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Supporting information for article:

Crystallographic characterization of three cathinone hydrochlorides new on the NPS market: 1-(4-methylphenyl)-2-(pyrrolidin-1-yl)hexan-1-one (4-MPHP), 4-methyl-1-phenyl-2-(pyrrolidin-1-yl)pentan-1-one (α-PiHP) and 2-(methylamino)-1-(4-methylphenyl)pentan-1-one (4-MPD)

Marcin Rojkiewicz, Piotr Kuś, Maria Książek and Joachim Kusz

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Crystallographic characterization of three, new on the NPS market cathinone hydrochlorides: 1-(4-methylphenyl)-2-(pyrrolidin-1-yl)-hexan-1-one (4-MPHP), 1-phenyl-2-(pyrrolidin-1-yl)-4-methylpentan-1-one (α-PiHP) and 2-(methylamino)-1-(4-methylphenyl)-pentan-1-one (4-MPD)

Marcin Rojkiewicz[a]*, Piotr Kuś[a], Maria Książek[b] and Joachim Kusz[b]

[a]Department of Organic Synthesis, Faculty of Science and Technology, University of Silesia, 9 Szkolna Street, Katowice, 40-006, Poland

[b]Institute of Physics, Faculty of Science and Technology, University of Silesia, 1 75 Pułku Piechoty Street, Chorzów, 41-500, Poland

Correspondence email: marcin.rojkiewicz@us.edu.pl

Instrumentation and analysis conditions of investigated samples

For the purpose of gas chromatography and electrospray ionization mass spectrometry, the 10-mg of each sample was dissolved in 1 mL methanol without the need for ultrasonication. An aliquot of 10 μL was collected from the solution and diluted one hundred-fold with methanol and analyzed by GC-MS and ESI-MS. For NMR spectroscopic analysis, 10 mg of each sample was dissolved in 0.6 mL deuterated dimethylsulfoxide (DMSO-d6). The IR and Raman analyses were performed without any further sample treatment, and for the UV-VIS analysis, the samples were dissolved and diluted in methanol. All analytical data for investigated compounds are provided in supporting information.

For GC-MS analysis, the Thermo Trace Ultra chromatograph was used, coupled with the Thermo DSQ mass spectrometer (Thermo Scientific). The analyses were carried out with use of the Rxi®-5Sil MS column (Restek, Bellefonte, PA, USA). The following working parameters were employed: injector temperature, 260^{o}C; oven temperatures, 100^{o}C for 2 min, ramp at 20^{o}C/min to 260^{o}C; the carrier gas (helium) flow rate, 1.2 mL min^{-1}; MS transfer line temperature, 250^{o}C; MS source temperature, 250^{o}C; the injection volume, 1 μL, the splitless mode.

For ESI-MS analysis Thermo TSQ Vantage mass spectrometer with electrospray ionization source (Thermo Scientific, Warsaw, Poland) was used. The following working parameters for the direct infusion ESI-MS experiment were employed: sheath gas pressure, 5 psi; H-ESI vaporizer temperature, 50^{o}C; spray voltage, 3500 V; ion transfer tube temperature, 50^{o}C; direct infusion syringe flow rate, 5 μL/min. The obtained data were processed using Xcalibur and TSQTune software (Thermo Scientific). The analytes were electrosprayed in the positive mode (ESI(+)-MS). In the ESI-MS^{2} mode the ESI-carrier and collision gases were nitrogen and argon, respectively.

The NMR spectra of the samples were recorded with use of the UltraShield 400 MHz apparatus (Bruker, Bremen, Germany) and deuterated dimethylsulfoxide (DMSO-d6) was used as a solvent. The data were collected with the chemical shift referenced to a residual solvent signal.

The infrared (IR) spectrum of the powder evidence material was acquired with use of the Nicolet iS50 FT-IR Spectrometer (Thermo Scientific), using the ATR technique, and the spectrum was collected in the wavenumber range 3500-400 cm-1. Raman measurements were made using a Thermo Scientific™ DXR™2xi Raman imaging microscope. The data were collected using a 780nm laser. The UV–Vis absorption spectra were recorded in the methanol solution using the Thermo Scientific Evolution 160 UV-VIS Spectrophotometer, and the spectrum was collected in range of wavelength 190-400 nm.

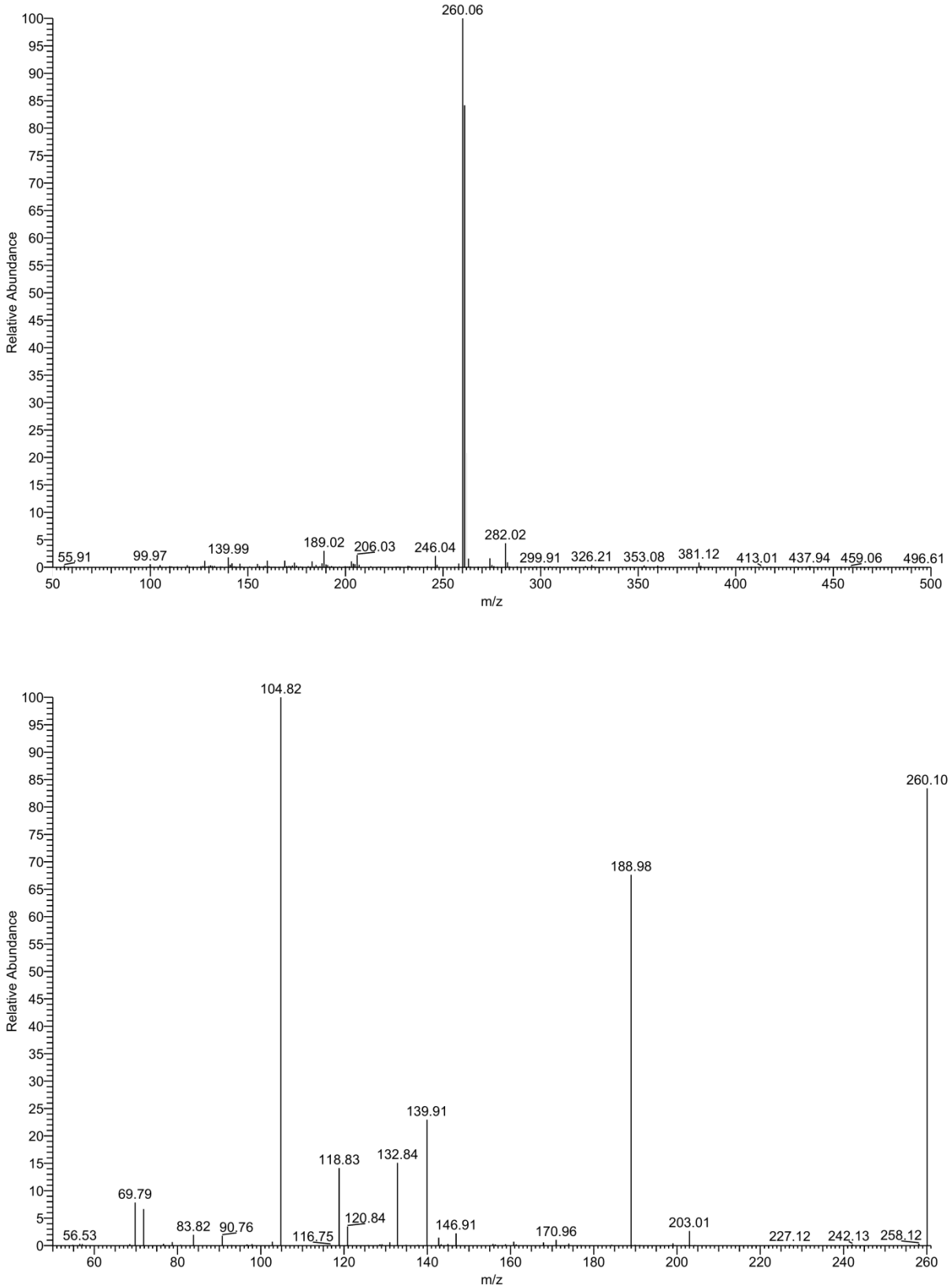

Figure S1 ESI-MS (upper) and ESI-MS/MS (lower) spectrum of compound **1** (MPHP)

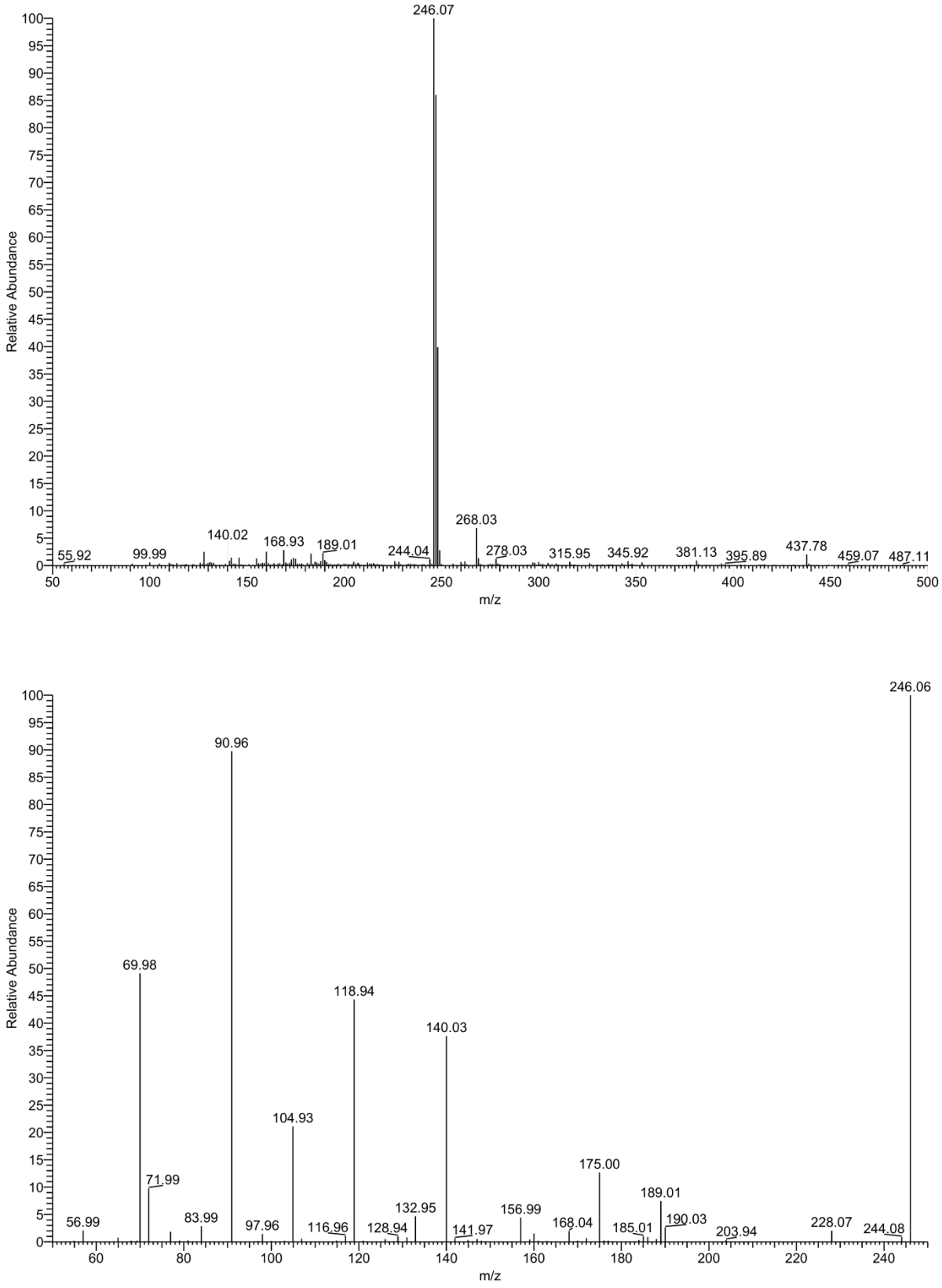

Figure S2 ESI-MS (upper) and ESI-MS/MS (lower) spectrum of compound **2** (α-PiHP)

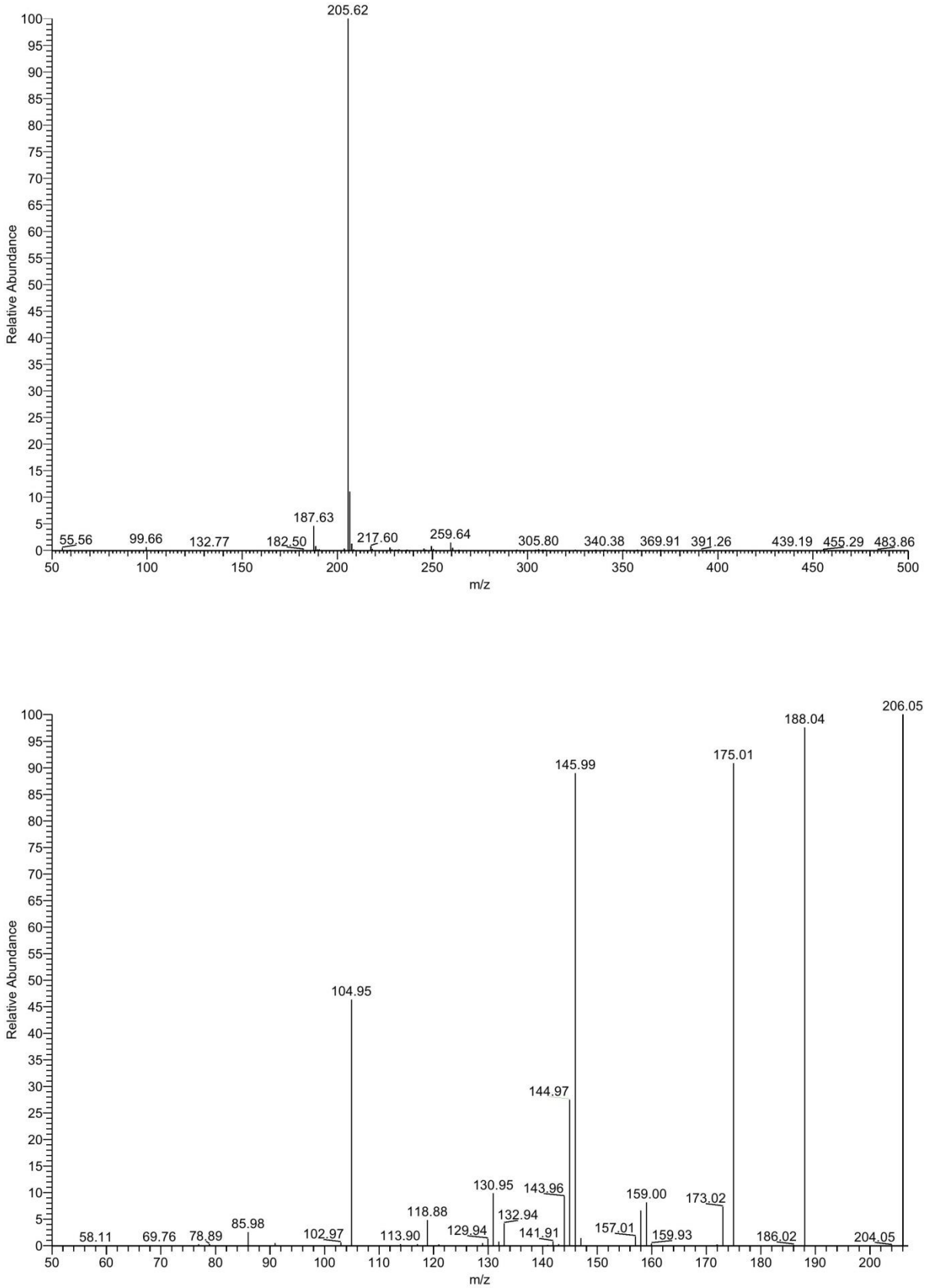

Figure S3 ESI-MS (upper) and ESI-MS/MS (lower) spectrum of compound **3** (4-MPD)

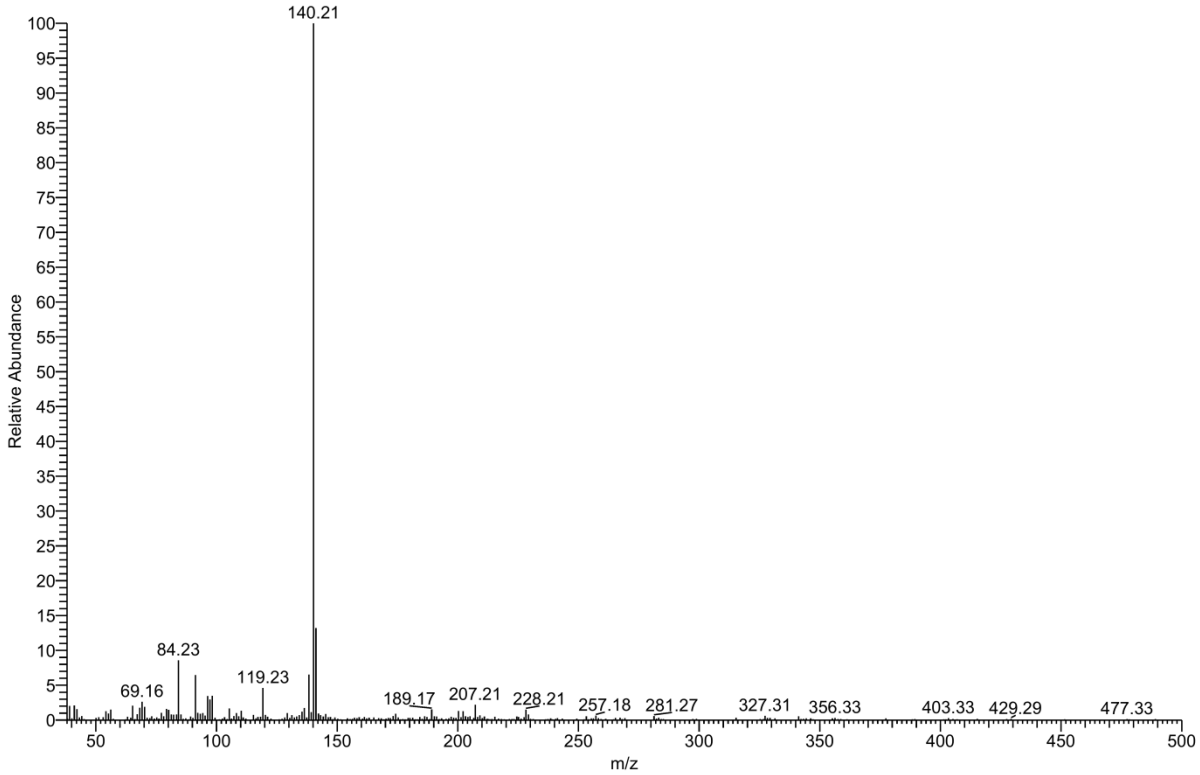

Figure S4 EI-MS spectrum of compound **1** (MPHP)

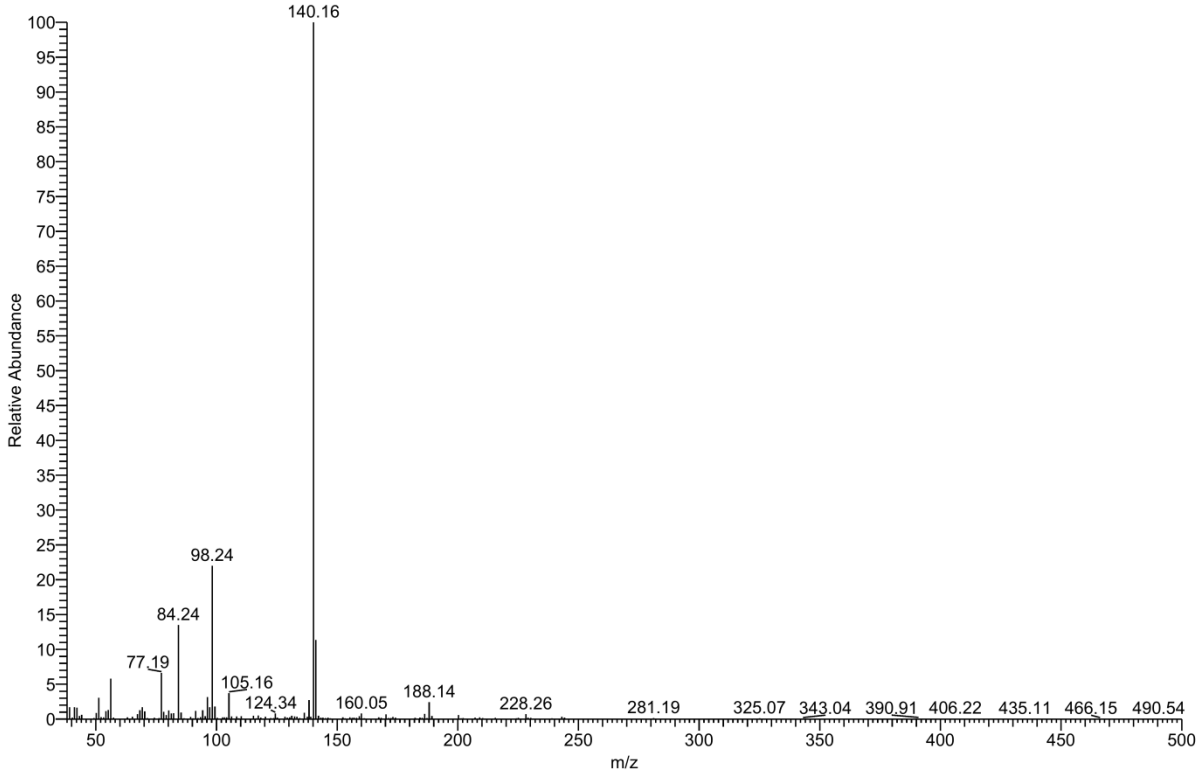

Figure S5 EI-MS spectrum of compound **2** (α-PiHP)

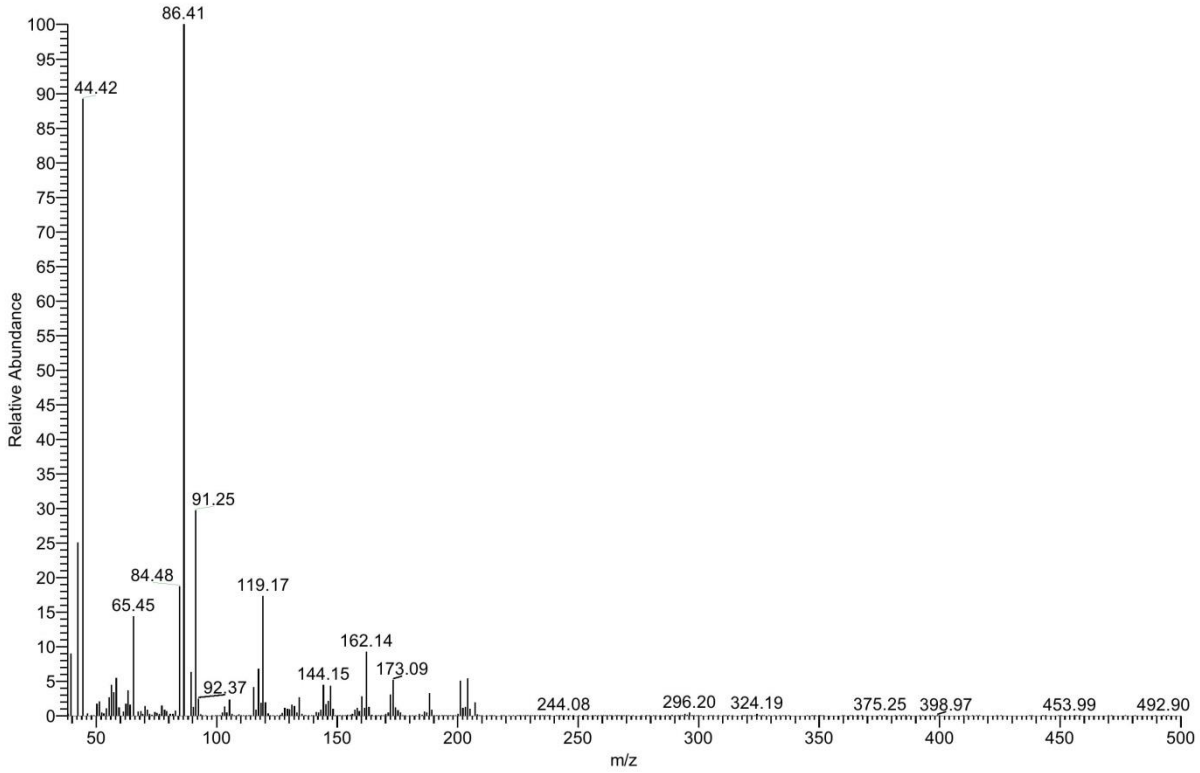

Figure S6 EI-MS spectrum of compound **3** (4-MPD)

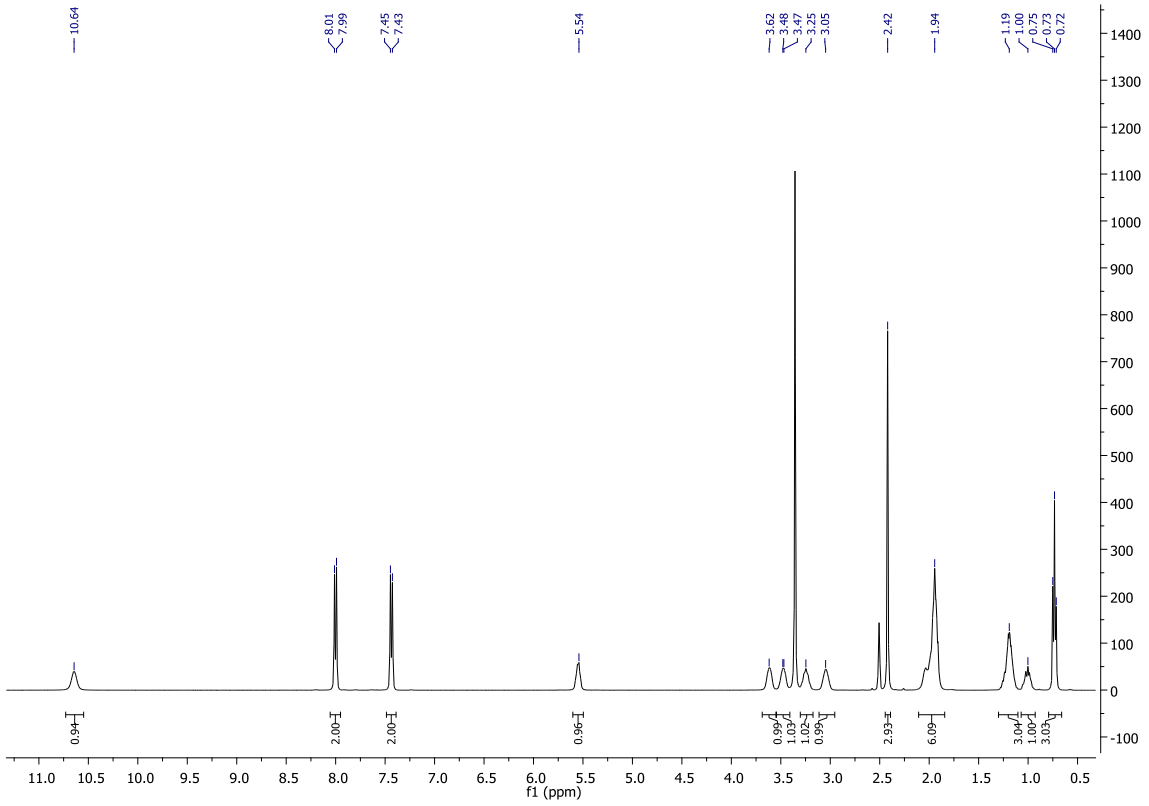

Figure S7 ^{1}H nuclear magnetic resonance spectrum of compound **1** (MPHP)

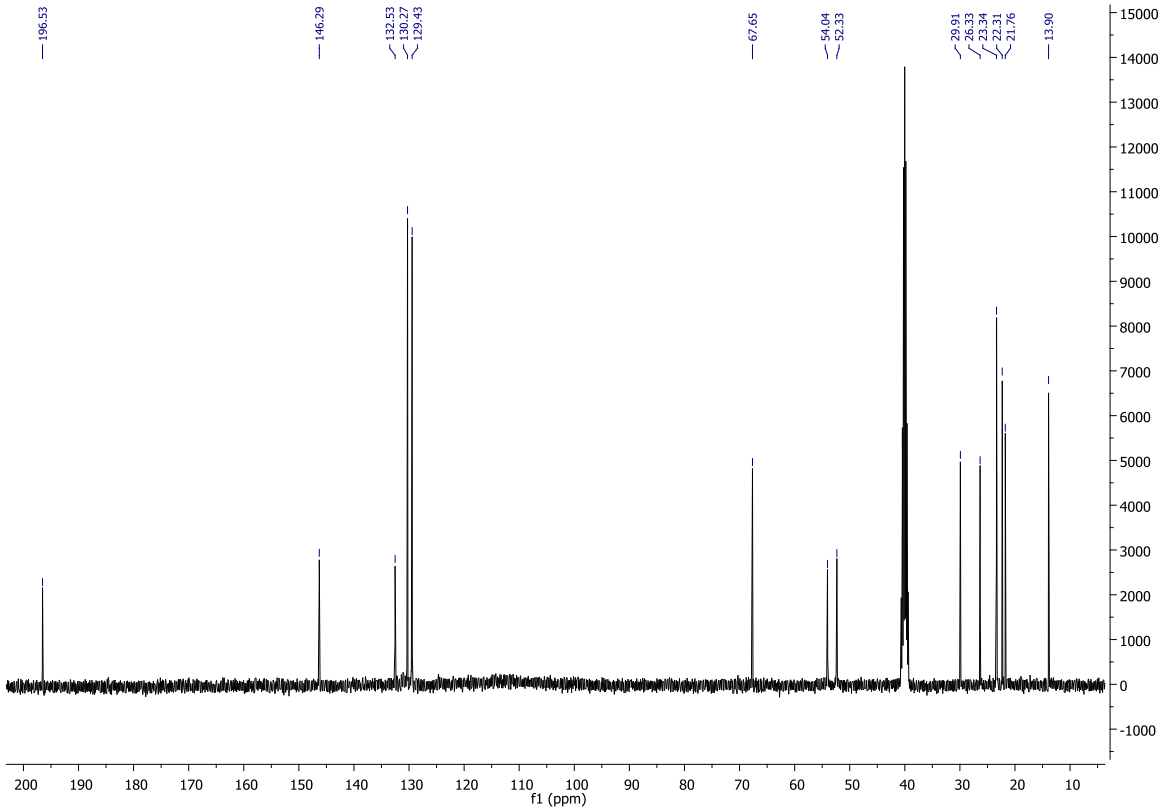

Figure S8 ^{13}C nuclear magnetic resonance spectrum of compound **1** (MPHP)

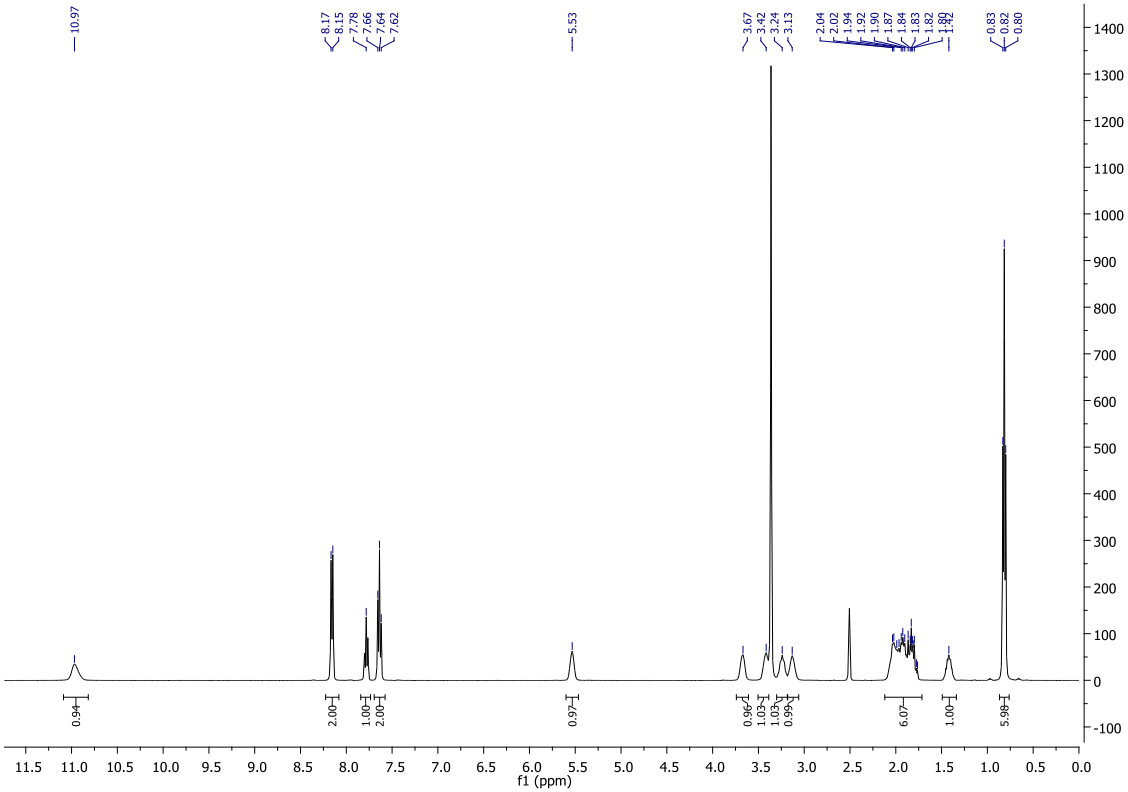

Figure S9 ^{1}H nuclear magnetic resonance spectrum of compound **2** (α-PiHP)

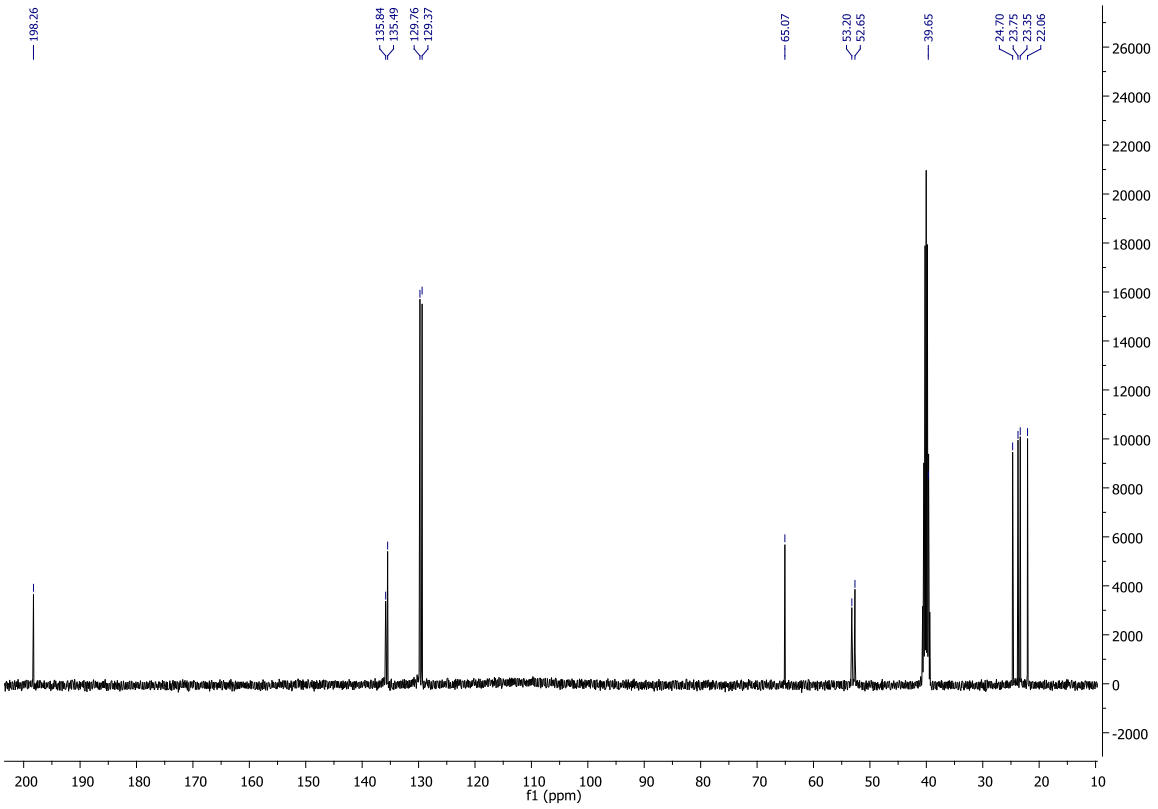

Figure S10 ^{13}C nuclear magnetic resonance spectrum of compound **1** (α-PiHP)

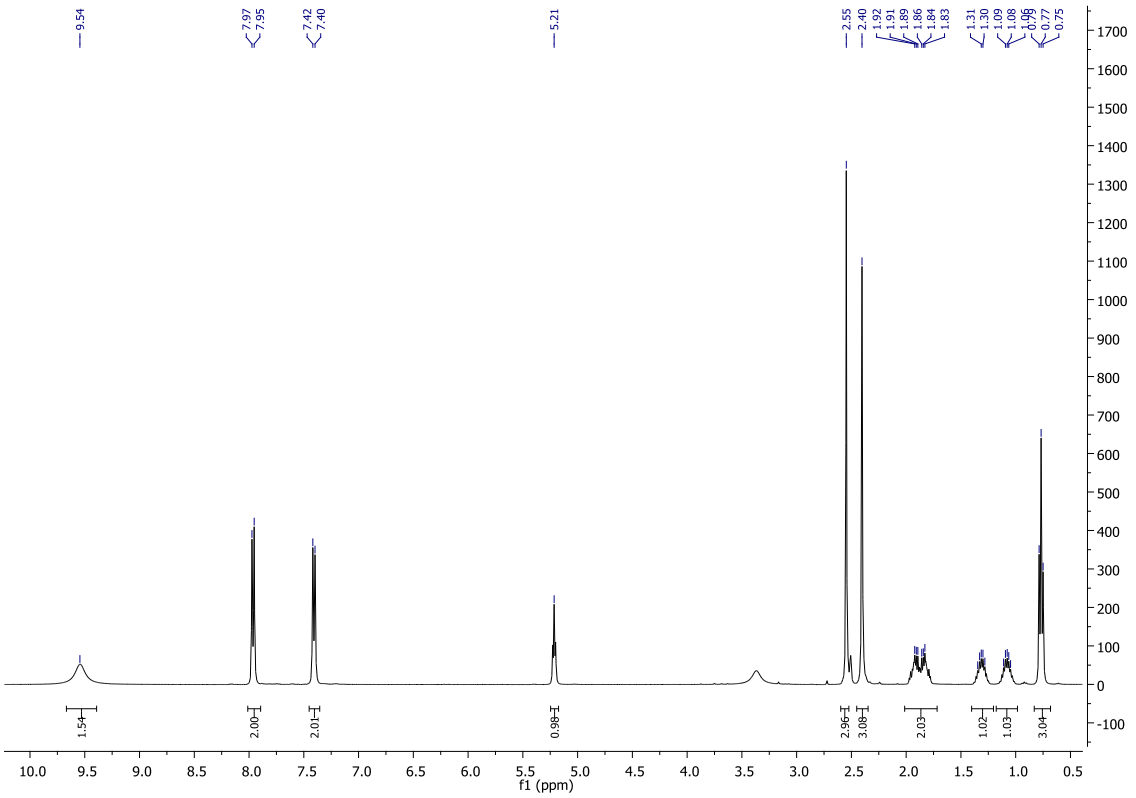

Figure S11 ^{1}H nuclear magnetic resonance spectrum of compound **3** (4-MPD)

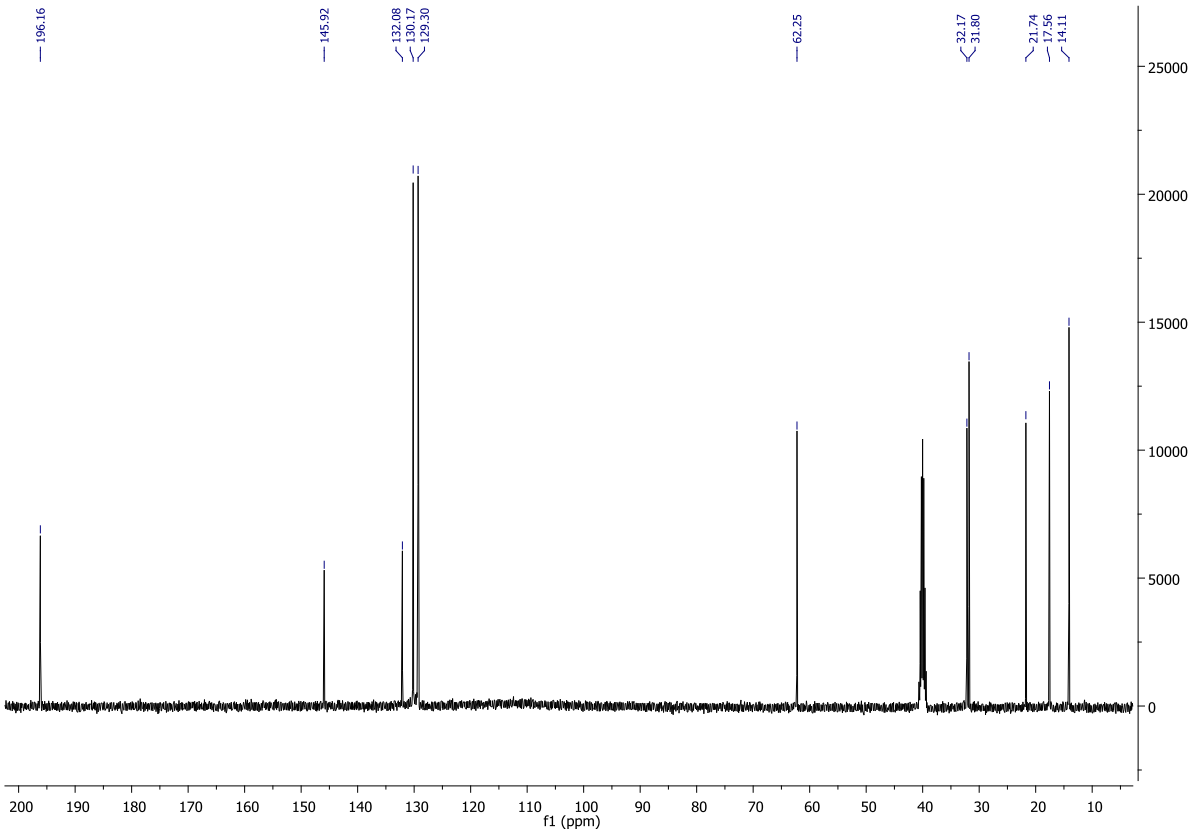

Figure S12 ^{13}C nuclear magnetic resonance spectrum of compound **3** (4-MPD)

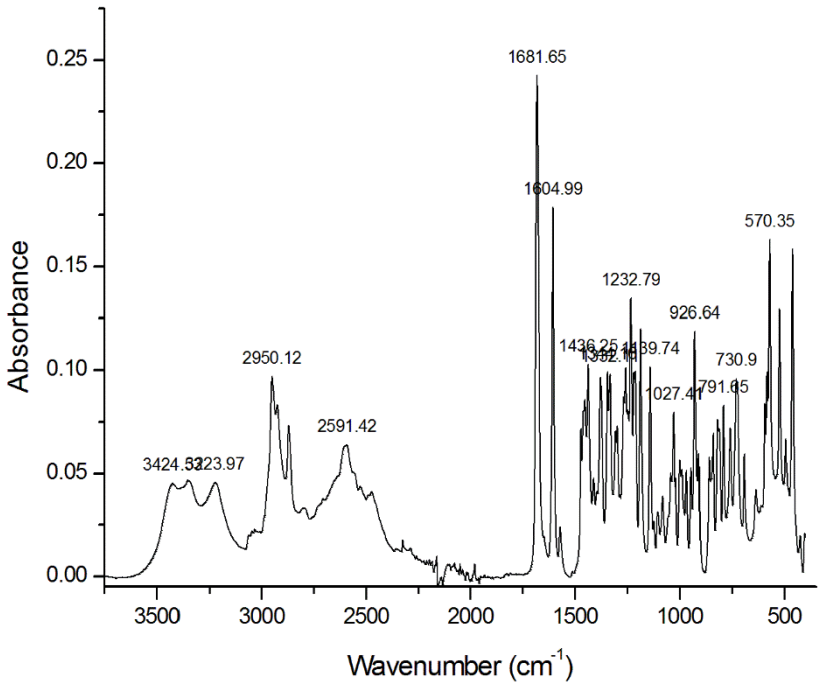

Figure S13 IR spectrum of compound **1** (MPHP)

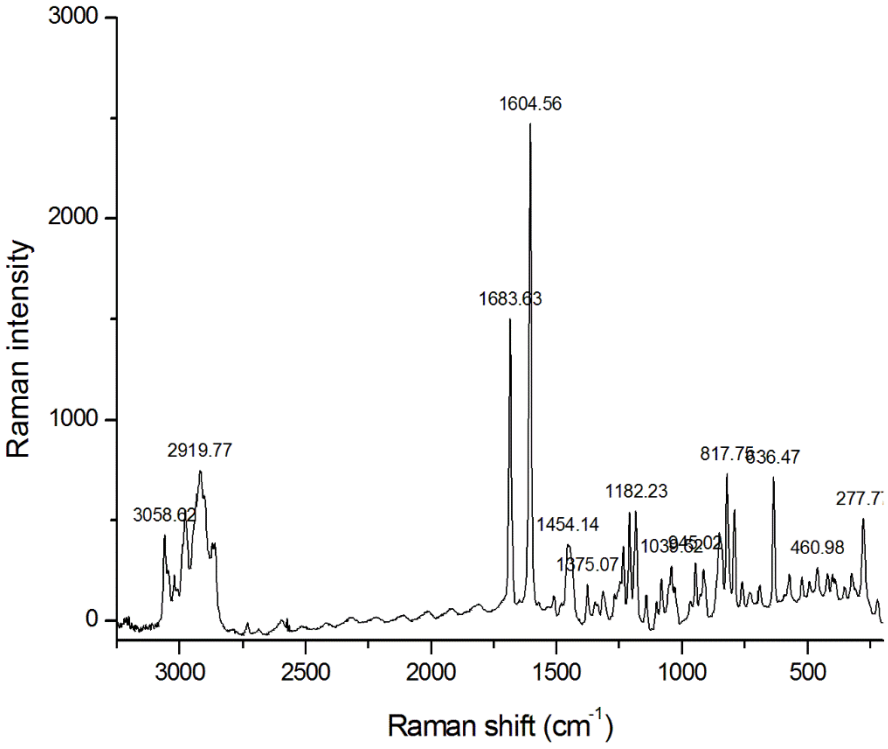

Figure S14 Raman spectrum of compound **1** (MPHP)

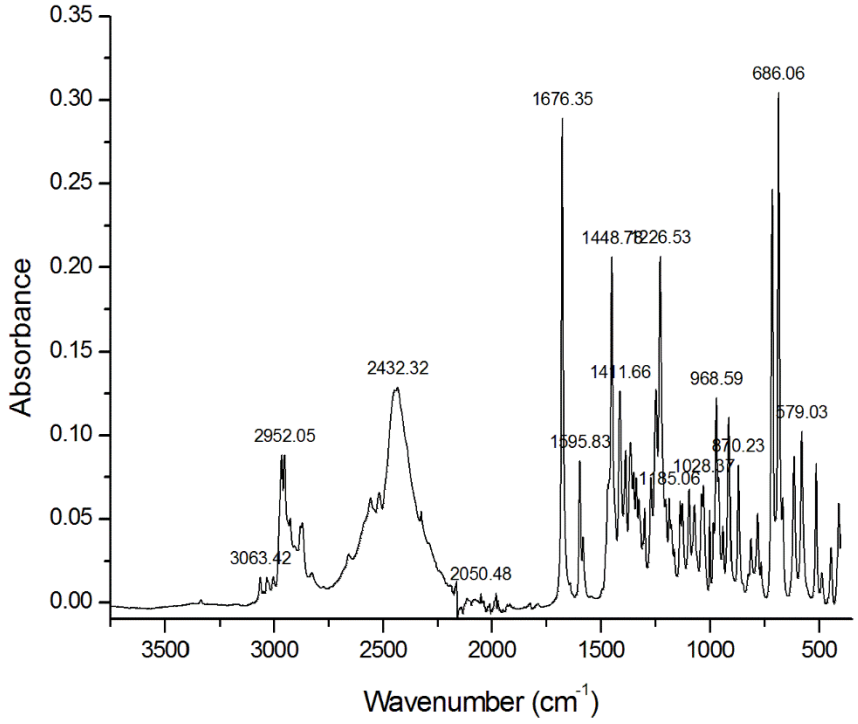

Figure S15 IR spectrum of compound **2** (α-PiHP)

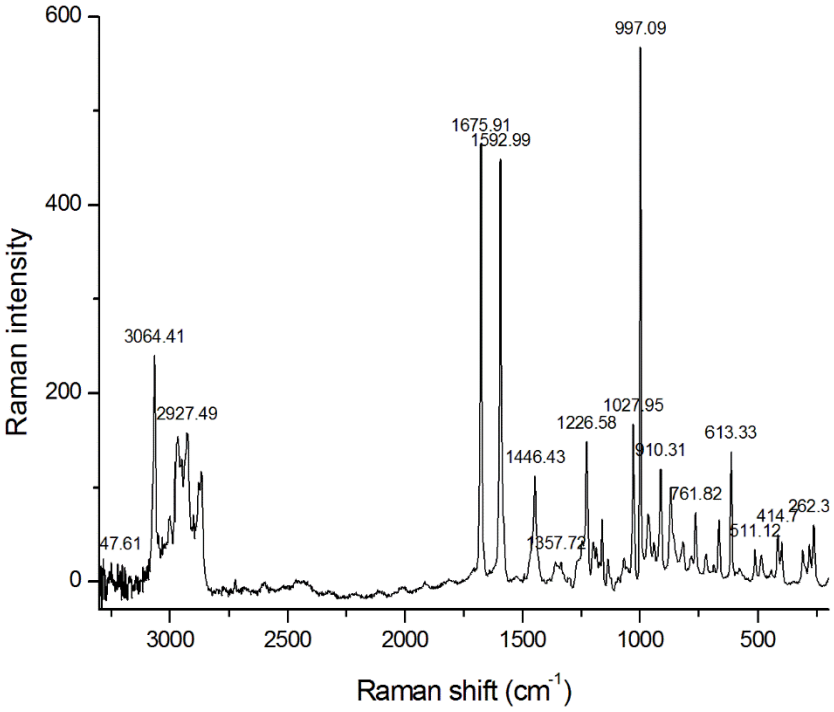

Figure S16 Raman spectrum of compound **2** (α-PiHP)

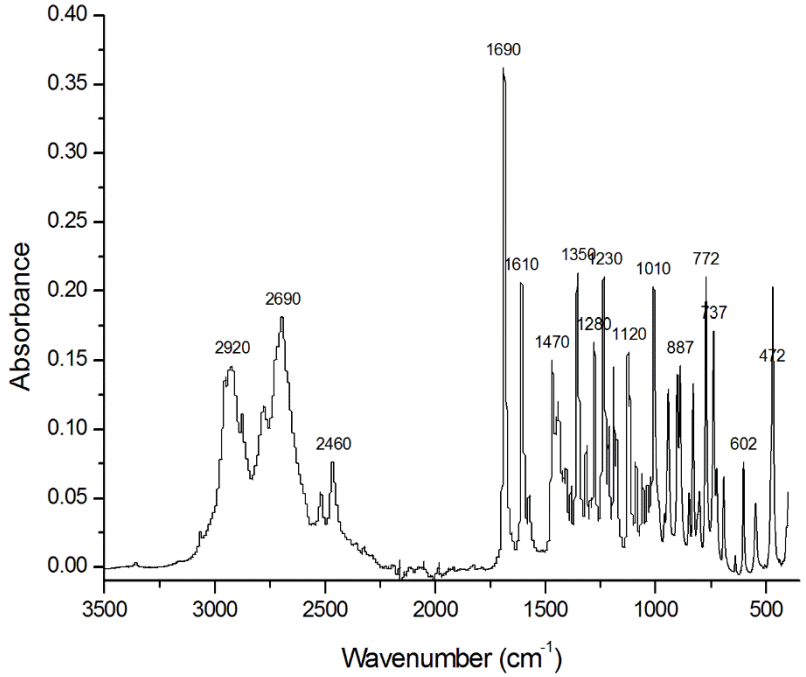

Figure S17 IR spectrum of compound **3** (4-MPD)

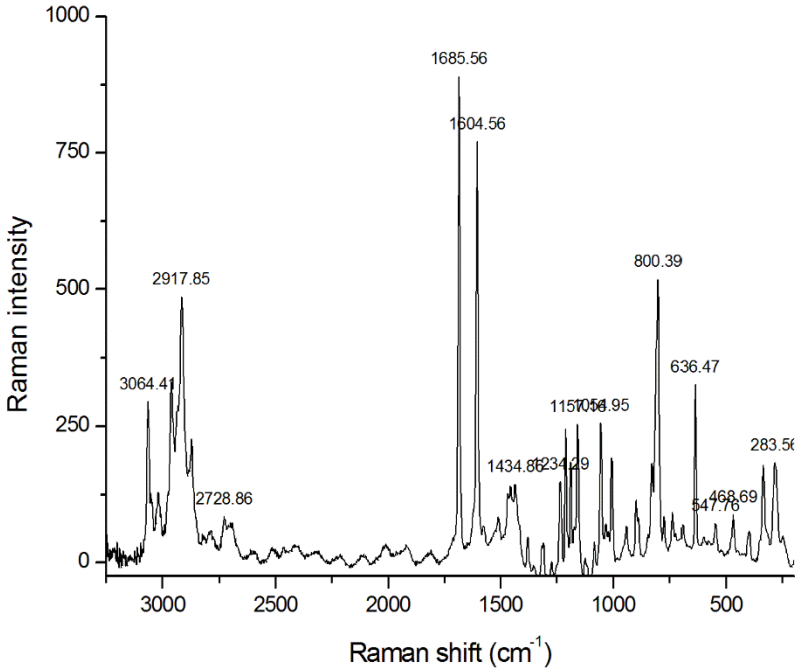

Figure S18 Raman spectrum of compound **3** (4-MPD)

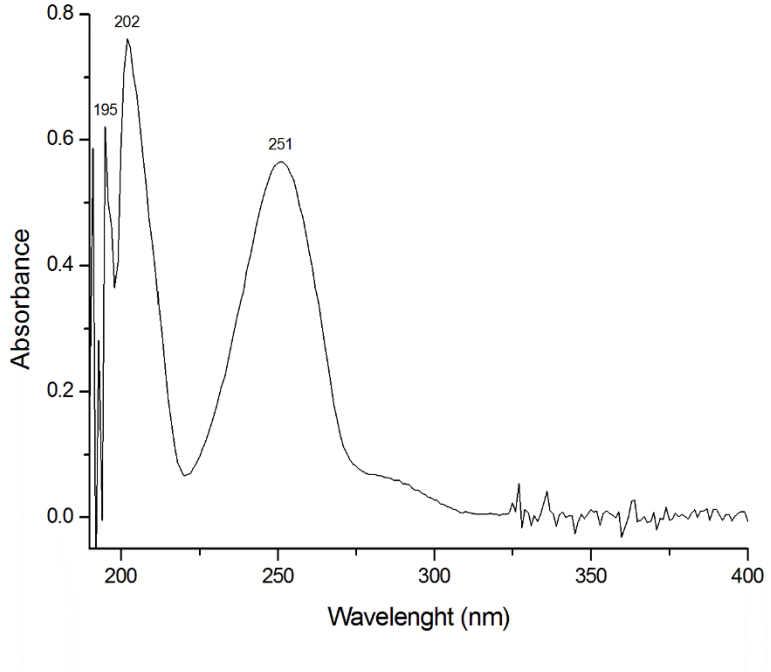

Figure S19 UV-VIS spectrum of compound **1** (MPHP)

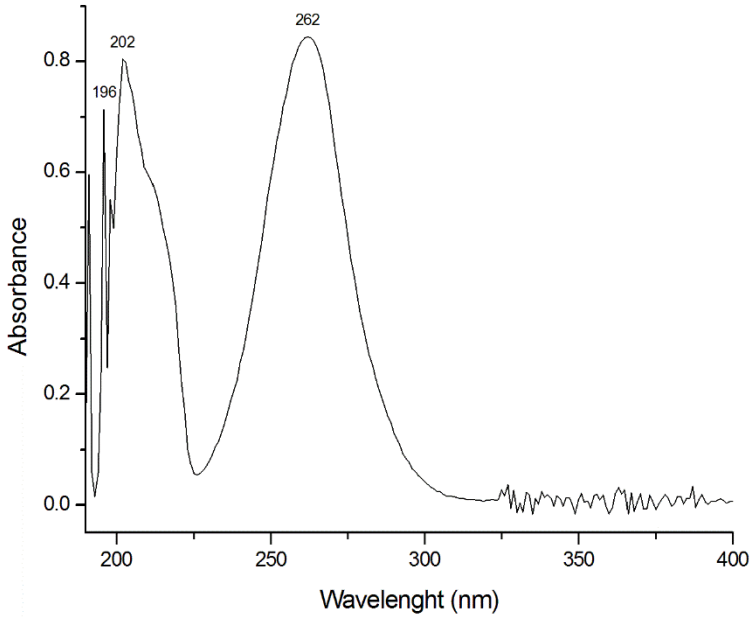

Figure S20 UV-VIS spectrum of compound **2** (α-PiHP)

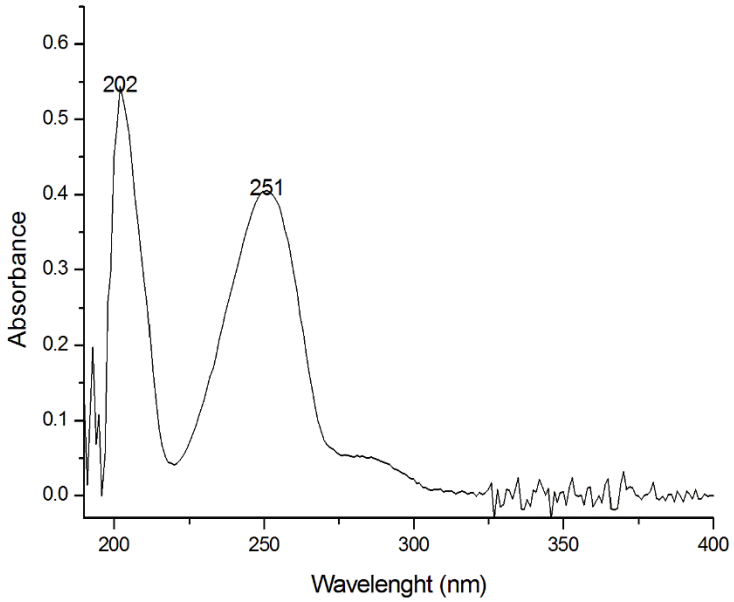

Figure S21 UV-VIS spectrum of compound **3** (4-MPD)

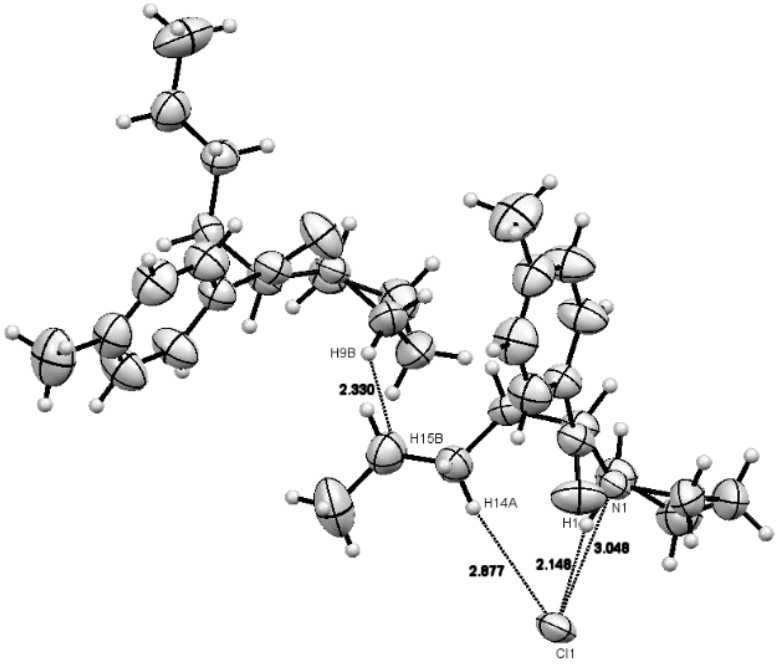

Figure S22 Intramolecular and intermolecular short distances in the structure of compound **1** (MPHP)

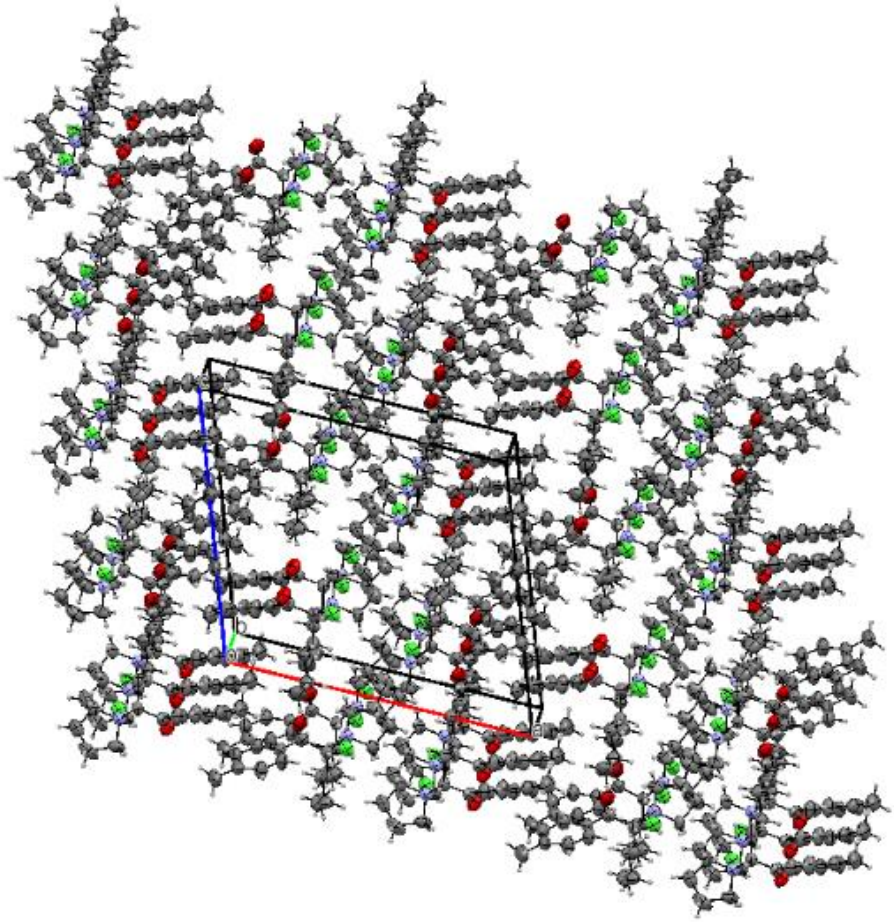

Figure S23 Expanded packing diagram of molecule **1** (MPHP)

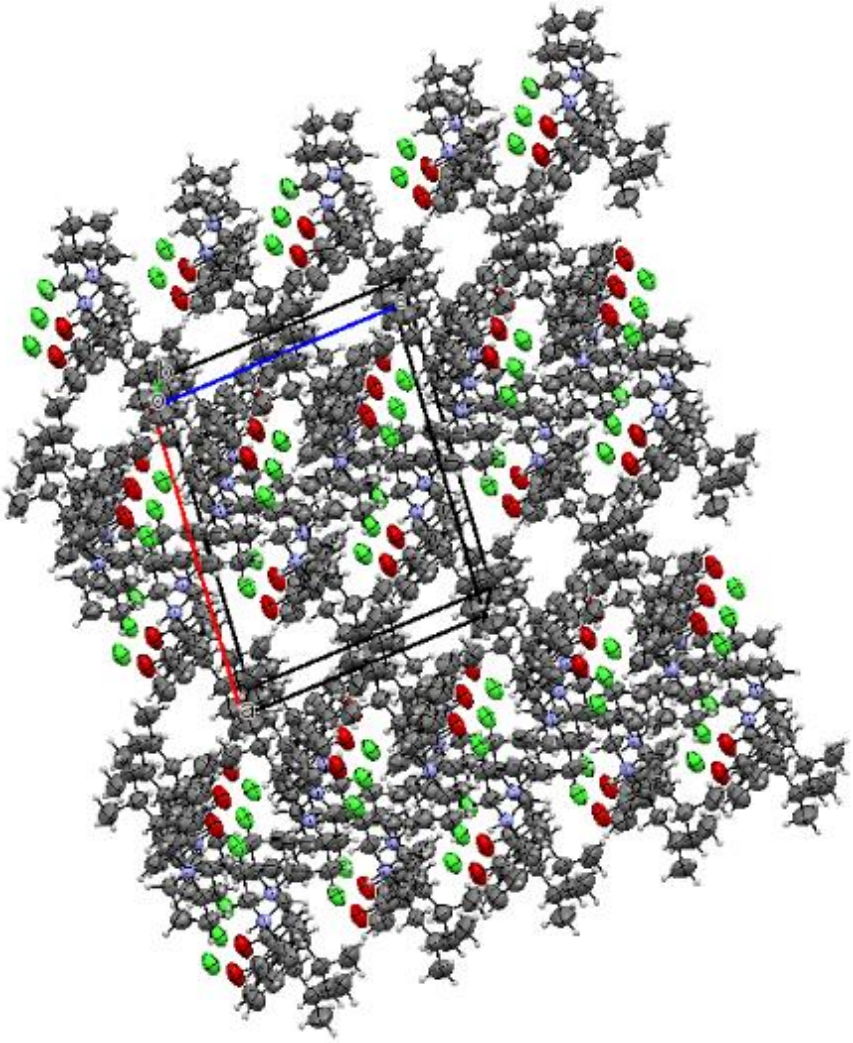

Figure S24 Expanded packing diagram of molecule **2** (α-PiHP)

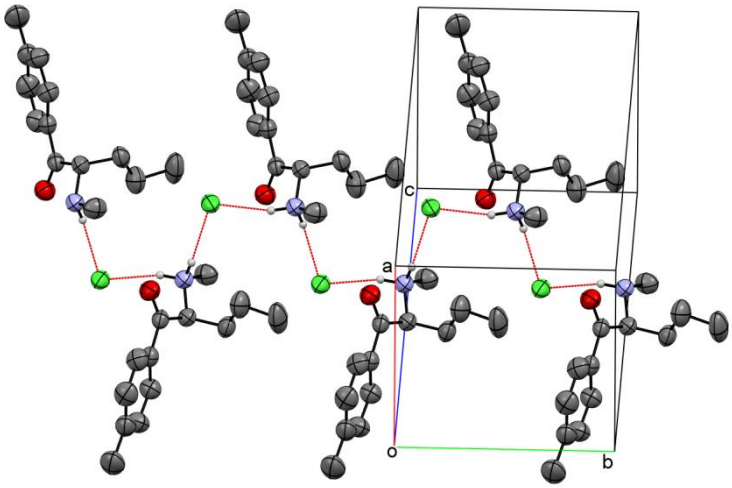

Figure S25 Fragment of the hydrogen bonds formed between chloride ions and ammonium ions creating chains of the molecules of compound **3** (4-MPD). Hydrogen atoms which not participate in these intermolecular interactions were omitted for clarity.