Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supplementary information

Synthesis and characterization of a new acid molten salt and the study of its thermal behavior and catalytic activity in Fischer esterification

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Materials and methods

The chemicals, reagents, and solvents were RA grade, ACS grade, or ReagentPlus® and purchased from Sigma Aldrich, Merck, Alfa Aesar, and Friendemann schmidt chemicals, and used as purchased. The FTIR spectra were recorded as neat films using the Perkin Elmer RX1FT-IR Spectrophotometer in the range of 4000-450 cm−1. Bruker Avance 600 MHz instrument was used to record the ^{1}H and ^{13}C NMR spectra of samples using a deuterated solvent, and all chemical shifts were quoted in ppm relative to TMS. Relaxation delay and acquisition time was 1.0 sec and 2.7 sec., and 2.0 sec. and 0.9 sec. for ^{1}H NMR and ^{13}C NMR, respectively. Agilent 6560 iFunnel Q-TOF LC-MS instrument was used to determine the mass spectra in positive and negative ESI mode in a mass range of 30-1000 m/z at 2 GHz, at drying gas temperature and flow rate 250 °C and 10 L/min, respectively. Büchi B-545 apparatus was employed to take melting points at a maximum rate of heat rise 0.5-2.0 °C/min in open capillary tubes. The amount of water in the liquid salt was determined by Karl Fisher (KF) titration, using a Metrohm 831 KF coulometer in conditions of ambient humidity and room temperature. The ionic conductivity (σ) was measured using a Mettler Toledo Seven Easy conductivity meter. Differential scanning calorimetry (DSC) curves were obtained with the use of a DSC-Mettler Toledo DSC 822e calorimeter. The measurements were taken in the aluminum pans with a pierced lid under a dry nitrogen gas atmosphere (10 mL min^{-1}). Dynamic scans were performed at a heating rate of 10 °C min^{-1} in three temperature cycles. TGA/DTA curves were recorded with the use of a Mettler Toledo TGA/SDTA 851e. All measurements were taken in the Al_2O_3 crucible under the nitrogen atmosphere (10 mL min^{-1}). Dynamic scans were performed at the heating rate of 10 °C min^{-1} in the temperature range of 25-800 °C.

Figure S1. ^{1}H NMR of piperazine (600 MHz, DMSO-d_6).

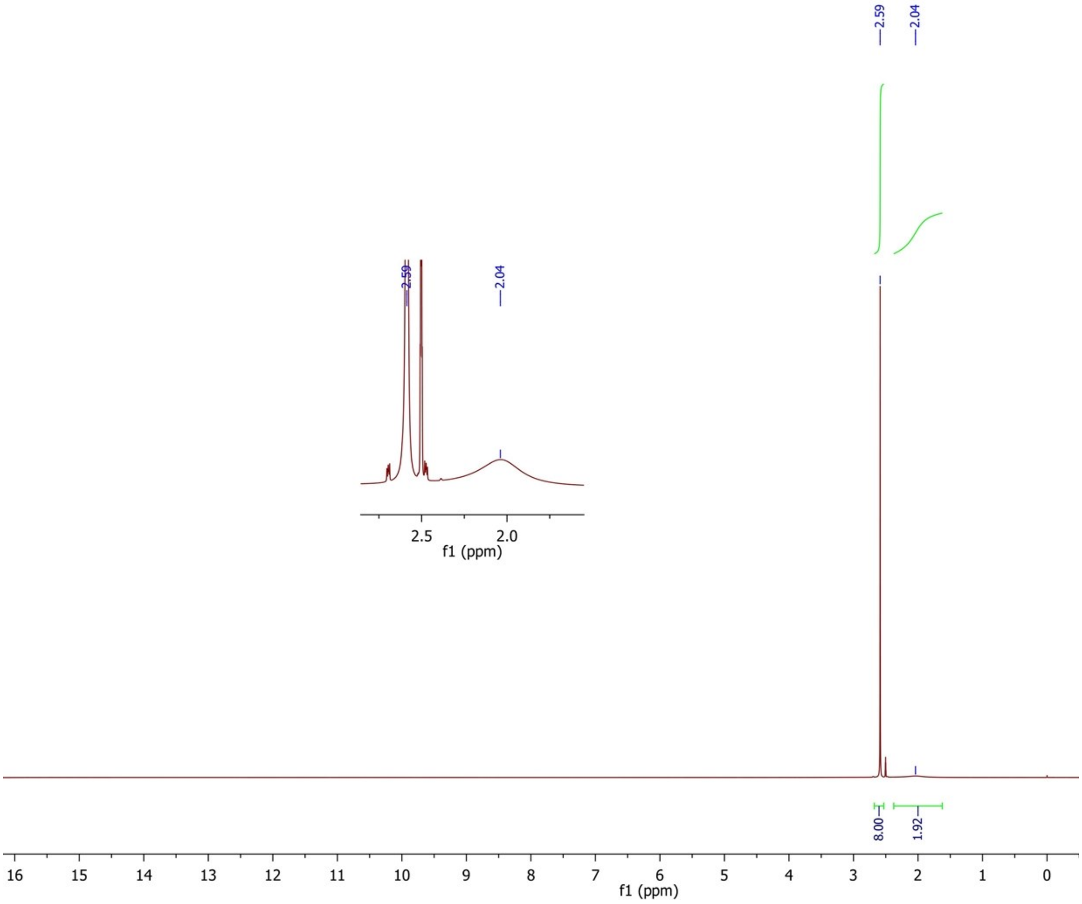


Figure S2. ^{13}C NMR of piperazine (600 MHz, DMSO-d_6).

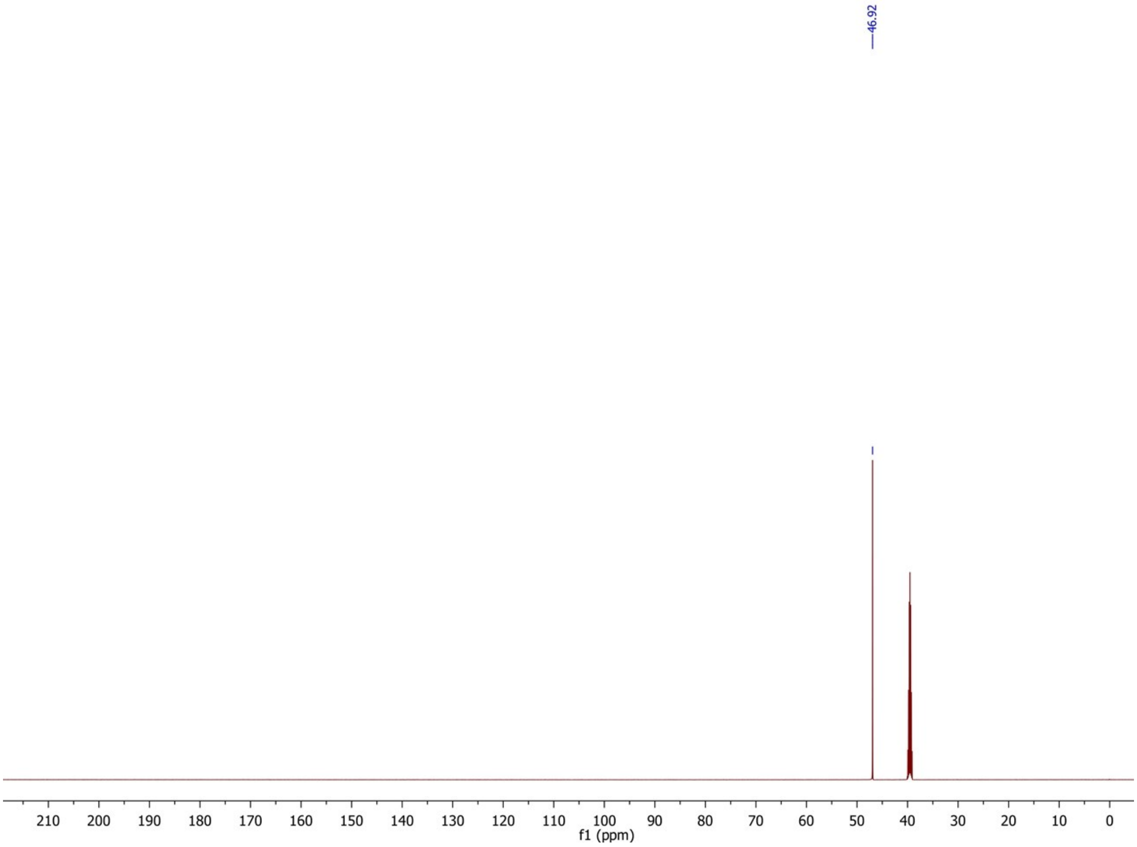


Figure S3. ^{1}H NMR of the PZ-SA(1:2) (600 MHz, DMSO-d_6).

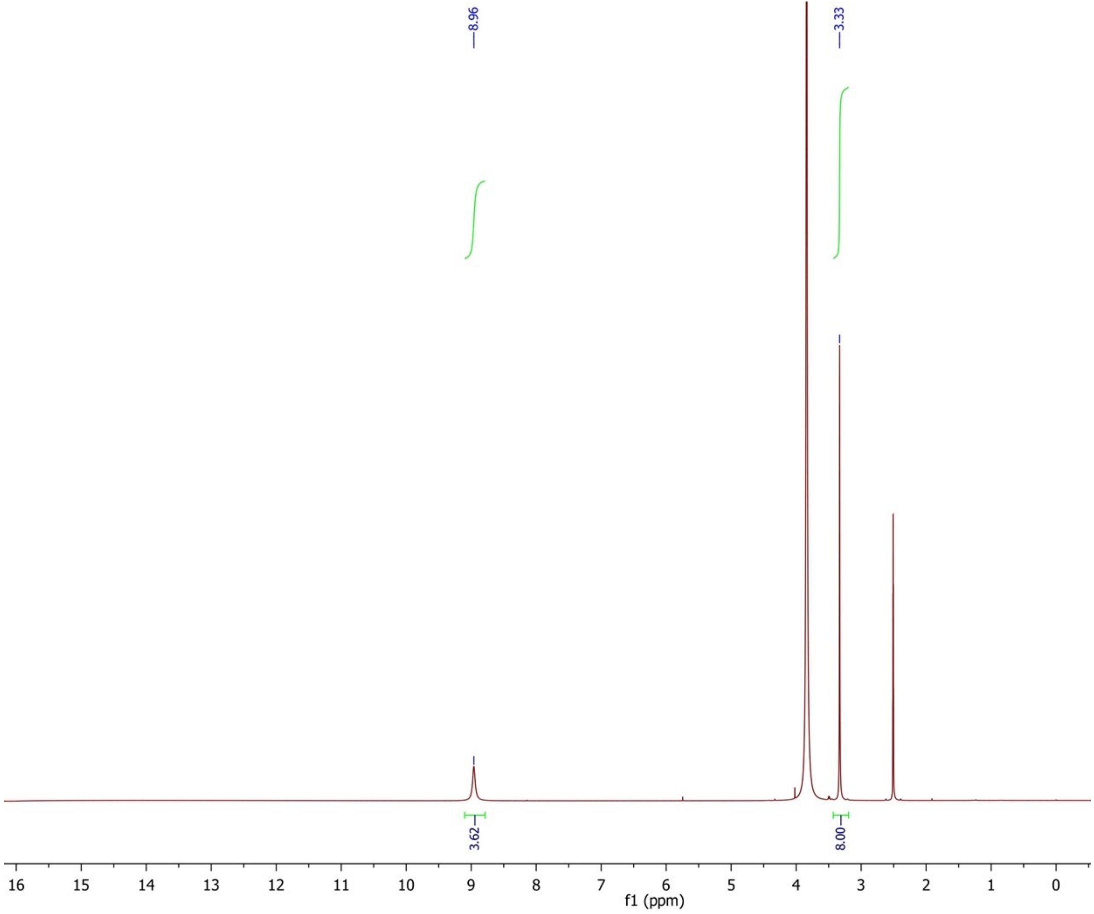


Figure S4. ^{13}C NMR of the PZ-SA(1:2) (600 MHz, DMSO-d_6).

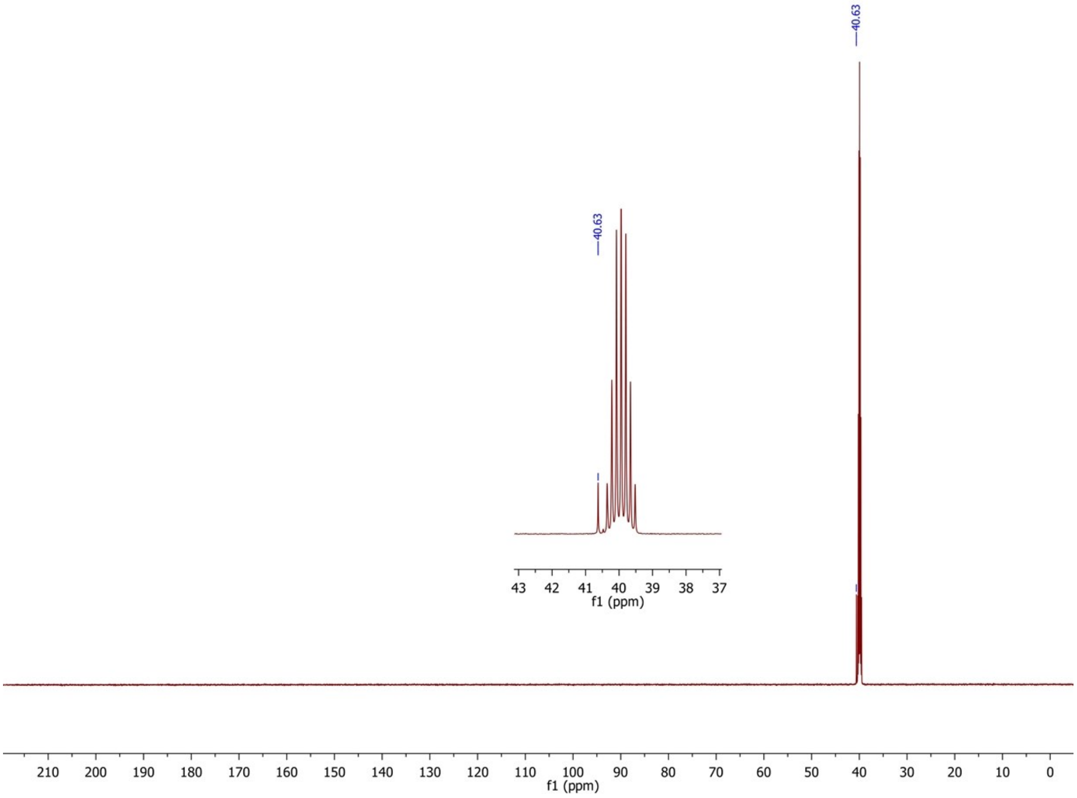


Figure S5. ^{1}H NMR of the PZ-SA(1:2) (600 MHz, D_2O).

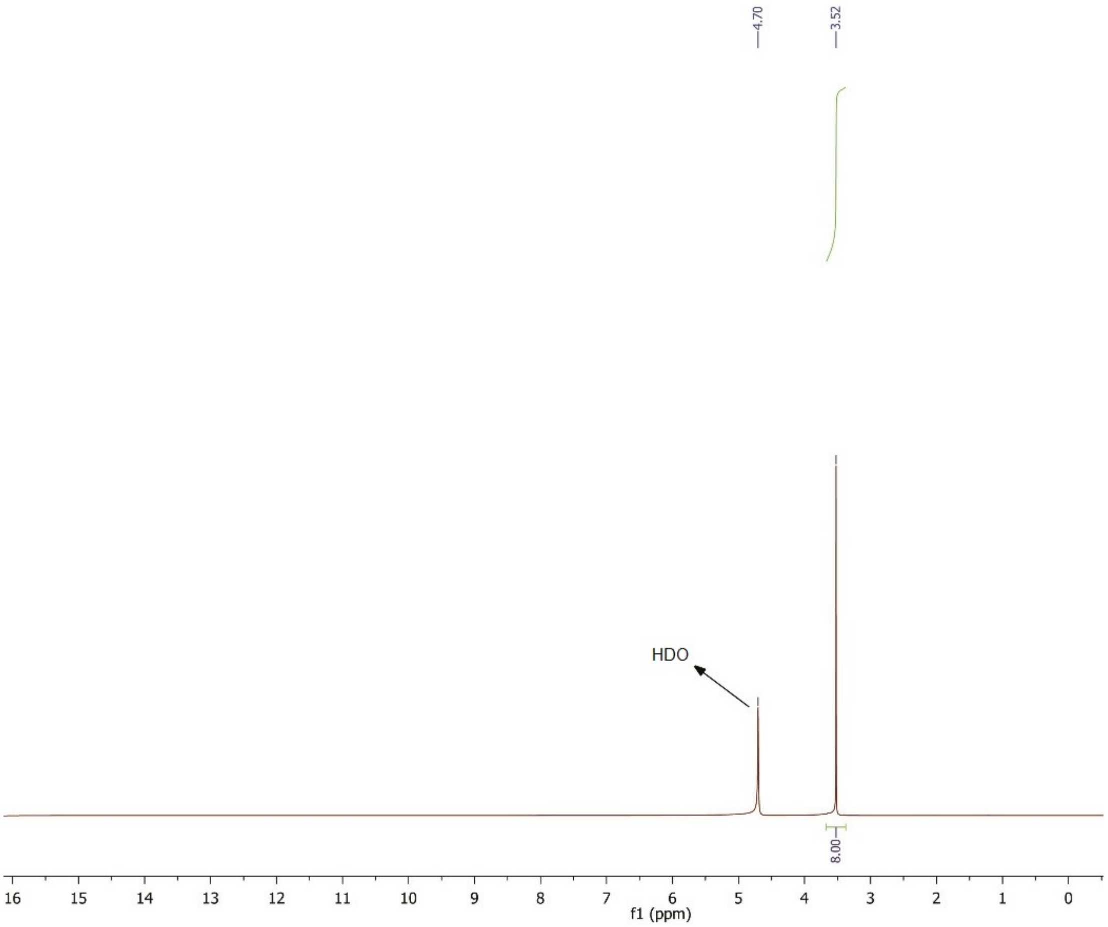


Figure S6. ^{13}C NMR of the PZ-SA(1:2) (150 MHz, D_2O).

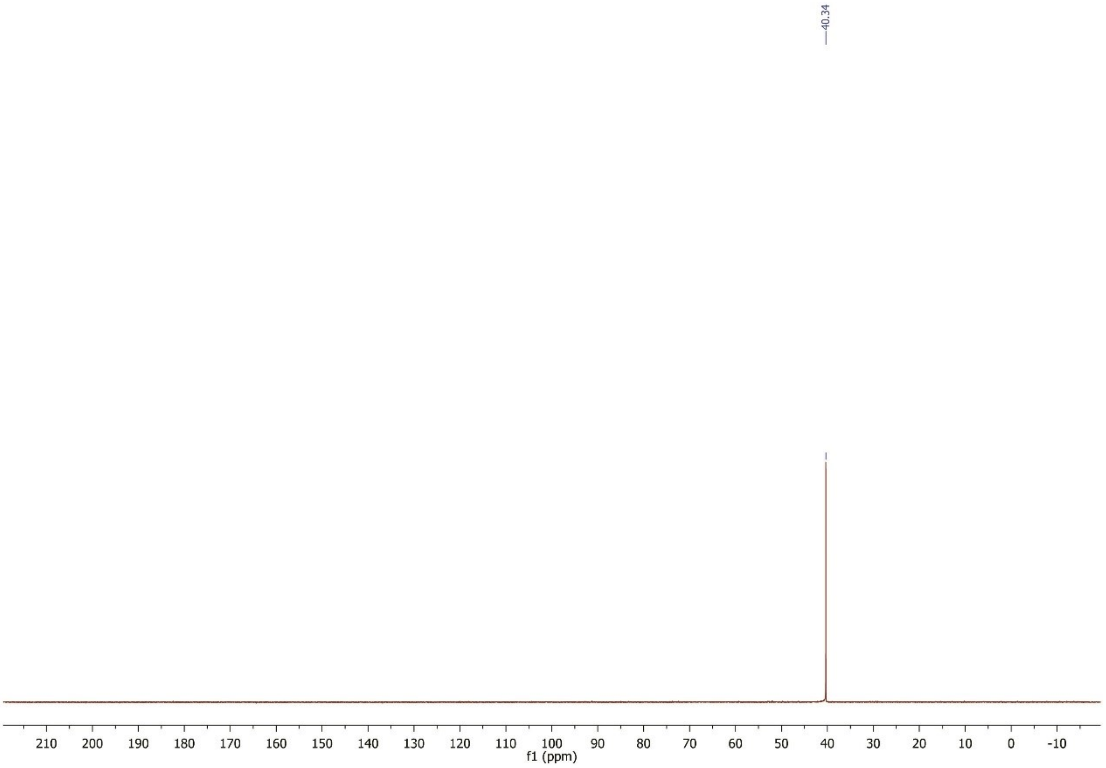


Figure S7. $^{1}H,^{1}H$-COSY of the PZ-SA(1:2) (600 MHz, DMSO-d_6).

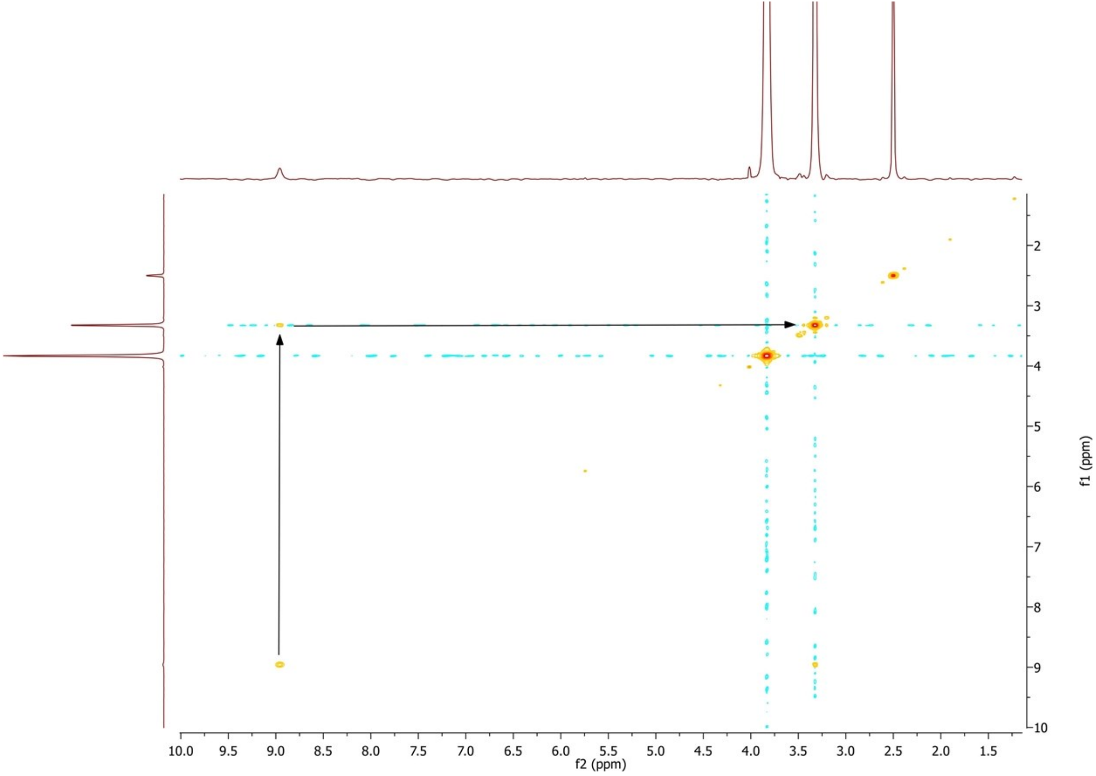
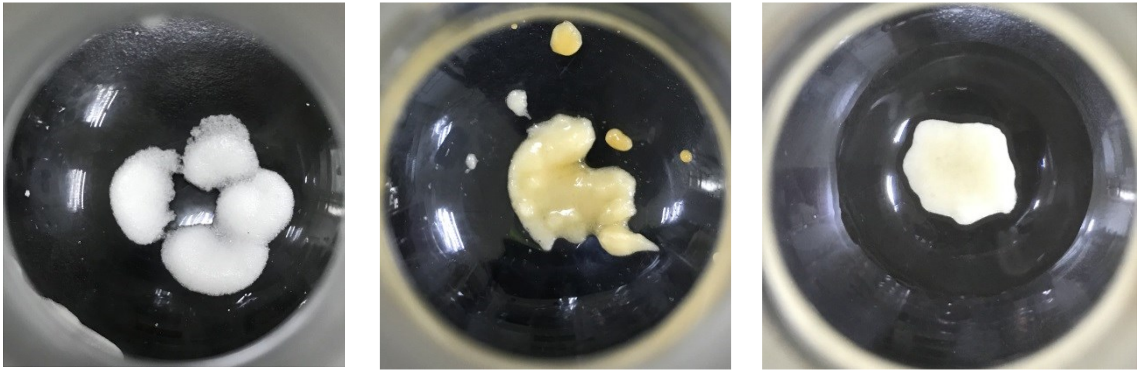


Figure S8. The image of the product $[PZH2]^{2+}$ $2Br^{-}$


After rotary evaporation of solvent at vacuum

After overnight at ambient conditions

After a repeat of the washing with CH_2Cl_2

Figure S9. ^{1}H NMR of the $[PZH_2]^{2+}2Br^{-}$ (600 MHz, DMSO-d_6).

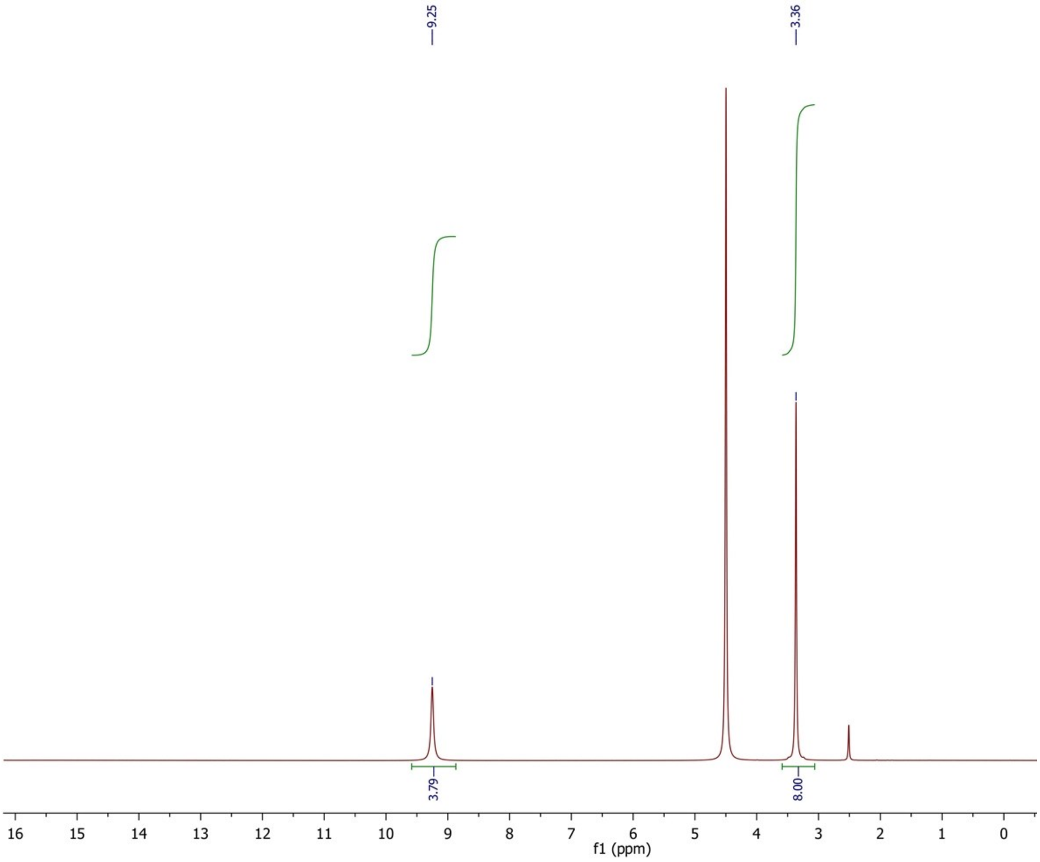


Figure S10. ^{13}C NMR of the $[PZH_2]^{2+}2Br^{-}$ (150 MHz, DMSO-d_6).

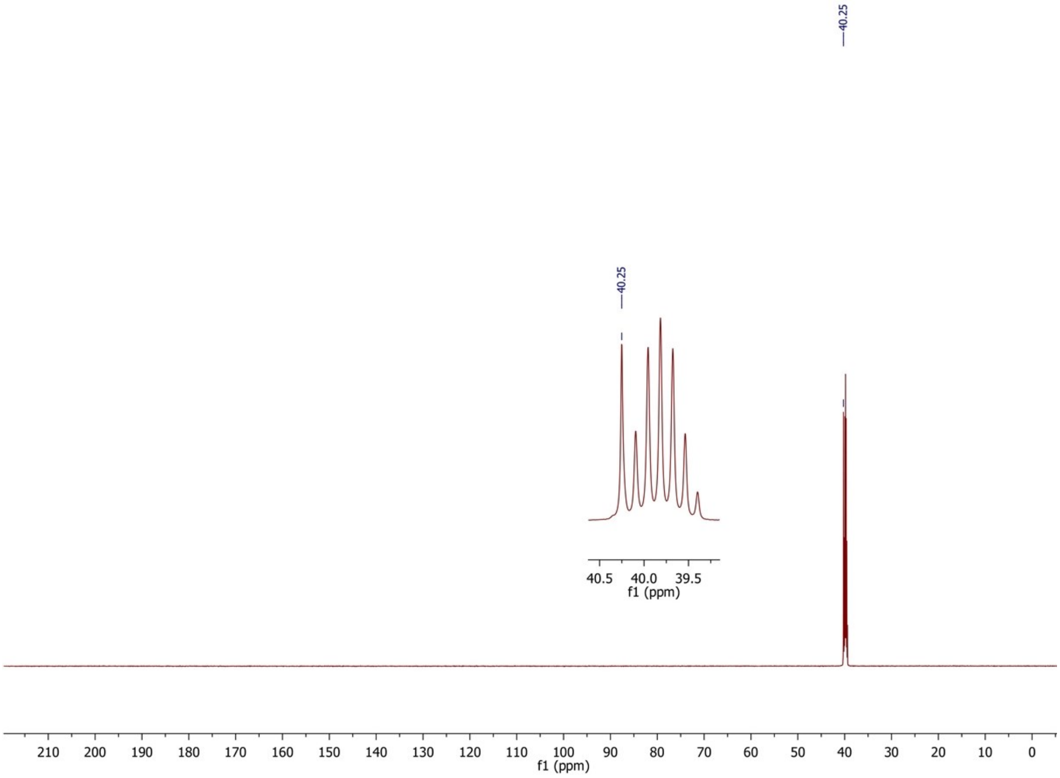


Figure S11. $^1H,^1H$-COSY of the $[PZH_2]^{2+}2Br^-$ (600 MHz, DMSO-d_6).

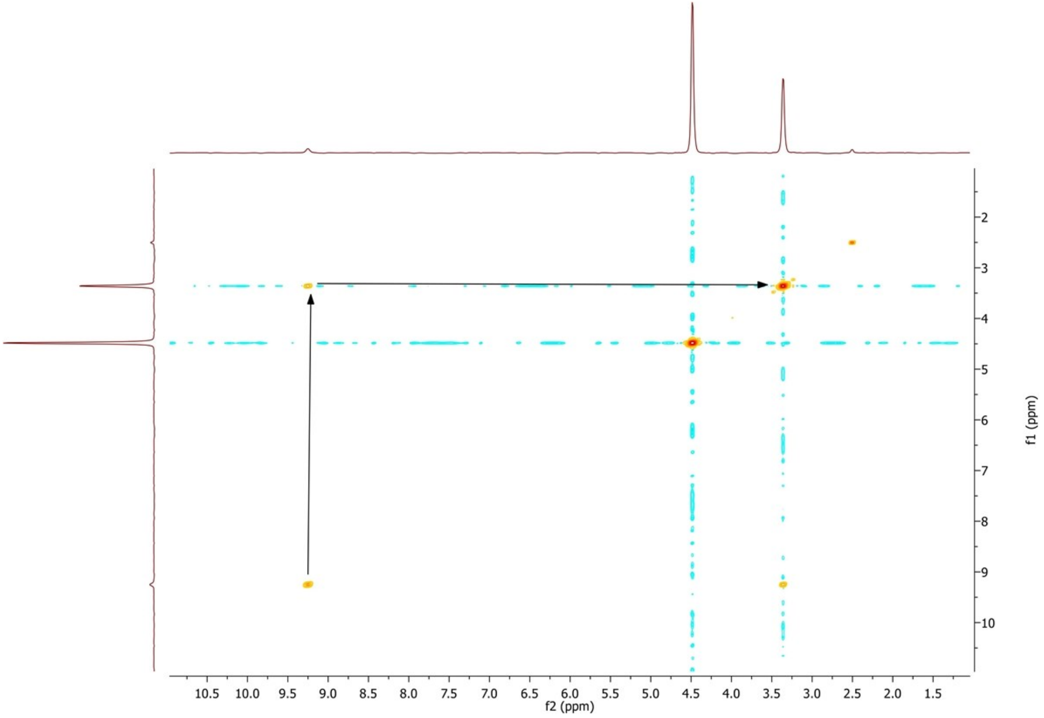


Figure S12. ^1H NMR of SA (98%) (600 MHz, DMSO-d_6).

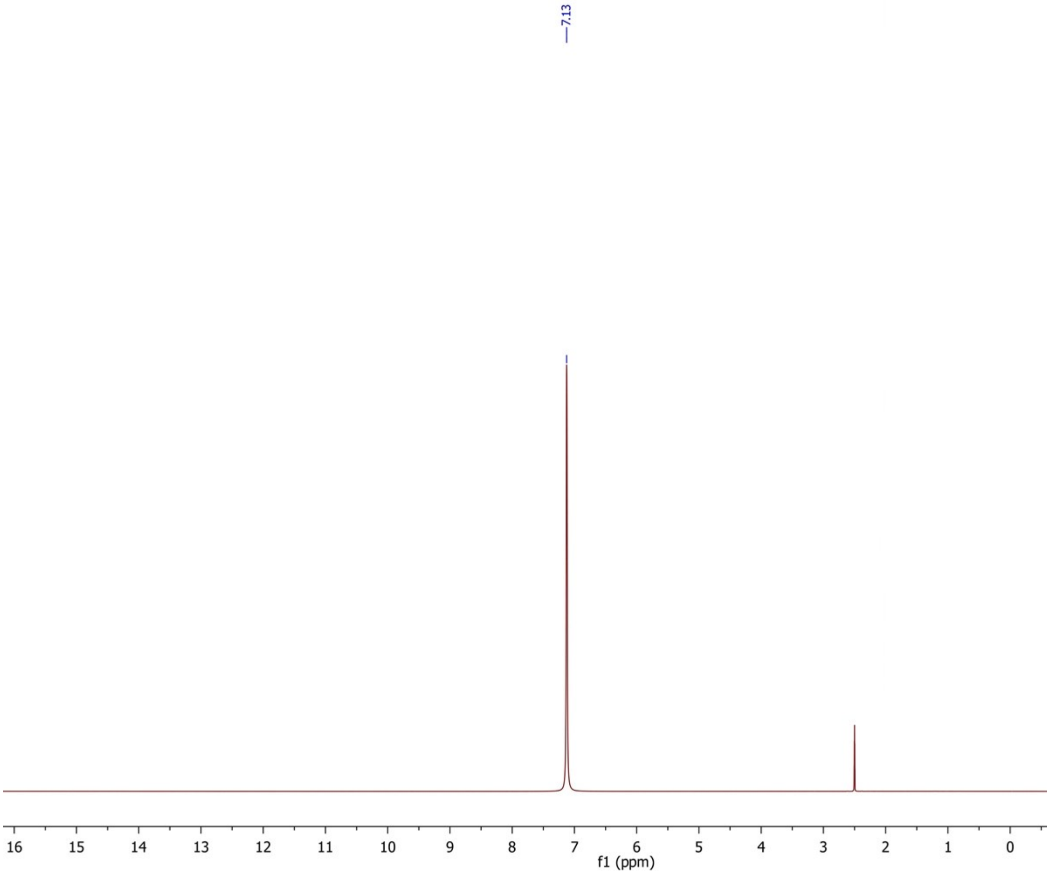


Figure S13. ^{1}H NMR of PZ-SA(1:2) containing 1.0 mmol excess of SA (98%) (600 MHz, DMSO-d_6).

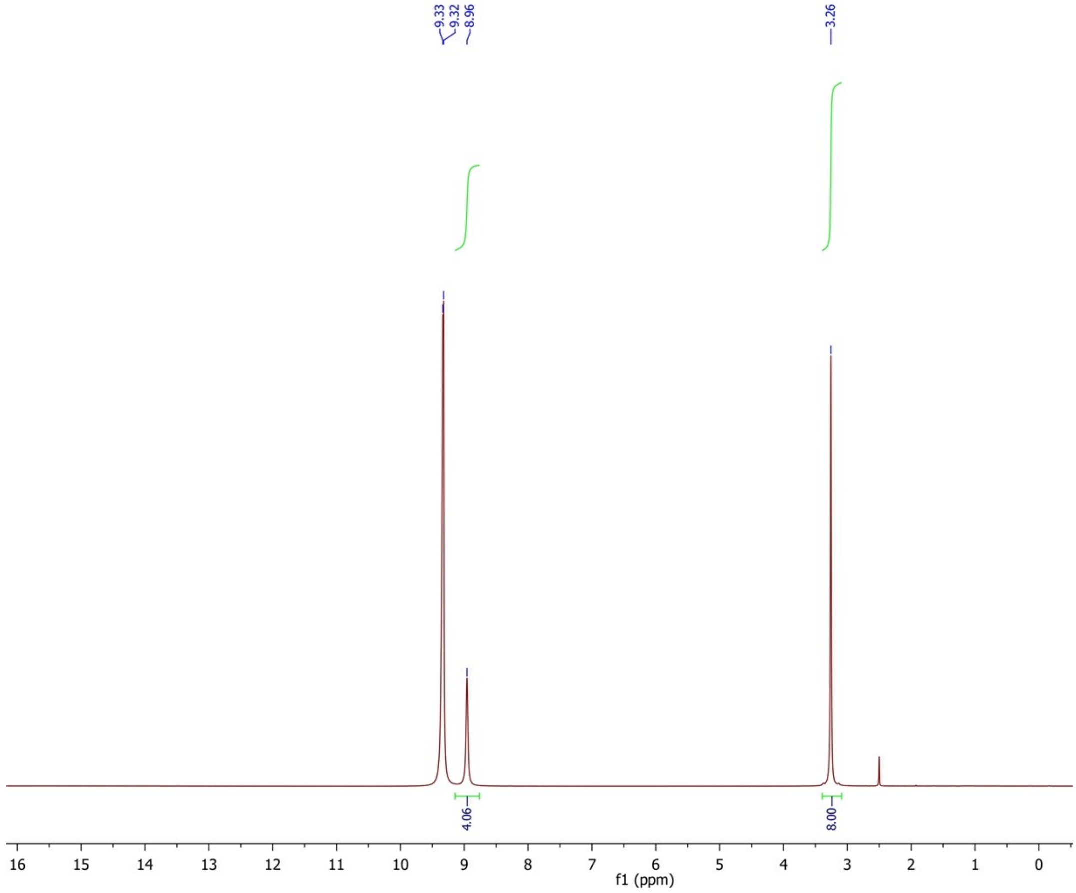


Figure S14. Titrimetric analysis of PZ-SA(1:2).

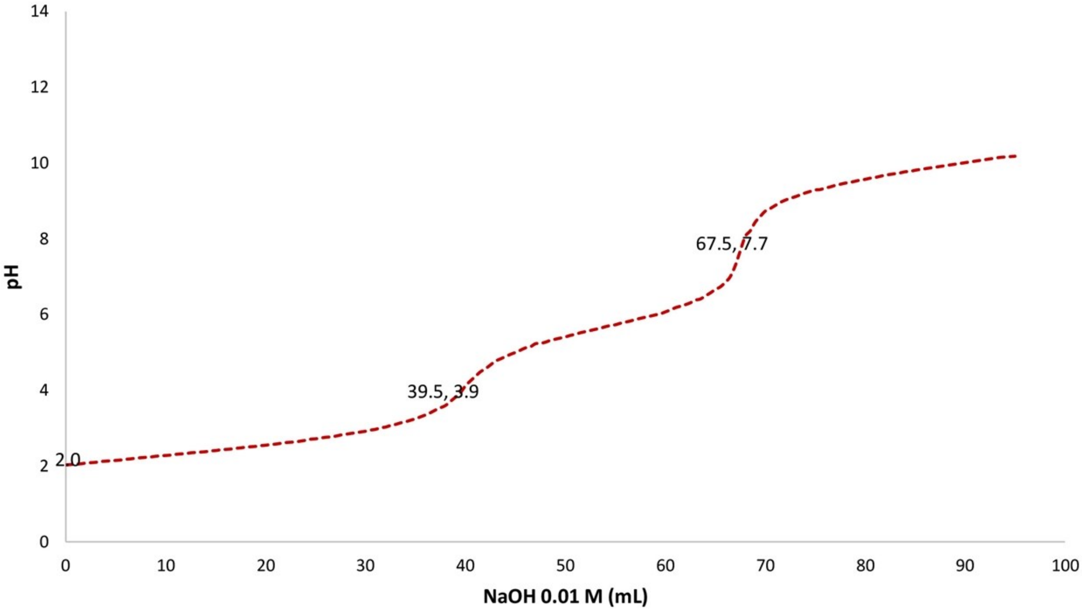


Figure S15. ^{1}H NMR of crude upper phase after four hours (600 MHz, DMSO-d_6).

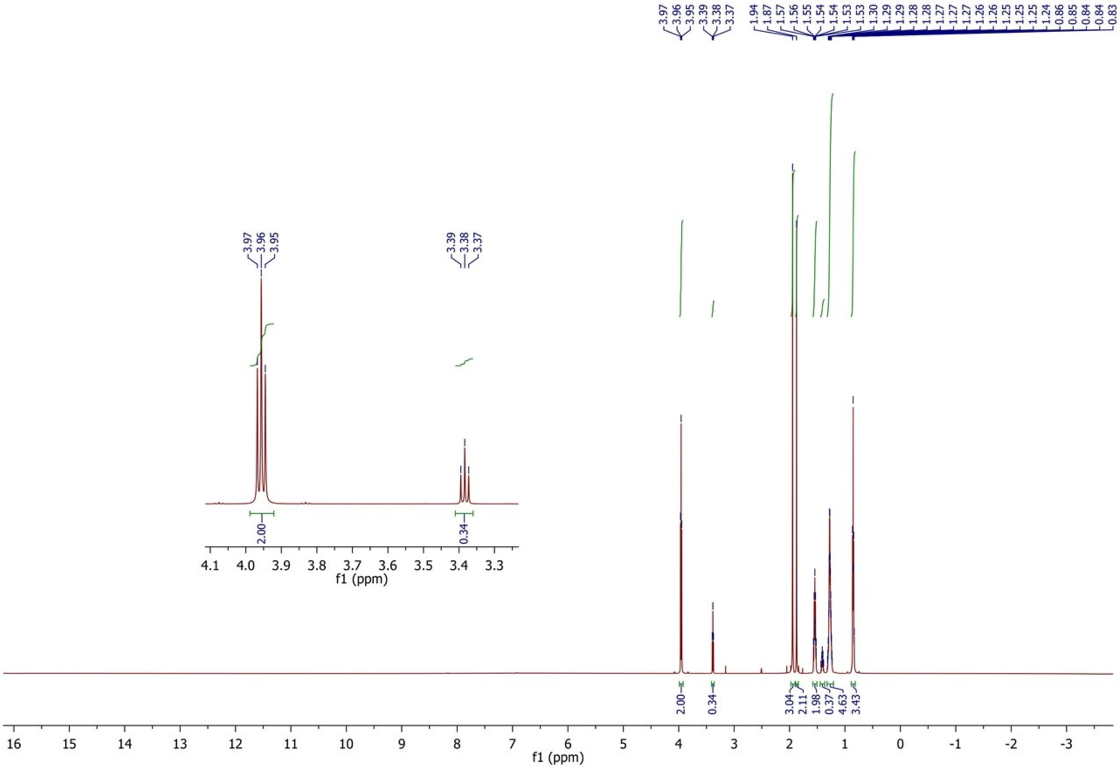


Figure S16. ^{1}H NMR of pure pentyl acetate after simple workup (600 MHz, DMSO-d_6).