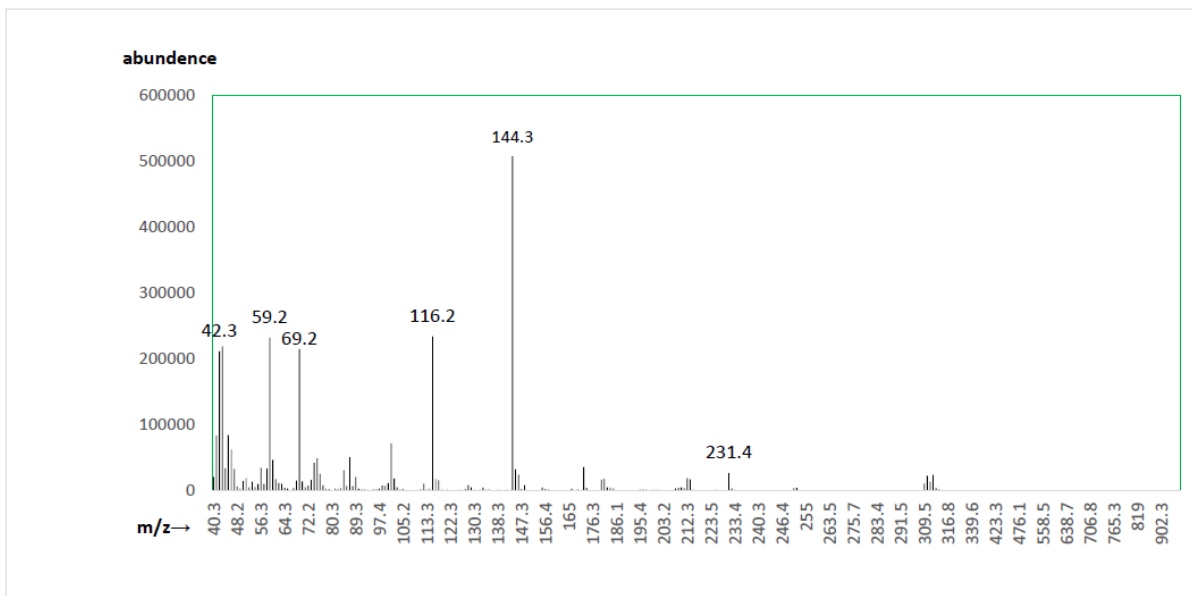
**Fig. S22** The structure and  $^1\text{H}$  NMR spectrum of product **16a**



**Fig. S23** The structure and  $^{13}\text{C}$  NMR spectrum of product **16a**



**Fig. S24** The structure and FT-IR spectrum of product **16a**



**Fig. S25** The structure and mass spectrum of product **16a**