Supplementary Information (SI) for Green Chemistry.

Electronic Supplementary Information

Effect of phase transfer catalyst structure on the alkaline hydrolysis of poly(ethylene terephthalate)

Lee B. Anderson,[a] Conall Molloy,[a] Lorenzo Pedrini,[a] Ian Martin,[a] and Stephen J. Connon*[a]

[a] *School of Chemistry, Trinity Biomedical Sciences Institute, Trinity College Dublin, 152-160*
Pearse St., Dublin 2, Ireland

Table of Contents

1.0 General
2.0 Basic hydrolysis of PET

Terephthalic acid characterisation

3.0 Catalyst synthesis and characterisation

4.0 NMR Spectra of catalysts and products

5.0 References

1.0 General

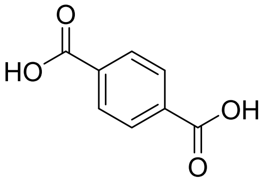
Proton Nuclear Magnetic Resonance (NMR) spectra were recorded on Bruker DPX 400 MHz and Bruker Avance II 600MHz spectrometers, using $CDCl_3$ and DMSO-d_6 as solvents, and referenced relative to residual $CHCl_3$ ($\delta = 7.26$ ppm) or DMSO ($\delta = 2.50$ ppm). Carbon NMR spectra were recorded on the same instruments (101 MHz and 151 MHz respectively) with total proton decoupling. Phosphorus NMR spectra were recorded on the Bruker DPX400 machine (162 MHz). Infrared spectra were obtained on a Perkin Elmer Spectrum 100 FT-IR spectrometer equipped with a universal ATR sampling accessory. ESI mass spectra were acquired using a Waters Micromass LCT- time of flight mass spectrometer (TOF), interfaced to a Waters 2690 HPLC. The instrument was operated in either positive or negative mode as required. APCI experiments were carried out on a Bruker microTOF-Q III spectrometer interfaced to a Dionex UltiMate 3000 LC or direct insertion probe. Agilent tuning mix APCI-TOF was used to calibrate the system. Hydrolysis experiments were carried out with Radleys Carousel 12 Plus Reaction Station. HPLC grade water was purchased from Sigma-Aldrich and used as the reaction medium for the basic hydrolysis of PET. Colourless polyethylene terephthalate bottles were purchased from a local large supermarket chain and cut into flakes (*ca*. 5 mm square). Unless otherwise noted, all commercially available compounds were used as provided, without any further purification.

2.0 Basic PET hydrolysis procedure

An oven dried carousel flask was charged with water (5 mL), sodium hydroxide (0.500 g) and catalyst (5 mol %).[a] The flask was placed under an atmosphere of nitrogen (balloon), and the reaction was stirred for 20 min at 90 °C. Polyethylene terephthalate flakes (*ca*. 5 mm squares, 0.500 g) were added and the mixture was stirred (500 RPM) for 3 h. The flask was removed from the carousel and cooled in an ice-bath for 10 min. The reaction mixture was then filtered, and the residue was washed with water (5 mL x 2). The filtrate was adjusted, with cooling, to pH 2-3 using dilute HCl and the resulting precipitate was filtered. The solids were washed with water (5 mL) and the product was dried for 4 h in a vacuum oven at 60 ˚C. The yield of terephthalic acid was determined by ^{1}H NMR spectroscopy using 4-iodoanisole as an internal standard: known masses of product (> 15 mg) and 4-iodoanisole (> 15 mg) were weighed into a sample vial and dissolved in DMSO-d_6. The integrals corresponding to both TPA and isophthalic acid (a common additive utilised in PET production) were used to determine yield.

[a] Mol% of catalyst is related to the number of moles of monomeric units (2.6 mmol) present in 0.500 g of polymer.

terephthalic acid (TPA)



An oven dried carousel flask was charged with water (2.5 mL), sodium hydroxide (0.500 g, 12.50 mmol) and catalyst **17** (0.0084 g, 0.026 mmol, 1 mol%).[a] The flask was placed under an atmosphere of nitrogen (balloon), and the reaction was stirred for 20 min at 90 °C. Polyethylene terephthalate flakes (*ca.* 5 mm squares, 0.500 g) were added and the mixture was stirred (500 RPM) for 3 h. The flask was removed from the carousel and cooled in an ice-bath for 10 min. The reaction mixture was then filtered, and the residue was washed with water (5 mL x 2). The filtrate was adjusted, with cooling, to pH 2-3 using dilute HCl and the resulting precipitate was filtered. The solids were washed with water (5 mL x 2) and the product was dried for 4 h in a vacuum oven at 60 ˚C. TPA was isolated as a white solid (0.43 g, 99%). M.p. 299-301 °C (lit. M.p. 300 °C)[1]

[a] Mol% of catalyst is related to the number of moles of monomeric units (2.6 mmol) present in 0.500 g of polymer.

δ_H (400 MHz, DMSO-d_6): 13.29 (br s, 2H), 8.04 (s, 4H) ppm.

δ_C (101 MHz, DMSO-d_6): 166.6, 134.4, 129.4 ppm.

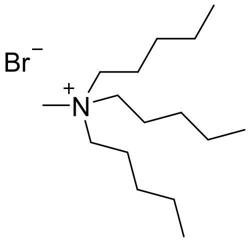
$\nu_{\max}$ (neat)/cm^{-1}: 2827, 2661, 2546, 1673, 1423, 1278, 928, 726.

Note: Isophthalic acid could be identified by characteristic ^{1}H resonances:

δ_H (400 MHz, DMSO-d_6): 8.48 (s, 1H), 8.16 (d, J = 7.7 Hz, 2H), 7.64 (t, J = 7.7 Hz, 1H)

3.0 Catalysis synthesis and characterisation

Compound 11



A 25 mL round-bottomed flask equipped with a stirring bar was charged with methylamine (2.5 mL, 2.0 M in THF, 5.0 mmol), acetonitrile (5 mL) and potassium carbonate (2.21 g, 16.0 mmol). 1-Bromopentane (1.86 mL, 15.0 mmol) was added dropwise at room temperature. The flask was equipped with a condenser and the solution was heated under reflux for 24 h before the mixture was filtered. The solvent was removed under reduced pressure and the product was triturated in diethyl ether. The product was dried *in vacuo* yielding **11** as a colourless oil (0.95 g, 59%).

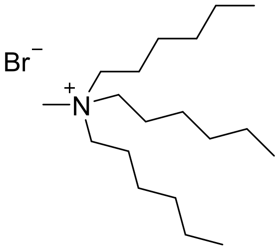
δ_H (400 MHz, DMSO-d_6): 3.27-3.14 (m, 6H), 2.95 (s, 3H), 1.71-1.53 (m, 6H), 1.42-1.19 (m, 12H), 0.89 (t, J = 7.2 Hz, 9H) ppm.

δ_C (101 MHz, DMSO-d_6): 60.5, 47.4, 27.9, 21.6, 21.0, 13.7 ppm.

HRMS (*m/z* – ESI$^+$): Found: 242.2847 (M$^+$) $C_{16}H_{36}N^+$ Requires: 242.2842.

ν_{max} (neat)/cm^{-1}: 2955, 2931, 2809, 1464, 1379, 1037, 935, 851, 774, 732.

Compound 12



A 25 mL round-bottomed flask equipped with a stirring bar was charged with methylamine (2.0 mL, 2.0 M in THF, 4.0 mmol), acetonitrile (4 mL) and potassium carbonate (2.21 g, 16 mmol). 1-Bromohexane (1.6 mL, 14.0 mmol) was added dropwise at room temperature. The flask was equipped with a condenser and the solution was heated under reflux for 24 h before the mixture was filtered. The solvent was removed under reduced pressure and the product was triturated in diethyl ether. The product was dried *in vacuo* yielding **12** as a brown oil (0.53 g, 37%).

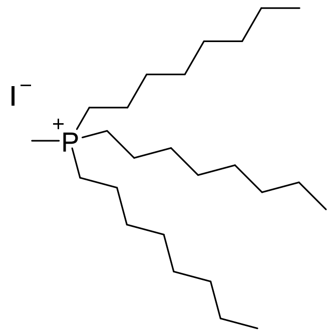
δ_H (400 MHz, DMSO-d_6): 3.23-3.13 (m, 6H), 2.93 (s, 3H), 1.69-1.53 (m, 6H), 1.39-1.21 (m, 18H), 0.88 (t, J = 6.3 Hz, 9H) ppm.

δ_C (101 MHz, DMSO-d_6): 60.5, 47.5, 30.6, 25.4, 21.8, 21.3, 13.8 ppm.

HRMS (*m/z* – ESI$^+$): Found: 284.3319 (M$^+$) $C_{19}H_{42}N^+$ Requires: 284.3312.

ν_{max} (neat)/cm^{-1}: 2955, 2926, 2859, 1464, 1379, 1058, 939, 877, 728.

Compound 14



A 25 mL round-bottomed flask equipped with a stirring bar was charged with trioctylphosphine (1.34 mL, 3.0 mmol) and acetonitrile (3 mL). Iodomethane (0.31 mL, 5.0 mmol) was added dropwise at 0 ºC. The reaction mixture was stirred at room temperature for 1 h. The solvent was removed under reduced pressure and the product was triturated in diethyl ether. The product was dried *in vacuo* yielding **14** as a colourless gum (1.31 g, 85%).

Spectral data for this compound were consistent with those in literature.[2]

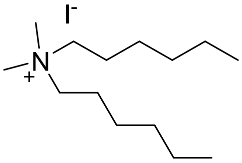
δ_H (400 MHz, $CDCl_3$): 2.50-2.43 (m, 6H), 2.11 (d, J = 13.3 Hz, 3H), 1.62-1.41 (m, 12H), 1.38-1.17 (m, 24H), 0.86 (t, J = 6.6 Hz, 9H) ppm.

δ_P (162 MHz, $CDCl_3$): 31.2 ppm.

HRMS (*m/z* – ESI$^+$): Found: 385.3965 (M$^+$) $C_{25}H_{54}P^+$ Requires: 385.3958.

ν_{max} (neat)/cm^{-1}: 2955, 2923, 2855, 1459, 1306, 906, 720.

Compound 16



A 100 mL round-bottomed flask equipped with a stirring bar was charged with dihexylamine (1.5 mL, 6.4 mmol), acetonitrile (20 mL) and potassium carbonate (5.30 g, 38.6 mmol). Iodomethane (1.6 mL, 25.7 mmol) was added dropwise at 0 ºC. The reaction mixture was stirred at room temperature for 30 min before the mixture was filtered. The solvent was removed under reduced pressure and the product was triturated in diethyl ether. The product was dried *in vacuo* yielding **16** as an off-white solid (1.51 g, 69%). M.p. 58-62 °C.

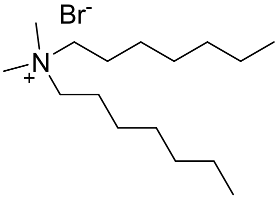
δ_H (400 MHz, DMSO-d_6): 3.26 – 3.22 (m, 4H), 3.00 (s, 6H), 1.73 – 1.57 (m, 4H), 1.40 – 1.22 (m, 12H), 0.89 (t, J = 6.4 Hz, 6H) ppm.

δ_c (101 MHz, DMSO-d_6): 62.9, 49.9, 30.6, 25.4, 21.8, 21.6, 13.8 ppm.

HRMS (m/z – ESI$^+$): Found: 214.2532 (M$^+$) $C_{14}H_{32}N^+$ Requires: 214.2529.

$\nu_{\max}$ (neat)/cm^{-1}: 3003, 2955, 2925, 2858, 1483, 1467, 936, 885, 727.

Compound 17



A 25 mL round-bottomed flask equipped with a stirring bar was charged with dimethylamine (3.0 mL, 2.0 M in THF, 6.0 mmol), acetonitrile (6 mL) and potassium carbonate (4.15 g, 30.0 mmol). 1-Bromoheptane (2.8 mL, 17.8 mmol) was added dropwise at room temperature. The flask was equipped with a condenser and the solution was heated under reflux for 24 h before the mixture was filtered. The solvent was removed under reduced pressure and the product was triturated in diethyl ether. The product was dried *in vacuo* yielding **17** as a colourless gum (1.41 g, 73%).

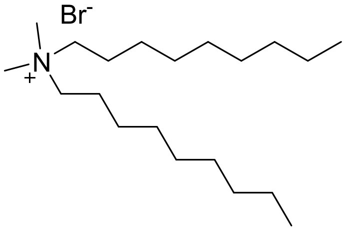
δ$_H$ (400 MHz, DMSO-*d*$_6$): 3.26 – 3.18 (m, 4H), 2.98 (s, 6H), 1.70 – 1.57 (m, 4H), 1.37 – 1.19 (m, 16H), 0.87 (t, *J* = 6.3 Hz, 6H) ppm.

δ$_H$ (101 MHz, DMSO-*d*$_6$): 62.9, 49.9, 31.0, 28.1, 25.7, 21.9, 21.6, 13.9 ppm.

HRMS (*m/z* – ESI$^+$): Found: 242.2844 (M$^+$) $C_{16}H_{36}N^+$ Requires: 242.2842.

ν$_{max}$ (neat)/cm^{-1}: 3011, 2924, 2858, 1467, 1275, 904, 729, 697.

Compound 19



A 25 mL round bottom flask equipped with a stirring bar was charged with dimethylamine (2.0 mL, 2.0 M in THF, 4.0 mmol), acetonitrile (4 mL) and potassium carbonate (2.21 g, 16.0 mmol). 1-Bromononane (2.7 mL, 14.1 mmol) was added and the solution was stirred at room temperature for 16 h before the mixture was filtered and the filtrate was dried *in vacuo*. The product was purified by flash column chromatography (CH_2Cl_2 to CH_2Cl_2/MeOH (95:5)) to afford **19** as a yellow oil (0.271 g, 20% yield).

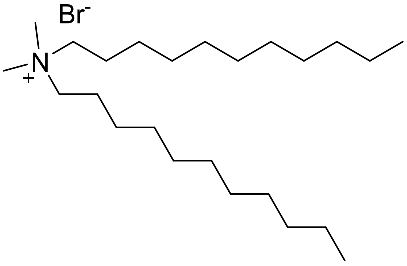
δ$_H$ (400 MHz, $CDCl_3$): 3.56-3.47 (m, 4H), 3.40 (s, 6H), 1.77-1.63 (m, 4H), 1.48-1.18 (m, 24H), 0.88 (t, *J* = 7.0 Hz, 6H) ppm.

δ$_C$ (400 MHz, $CDCl_3$): 64.1, 51.4, 31.9, 29.4, 29.3, 29.2, 26.4, 22.9, 22.7, 14.2 ppm.

HRMS (m/z -ES): Found: 289.3468 (M$^+$) $C_{20}H_{44}N^+$ Requires: 298.3468.

ν$_{max}$ (neat)/cm^{-1}: 2922, 2854, 1467, 927, 722.

Compound 20



A 25 mL round bottomed flask was charged with acetonitrile (2 mL), dimethylamine (2.0 mL, 2.0 M solution in THF, 4.0 mmol) and potassium carbonate (2.21 g, 16.0 mmol). 1-Bromoundecane (3.12 mL, 14.0 mmol) was diluted with anhydrous acetonitrile (2 mL) and added dropwise to the reaction mixture at room temperature. The flask was equipped with a condenser and the solution was heated under reflux for 24 h before the mixture was filtered. The residual white solid was washed with acetonitrile (3 x 5 mL) and the filtrate was concentrated under reduced pressure. The resulting oil was purified by flash chromatography (CH_2Cl_2 to CH_2Cl_2/MeOH (9:1)) to yield **20** as a white solid (1.14 g, 66%). M.p. 159-161 °C.

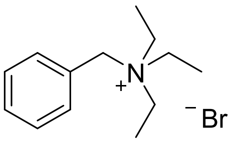
δ_H (400 MHz, $CDCl_3$): 3.57-3.46 (m, 4H), 3.41 (s, 6H), 1.74-1.63 (m, 4H), 1.43-1.20 (m, 32H), 0.87 (t, 6H, $J = 6.8$ Hz) ppm.

δ_C (100 MHz, $CDCl_3$): 64.0, 51.4, 32.0, 29.7, 29.6, 29.5, 29.4, 29.3, 26.4, 22.9, 22.8, 14.2 ppm.

HRMS (*m/z* – ESI$^+$): Found: 354.4098 (M$^+$) $C_{24}H_{52}N^+$ Requires: 354.4094.

νmax (neat)/ cm^{-1}: 2951, 2921, 2853, 1466, 1455, 889, 720.

Compound 31



A 25 mL round bottom flask equipped with a stirring bar was charged with triethylamine (1.4 mL, 10.0 mmol), THF (5 mL) and benzyl bromide (0.6 g, 5.1 mmol). The reaction mixture was stirred at room temperature for 48 h and the resulting precipitate was filtered. The solid was dissolved in the minimum volume of CH_2Cl_2 before being added to ice cold diethyl ether. The resulting suspension was filtered and the solid was washed with diethyl ether (3 X 5 mL) and dried *in vacuo* to give **31** as a white solid (1.21 g, 89%). M.p. 196-198 °C.

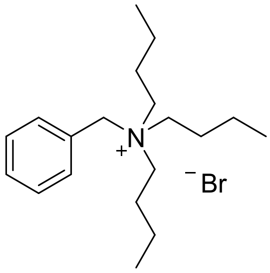
δ_H (400 MHz, $CDCl_3$): 7.53-7.28 (m, 5H), 4.67 (s, 2H), 3.35 (q, *J* = 7.2 Hz, 6H), 1.36 (t, *J* = 7.2 Hz, 9H) ppm.

δ_C (101 MHz, $CDCl_3$): 132.4, 130.6, 129.3, 127.1, 61.3, 52.9, 8.6 ppm.

HRMS (*m/z* – ESI^+): Found: 192.1748 (M^+) $C_{13}H_{22}N^+$ Requires: 192.1747.

vmax (neat)/ cm^{-1}: 2970, 2014, 1867, 1457, 1455, 766, 713.

Compound 32



A 25 mL round bottom flask equipped with a stirring bar was charged with tributylamine (1.8 mL, 7.6 mmol), THF (5 mL) and benzyl bromide (0.60 g, 5.1 mmol). The reaction mixture was stirred at room temperature for 48 h and the resulting precipitate was filtered. The solid was dissolved in the minimum volume of CH_2Cl_2 before being added to ice cold diethyl ether. The resulting suspension was filtered and the solid was washed with diethyl ether (3 x 5 mL) and dried *in vacuo* to give **32** as a white solid (1.67 g, 93%).

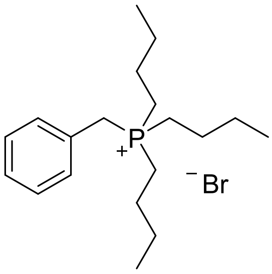
δ_H (400 MHz, DMSO-d_6): 7.54-7.48 (m, 5H), 4.52 (s, 2H), 3.12-3.05 (m, 6H), 1.77-1.66 (m, 6H), 1.36-1.26 (m, 6H), 0.95 (t, *J* = 7.5 Hz, 9H) ppm.

δ_C (101 MHz, DMSO-d_6): 132.5, 130.4, 129.1, 127.8, 61.2, 57.5, 23.3, 19.2, 13.5 ppm.

HRMS (*m/z* – ESI^+): Found: 276.2686 (M^+) $C_{19}H_{34}N^+$ Requires: 276.2686.

vmax (neat)/ cm^{-1}: 2960, 2872, 1476, 1458, 723, 702.

Compound 33



A 50 mL round bottomed flask was charged with anhydrous acetonitrile (10 mL) and tributylphosphine (2.02 g, 10.0 mmol). The emulsion was kept under an argon atmosphere, stirred vigorously for 5 min, and cooled to 0 °C. Benzyl bromide (1.54 g, 9.0 mmol) was added dropwise to the emulsion and the resulting mixture was stirred at room temperature for 16 h. The crude mixture was evaporated under reduced pressure. Diethyl ether (30 mL) was added and the resulting mixture was filtered. The solid was washed with diethyl ether (3 x 20 mL) to yield **33** as a white solid (3.28 g, 98%). M.p. 143.0-144.0 °C. (Lit.,[2] 143 °C)

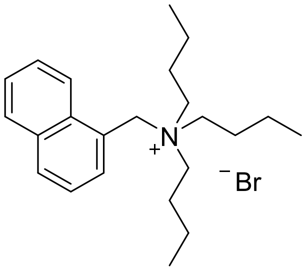
Spectral data for this compound were consistent with those in literature.[4]

δ_H (400 MHz, DMSO-d_6): 7.46-7.33 (m, 5H), 3.83 (d, J = 15.5 Hz, 2H), 2.21-2.10 (m, 6H), 1.48-1.31 (m, 12H), 0.88 (t, J = 7.0 Hz, 9H) ppm.

δ_C (100 MHz, DMSO-d_6): 130.0 (d, J = 4.8 Hz), 129.3, 129.2 (d, J = 3.1 Hz), 128.0 (d, J = 3.4 Hz), 25.4 (d, J = 44.5 Hz), 23.3 (d, J = 15.7 Hz), 22.5 (d, J = 4.5 Hz), 17.4 (d, J = 47.1 Hz), 13.2 ppm.

HRMS (m/z – ESI$^+$): Found: 293.2397 (M$^+$) $C_{19}H_{34}P^+$ Requires: 293.2393.

Compound 34



A 25 mL round bottom flask equipped with a stirring bar was charged with tributylamine (3.6 mL, 15.1 mmol), THF (5 mL) and (1-bromomethyl)naphthalene (1.11 g, 5.0 mmol). The reaction mixture was stirred at room temperature for 48 h and the resulting precipitate was filtered. The solid was dissolved in the minimum volume of CH_2Cl_2 before being added to ice cold diethyl ether. The resulting suspension was filtered and the solid was washed with diethyl ether (3 x 5 mL) and dried *in vacuo* to give **34** as a white solid (1.67 g, 60%). M.p. 141.5-142.5 °C

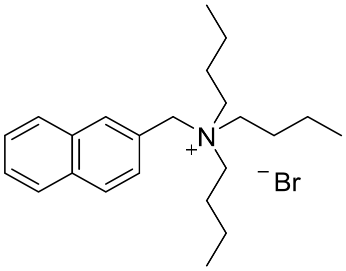
δ_H (600 MHz, DMSO-d_6): 8.50 (d, 1H, J = 8.5 Hz), 8.15 (d, 1H, J = 8.2 Hz), 8.07 (dd, 1H, J = 8.1, 0.9 Hz), 7.78 (dd, J = 7.2, 0.9 Hz, 1H), 7.72-7.63 (m, 3H), 5.08 (s, 2H), 3.30-3.25 (m, 6H), 1.62-1.53 (m, 6H), 1.22-1.14 (m, 6H), 0.83 (t, 9H, J = 7.4 Hz) ppm.

δ_C (151 MHz, DMSO-d_6): 133.6, 132.8, 132.7, 131.3, 129.1, 127.2, 126.4, 125.3, 124.7, 123.5, 60.1, 58.6, 23.8, 19.3, 13.4 ppm.

HRMS (m/z – ESI$^+$): Found: 326.2849 (M$^+$) $C_{23}H_{36}N^+$ Requires: 326.2842.

vmax (neat)/ cm^{-1}: 3044, 2955, 3937, 3872, 1515, 1462, 1379, 1236, 1170, 1024, 875, 812, 791, 731, 657.

Compound 35



A 25 mL round bottom flask equipped with a stirring bar was charged with tributylamine (3.0 mL, 12.6 mmol), THF (5 mL) and (2-bromomethyl)naphthalene (1.11 g, 5.0 mmol). The reaction mixture was stirred at room temperature for 24 h and the resulting precipitate was filtered. The solid was dissolved in the minimum volume of CH_2Cl_2 before being added to ice cold diethyl ether. The resulting suspension was filtered and the solid was washed with diethyl ether (3 X 5 mL) and dried *in vacuo* to give **35** as a white solid (1.01 g, 49%). M.p. 137-139 °C.

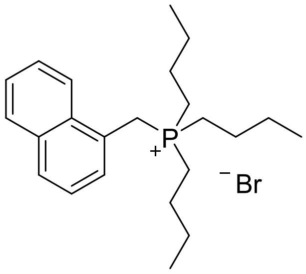
δ_H (600 MHz, DMSO-d_6): 8.08-7.97 (m, 4H), 7.66-7.54 (m, 3H), 4.68 (s, 2H), 3.18-3.09 (m, 6H), 1.81-1.71 (m, 6H), 1.37-1.28 (m, 6H), 0.94 (t, J = 7.7 Hz, 9H) ppm.

δ_C (151 MHz, DMSO-d_6): 133.8, 133.4, 132.9, 128.4, 129.1, 128.7, 128.1, 128.1, 127.5, 125.8, 61.7, 58.0, 23.8, 19.7, 14.0 ppm.

HRMS (*m/z* – ESI$^+$): Found: 326.2850 (M$^+$) $C_{23}H_{36}N^+$ Requires: 326.2842.

νmax (neat)/ cm^{-1}: 2962, 1484, 1371, 840, 787, 771.

Compound 36



A 25 mL round bottomed flask was charged with anhydrous acetonitrile (3 mL) and tributylphosphine (1.01 g, 5.0 mmol). The emulsion was kept under an argon atmosphere, stirred vigorously for 5 min, and cooled to 0 °C. 1-(Bromomethyl)naphthalene (1.11 g, 5.0 mmol) was dissolved in anhydrous acetonitrile (2 mL), added dropwise to the emulsion and the resulting mixture was stirred at room temperature for 16 h. The crude mixture was evaporated under reduced pressure. Diethyl ether (15 mL) was added to initiate product precipitation. The product was filtered and the resulting solid was washed with diethyl ether (3 x 10 mL) to yield **36** as a white solid (1.64 g, 86%). M.p. 100-102 °C.

δ_H (600 MHz, DMSO-d_6): 8.03-7.87 (m, 4H), 7.61-7.52 (m, 2H), 7.52-7.44 (m, 1H), 4.01 (d, J = 15.5 Hz, 2H), 2.27-2.12 (m, 6H), 1.53-1.30 (m, 12H), 0.87 (t, J = 7.2 Hz, 9H) ppm.

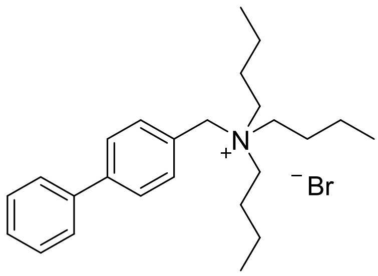
δ_C (151 MHz, DMSO-d_6): 132.9 (d, J = 2.9 Hz), 132.2 (d, J = 2.5 Hz), 128.9 (d, J = 6.5 Hz), 128.8 (d, J = 2.3 Hz), 127.7, 127.6 (d, J = 3.9 Hz), 127.5, 126.9, 126.8, 126.5, 25.6 (d, J = 44.2 Hz), 23.3 (d, J =15.7 Hz), 22.5 (d, J = 4.5 Hz) , 17.4 (d, J = 46.8 Hz), 13.2 ppm.

δ_P (162 MHz, DMSO-d_6): 33.0 ppm.

HRMS (*m/z* – ESI$^+$): Found: 343.2551 (M$^+$) $C_{23}H_{36}P^+$ Requires: 343.2549.

νmax (neat)/ cm^{-1}: 2957, 2929, 2871, 1465, 1094, 917, 861, 828, 744.

Compound 37



A 25 mL round bottom flask equipped with a stirring bar was charged with tributylamine (2.4 mL, 10.1 mmol), THF (5 mL) and 4-(bromomethyl)-1,1’-biphenyl (1.24 g, 5.0 mmol). The reaction mixture was stirred at room temperature for 24 h and the resulting precipitate was filtered. The solid was dissolved in the minimum volume of CH_2Cl_2 before being added to ice cold diethyl ether. The resulting suspension was filtered and the solid was washed with diethyl ether (3 x 5 mL) and dried *in vacuo* to give **37** as a white powder (1.01 g, 49%). M.p. 158-159 °C.

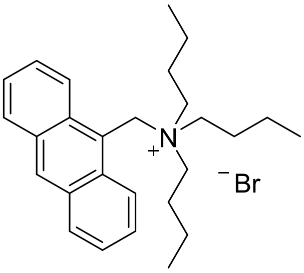
δ_H (600 MHz, DMSO-d_6): 7.87 (m, 2H), 7.75-7.72 (m, 2H), 7.60-7.56 (m, 2H), 7.52-7.48 (m, 2H), 7.44-7.40 (m, 1H), 4.57 (s, 2H), 3.16-3.09 (m, 6H), 1.80-1.70 (m, 6H), 1.38-1.29 (m, 6H), 0.96 (t, J = 7.4 Hz, 9H) ppm.

δ_C (151 MHz, DMSO-d_6): 141.8, 138.8, 133.2, 129.1, 128.1, 127.1, 126.9, 126.8, 60.9, 57.5, 23.3, 19.2, 13.5 ppm.

HRMS (*m/z* – ESI$^+$): Found: 352.2999 (M$^+$) $C_{25}H_{38}N^+$ Requires: 352.2999.

νmax (neat)/ cm^{-1}: 2960, 2872, 1483, 1461, 869, 765, 703.

Compound 38



A 25 mL round bottom flask equipped with a stirring bar was charged with tributylamine (2.6 mL, 11.1 mmol), THF (5 mL) and 9-(bromomethyl)anthracene (1.00 g, 3.7 mmol). The reaction mixture was stirred at room temperature for 24 h and the resulting precipitate was filtered. The solid was dissolved in the minimum volume of CH_2Cl_2 before being added to ice cold diethyl ether. The resulting suspension was filtered and the solid was washed with diethyl ether (3 x 5 mL) and dried *in vacuo* to give **38** as a white powder (1.43 g, 79%). M.p. 137-139 °C.

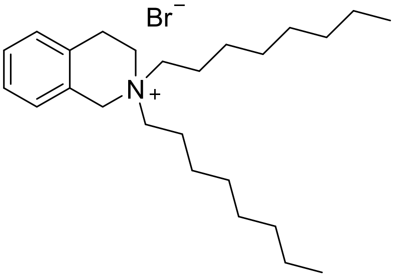
δ_H (600 MHz, DMSO-d_6): 8.93 (s, 1H), 8.72 (app. d, 2H), 8.23 (app. d, 2H), 7.73 (app. t, J = 8.2 Hz, 2H), 7.63 (app. t, J = 7.7 Hz, 2H), 5.73 (s, 2H), 3.4-3.28 (m, 6H), 1.28-1.14 (m, 6H), 0.96 (m, 6H), 0.67 (t, J = 7.3 Hz, 9H) ppm.

δ_C (151 MHz, DMSO-d_6):132.4, 131.5, 130.9, 129.4, 127.5, 125.7, 124.4, 120.6, 60.0, 58.4, 24.0, 19.2, 13.1 ppm.

HRMS (m/z – ESI$^+$): Found: 376.2999 (M$^+$) $C_{27}H_{38}N^+$ Requires: 376.2999.

νmax (neat)/ cm^{-1}: 2959, 2873, 1467, 1378, 858, 746.

Compound 39



A 25 mL round bottomed flask under argon atmosphere, equipped with a stirring bar, was charged with acetonitrile (3 mL), potassium carbonate (0.70 g, 5.1 mmol) and tetrahydroisoquinoline (0.32 mL, 2.5 mmol). 1-Bromooctane (0.70 mL, 4.0 mmol) was added dropwise. The flask was equipped with a condenser and the solution was heated under reflux for 16 h. After cooling to room temperature, the solution was filtered and the filtrate was concentrated under reduced pressure. The resulting yellow oil was then dissolved in CH_2Cl_2 (1 mL) before being added dropwise to ice cold diethyl ether (15 mL) yielding a yellow precipitate. The product was purified by flash column chromatography (CH_2Cl_2 to CH_2Cl_2/MeOH (95:5)) to afford **39** as a white solid (0.184 g, 17% yield). M.p. 144-146 °C.

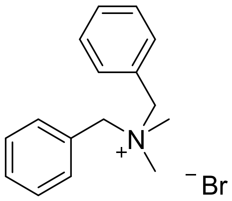
δ_H (400 MHz, $CDCl_3$): 7.29-7.14 (m, 4H), 4.90 (s, 2H), 4.10 (t, 2H, J = 6.5 Hz), 3.62-3.38 (m, 4H), 3.14 (t, 2H, J = 6.5 Hz), 1.84-1.64 (m, 4H), 1.38-1.12 (m, 20H), 0.83 (t, 6H, J = 6.8 Hz) ppm.

δ_C (100 MHz, $CDCl_3$): 129.2, 128.9, 128.8, 127.8, 127.7, 126.2, 61.0, 58.5, 56.2, 31.6, 29.1, 29.0, 26.4, 23.9, 22.6, 22.2, 14.1 ppm.

HRMS (m/z -ESI$^+$): Found: 358.3476 (M$^+$) $C_{25}H_{44}N^+$ Requires: 358.3468.

ν_{max} (neat)/cm^{-1}: 2955, 2923, 2854, 1494, 1453, 748, 724.

Compound 40



A 25 mL round-bottomed flask equipped with a stirring bar was charged with dimethylamine (5.0 mL, 2.0 M in THF, 5.0 mmol), acetonitrile (5 mL) and potassium carbonate (2.07 g, 15.0 mmol). Benzyl bromide (1.78 mL, 15.0 mmol) was added dropwise at room temperature. The reaction mixture was stirred for 24 h before the mixture was filtered. The solvent was removed under reduced pressure and the product was triturated in diethyl ether. The product was dried *in vacuo* yielding **40** as a white solid (1.45 g, 95%). M.p. 196-198 °C.

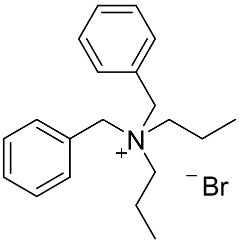
δ_H (400 MHz, DMSO-d_6): 7.66-7.48 (m, 10H), 4.62 (s, 4H), 2.87 (s, 6H) ppm.

δ_C (101 MHz, DMSO-d_6): 133.1, 130.2, 128.9, 128.1, 66.7, 48.0 ppm.

HRMS (m/z – ESI$^+$): Found: 226.1594 (M$^+$) $C_{16}H_{20}N^+$ Requires: 226.1590.

ν_{max} (neat)/cm^{-1}: 3034, 2960, 2854, 2013, 1827, 1495, 847, 757, 731.

Compound 41



A 25 mL round-bottomed flask equipped with a stirring bar was charged with dipropylamine (0.69 mL, 5.0 mmol), acetonitrile (5 mL) and potassium carbonate (1.73 g, 12.5 mmol). Benzyl bromide (1.34 mL, 11.3 mmol) was added dropwise at room temperature. The reaction mixture was stirred for 48 h before the mixture was filtered. The solvent was removed under reduced pressure and the product was triturated in diethyl ether. The product was dried *in vacuo* yielding **41** as a white solid (1.31 g, 72%). M.p. 159-160 °C.

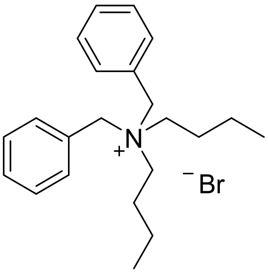
δ_H (400 MHz, DMSO-d_6): 7.65-7.48 (m, 10H), 4.64 (s, 4H), 3.15-3.03 (m, 4H), 1.89-1.74 (m, 4H), 0.84 (t, J = 7.1 Hz, 6H) ppm.

δ_C (101 MHz, DMSO-d_6): 132.8, 130.2, 129.0, 128.1, 62.8, 58.8, 15.8, 10.4 ppm.

HRMS (m/z – ESI$^+$): Found: 282.2220 (M$^+$) $C_{20}H_{28}N^+$ Requires: 282.2216.

ν_{max} (neat)/cm^{-1}: 2988, 2962, 2876, 1473, 1460, 963, 743, 729, 702.

Compound 42



A 25 mL round-bottomed flask equipped with a stirring bar was charged with dibutylamine (0.84 mL, 5.0 mmol), acetonitrile (5 mL) and potassium carbonate (1.728 g, 12.5 mmol). Benzyl bromide (1.48 mL, 12.5 mmol) was added dropwise at room temperature. The reaction mixture was stirred for 48 h before the mixture was filtered. The solvent was removed under reduced pressure and the product was triturated in diethyl ether. The product was dried *in vacuo* yielding **42** as a white solid (1.72 g, 88%). M.p. 140-141 °C.

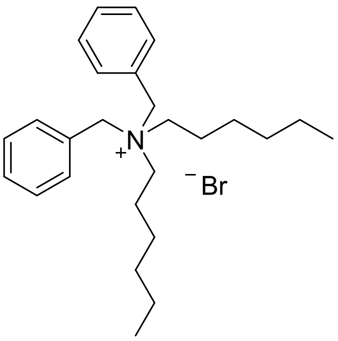
δ_H (400 MHz, DMSO-d_6): 7.64-7.47 (m, 10H), 4.60 (s, 4H), 3.16-3.05 (m, 4H), 1.86-1.72 (m, 4H), 1.34-1.18 (m, 4H), 0.90 (t, J = 7.4 Hz, 6H) ppm.

δ_C (101 MHz, DMSO-d_6): 132.9, 130.3, 129.1, 128.0, 62.8, 57.4, 23.9, 19.3, 13.5 ppm.

HRMS (m/z – ESI$^+$): Found: 310.2529 (M$^+$) $C_{22}H_{32}N^+$ Requires: 310.2529.

ν_{max} (neat)/cm^{-1}: 2955, 2868, 1498, 1476, 872, 756, 725.

Compound 43



A 25 mL round-bottomed flask equipped with a stirring bar was charged with dihexylamine (0.93 mL, 7.1 mmol), acetonitrile (5 mL) and potassium carbonate (1.73 g, 12.5 mmol). Benzyl bromide (1.92 mL, 16.1 mmol) was added dropwise at room temperature. The reaction mixture was stirred for 48 h before the mixture was filtered. The solvent was removed under reduced pressure and the product was triturated in diethyl ether. The product was dried *in vacuo* yielding **43** as a white solid (1.52 g, 48%). M.p. 145-146 °C.

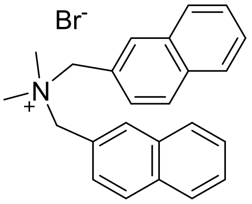
δ_H (400 MHz, DMSO-d_6): 7.63-7.47 (m, 10H), 4.62 (s, 4H), 3.19-3.02 (m, 4H), 1.87-1.70 (m, 4H), 1.39-1.15 (m, 12H), 0.87 (t, J = 6.6 Hz, 6H) ppm.

δ_C (101 MHz, DMSO-d_6): 132.8, 130.3, 129.0, 128.1, 62.9, 57.6, 30.6, 25.5, 22.0, 21.8, 13.7 ppm.

HRMS (m/z – ESI$^+$): Found: 366.3162 (M$^+$) $C_{26}H_{40}N^+$ Requires: 366.3155.

ν_{max} (neat)/cm^{-1}: 2950, 2927, 2858, 1985, 1924, 1851, 1497, 1482, 756, 711.

Compound 44



A 25 mL round-bottomed flask equipped with a stirring bar was charged with dimethylamine (2.53 mL, 2.0 M in THF, 5.1 mmol), acetonitrile (5 mL) and potassium carbonate (2.07 g, 15.0 mmol). 2-(bromomethyl)naphthalene (2.76 mL, 12.5 mmol) was added dropwise at room temperature. The reaction mixture was stirred for 24 h before the mixture was filtered. The solvent was removed under reduced pressure and the product was triturated in diethyl ether. The product was dried *in vacuo* yielding **44** as a white solid (0.166 g, 8%). M.p. 228-230 °C.

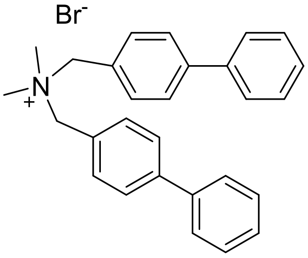
δ_H (400 MHz, DMSO-d_6): 8.21 (s, 2H), 8.1-7.94 (m, 6H), 7.70 (d, J = 8.2, 2H), 7.66-7.57 (m, 4H), 4.87 (s, 4H), 3.00 (s, 6H) ppm.

δ_C (101 MHz, DMSO-d_6): 133.4, 133.3, 132.5, 129.6, 128.4, 128.3, 127.6, 127.5, 126.8, 125.5, 67.2, 48.4 ppm.

HRMS (m/z – ESI$^+$): Found: 326.1903 (M$^+$) $C_{24}H_{24}N^+$ Requires: 326.1903.

ν_{max} (neat)/cm^{-1}: 3030, 2961, 2868, 1487, 1474, 860, 819, 782.

Compound 45



A 25 mL round-bottomed flask equipped with a stirring bar was charged with dimethylamine (0.67 mL, 2.0 M in THF, 1.4 mmol), acetonitrile (2 mL) and potassium carbonate (0.28 g, 2.0 mmol). 4-(bromomethyl)-1,1'-biphenyl (0.50 g, 2.0 mmol) was added dropwise at room temperature. The reaction mixture was stirred at reflux for 24 h before the mixture was filtered. The solvent was removed under reduced pressure and the product was triturated in diethyl ether. The product was dried *in vacuo* yielding **45** as a white solid (0.492 g, 80%). M.p. 230-232 °C.

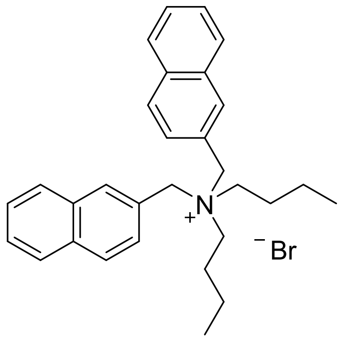
δ_H (400 MHz, DMSO-d_6): 7.83 (d, J = 8.0 Hz, 4H), 7.79 – 7.69 (m, 8H), 7.49 (app. t, J = 7.5 Hz, 4H), 7.43-7.41 (t, J = 7.2 Hz, 2H), 4.83 (s, 4H), 2.99 (s, 6H) ppm.

δ_C (101 MHz, DMSO-d_6): 141.7, 139.0, 133.8, 129.0, 128.0, 127.2, 127.0, 126.8, 66.4, 48.1 ppm.

HRMS (m/z – ESI$^+$): Found: 378.2220 (M$^+$) $C_{28}H_{28}N^+$ Requires: 378.2216.

ν_{max} (neat)/cm^{-1}: 3030, 2989, 2957, 1487, 1473, 853, 748.

Compound 46



A 25 mL round-bottomed flask equipped with a stirring bar was charged with dibutylamine (0.84 mL, 5.0 mmol), acetonitrile (5 mL) and potassium carbonate (2.07 g, 15.0 mmol). (2-bromomethyl)naphthalene (2.76 g, 12.5 mmol) was added dropwise at room temperature. The reaction mixture was stirred for 48 h before the mixture was filtered. The solvent was removed under reduced pressure and the product was triturated in diethyl ether. The product was dried *in vacuo* yielding **46** as a white solid (0.26 g, 11%). M.p. 166-167 °C.

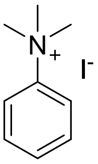
δ_H (400 MHz, DMSO-d_6): 8.22 (s, 2H), 8.07 (d, J = 8.6 Hz, 2H), 8.01 (t, J = 7.8 Hz, 4H), 7.71 (d, J = 8.6 Hz, 2H), 7.68-7.58 (m, 4H), 4.87 (s, 4H), 3.28-3.20 (m, 4H), 2.00-1.82 (m, 4H), 1.39-1.22 (m, 4H), 0.92 (t, J = 7.3 Hz, 6H) ppm.

δ_C (101 MHz, DMSO-d_6): 133.4, 133.3, 132.5, 129.4, 128.5, 128.3, 127.6, 127.6, 126.9, 125.5, 62.9, 57.4, 24.0, 19.4, 13.4 ppm.

HRMS (m/z – ESI$^+$): Found: 410.2848 (M$^+$) $C_{30}H_{36}N^+$ Requires: 410.2842.

ν_{max} (neat)/cm^{-1}: 3049, 2961, 2871, 2164, 1982, 1474, 1365, 762.

Compound 47



A 50 mL round-bottomed flask equipped with a stirring bar was charged with *N,N*-dimethylaniline (0.63 mL, 5.0 mmol) and CH_2Cl_2 (5 mL). Iodomethane (0.31 mL, 5.0 mmol) was added dropwise at 0 ºC. The reaction mixture was stirred at room temperature for 24 h. The solvent was removed under reduced pressure and the product was triturated in diethyl ether. The product was dried *in vacuo* yielding **47** as an off-white solid (0.47 g, 36%). M.p. 220-221 °C. (lit. M.p. 222-223)[4]

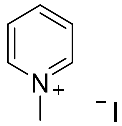
δ_H (400 MHz, $CDCl_3$):7.87 (d, J = 8.3 Hz, 2H), 7.70-7.62 (m, 2H), 7.62-7.55 (m, 1H), 4.02 (s, 9H) ppm.

δ_C (101 MHz, $CDCl_3$): 147.3, 131.1, 131.0, 119.8, 57.9 ppm.

HRMS (*m/z* – ESI$^+$): Found: 136.1120 (M$^+$) $C_9H_{14}N^+$ Requires: 136.1121.

ν_{max} (neat)/cm^{-1}: 3009, 1743, 1593, 1498, 1460, 939, 841, 765, 691.

Compound 48



A 100mL round bottomed flask under argon atmosphere, equipped with a stirring bar, was charged with anhydrous pyridine (0.40 mL, 5.0 mmol) and acetonitrile (50 mL). Methyl iodide (0.31 mL, 5.0 mmol) was added dropwise and the mixture was refluxed for 18 h before being concentrated under reduced pressure. The resulting yellow solid was then dissolved in acetonitrile (7 mL) and was added to a stirring solution of diethyl ether (20 mL) before being filtered and washed with diethyl ether (3 x 10 mL) yielding **48** as a light-yellow solid (0.97 g, 88%). M.p. 79-80 °C dec.

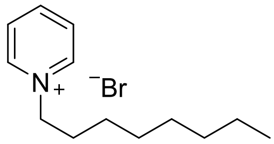
δ_H (400 MHz, DMSO-d_6): 8.99 (d, *J* = 5.6 Hz, 2H), 8.58 (t, *J* = 7.8 Hz, 1H), 8.14 (app. t, 2H), 4.36 (s, 3H) ppm.

δ_C (101 MHz, DMSO-d_6): 145.5, 145.04, 127.7, 47.9 ppm.

HRMS (*m/z* – ESI$^+$): Found: 94.0651 (M$^+$) $C_6H_8N^+$ Requires: 94.0651.

ν_{max} (neat)/cm^{-1}: 3033, 2989, 1629, 1497, 1482, 1284, 766, 673.

Compound 49



A 25 mL round bottomed flask under argon atmosphere, equipped with a stirring bar, was charged with anhydrous pyridine (1.05 mL, 13.0 mmol). 1-bromooctane (2.25 mL, 13.0 mmol) was added and the mixture was heated to 80 ºC for 3 h. The mixture was concentrated under reduced pressure. The resulting yellow oil was triturated in diethyl ether (3 x 10 mL) yielding **49** as a light-yellow oil (1.23 g, 35%).

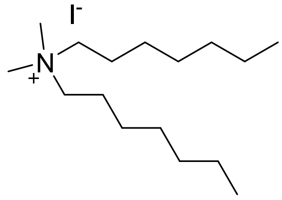
δ_H (600 MHz, DMSO-d_6): 9.12 (d, *J* = 5.3 Hz, 2H), 8.61 (t, *J* = 7.8 Hz, 1H), 8.14 (app. t, 2H), 4.60 (t, *J* = 7.2 Hz, 2H), 1.99-1.83 (m, 2H), 1.38-1.14 (m, 10H), 0.85 (t, *J* = 8.3 Hz, 3H) ppm.

δ_C (151 MHz, DMSO-d_6): 145.5, 144.7, 128.1, 60.7, 31.1, 30.7, 28.4, 28.3, 25.4, 22.0, 13.9 ppm.

HRMS (*m/z* – ESI$^+$): Found: 192.1750 (M$^+$) $C_{13}H_{22}N^+$ Requires: 192.1747.

ν_{max} (neat)/cm^{-1}: 3028, 2955, 2925, 2855, 1633, 1486, 1170, 774, 683.

Compound 52



A 100 mL round-bottomed flask equipped with a stirring bar was charged with diheptylamine (1.00 g, 4.7 mmol), acetonitrile (15 mL) and potassium carbonate (3.89 g, 28.1 mmol). Iodomethane (1.17 mL, 18.7 mmol) was added dropwise at 0 ºC. The reaction mixture was stirred at room temperature for 30 min before the mixture was filtered. The solvent was removed under reduced pressure and the product was triturated in diethyl ether. The product was dried *in vacuo* yielding **52** as an off-white solid (1.46 g, 84%). M.p. 66-68 °C.

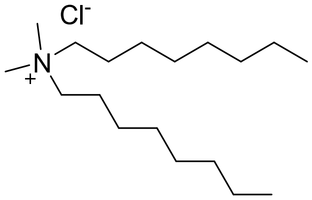
δ_H (400 MHz, DMSO-d_6): 3.24 – 3.20 (m, 4H), 2.98 (s, 6H), 1.68 – 1.55 (m, 4H), 1.36 – 1.16 (m, 16H), 0.85 (t, J = 6.5 Hz, 6H) ppm.

δ_C (101 MHz, DMSO-d_6): 62.9, 49.9, 30.9, 28.1, 25.7, 21.9, 21.7, 13.9 ppm.

HRMS (m/z – ESI$^+$): Found: 242.2842 (M$^+$) $C_{16}H_{36}N^+$ Requires: 242.2842.

ν_{max} (neat)/cm^{-1}: 2924, 2856, 1467, 1378, 902, 724.

Compound 53



A 100 mL round-bottomed flask equipped with a stirring bar was charged with Dowex® 1X8 Cl (5.0 g) and methanol (30 mL). *N,N*-dimethyl-*N*-octyloctan-1-aminium bromide (**18**, 0.50 g, 1.43 mmol) was added and the mixture was shaken for 20 h on an orbital shaker. The mixture was passed through a plug of Dowex® 1X8 Cl (5.0 g) and the solvent was removed under reduced pressure. The product was triturated in diethyl ether and dried *in vacuo* yielding **53** as an off-white solid (0.41 g, 94%). M.p. 58-61 °C.

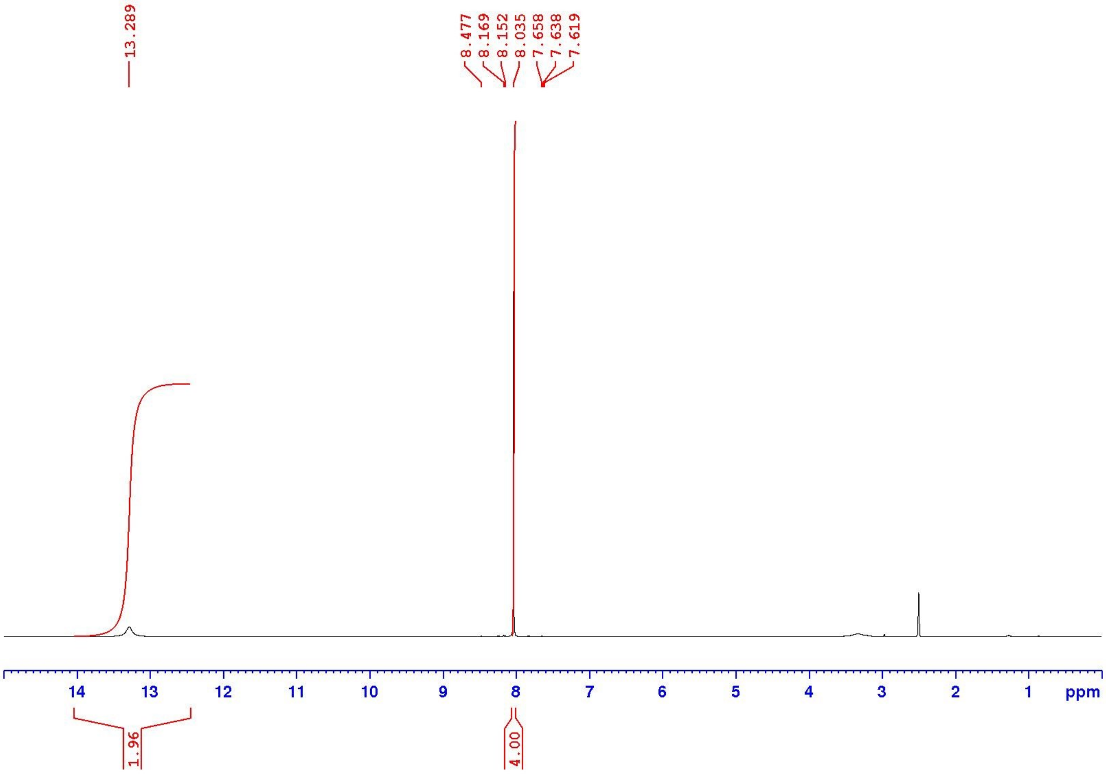
δ_H (400 MHz, DMSO-d_6): 3.31-3.21 (m, 4H), 3.02 (s, 6H), 1.71-1.56 (m, 4H), 1.38-1.16 (m, 20H), 0.86 (t, J = 6.5 Hz, 6H) ppm.

δ_C (101 MHz, DMSO-d_6): 62.7, 49.8, 31.1, 28.4, 28.4, 25.8, 22.0, 21.6, 13.9 ppm.

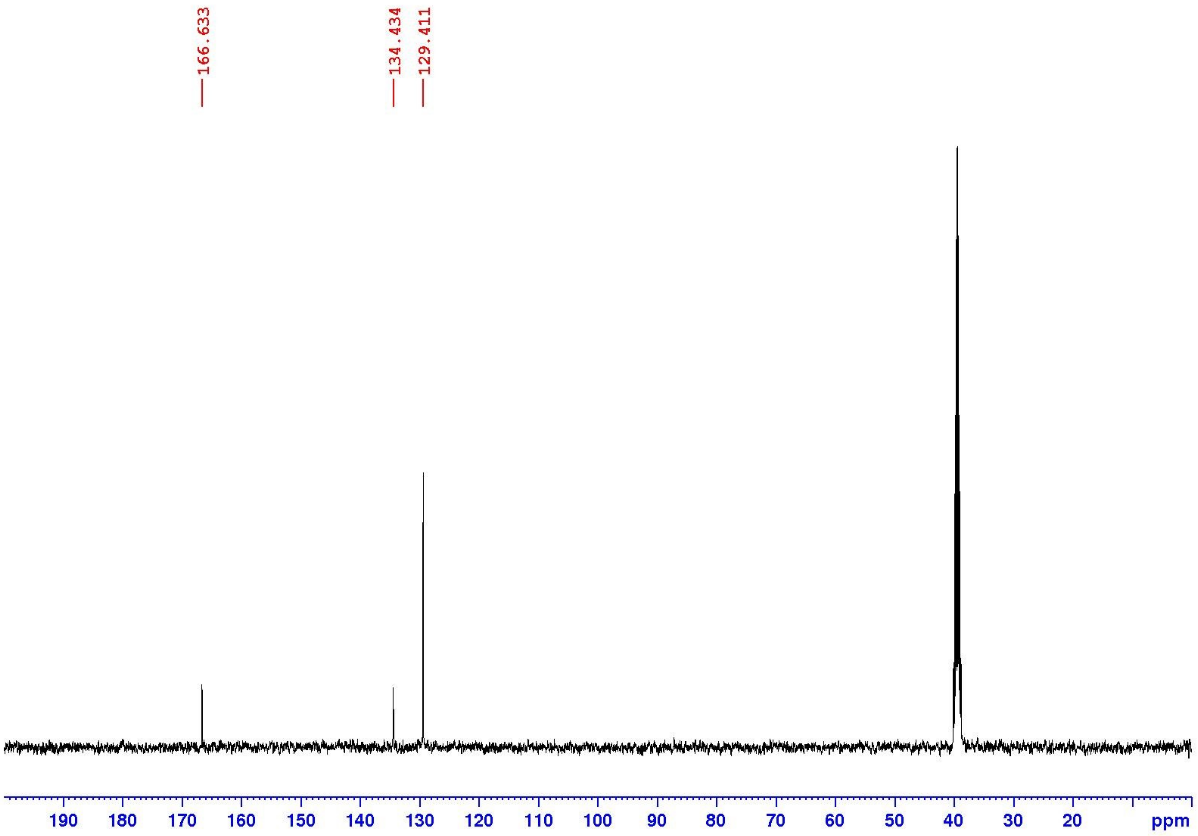
HRMS (m/z – ESI$^+$): Found: 354.4098 (M$^+$) $C_{24}H_{52}N^+$ Requires: 354.4094.

ν_{max} (neat)/cm^{-1}: 2998, 2921, 2871, 2854, 1470, 897, 725.

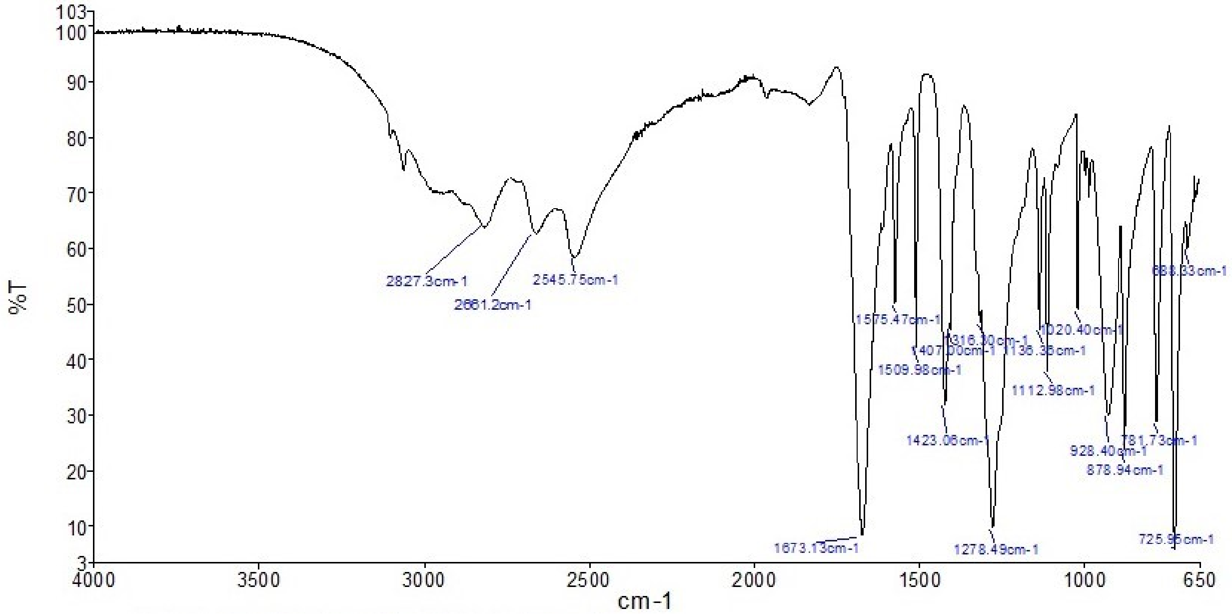
4.0 Analytical Spectra of catalysts and products



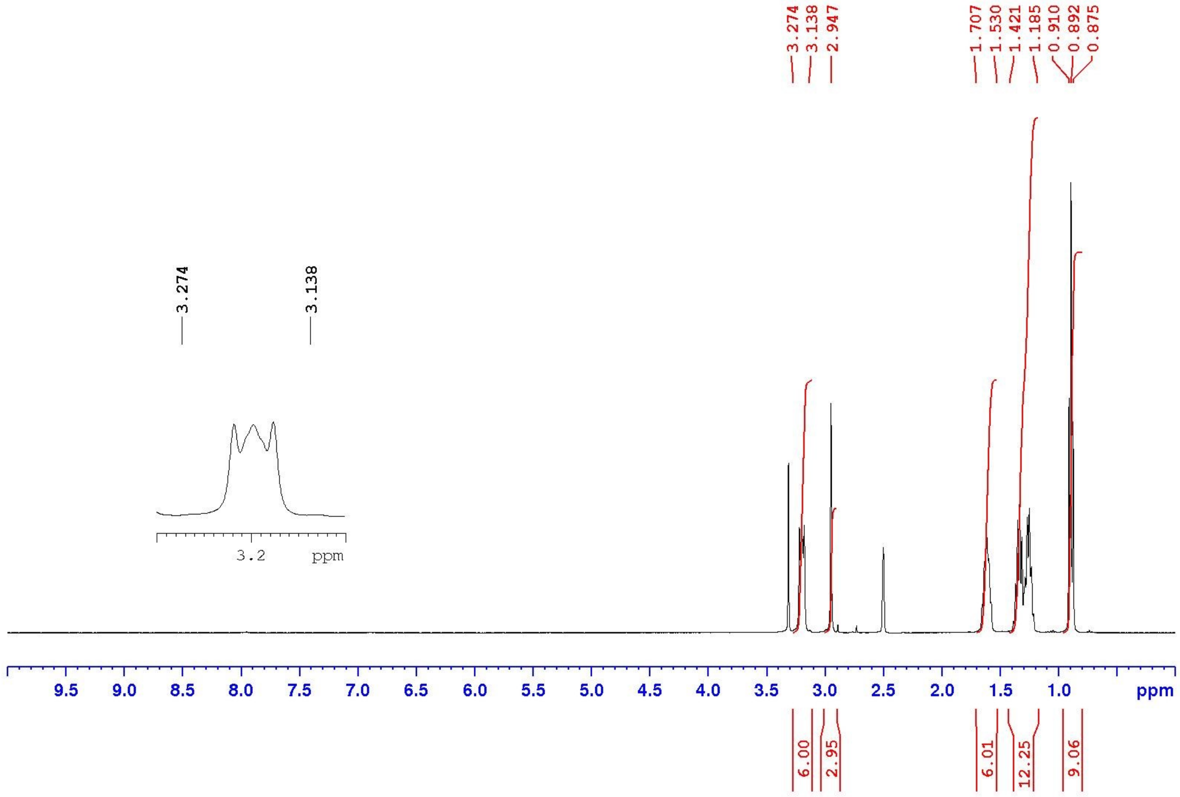
^{1}H NMR spectrum (400 MHz, DMSO-*d6*) of **TPA**



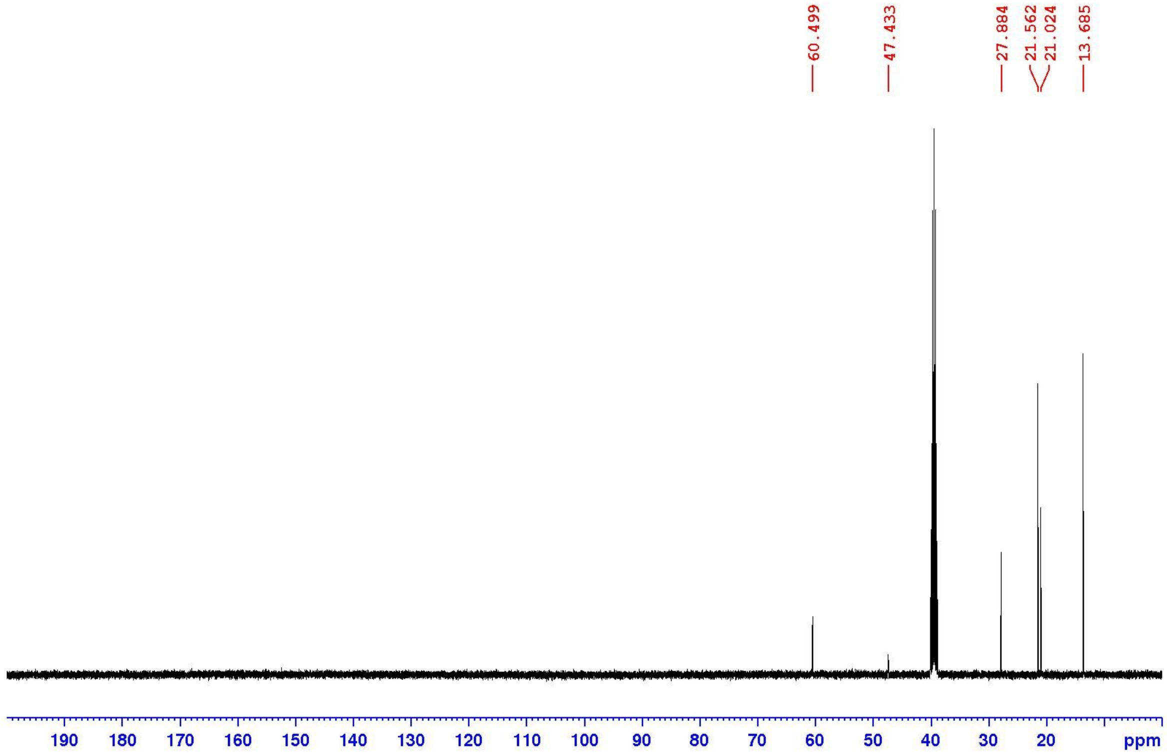
^{13}C NMR spectrum (101 MHz, DMSO-*d*6) of **TPA**



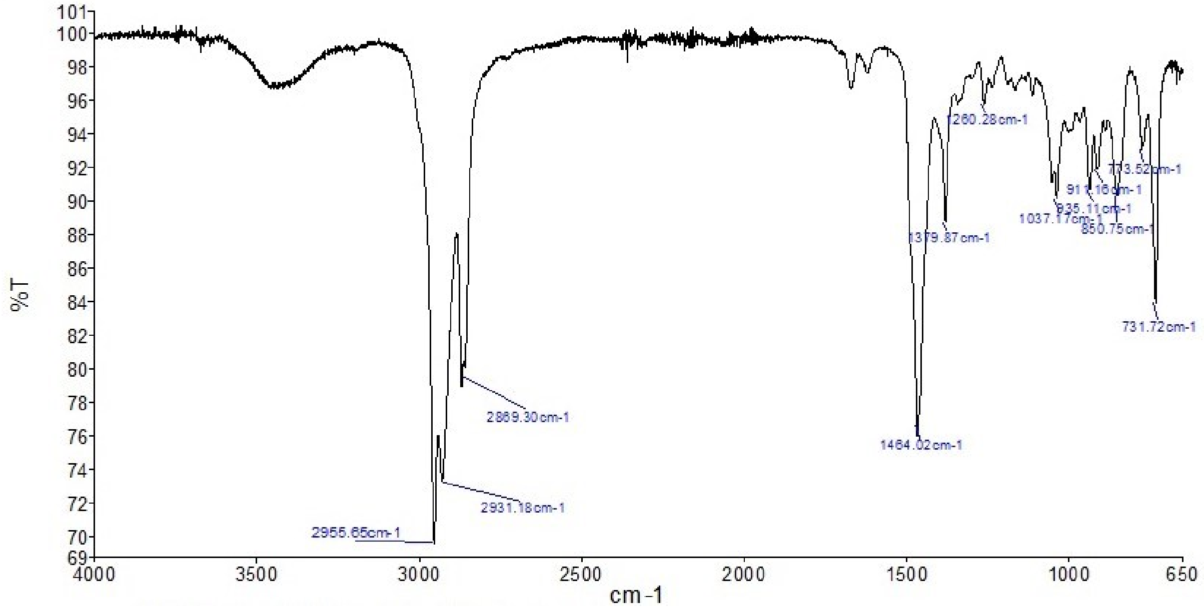
FT-IR spectrum of **TPA**



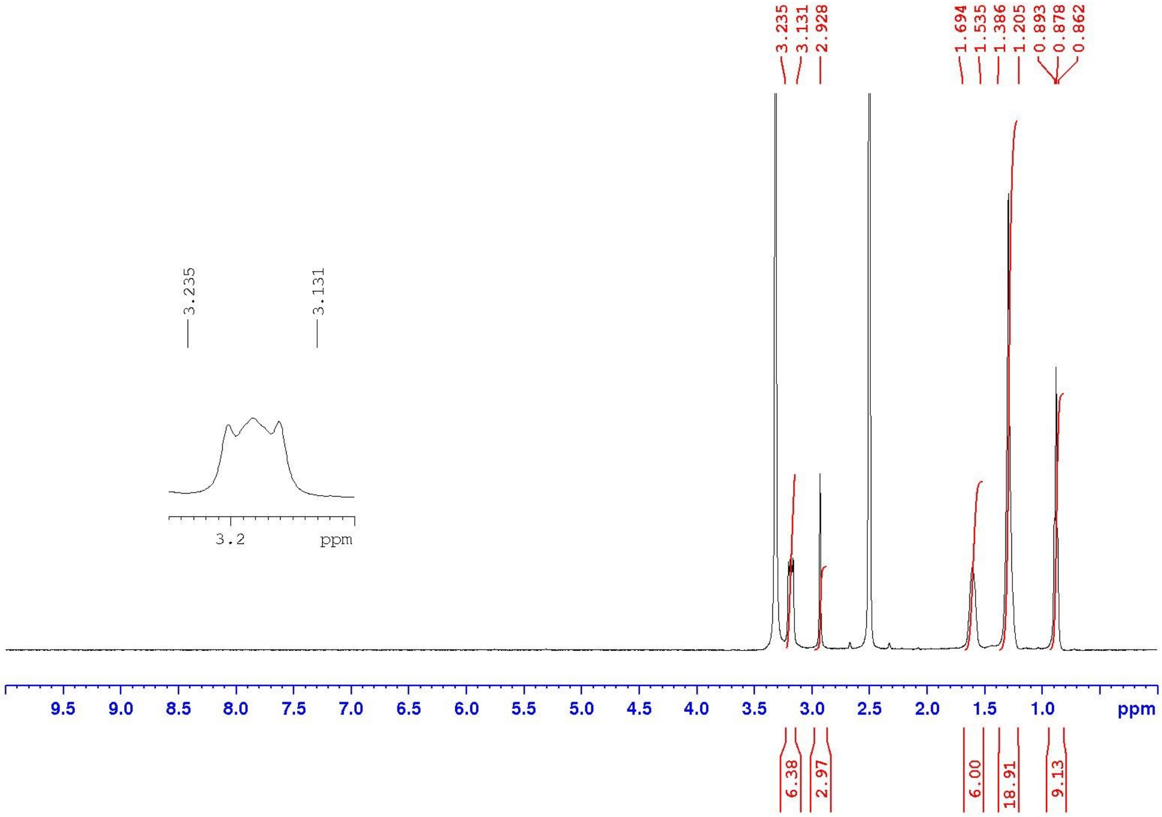
^{1}H NMR spectrum (400 MHz, DMSO-*d*6) of **11**



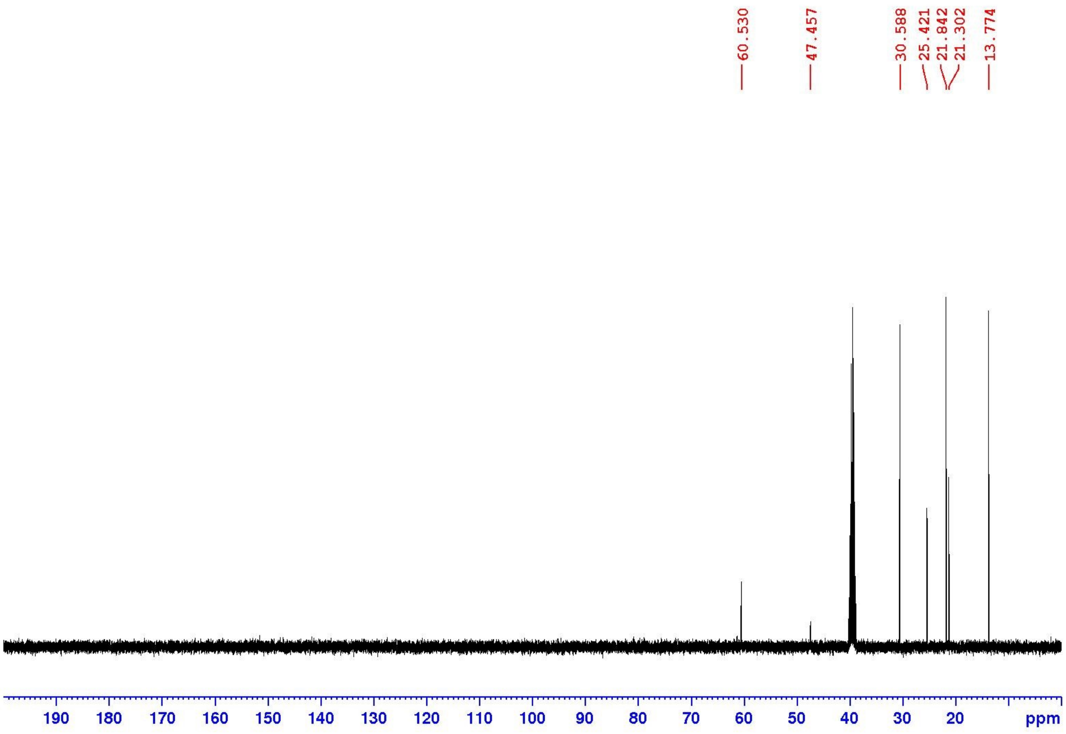
^{13}C NMR spectrum (101 MHz, DMSO-*d6*) of **11**



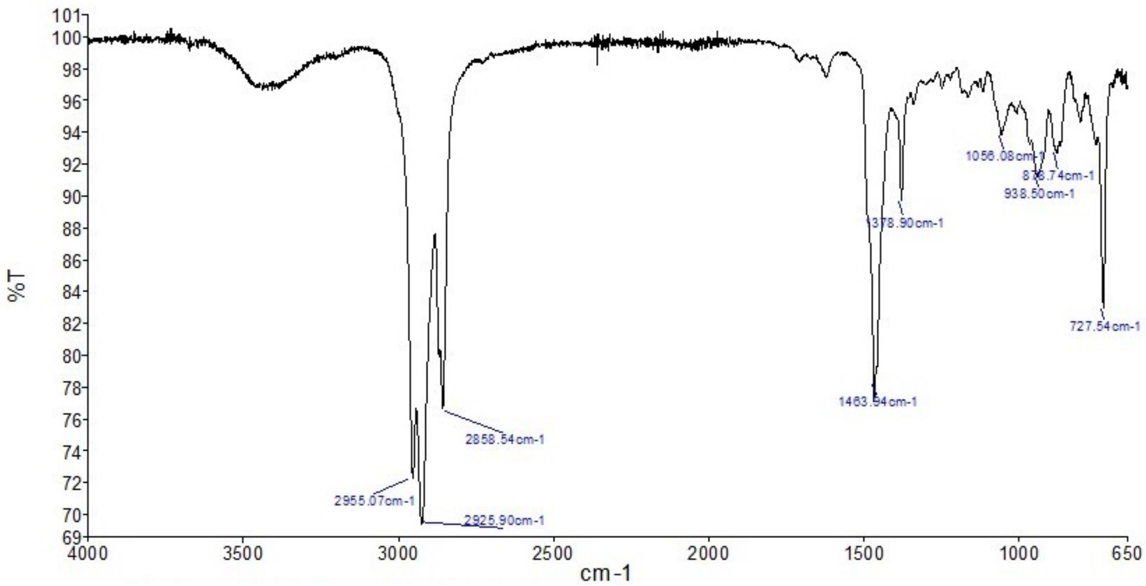
FT-IR spectrum of **11**



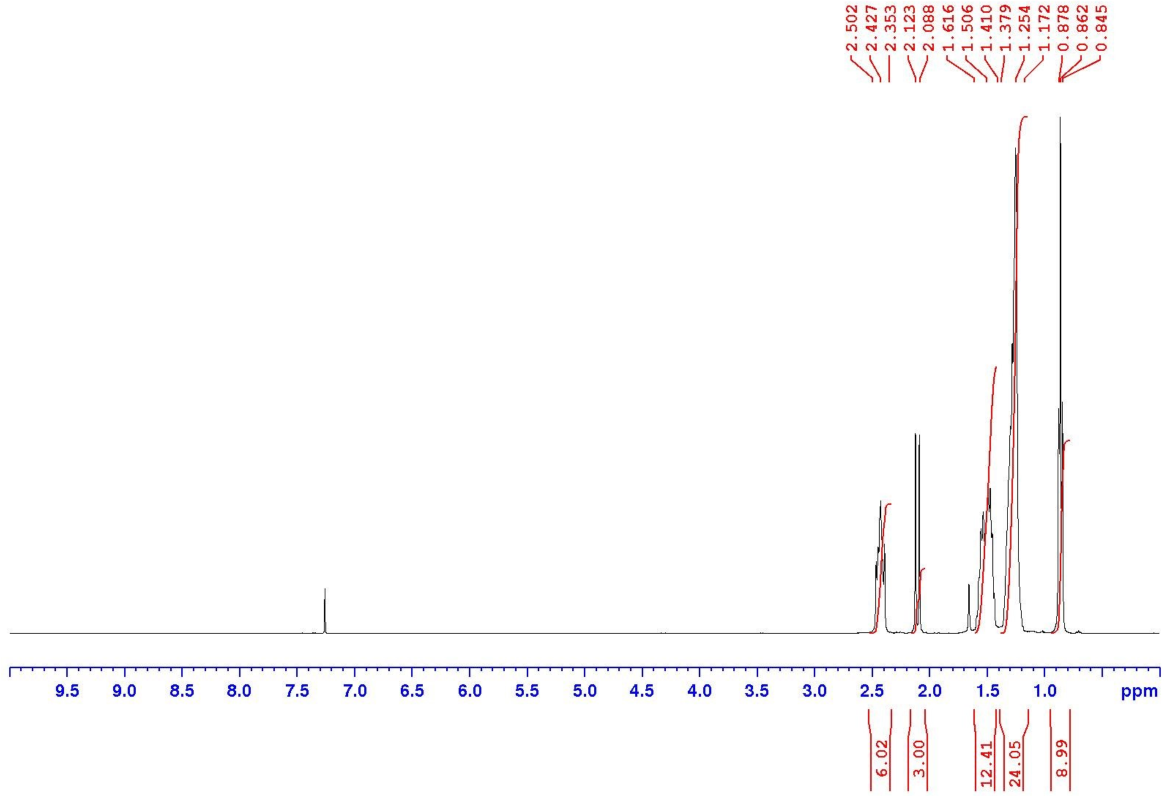
^{1}H NMR spectrum (400 MHz, DMSO-*d*6) of **12**



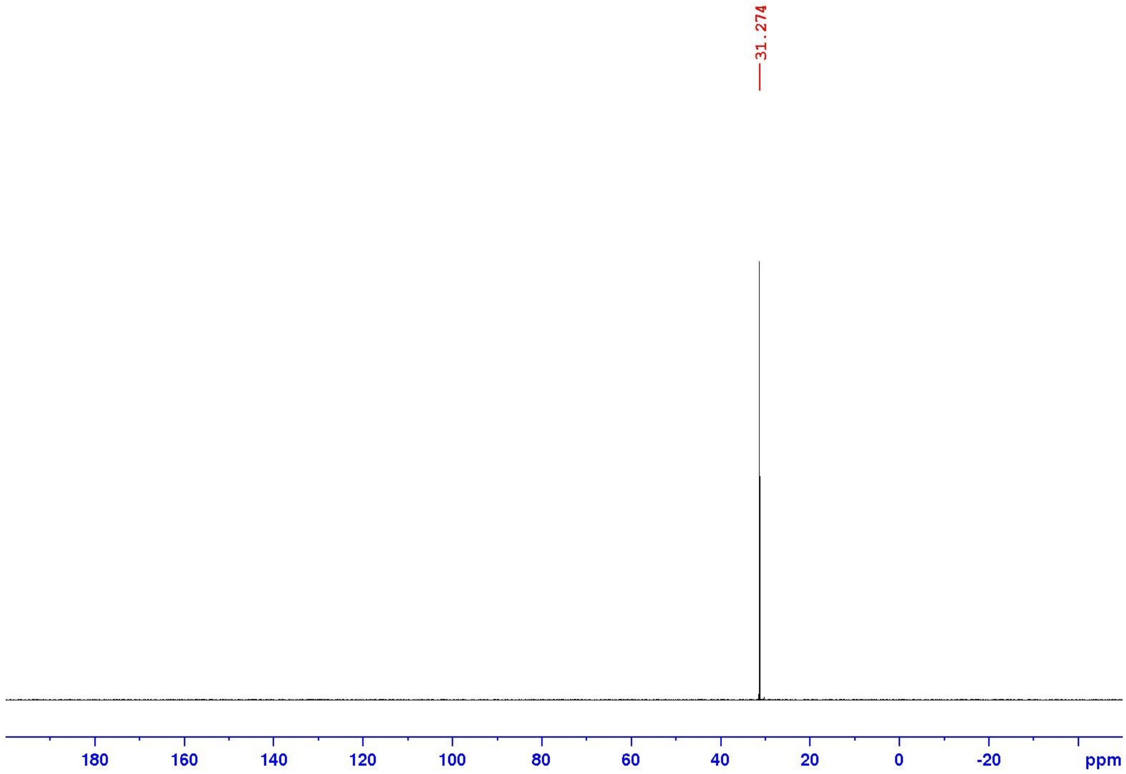
^{13}C NMR spectrum (101 MHz, DMSO-*d6*) of **12**



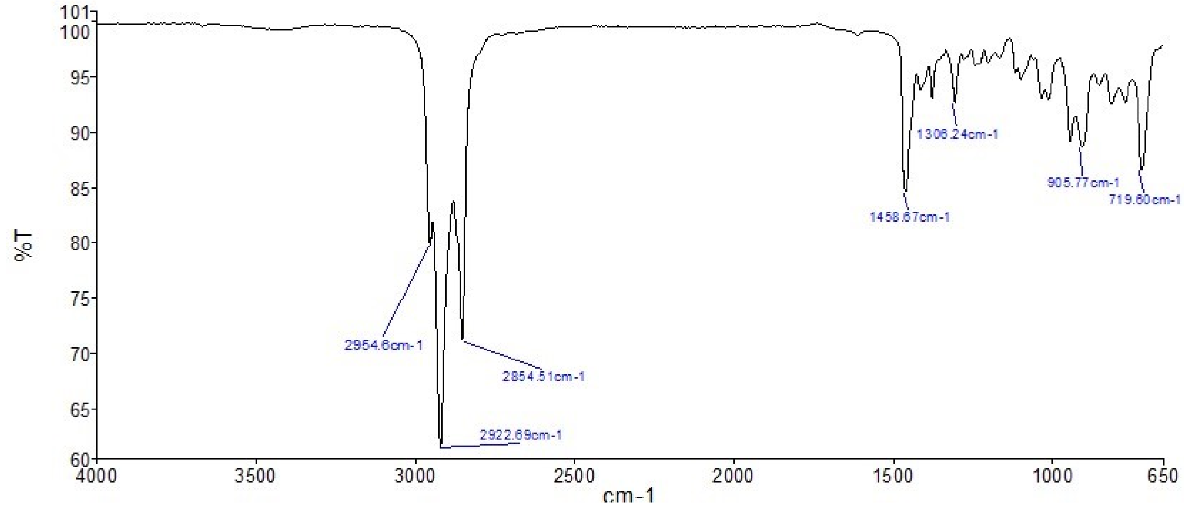
FT-IR spectrum of **12**



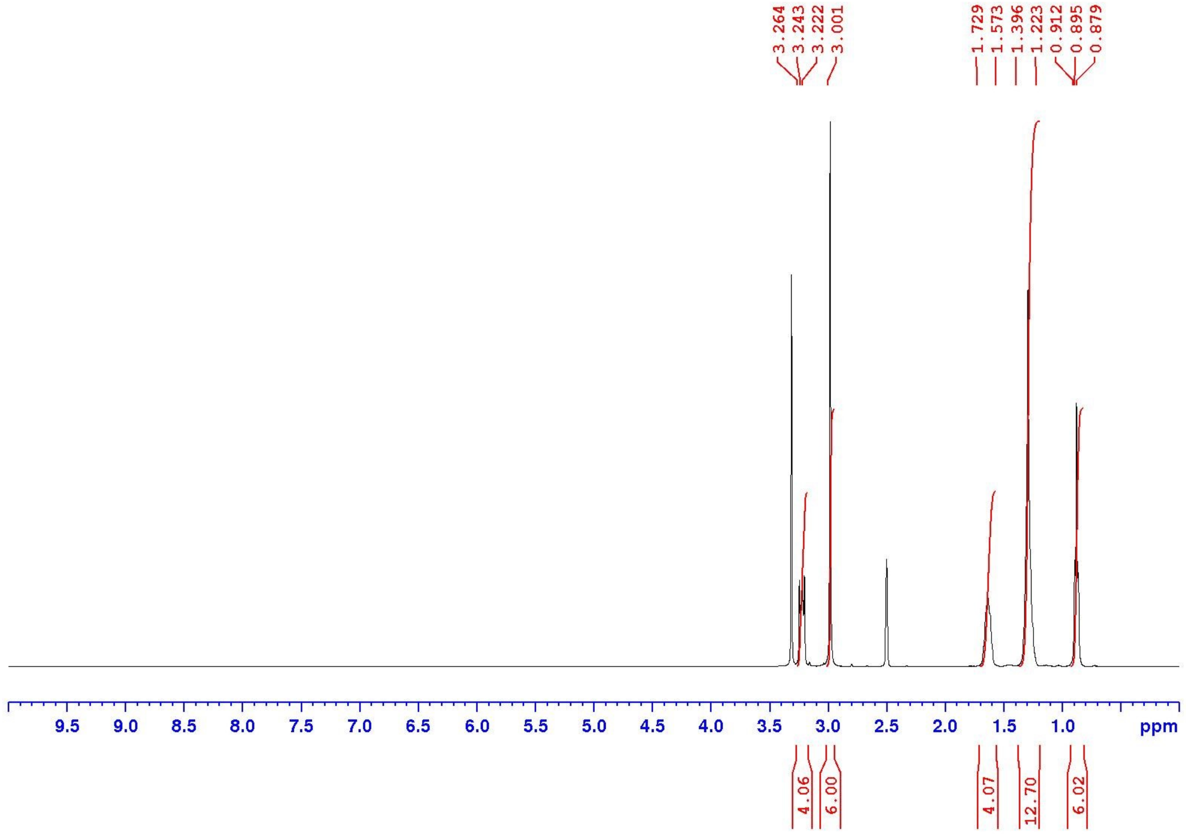
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **14**



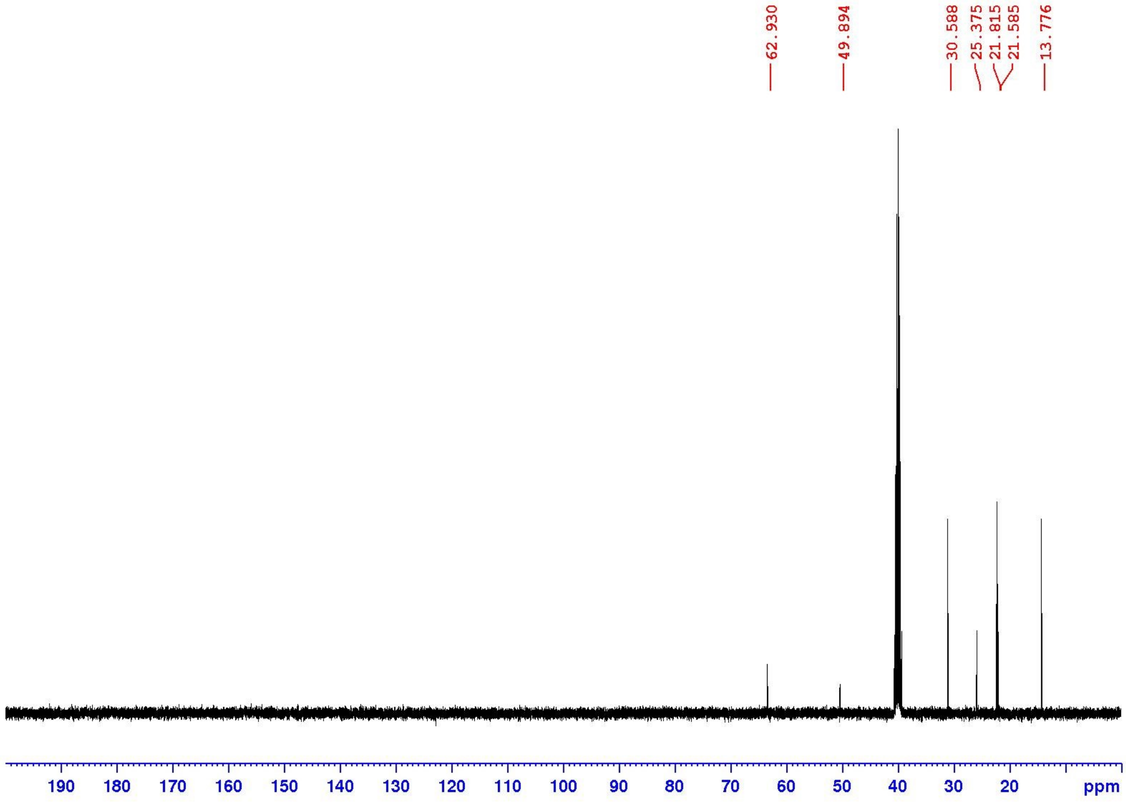
^{31}P NMR spectrum (162 MHz, CDCl$_3$) of **14**



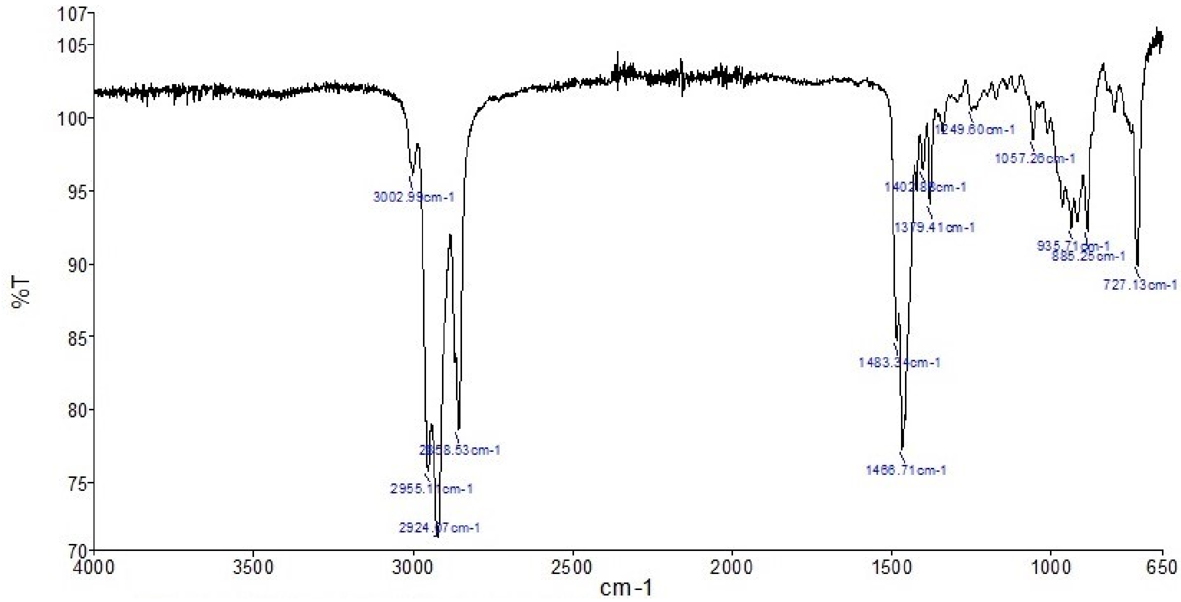
FT-IR spectrum of **14**



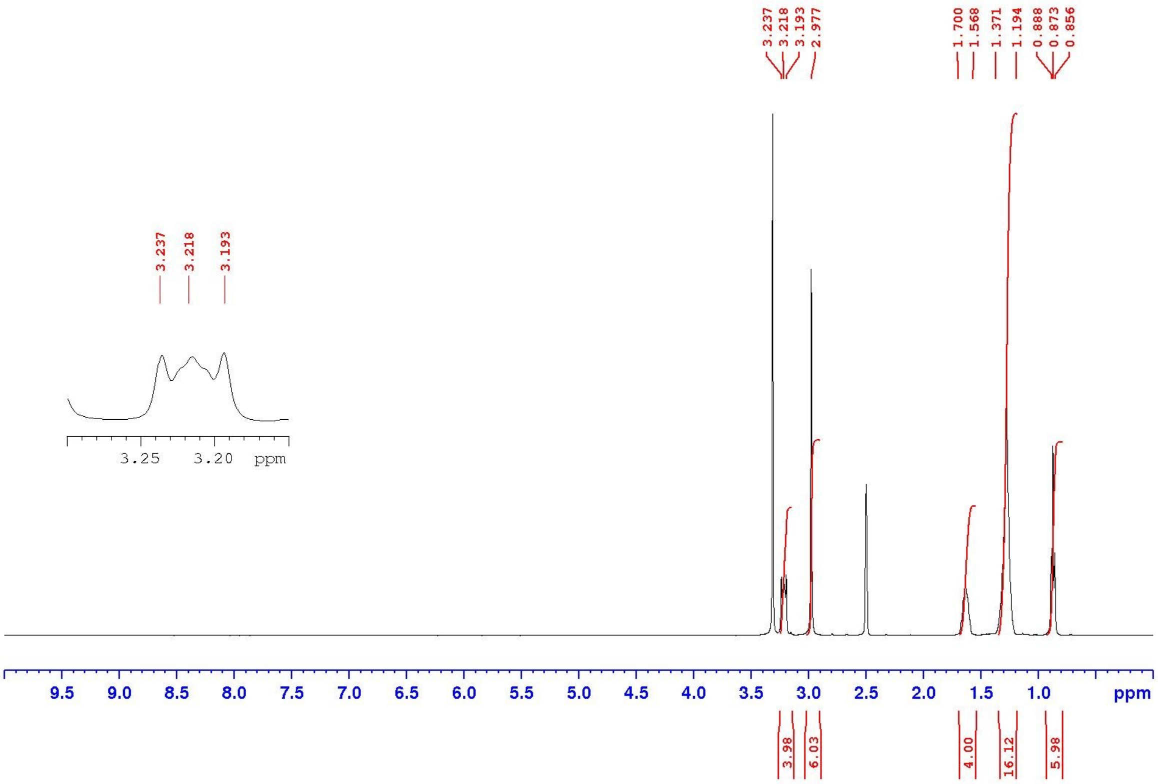
^{1}H NMR spectrum (400 MHz, DMSO-*d*6) of **16**



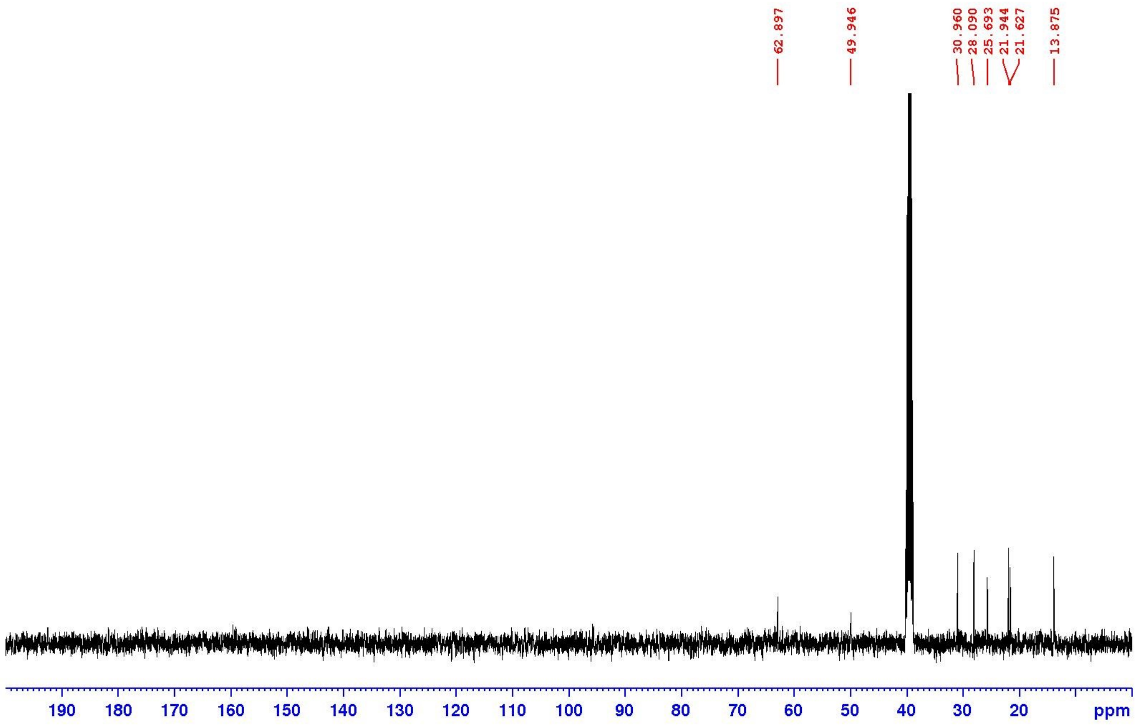
^{13}C NMR spectrum (101 MHz, DMSO-*d*6) of **16**



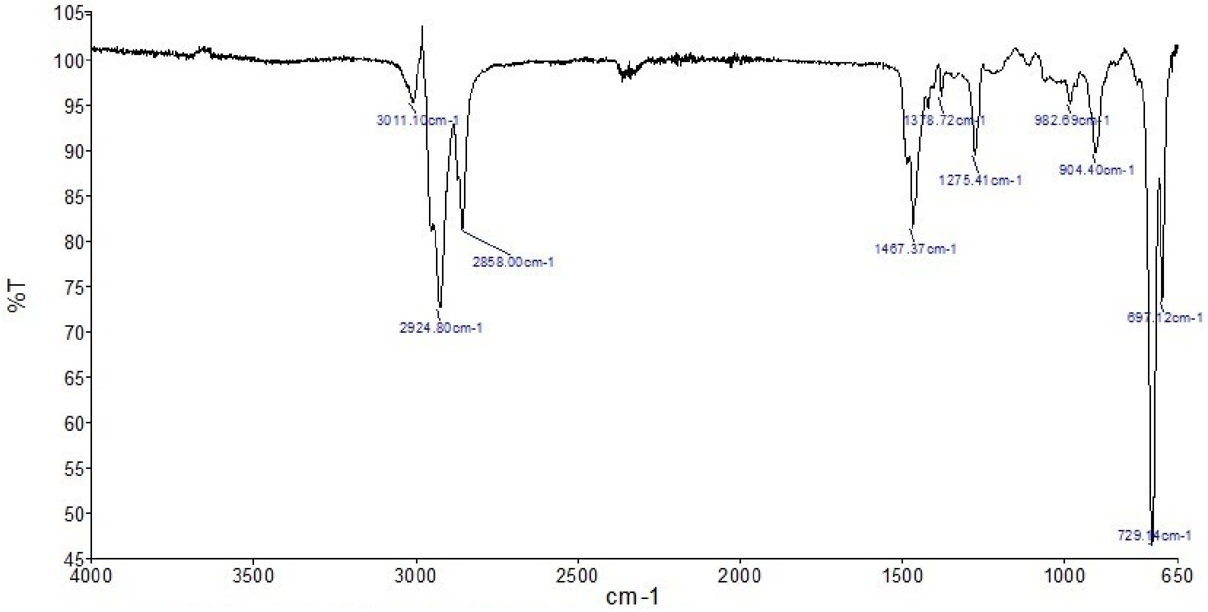
FT-IR spectrum of **16**



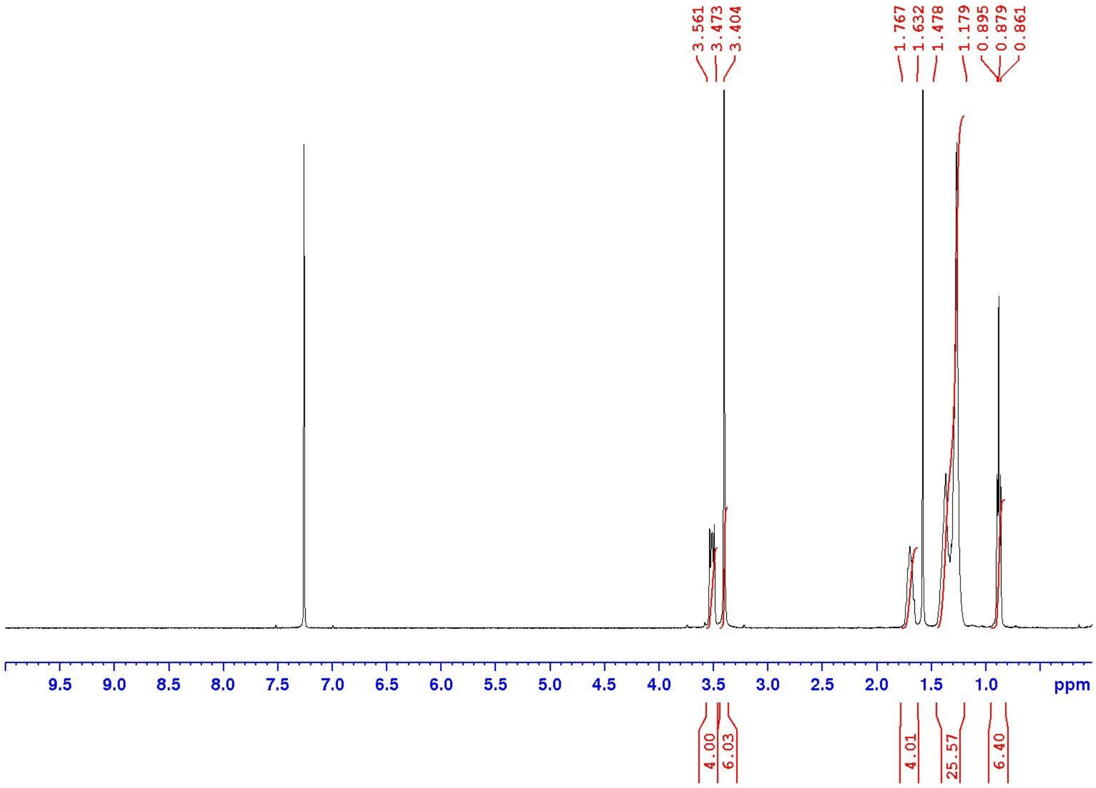
^{1}H NMR spectrum (400 MHz, DMSO-*d*6) of **17**



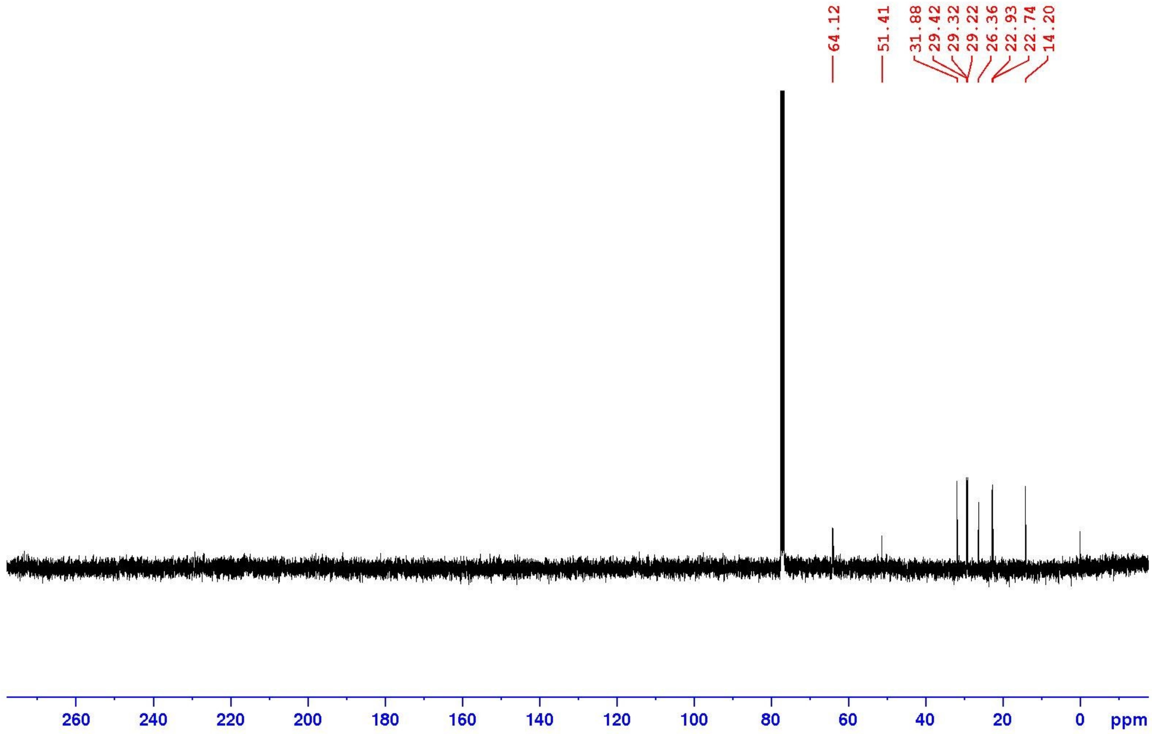
^{13}C NMR spectrum (101 MHz, DMSO-*d*6) of **17**



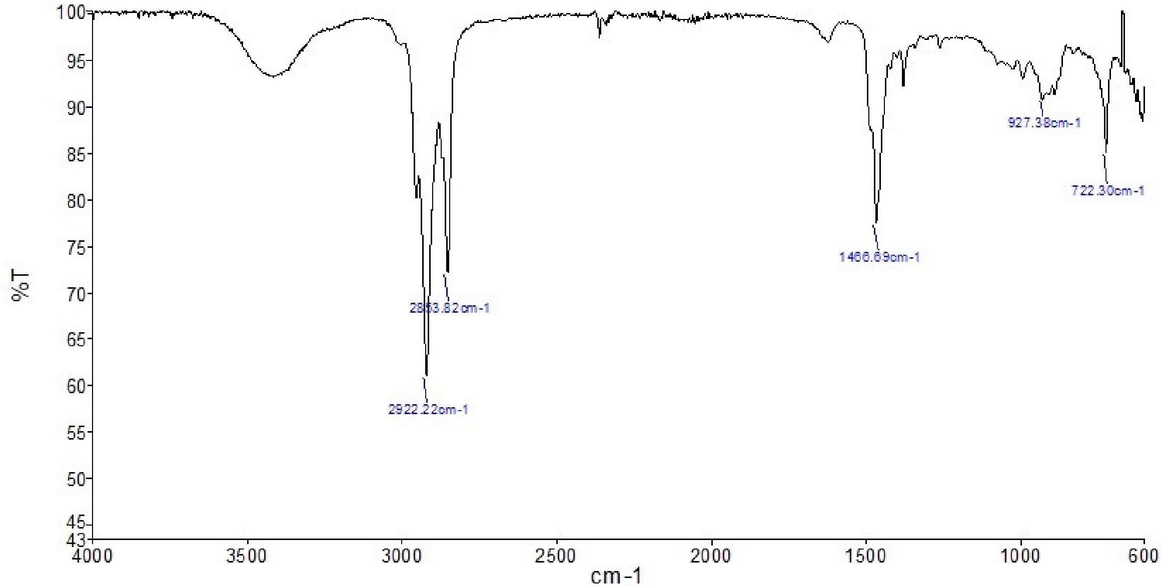
FT-IR spectrum of **17**



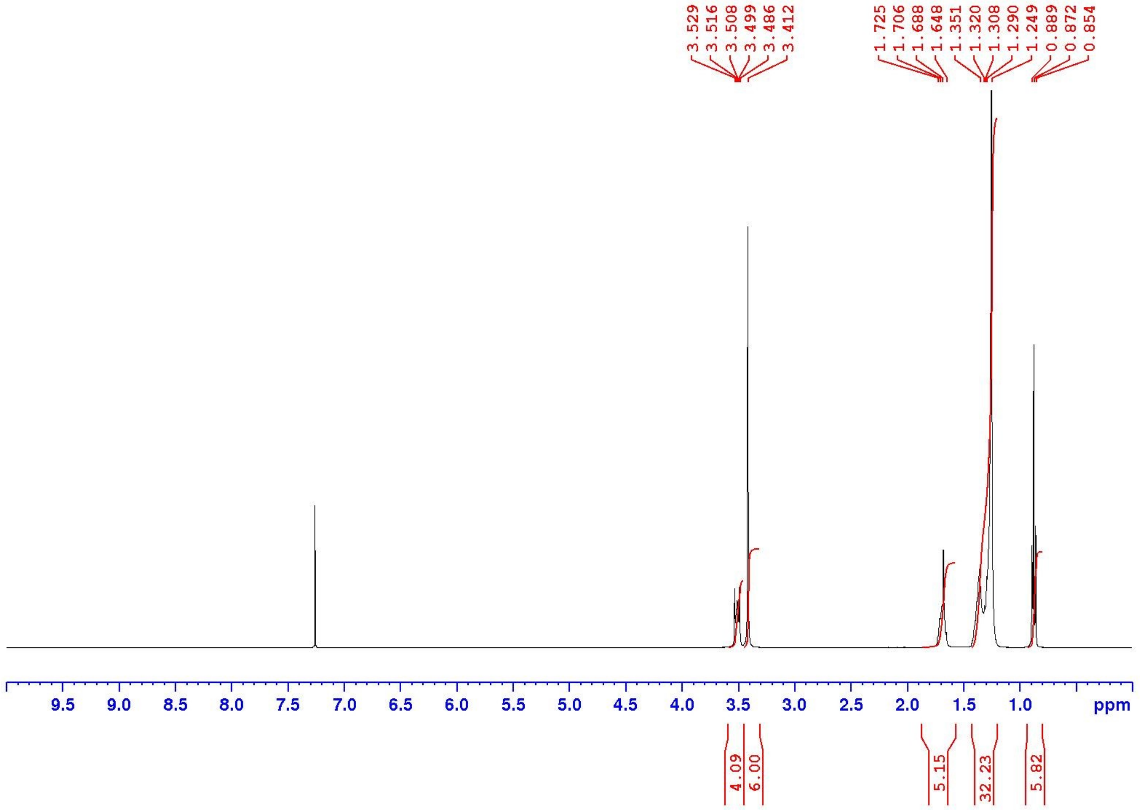
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **19**



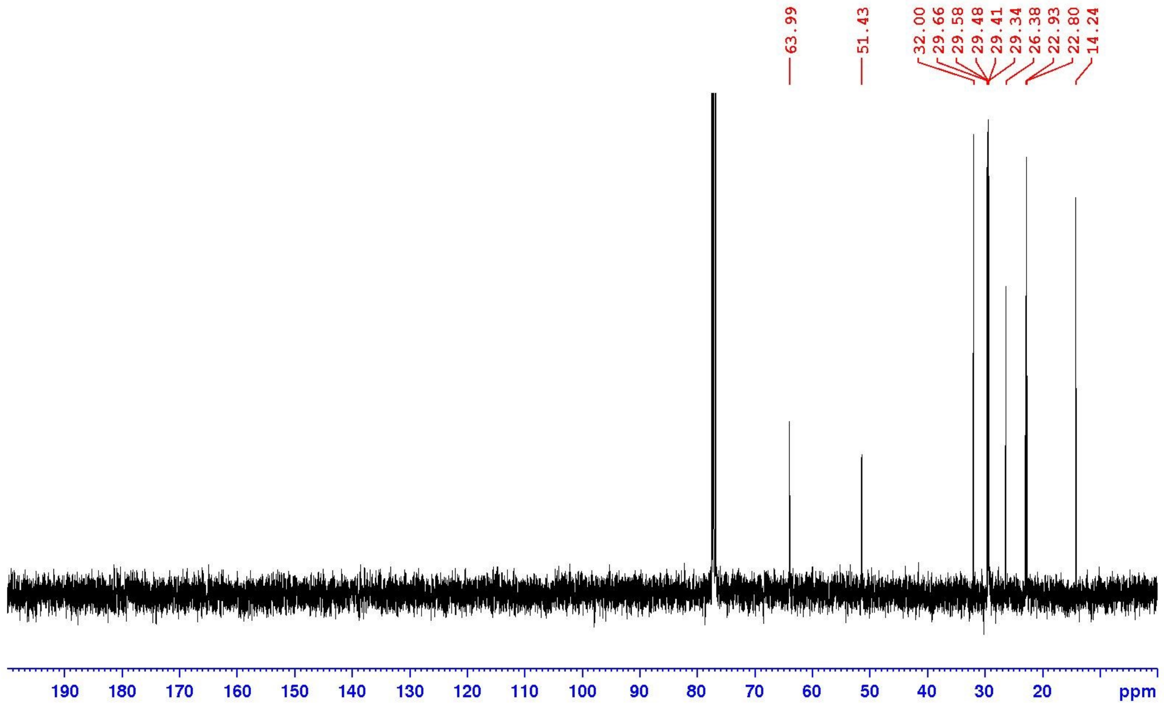
^{13}C NMR spectrum (400 MHz, CDCl$_3$) of **19**



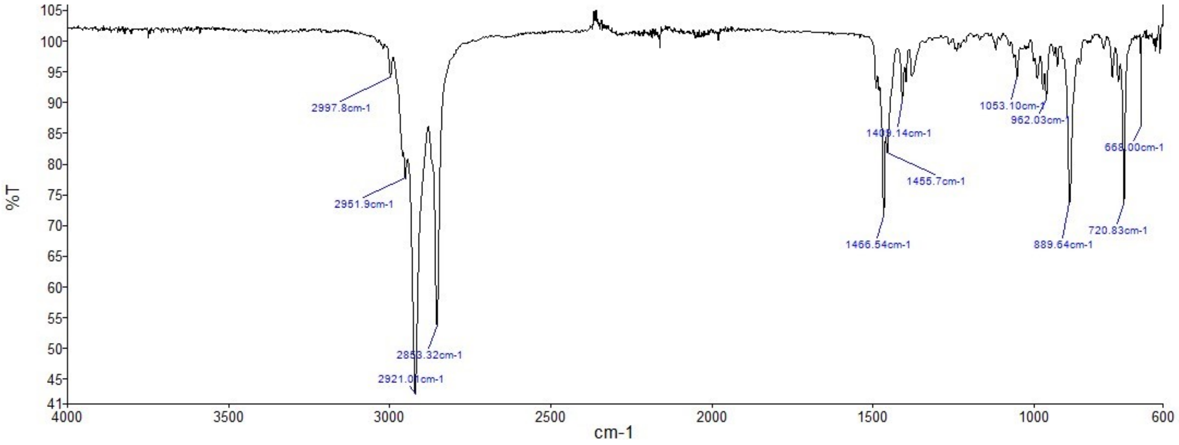
FT-IR spectrum of **19**



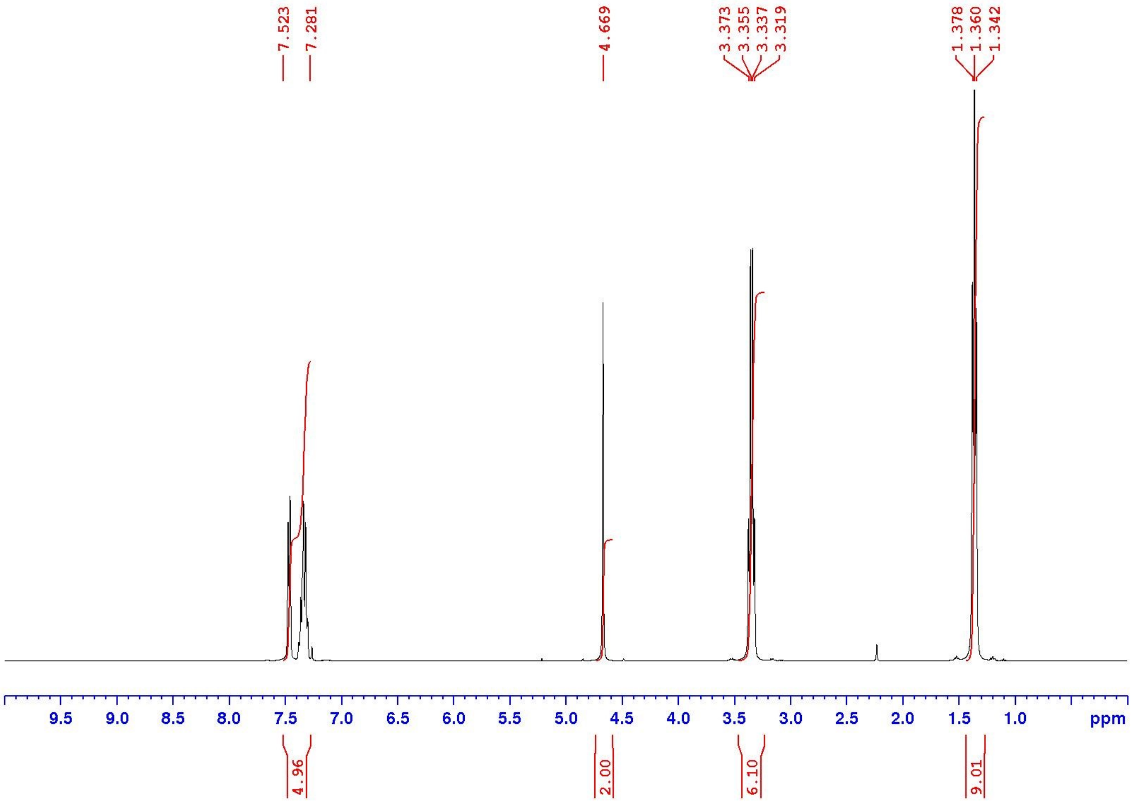
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **20**



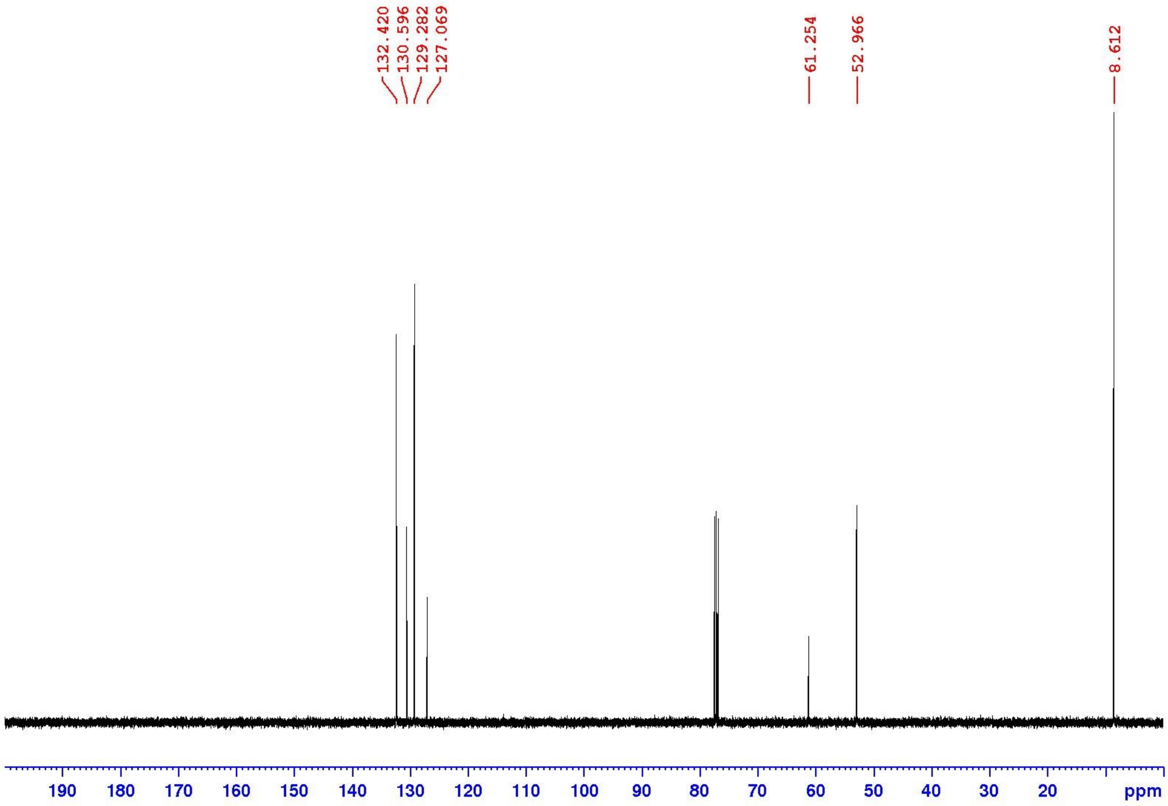
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **20**



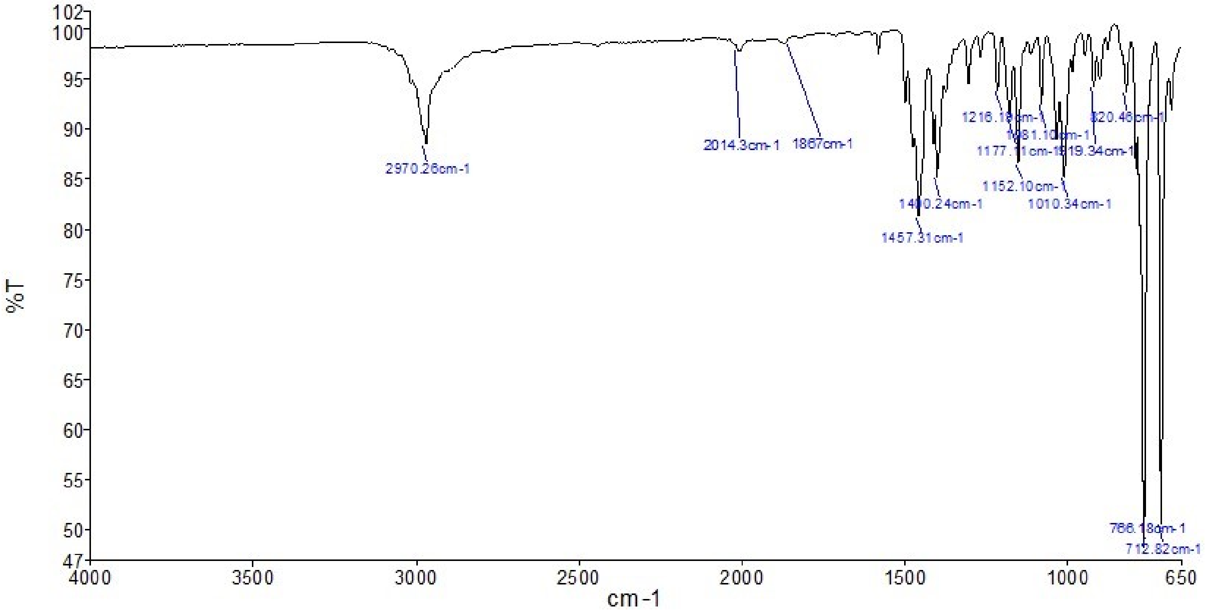
FT-IR spectrum of **20**



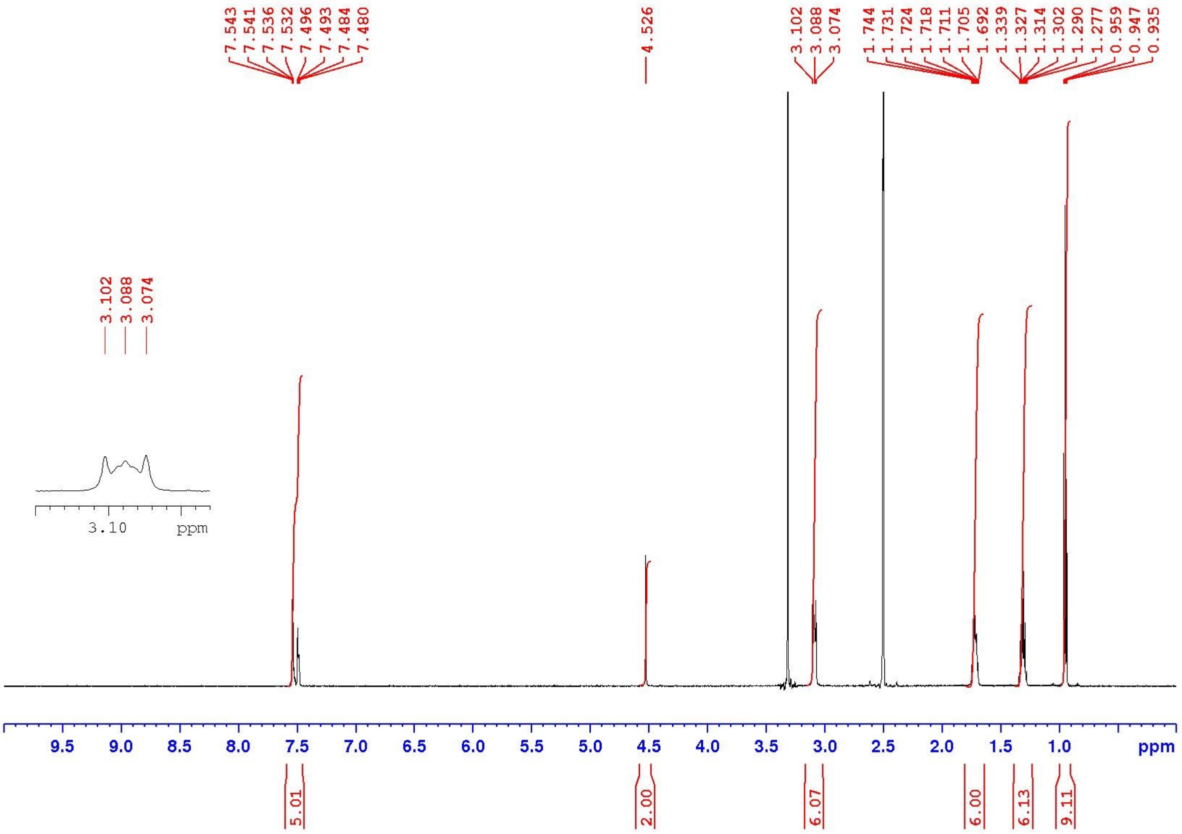
^{1}H NMR spectrum (400 MHz, DMSO-*d*6) of **31**



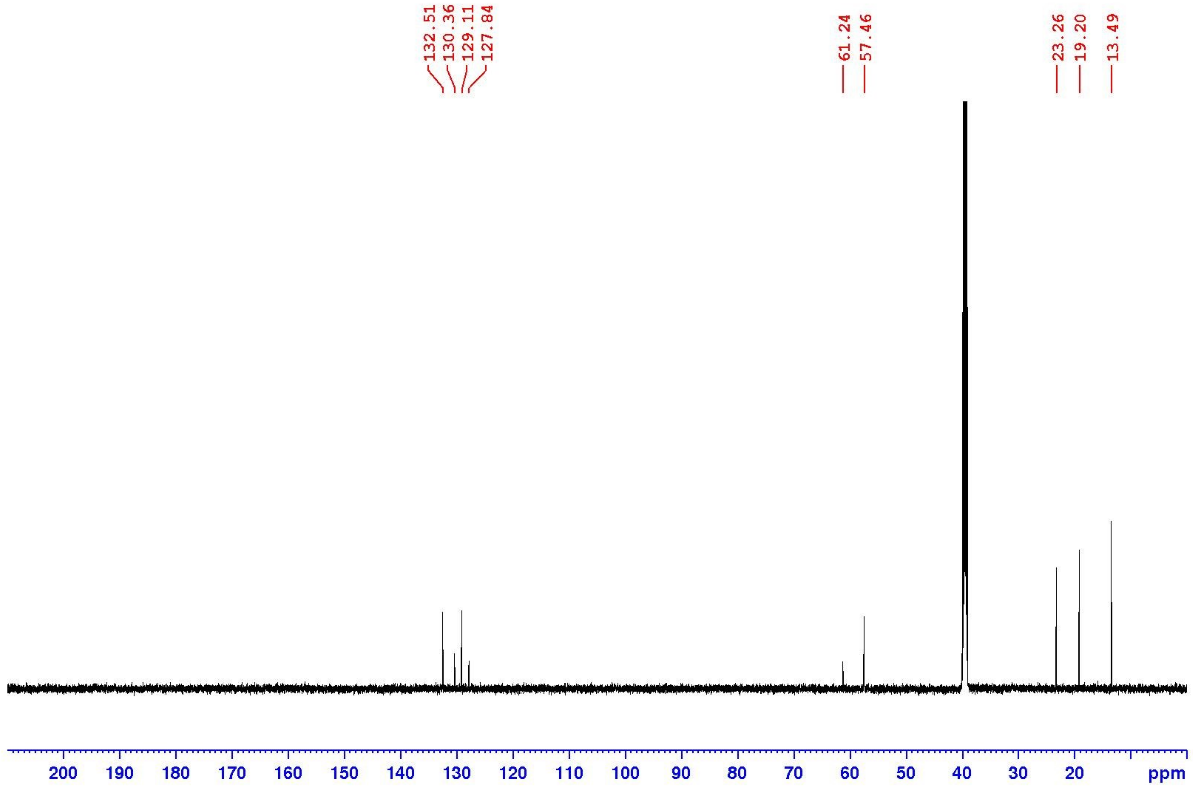
^{13}C NMR spectrum (101 MHz, DMSO-*d6*) of **31**



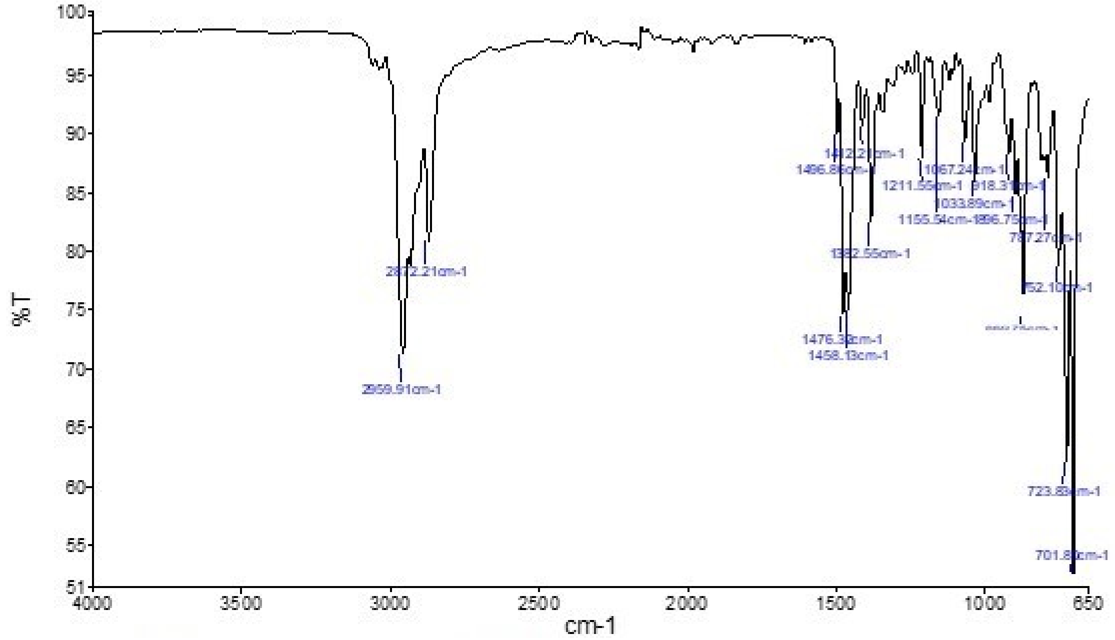
FT-IR spectrum of **31**



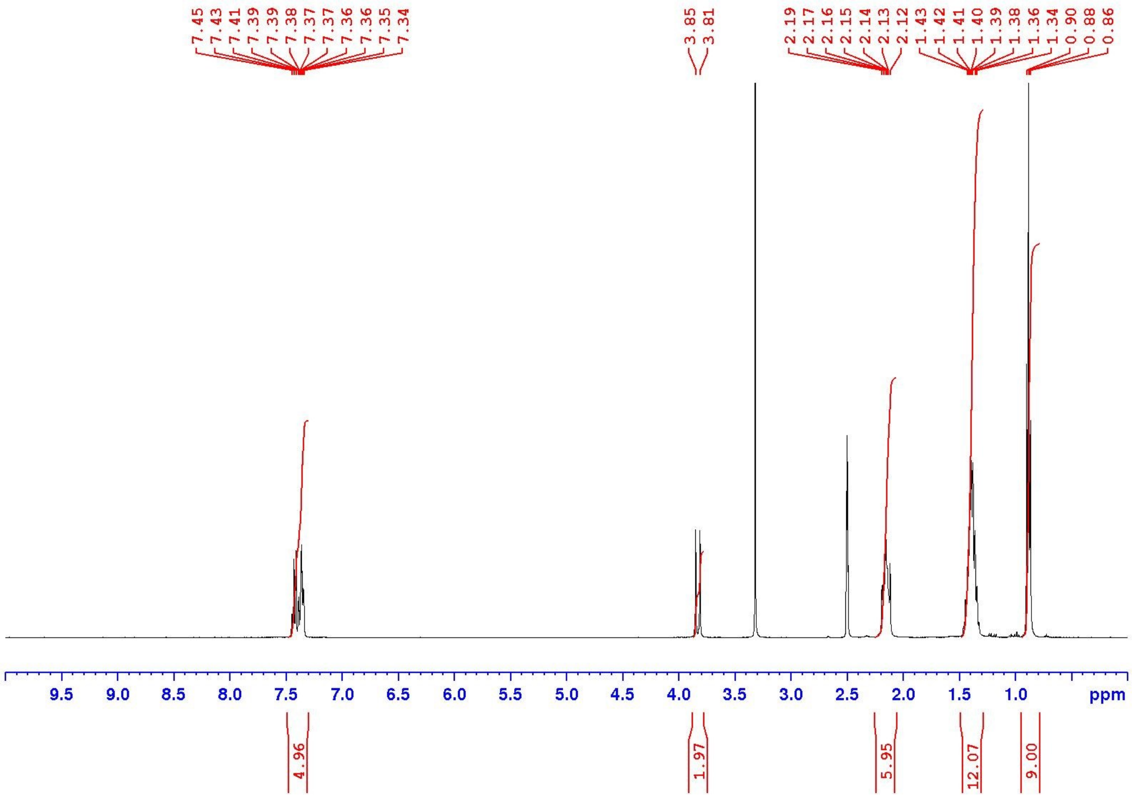
^{1}H NMR spectrum (400 MHz, DMSO-*d*6) of **32**



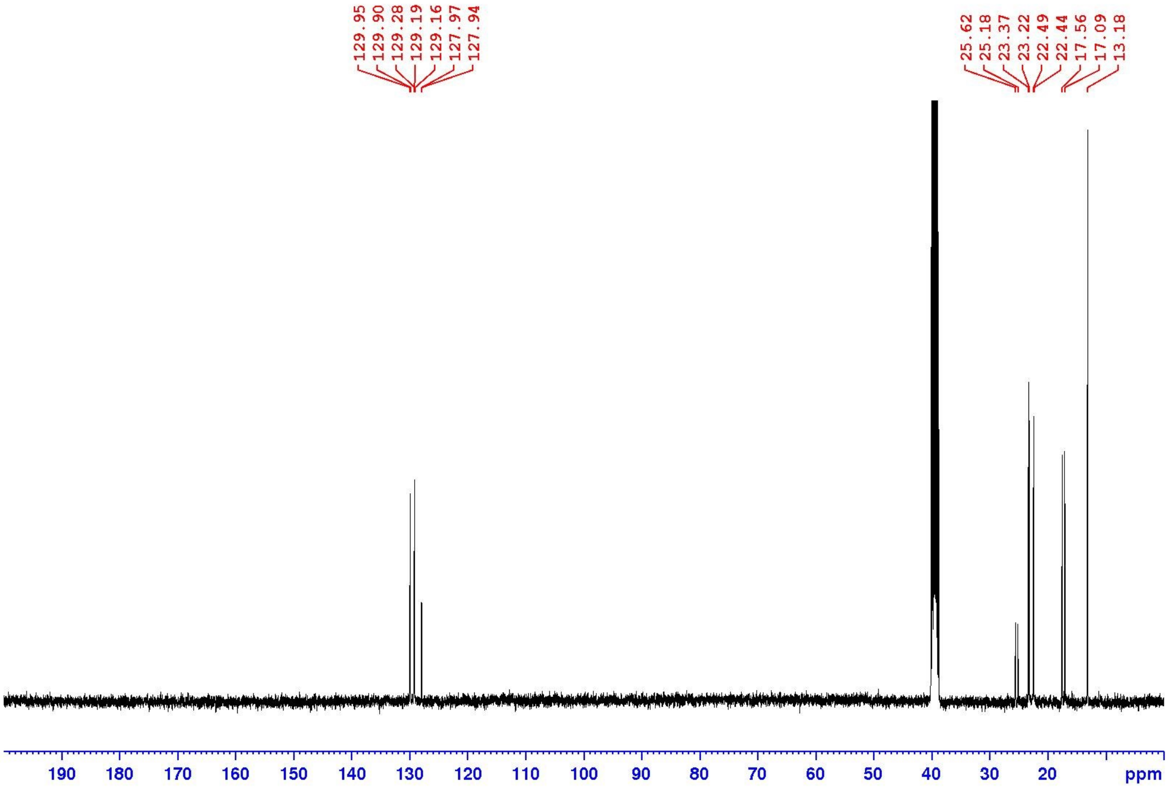
^{13}C NMR spectrum (101 MHz, DMSO-*d*6) of **32**



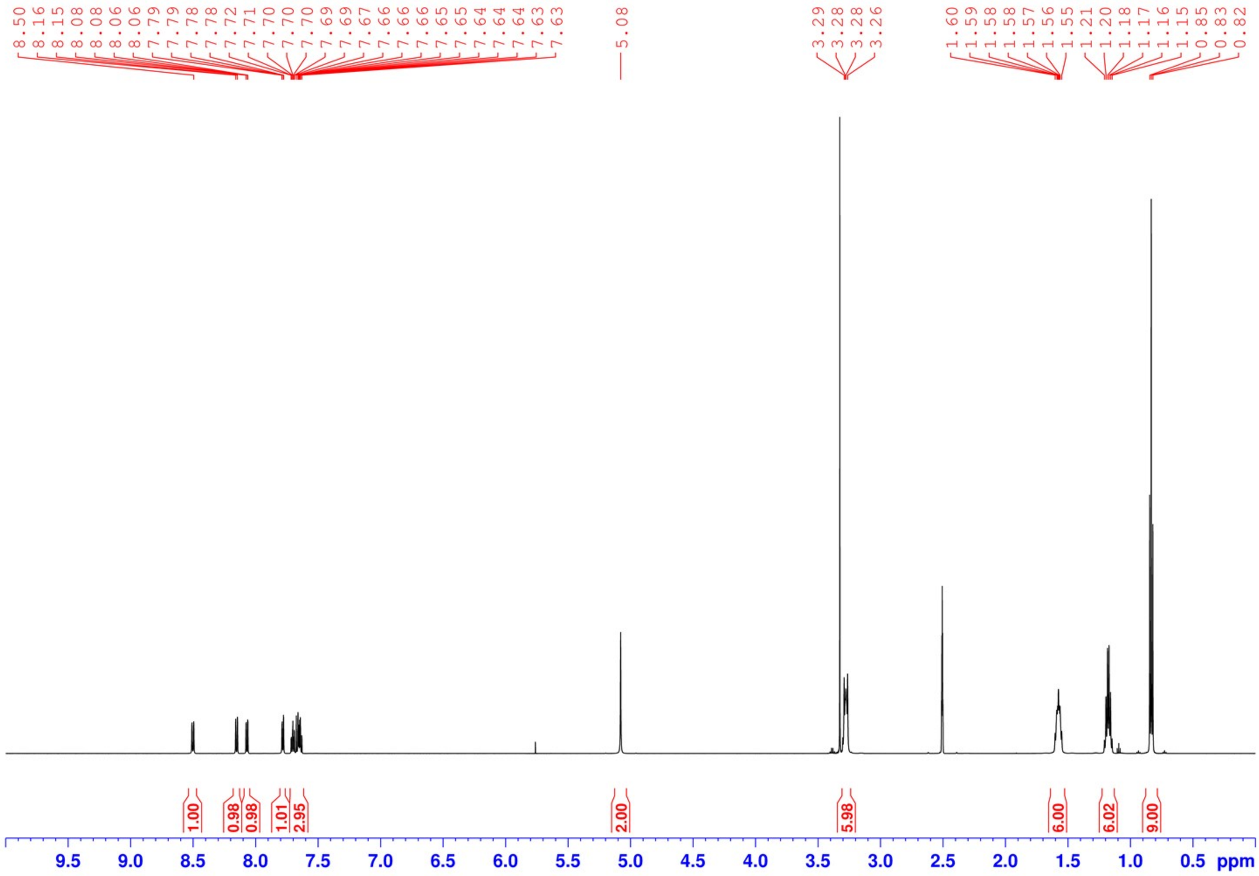
FT-IR spectrum of **32**



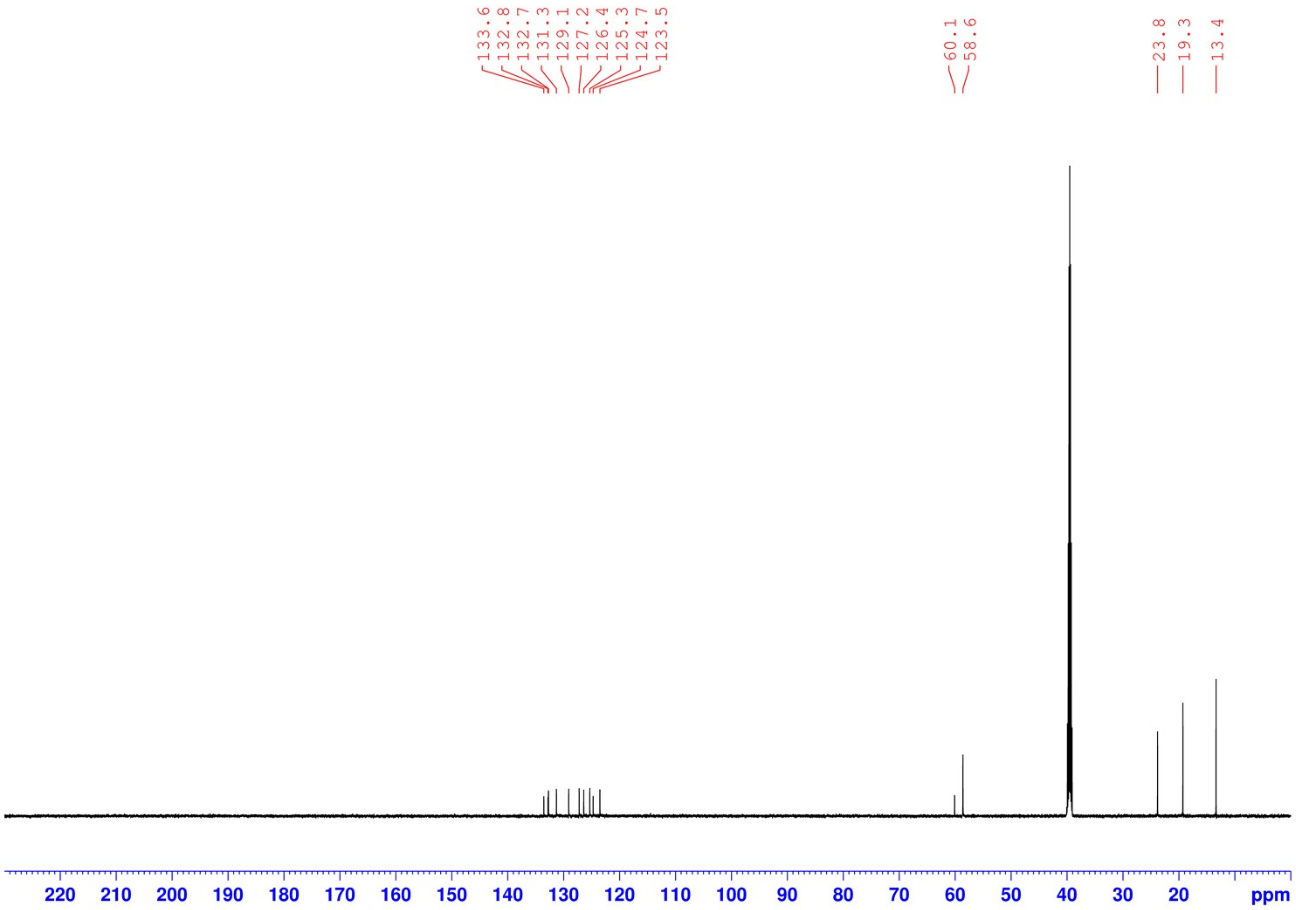
^{1}H NMR spectrum (400 MHz, DMSO-*d*6) of **33**



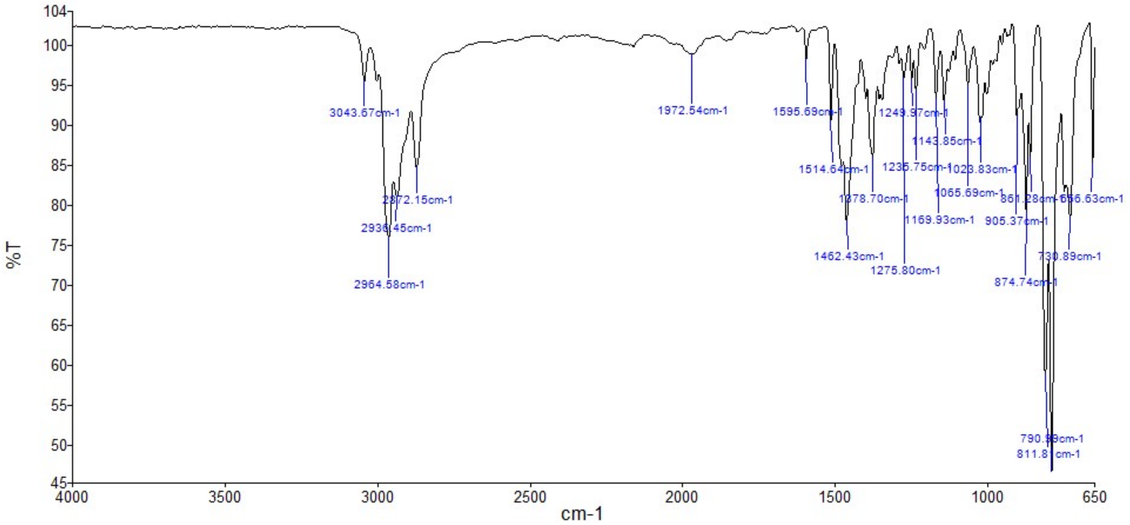
^{13}C NMR spectrum (101 MHz, DMSO-*d6*) of **33**



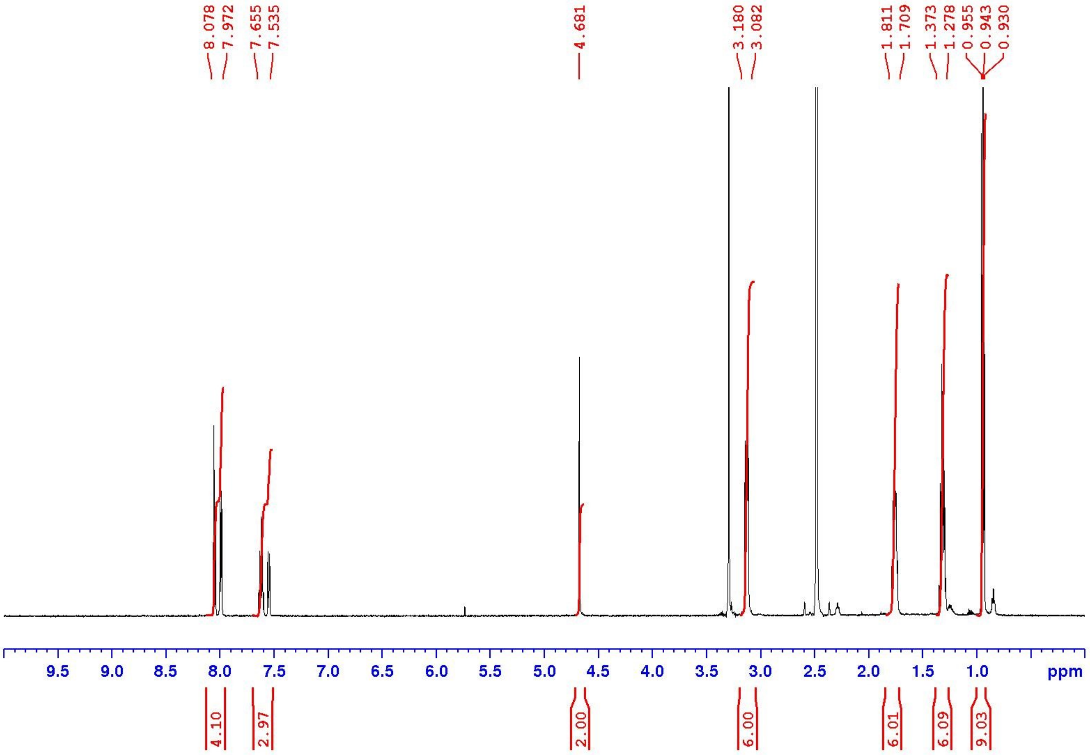
^{1}H NMR spectrum (400 MHz, DMSO-*d*6) of **34**



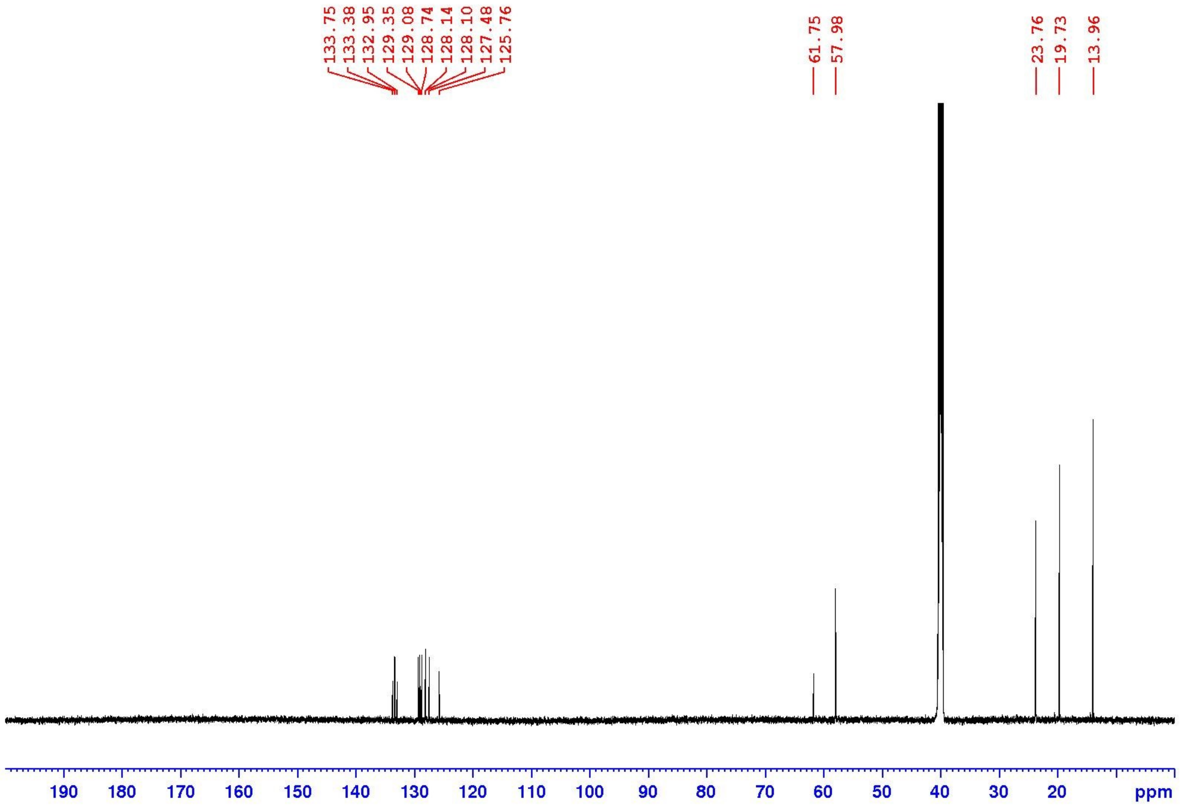
^{13}C NMR spectrum (101 MHz, DMSO-*d*6) of **34**



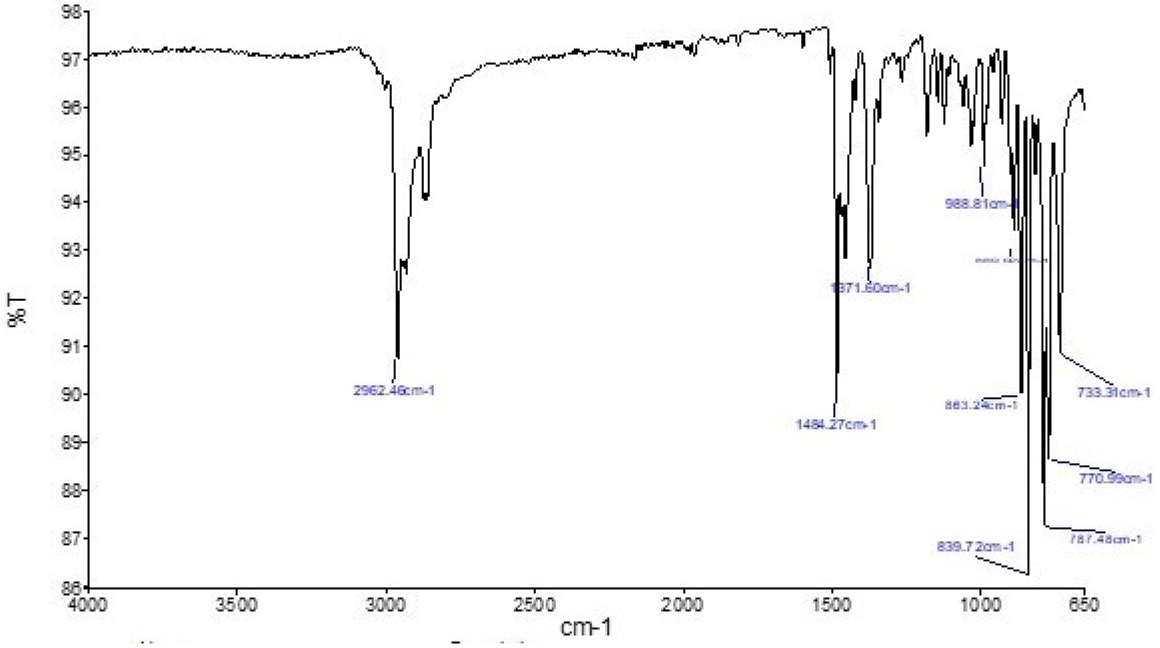
FT-IR spectrum of **34**



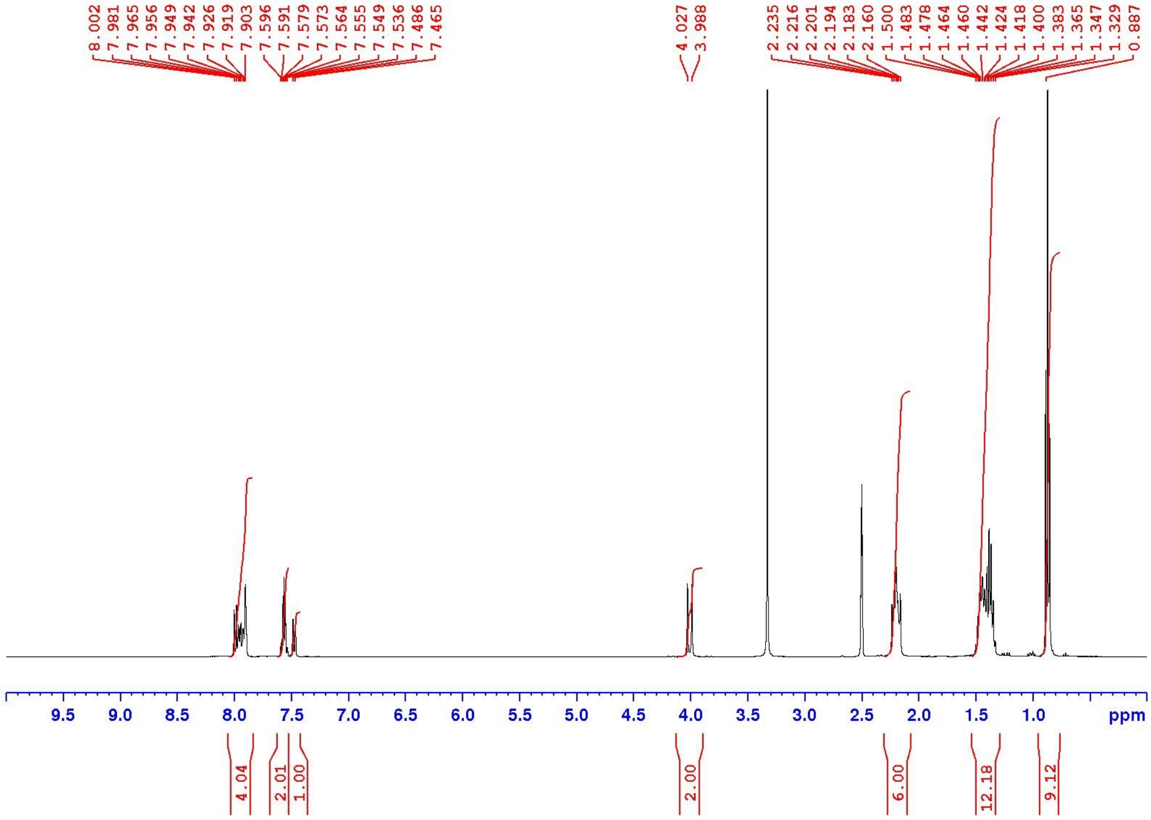
^{1}H NMR spectrum (600 MHz, DMSO-*d*6) of **35**



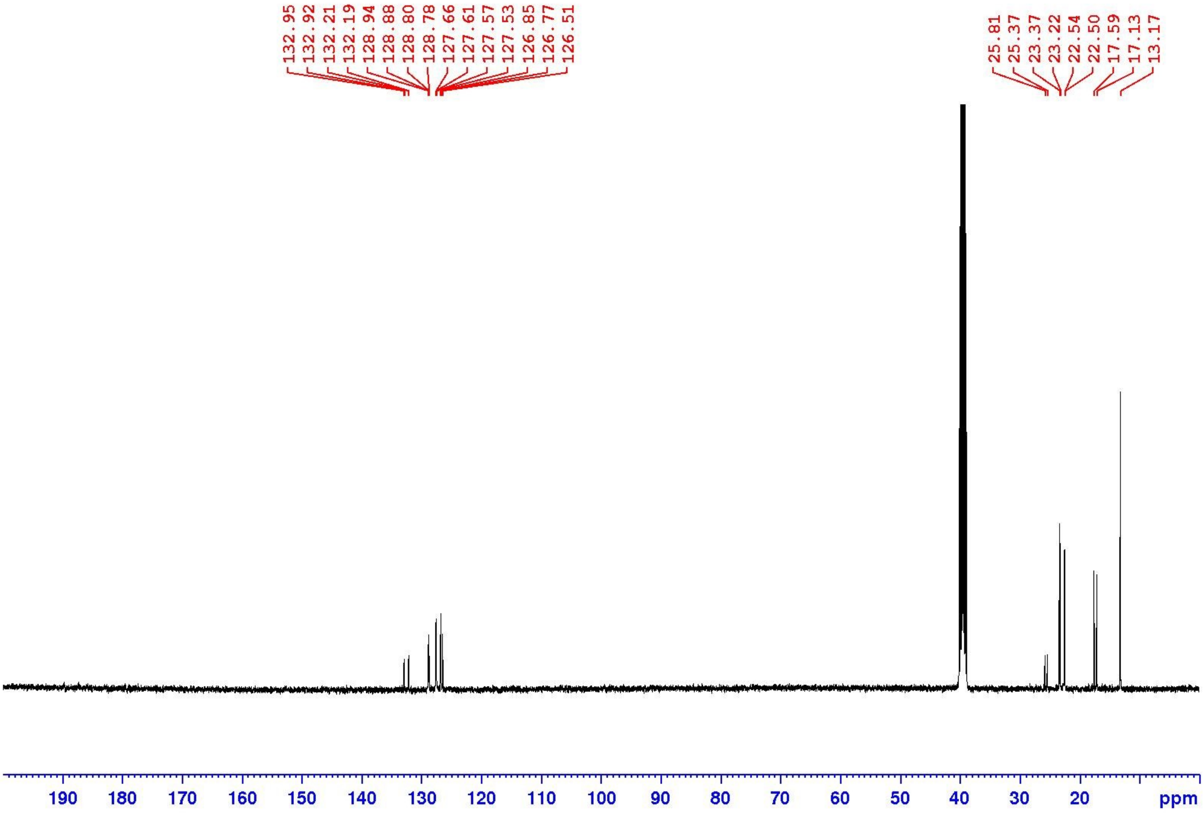
^{13}C NMR spectrum (101 MHz, DMSO-*d*6) of **35**



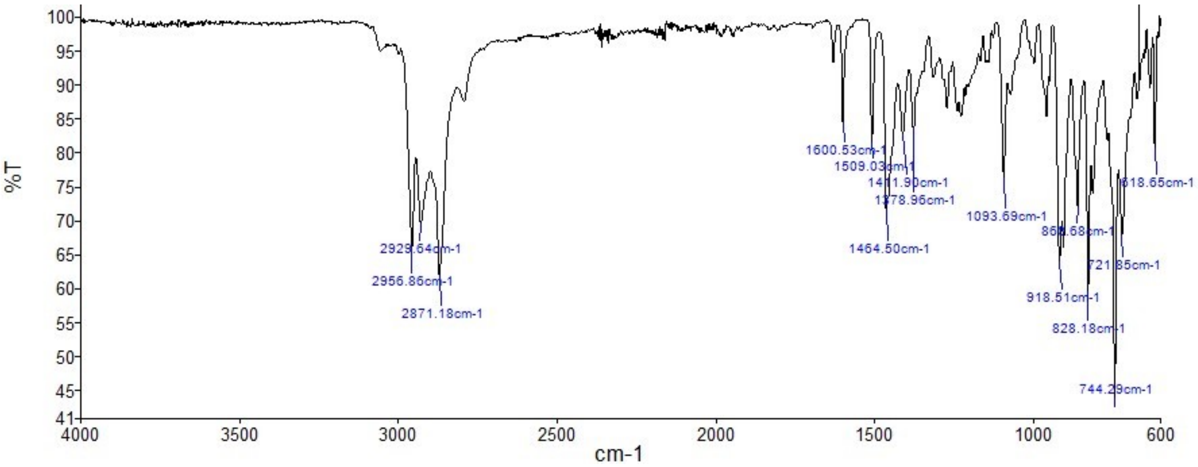
FT-IR spectrum of **35**



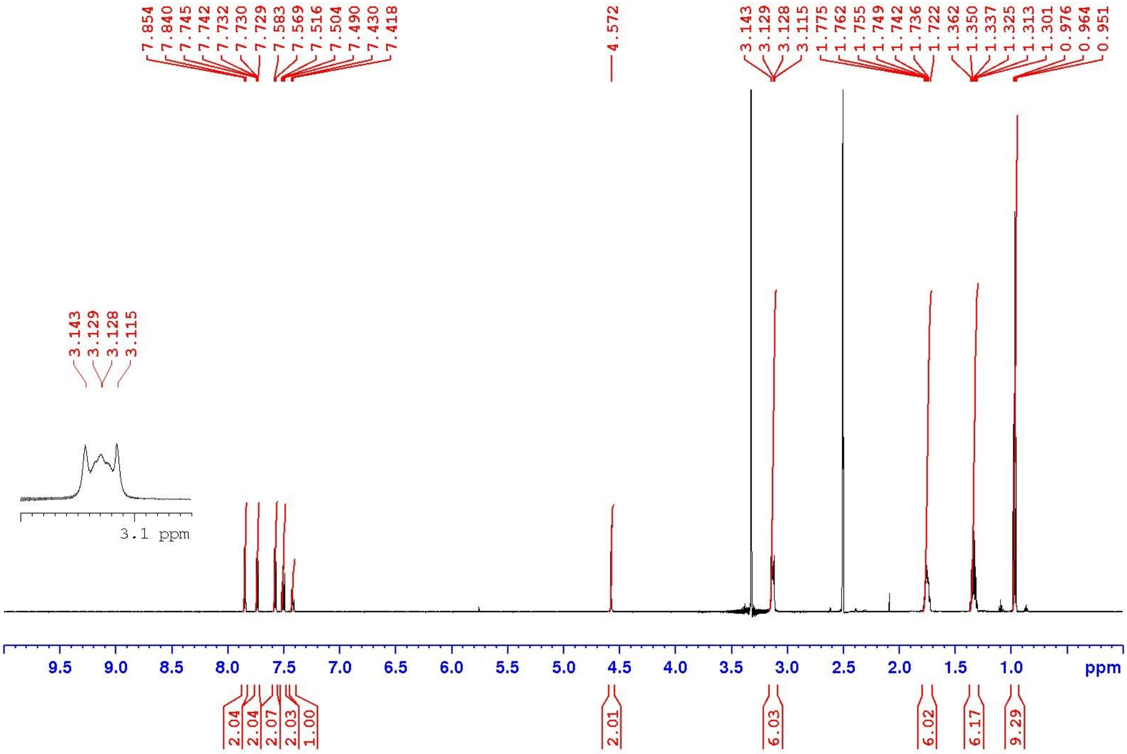
^{1}H NMR spectrum (600 MHz, DMSO-*d*6) of **36**



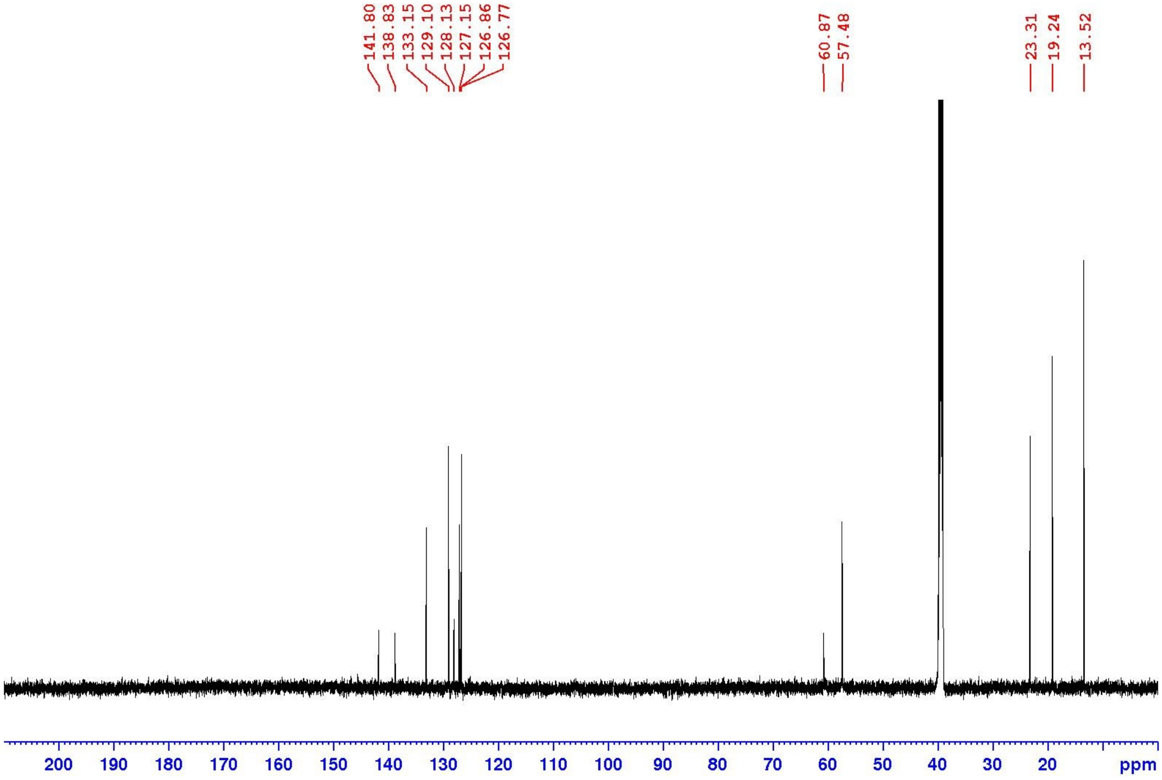
^{13}C NMR spectrum (151 MHz, DMSO-*d6*) of **36**



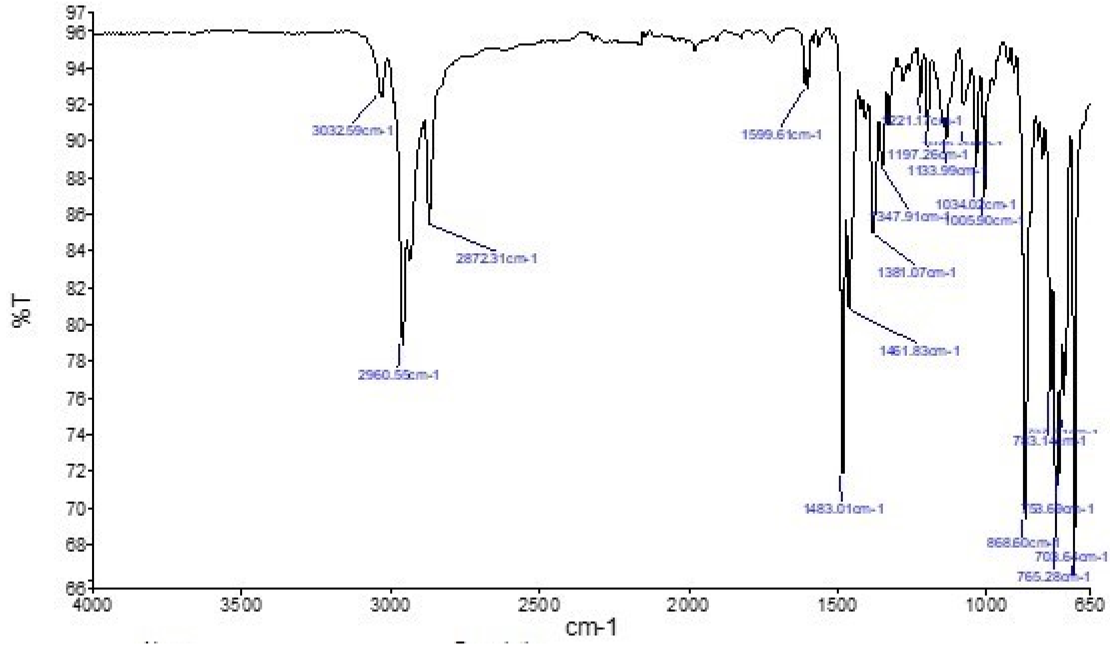
FT-IR spectrum of **36**



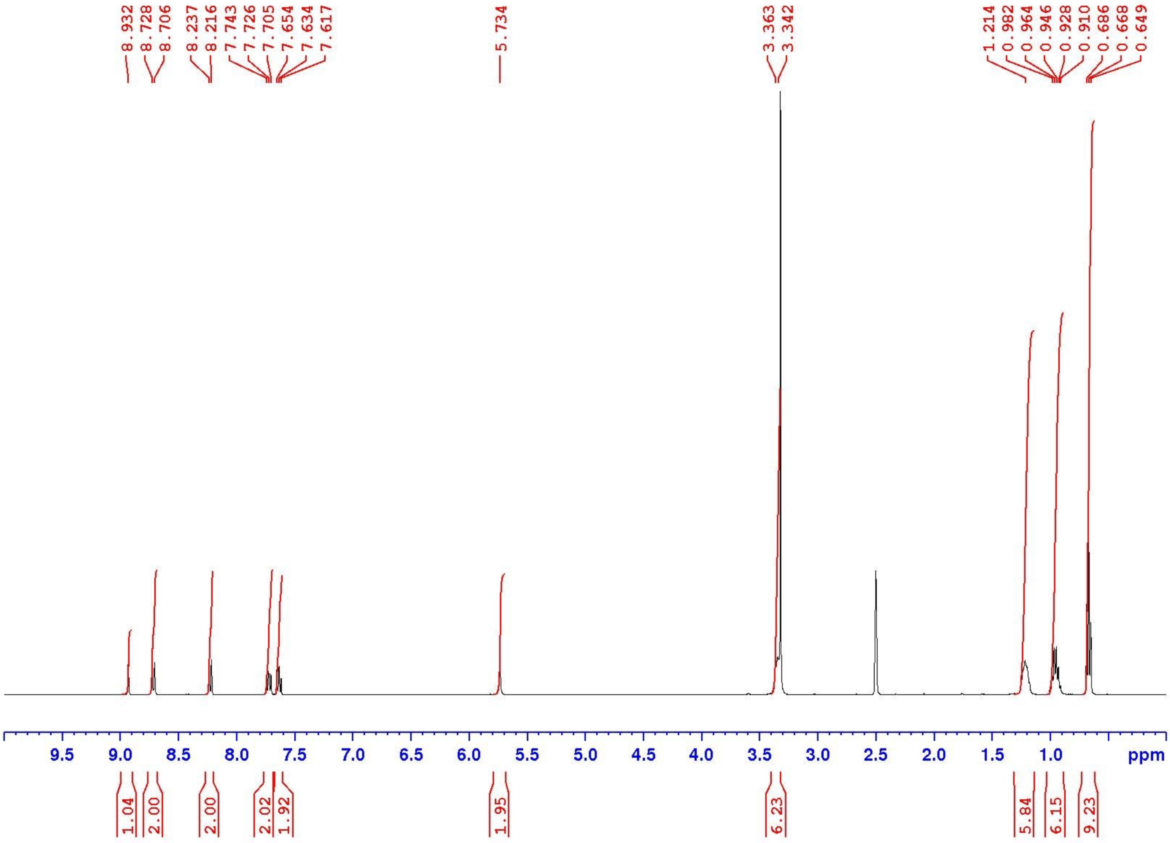
^{1}H NMR spectrum (400 MHz, DMSO-*d*6) of **37**



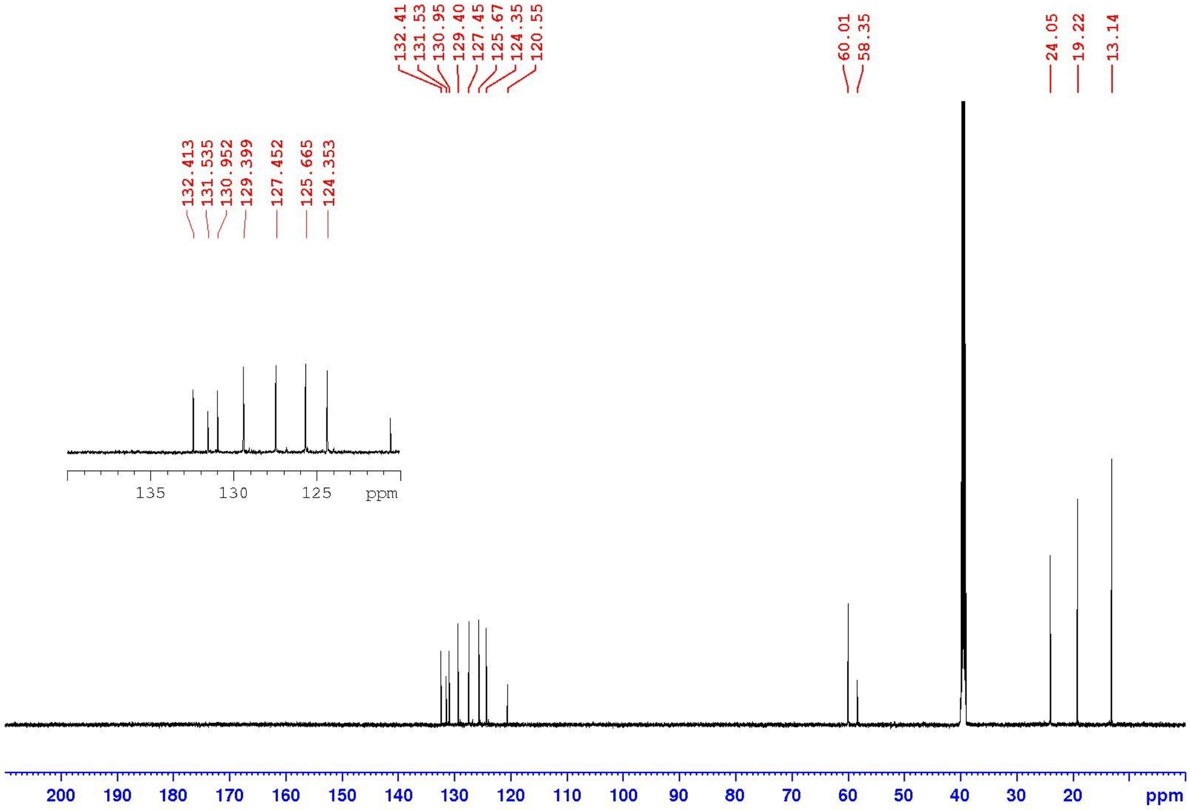
^{13}C NMR spectrum (101 MHz, DMSO-*d*6) of **37**



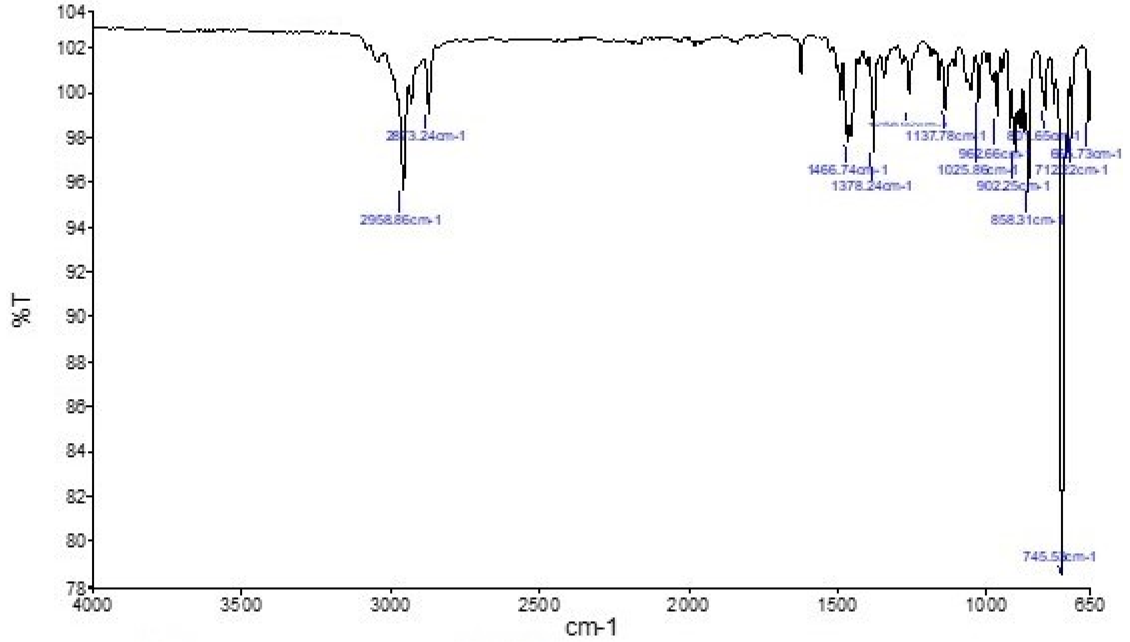
FT-IR spectrum of **37**



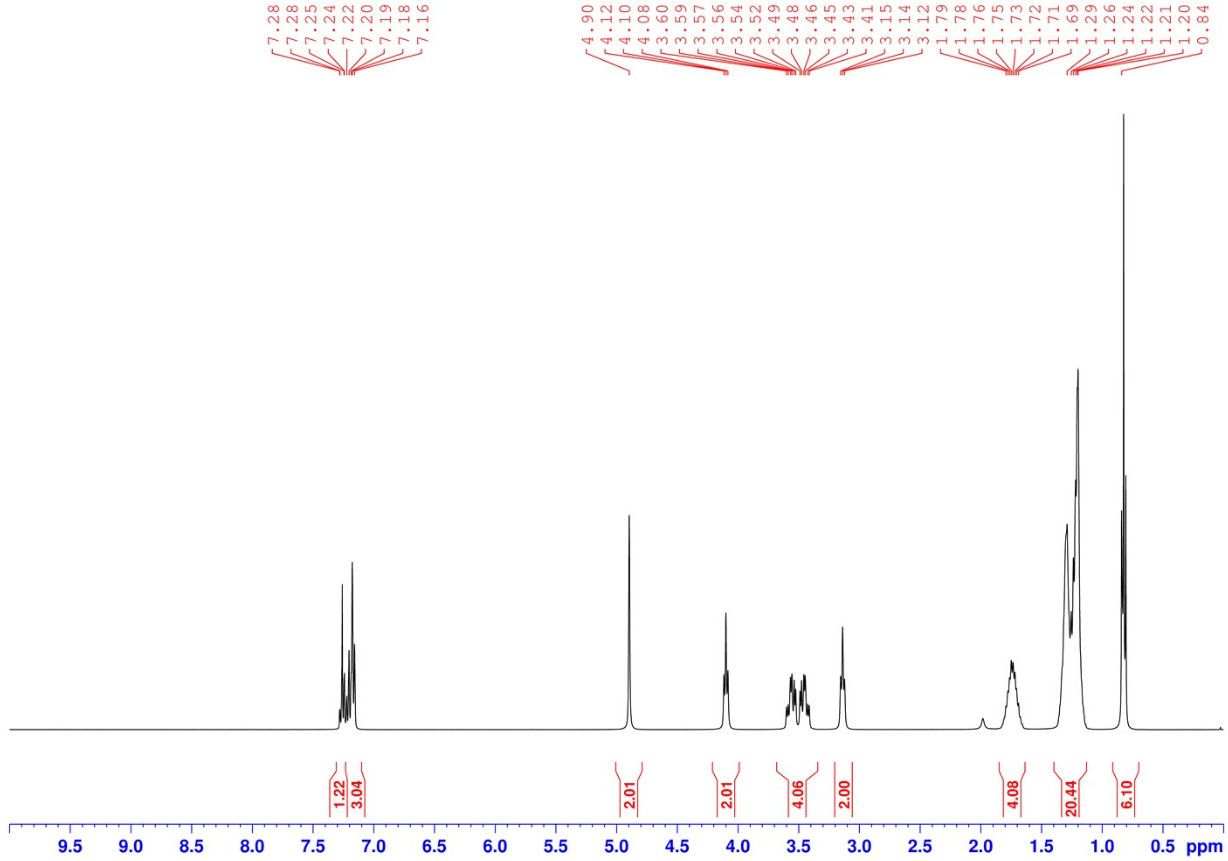
^{1}H NMR spectrum (400 MHz, DMSO-*d*6) of **38**



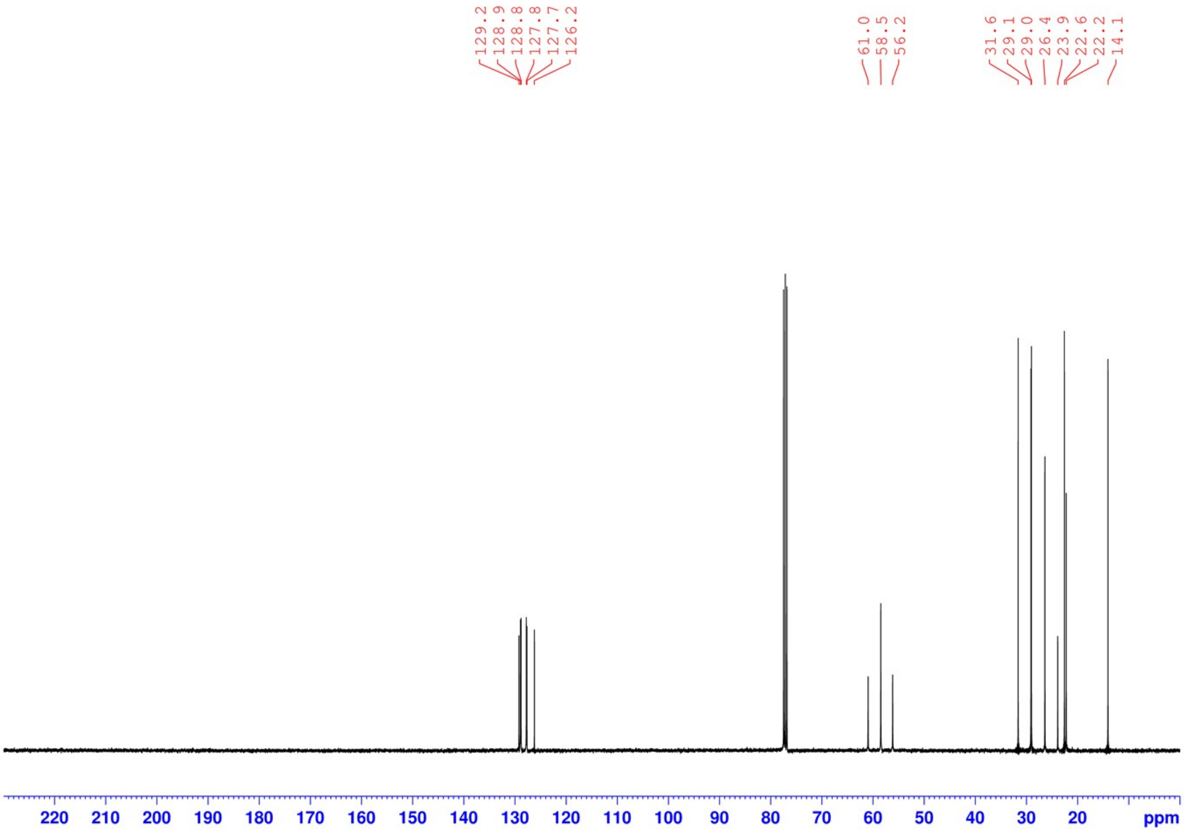
^{13}C NMR spectrum (101 MHz, DMSO-*d*6) of **38**



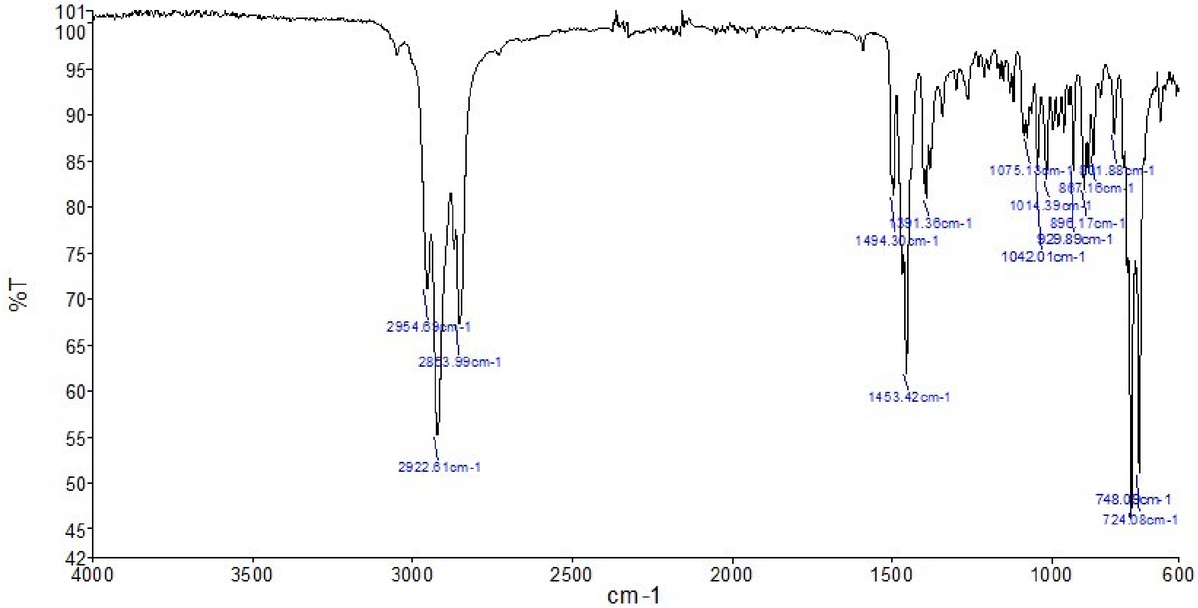
FT-IR spectrum of **38**



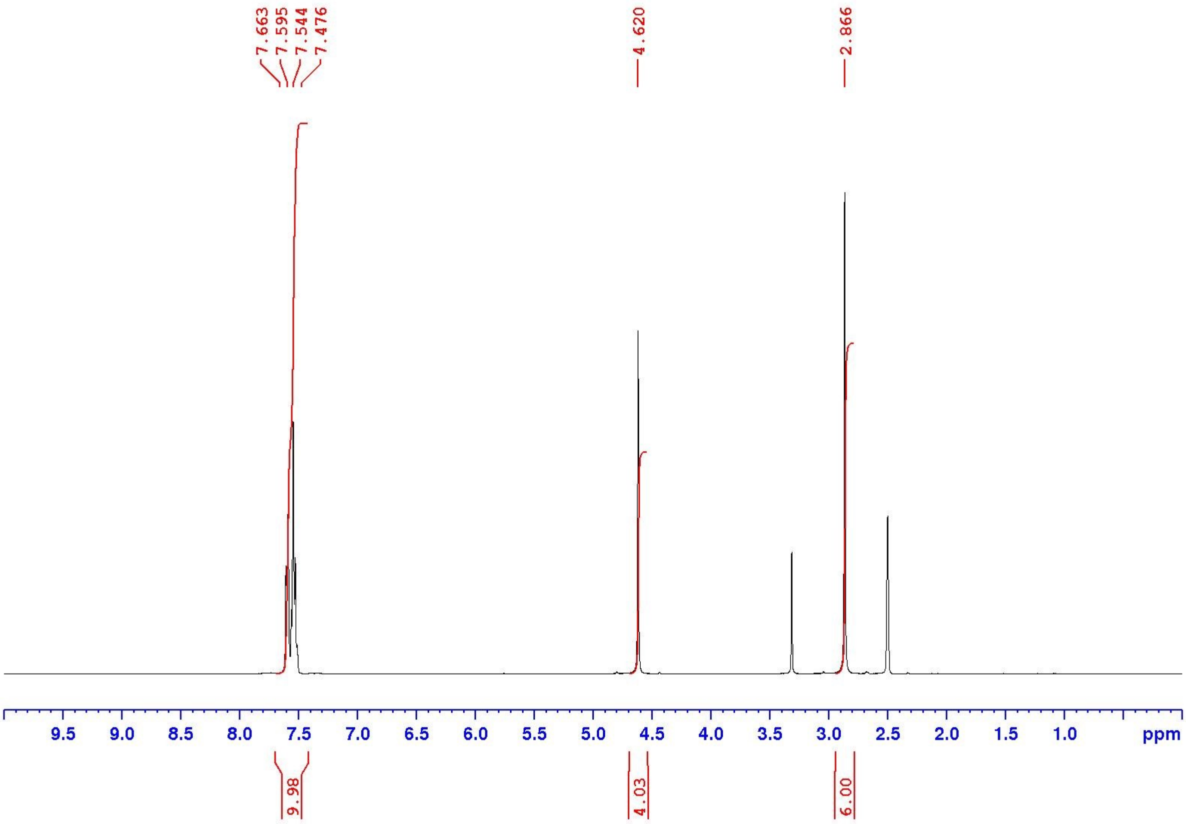
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **39**



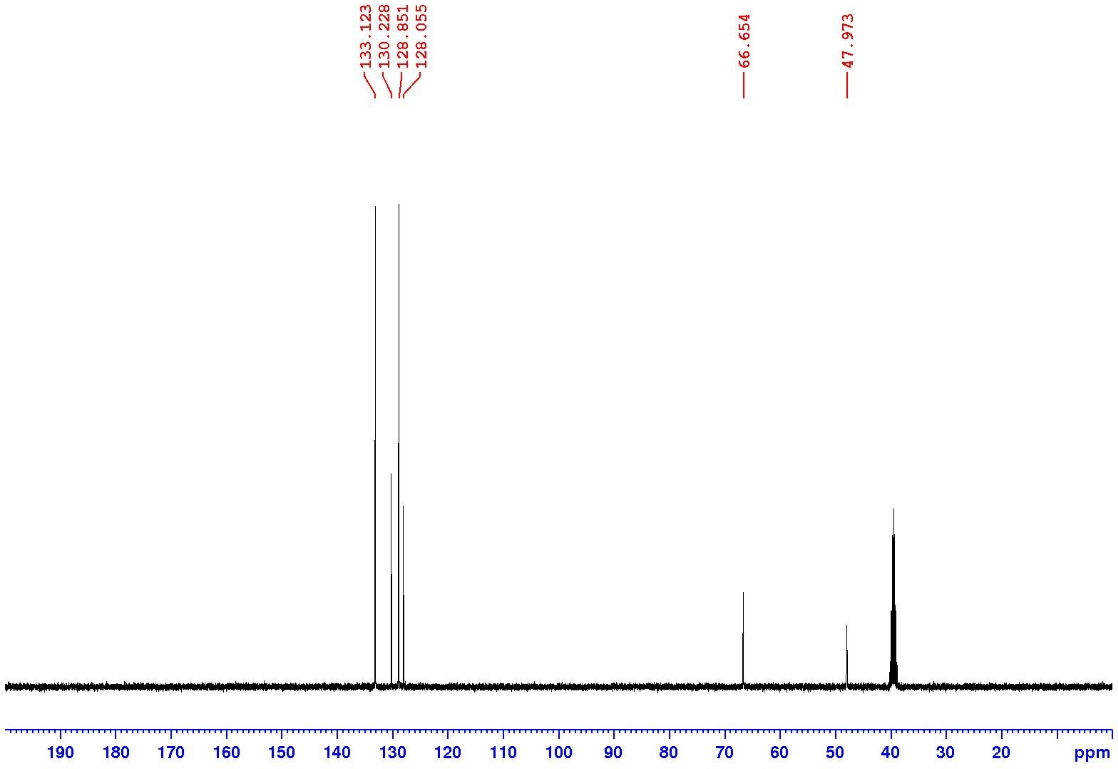
^{13}C NMR spectrum (100 MHz, CDCl$_3$) of **39**



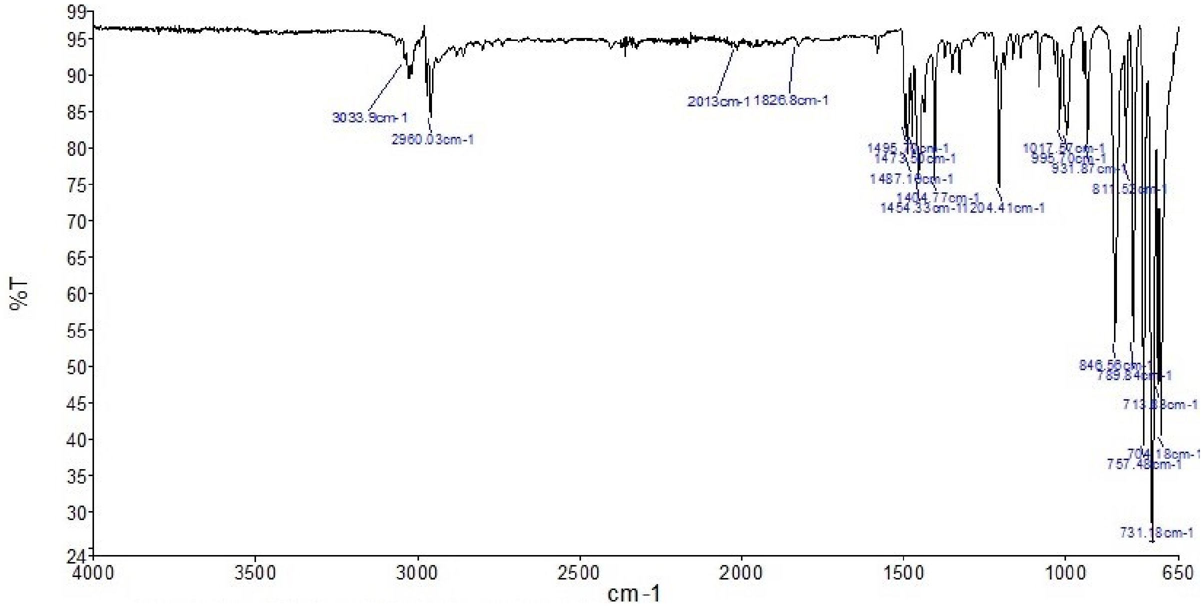
FT-IR spectrum of **39**



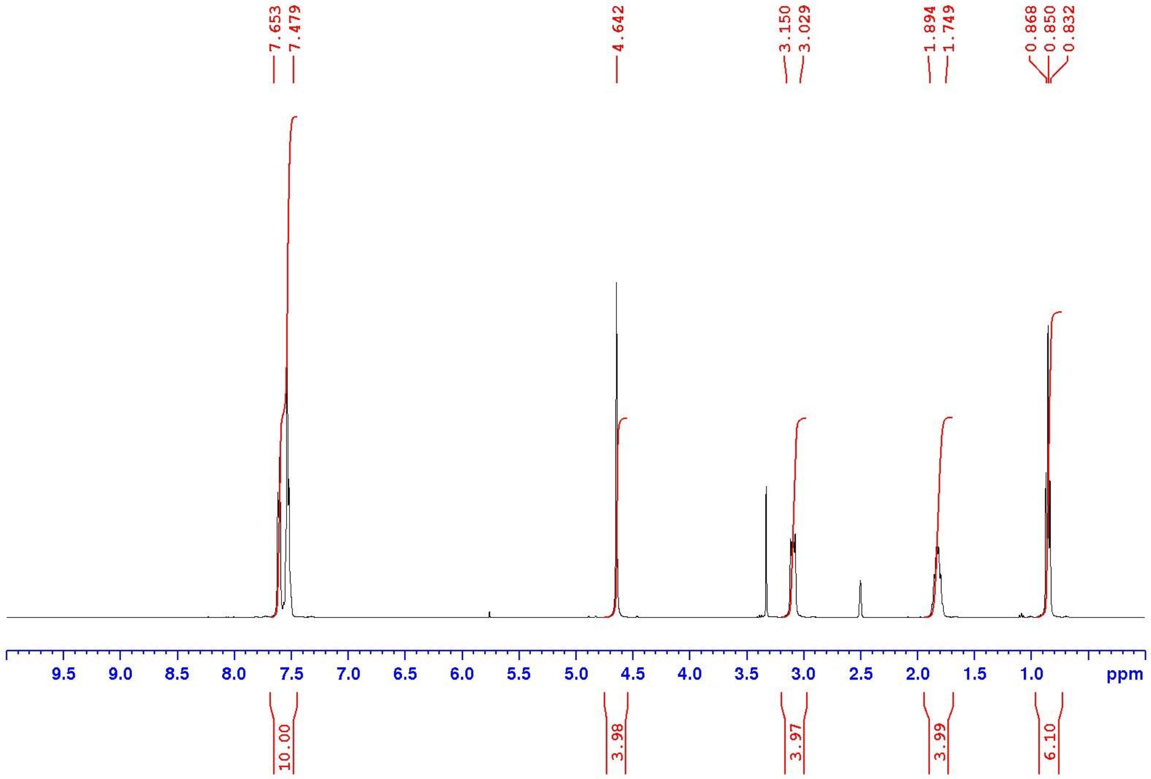
^{1}H NMR spectrum (400 MHz, DMSO-*d*6) of **40**



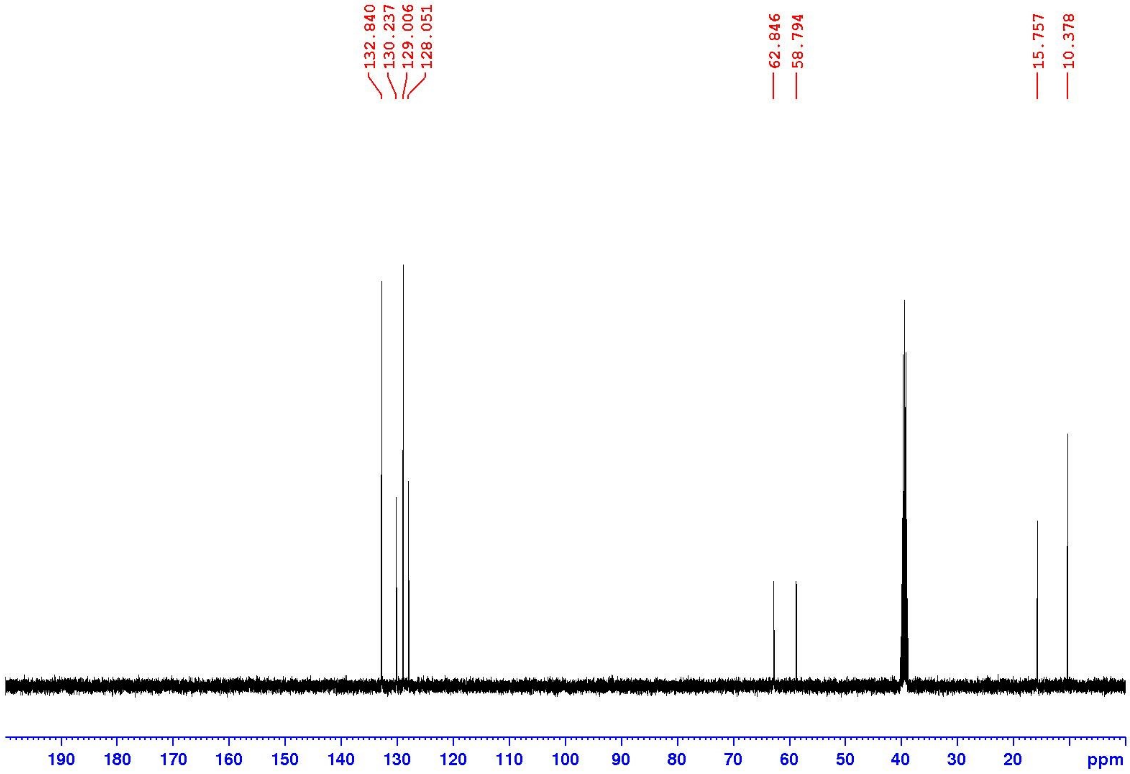
^{13}C NMR spectrum (101 MHz, DMSO-*d*6) of **40**



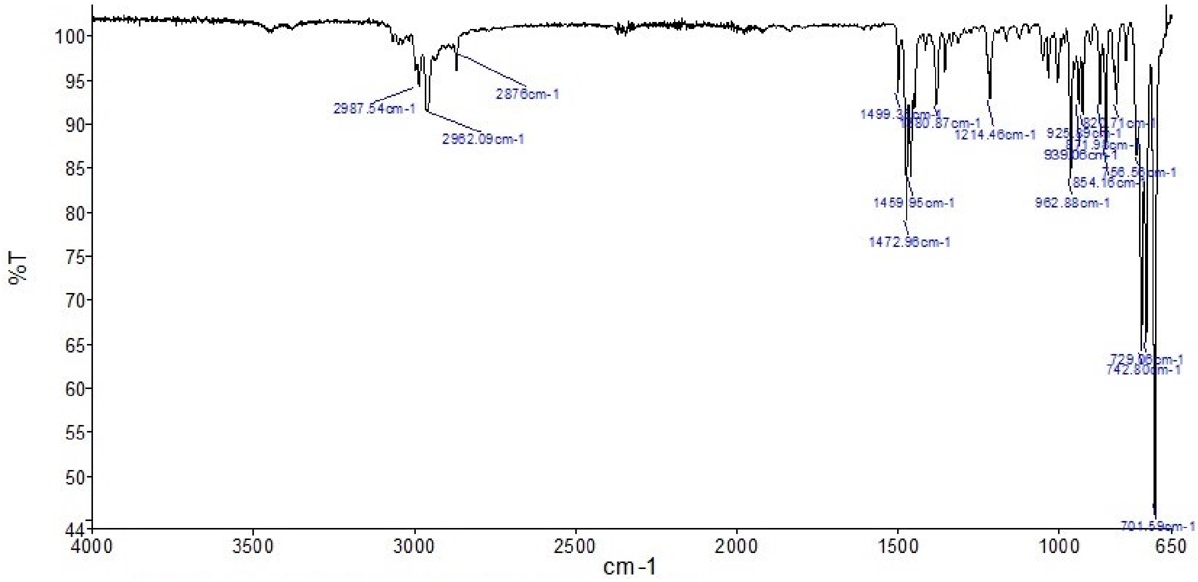
FT-IR spectrum of **40**



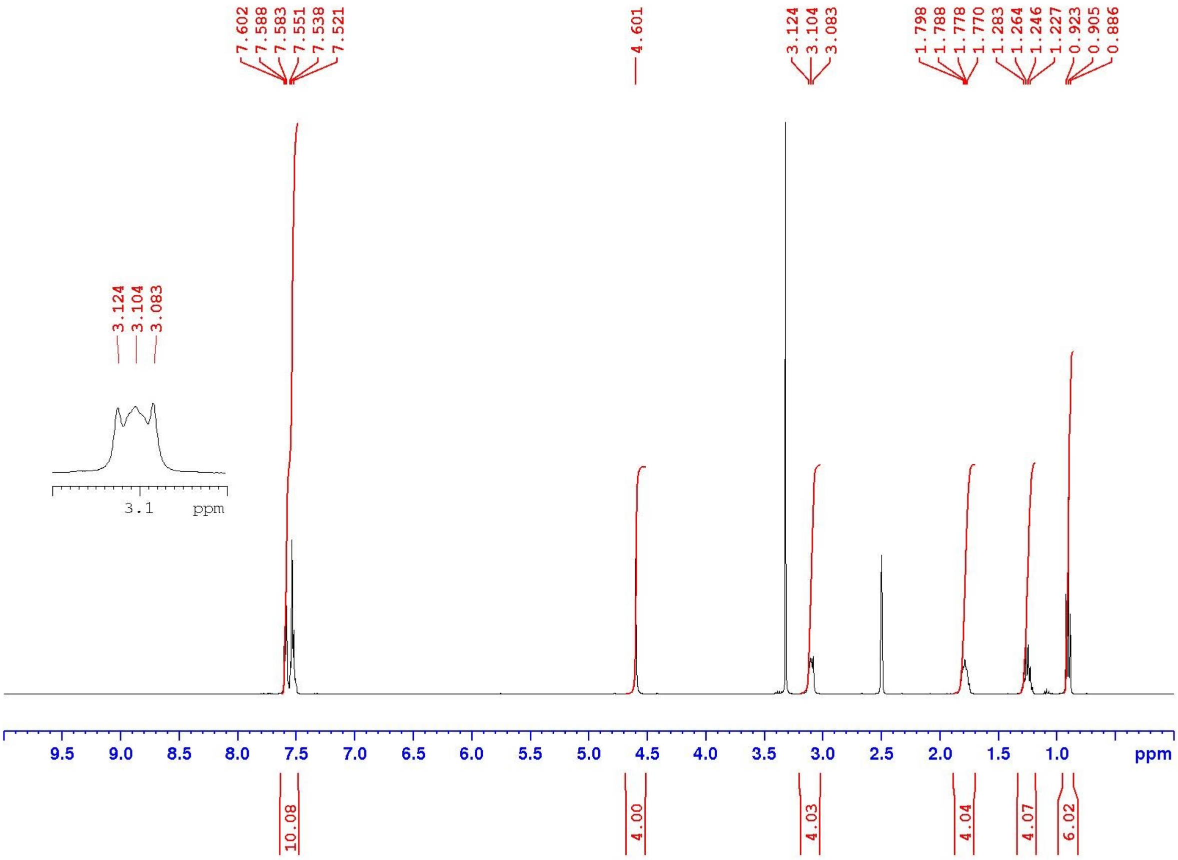
^{1}H NMR spectrum (400 MHz, DMSO-*d*6) of **41**



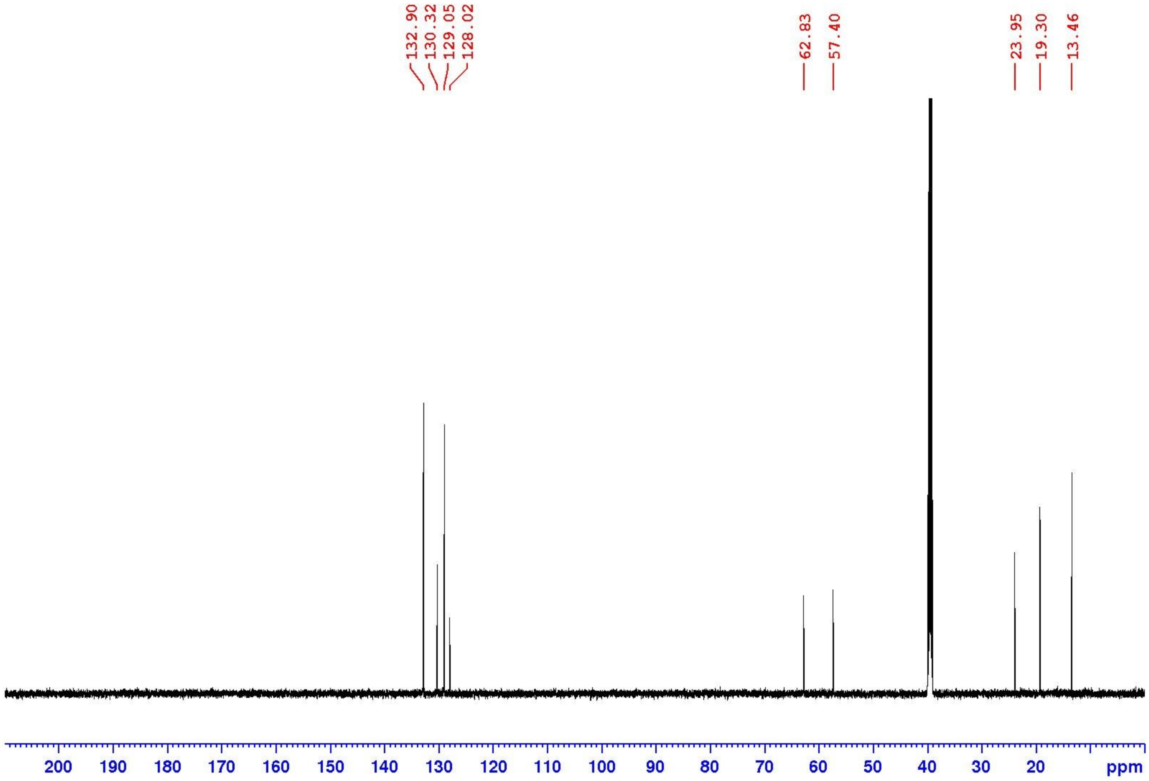
^{13}C NMR spectrum (101 MHz, DMSO-*d*6) of **41**



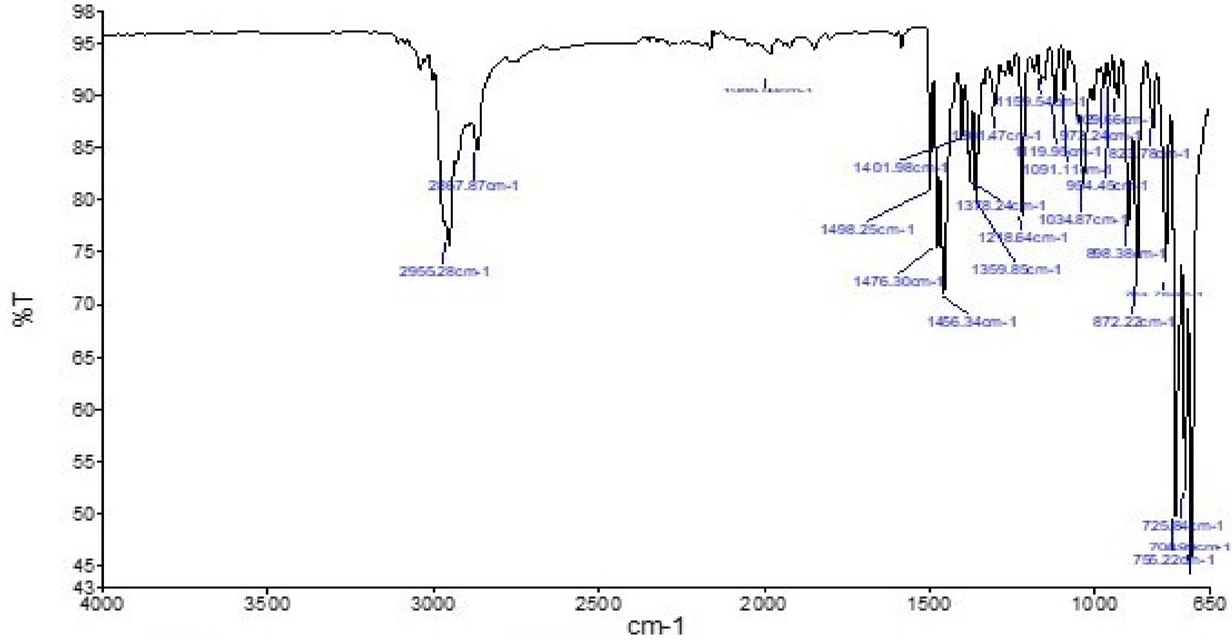
FT-IR spectrum of **41**



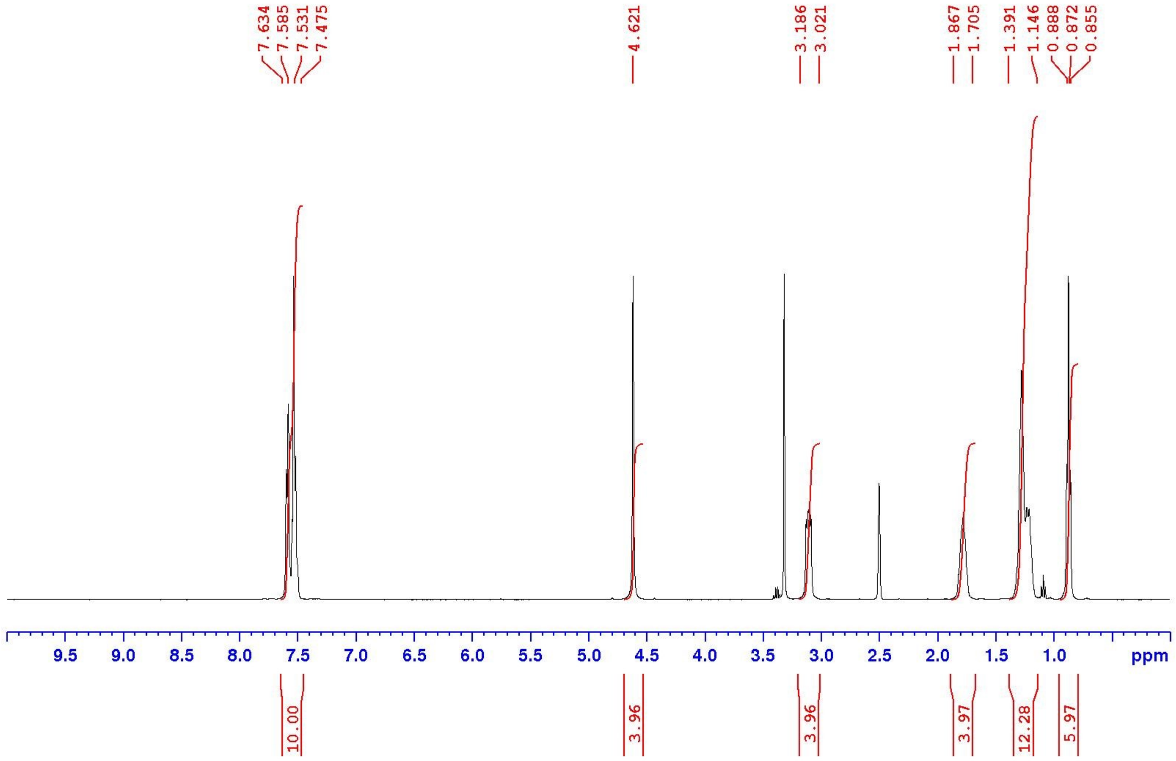
^{1}H NMR spectrum (400 MHz, DMSO-*d*6) of **42**



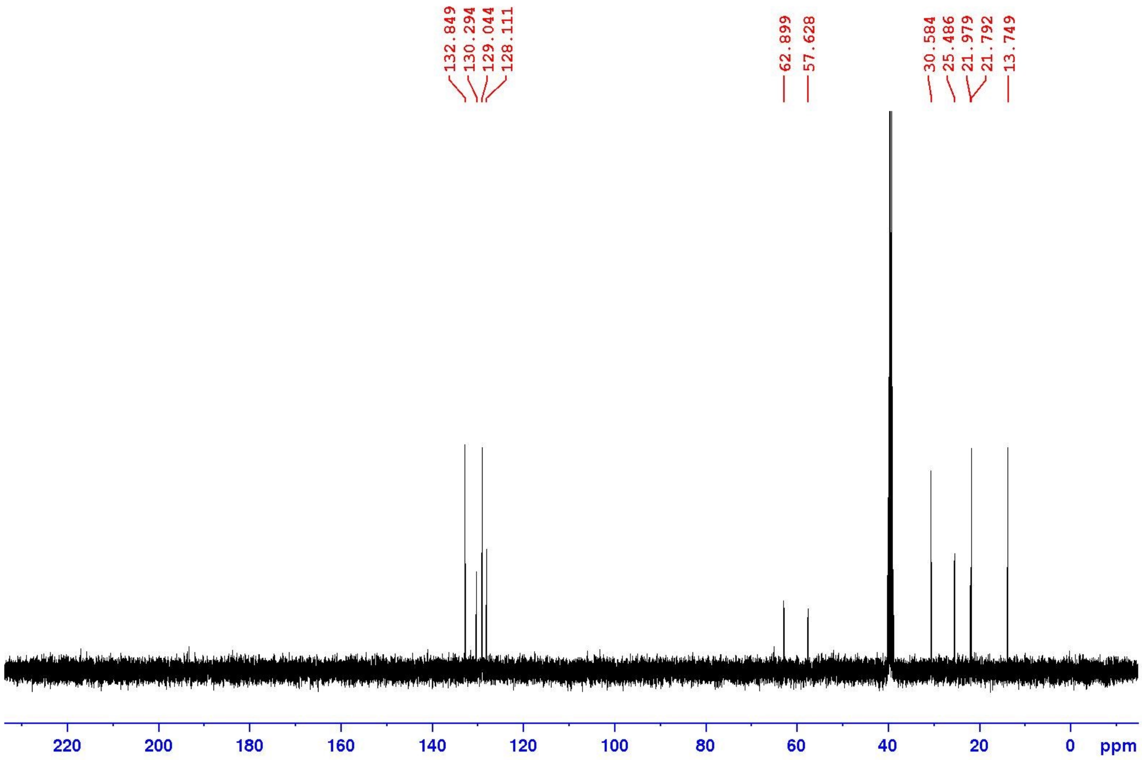
^{13}C NMR spectrum (101 MHz, DMSO-*d*6) of **42**



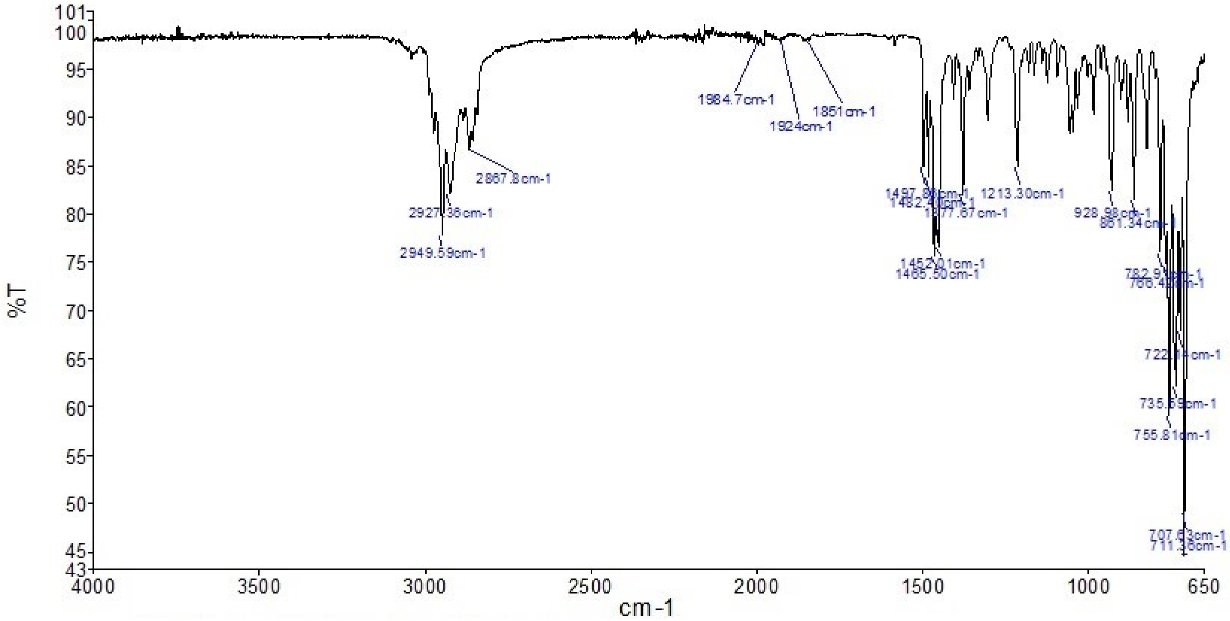
FT-IR spectrum of **42**



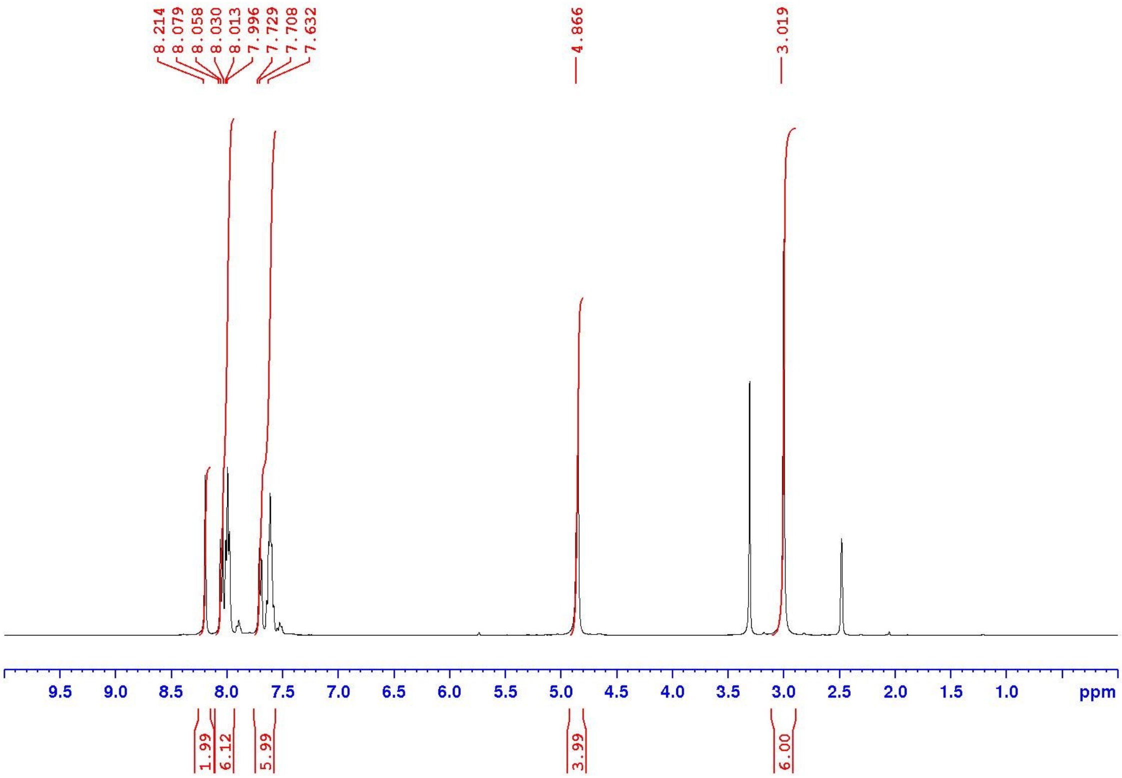
^{1}H NMR spectrum (400 MHz, DMSO-*d*6) of **43**



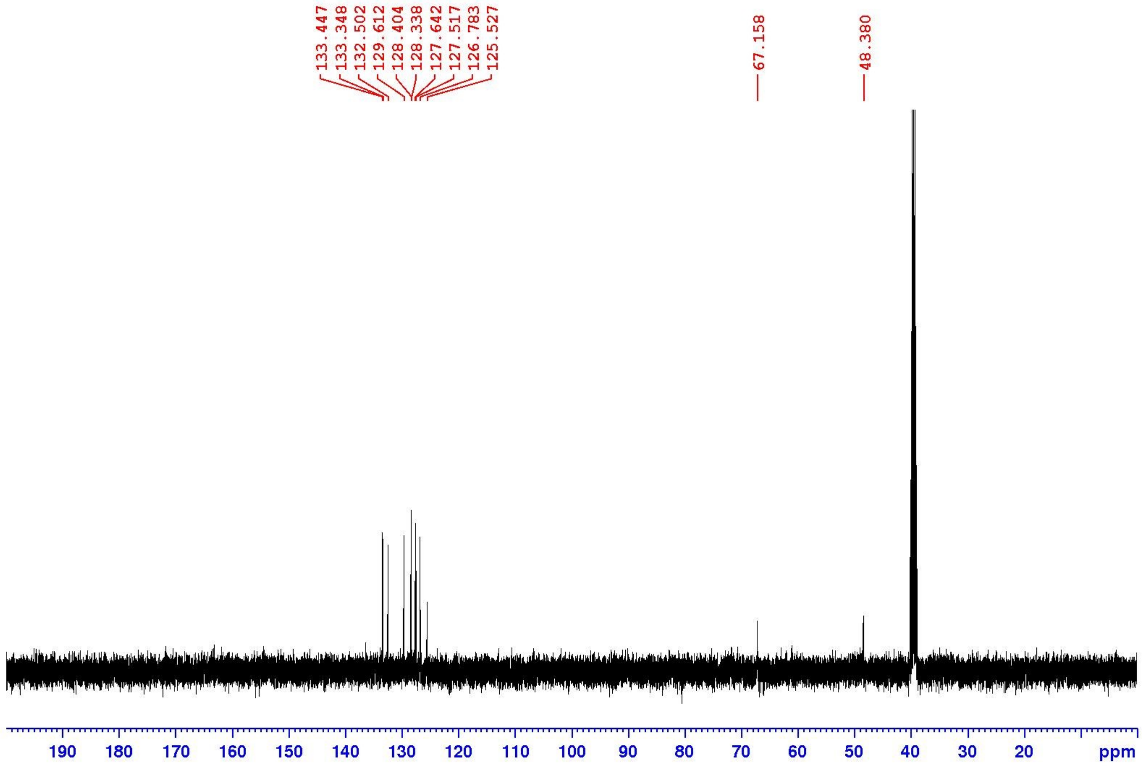
^{13}C NMR spectrum (101 MHz, DMSO-*d*6) of **43**



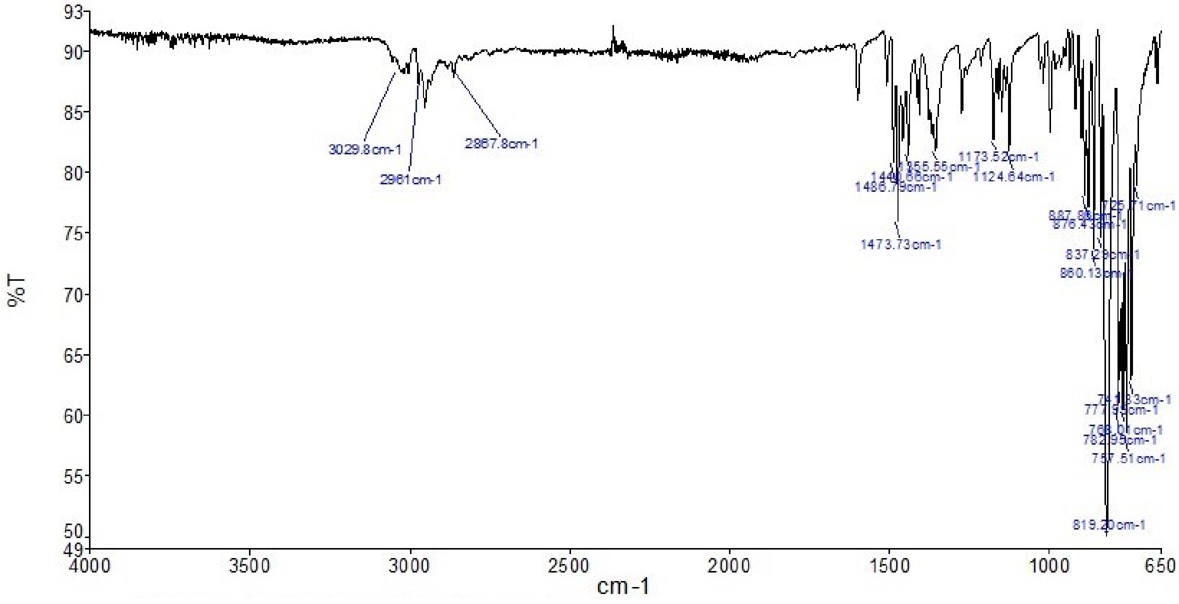
FT-IR spectrum of **43**



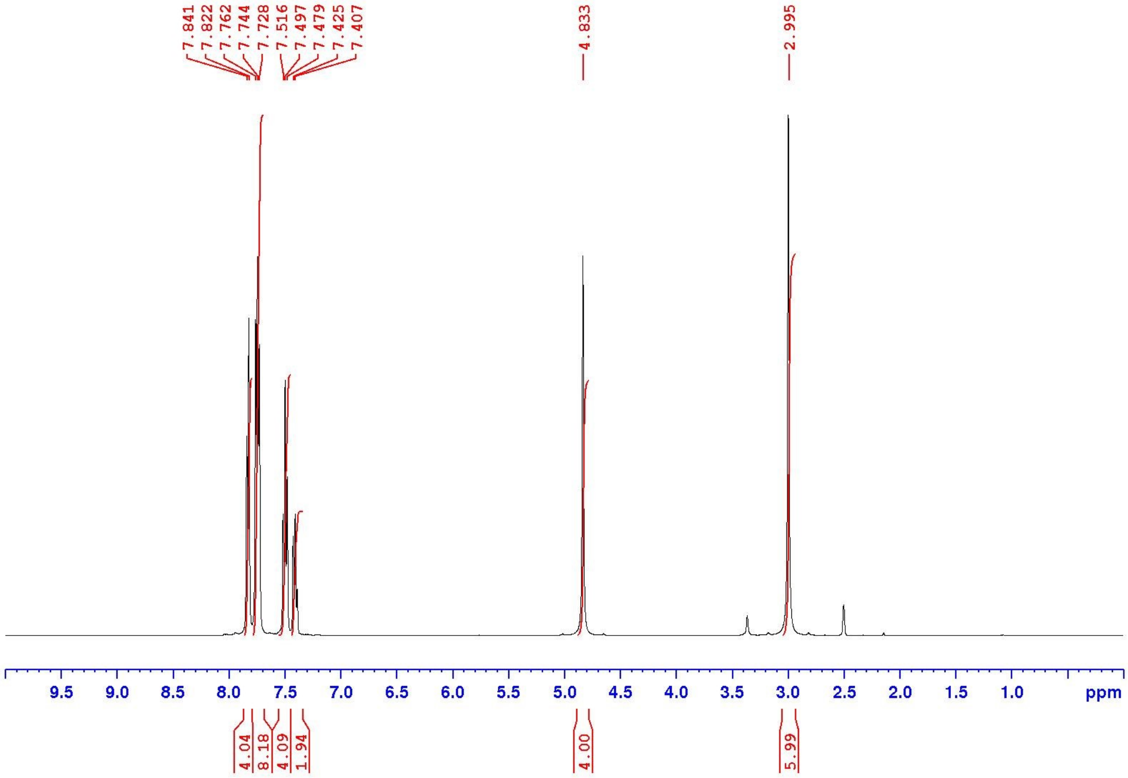
^{1}H NMR spectrum (400 MHz, DMSO-*d*6) of **44**



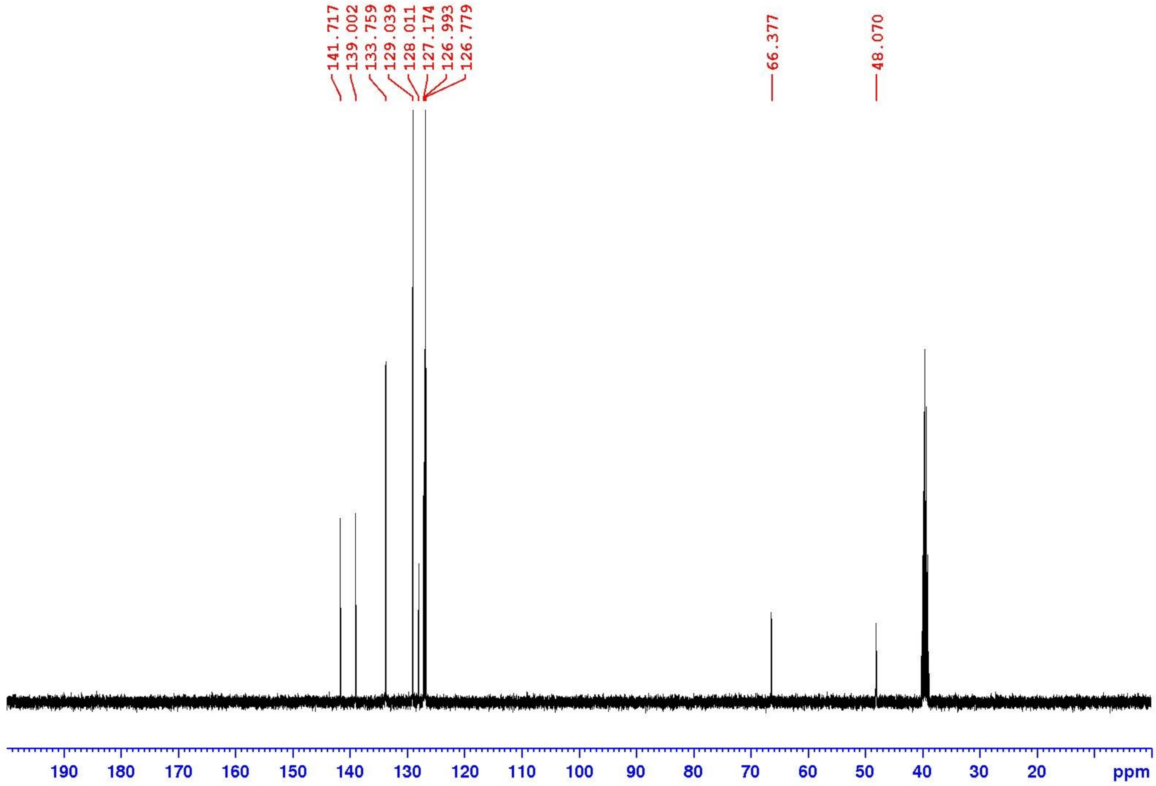
^{13}C NMR spectrum (101 MHz, DMSO-*d*6) of **44**



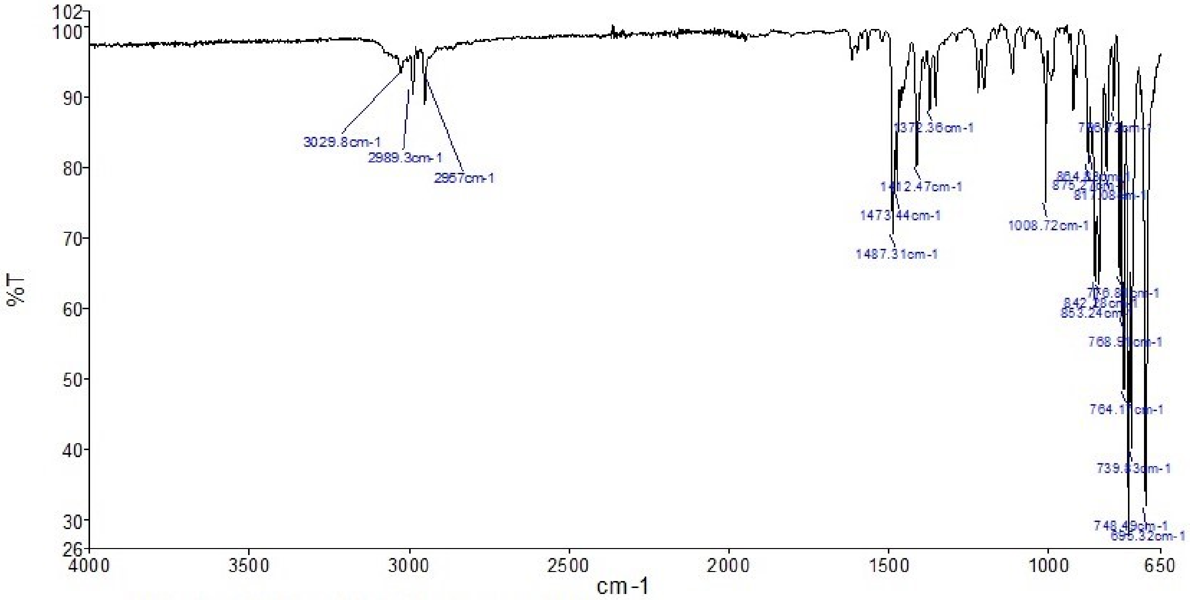
FT-IR spectrum of **44**



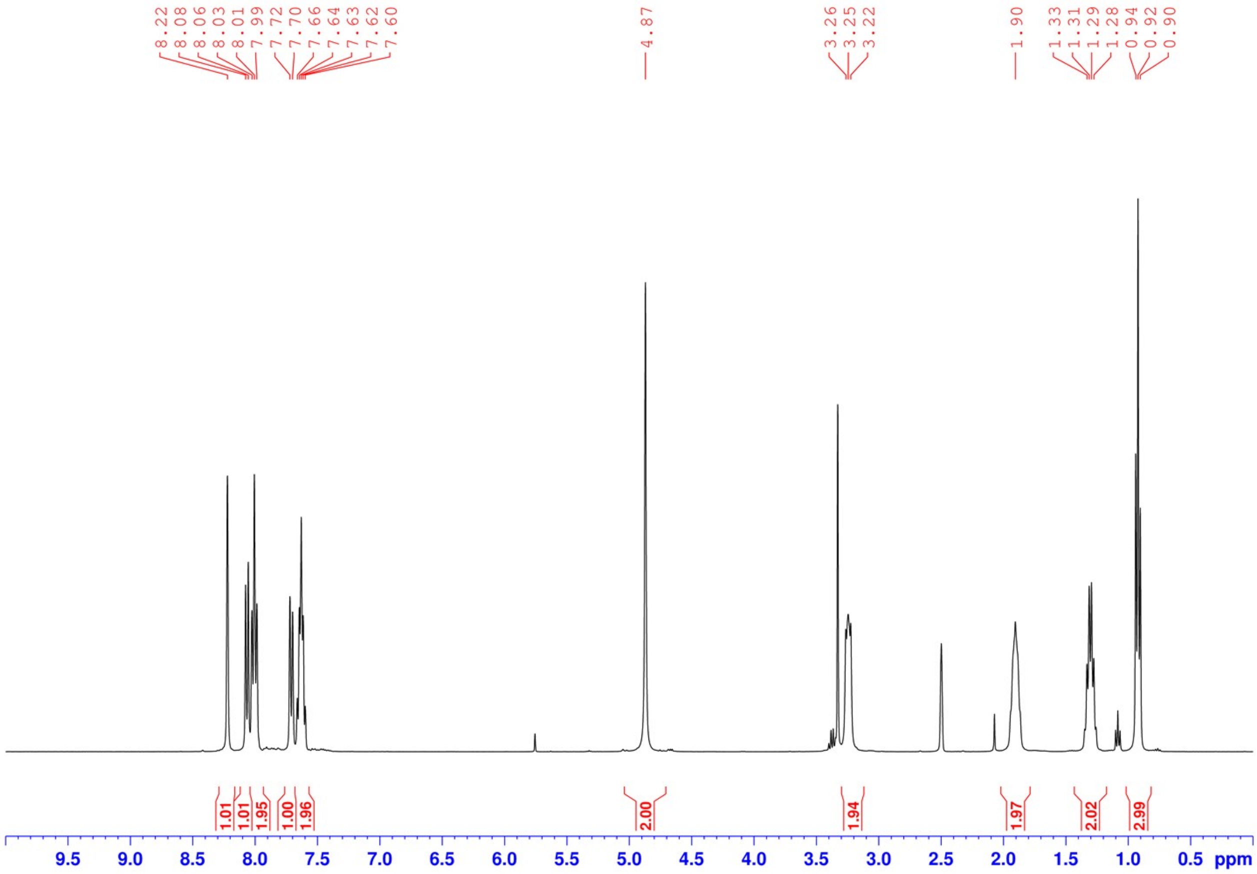
^{1}H NMR spectrum (400 MHz, DMSO-*d*6) of **45**



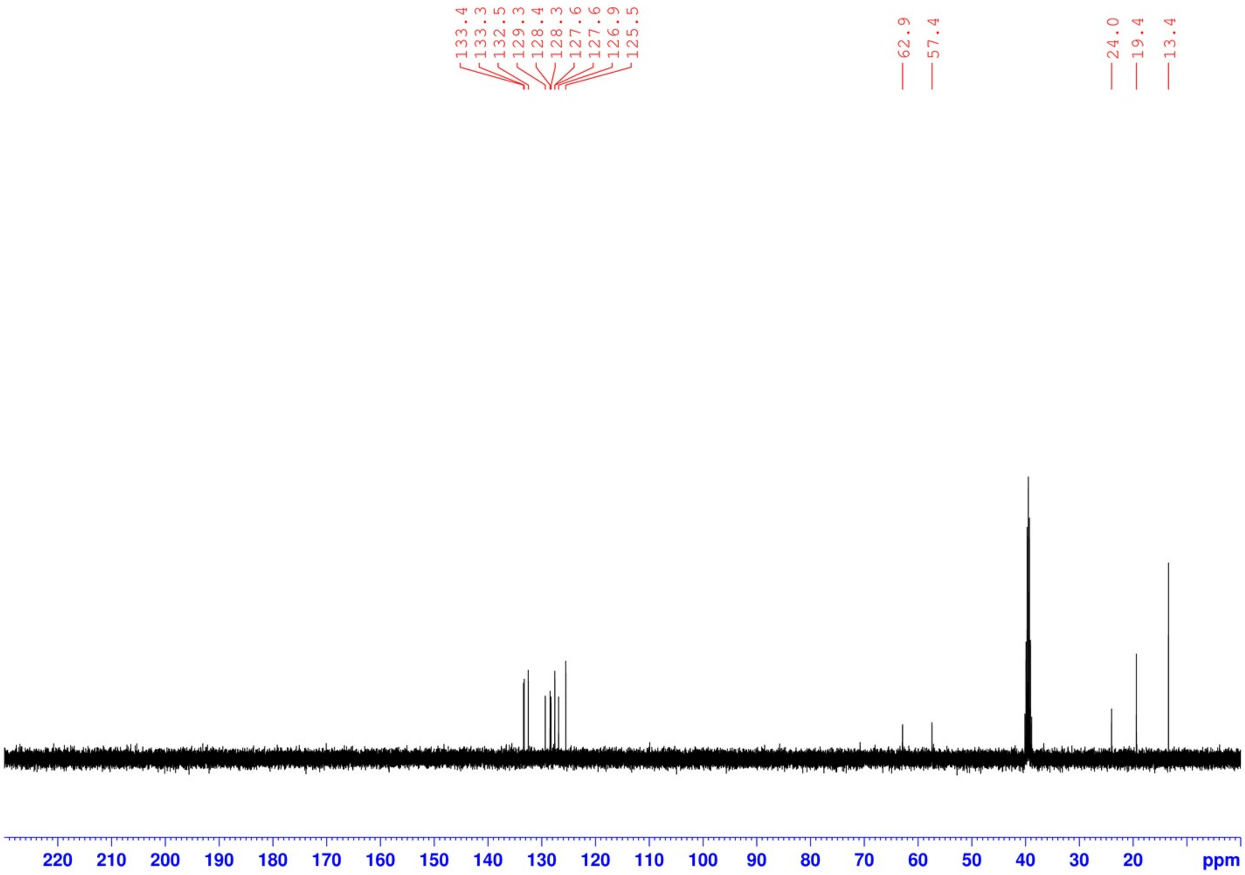
^{13}C NMR spectrum (101 MHz, DMSO-*d*6) of **45**



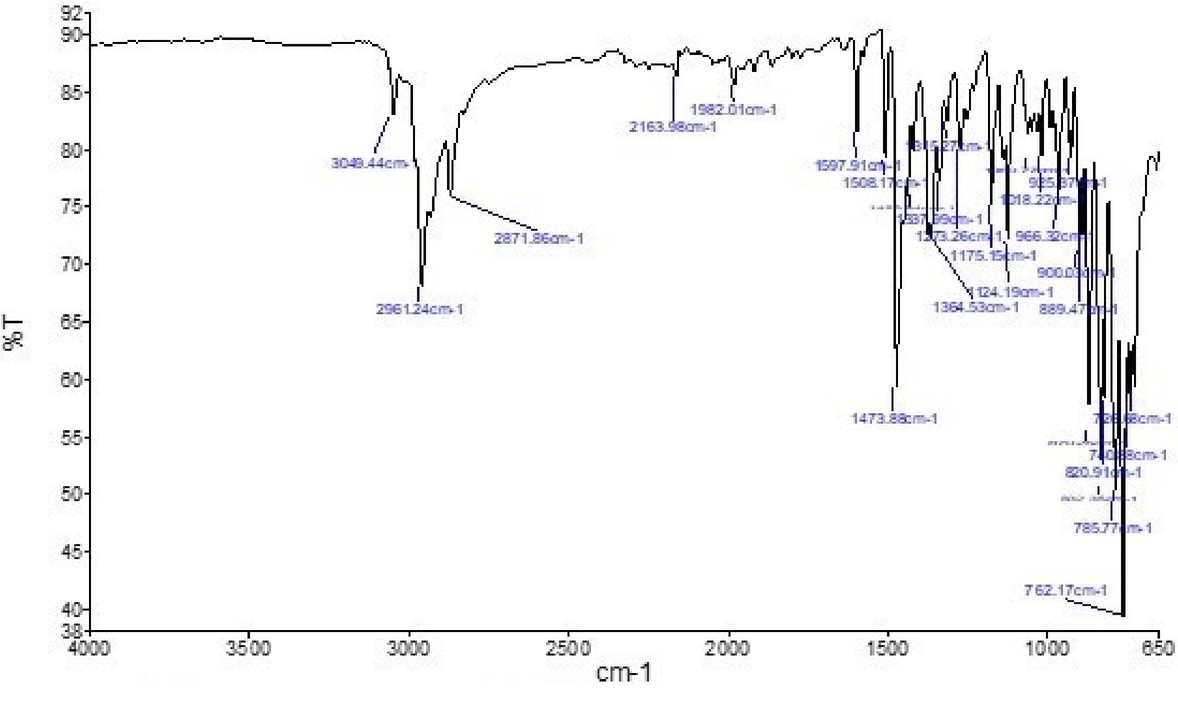
FT-IR spectrum of **45**



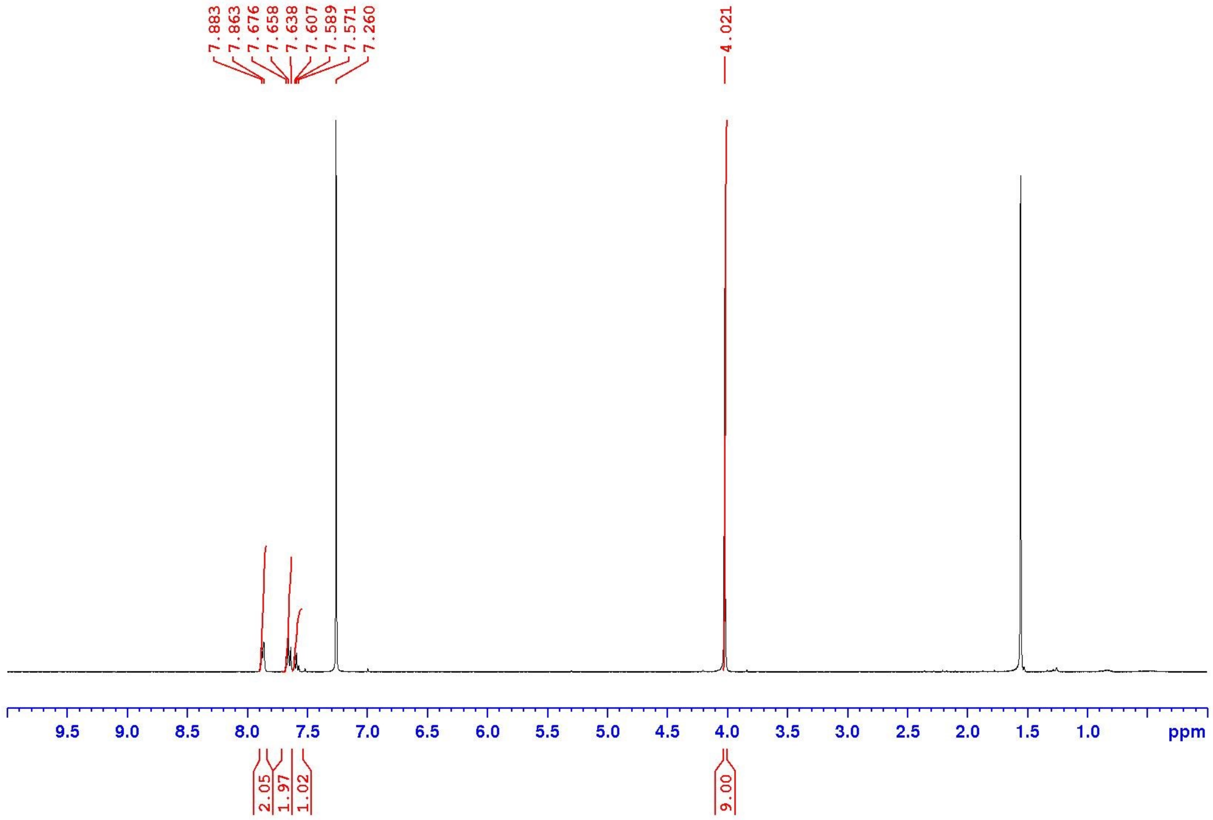
^{1}H NMR spectrum (400 MHz, DMSO-*d*6) of **46**



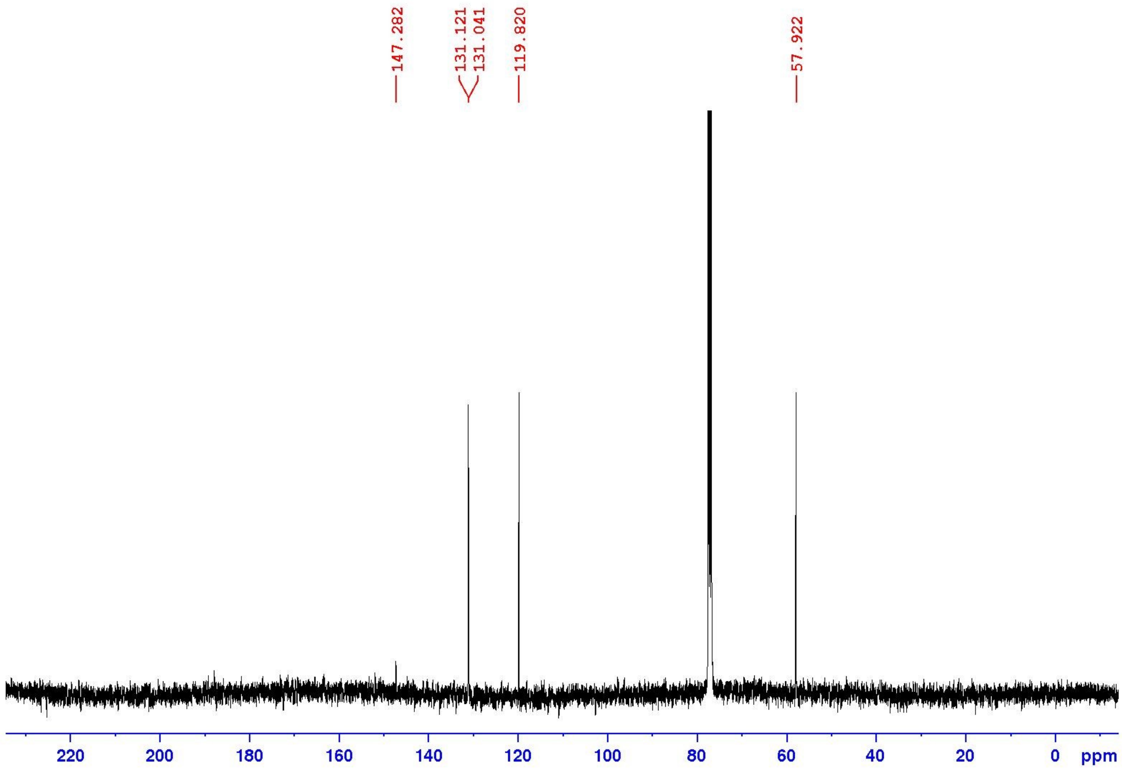
^{13}C NMR spectrum (101 MHz, DMSO-*d6*) of **46**



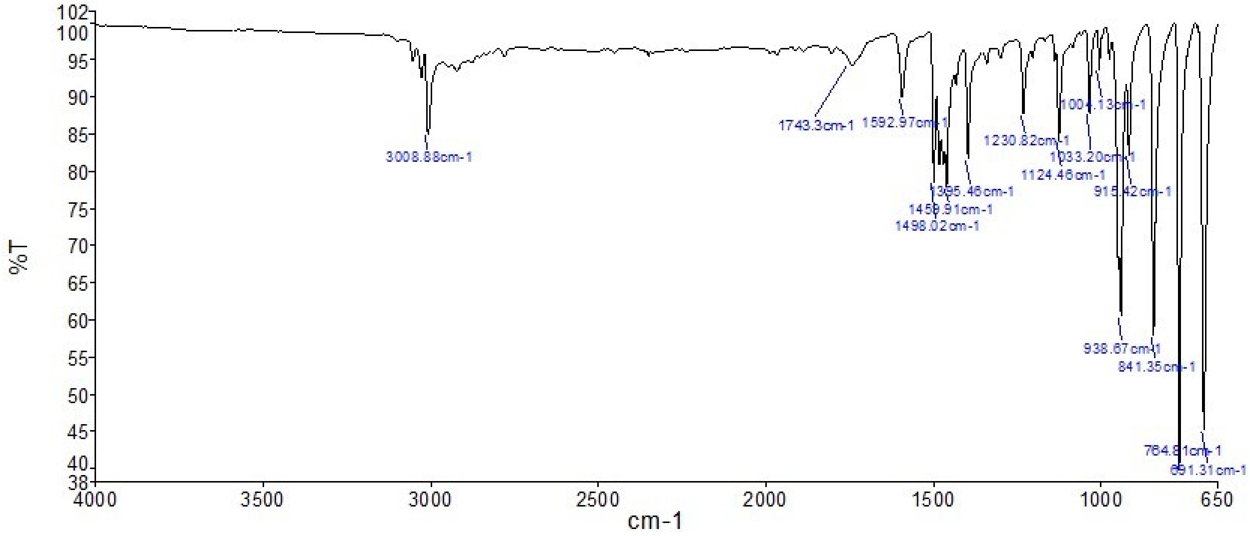
FT-IR spectrum of **46**



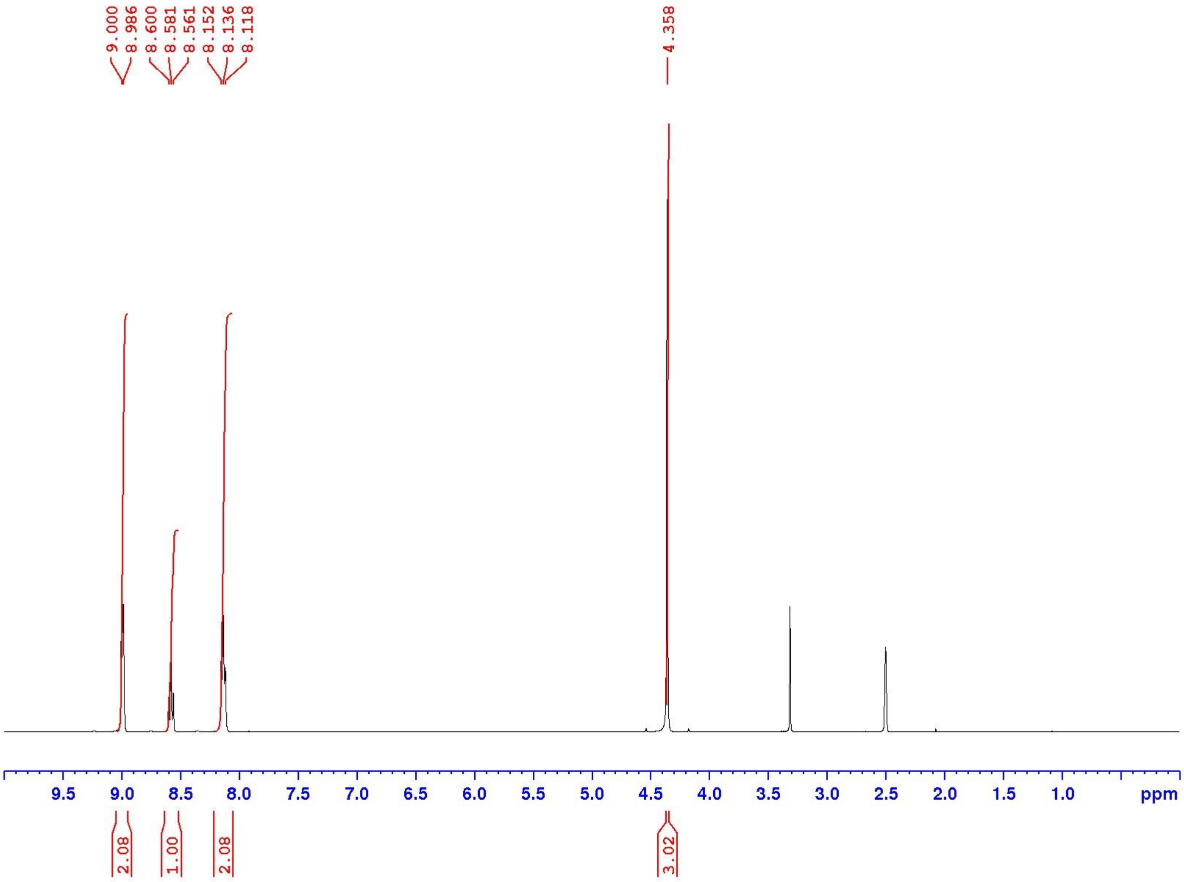
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **47**



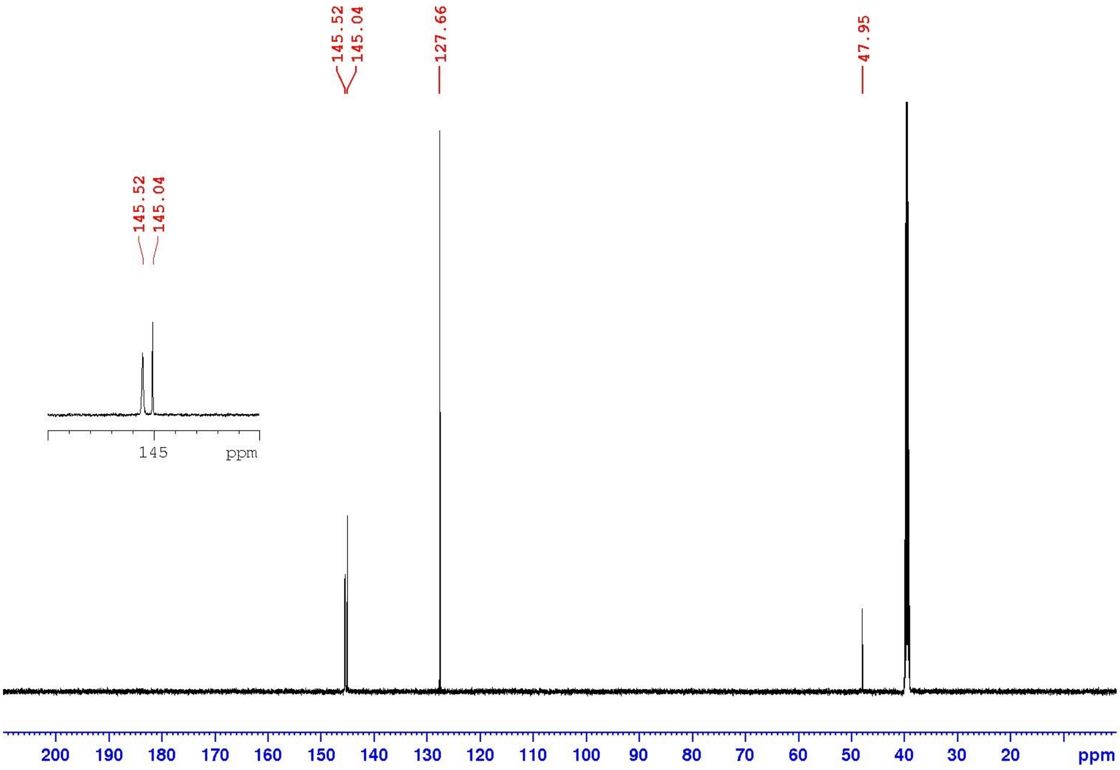
^{13}C NMR spectrum (101 MHz, CDCl$_3$) of **47**



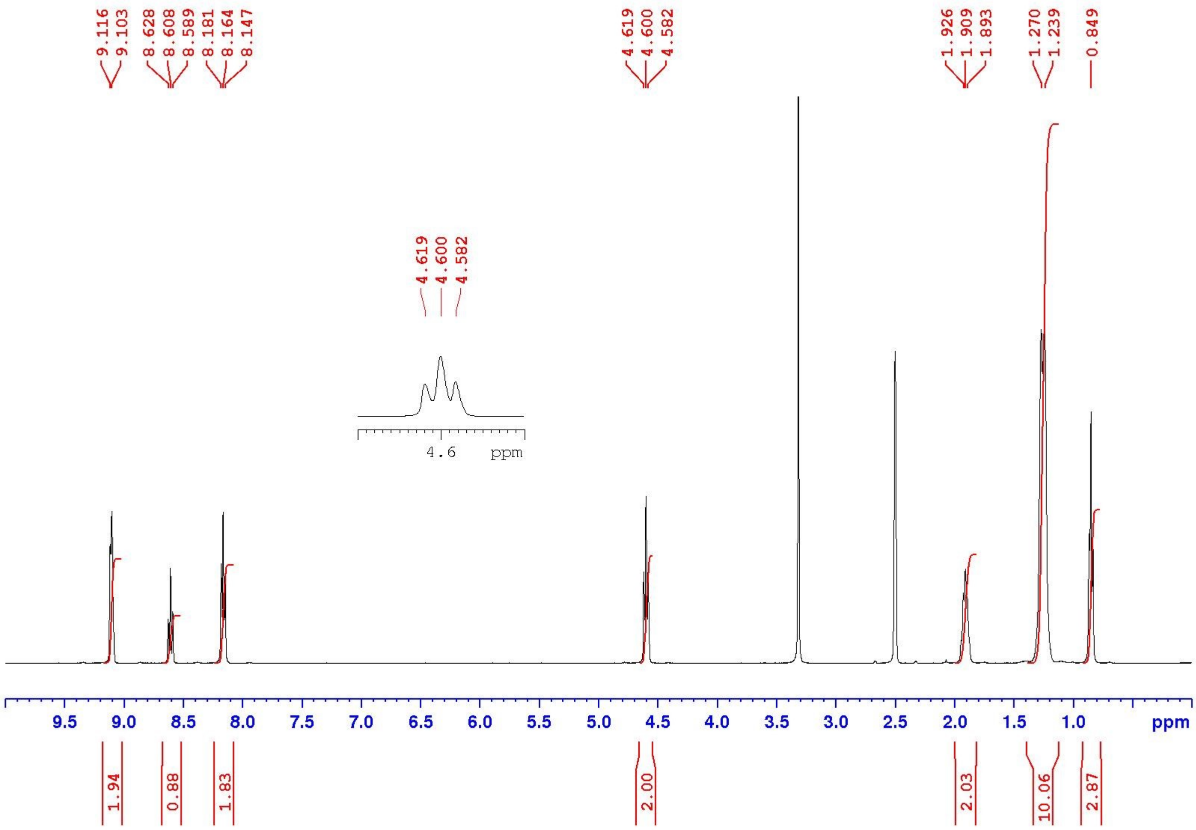
FT-IR spectrum of **47**



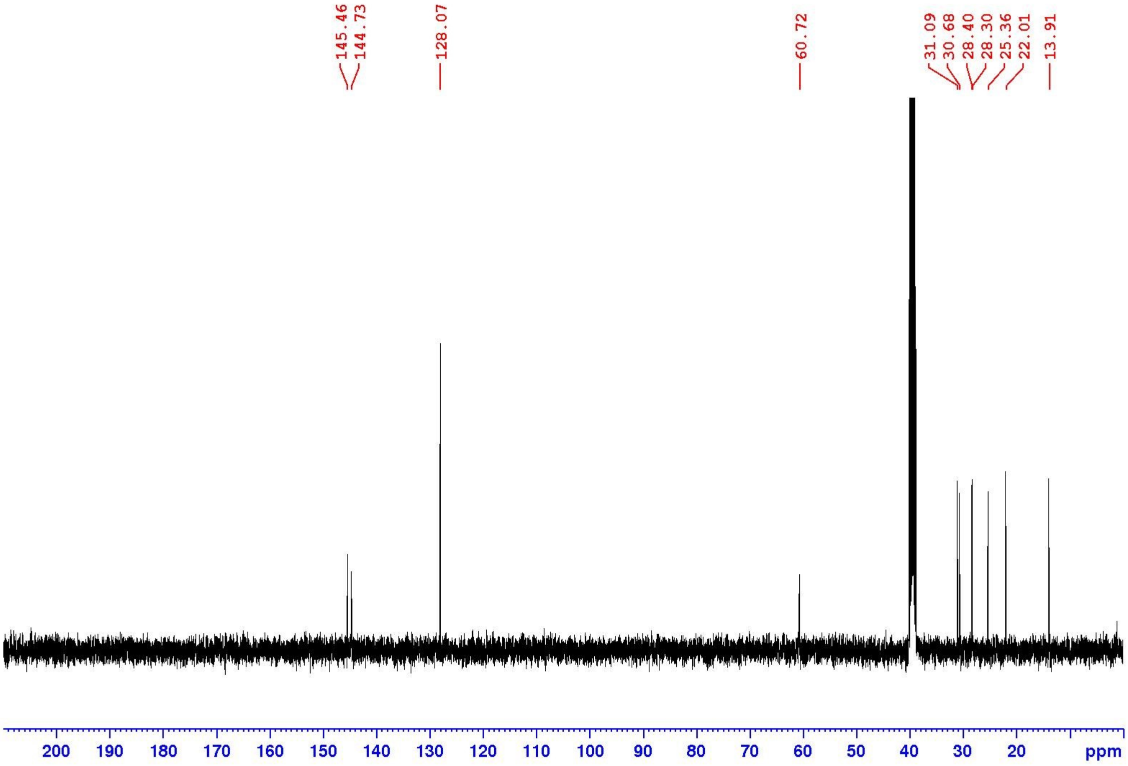
^{1}H NMR spectrum (400 MHz, DMSO-*d*6) of **48**



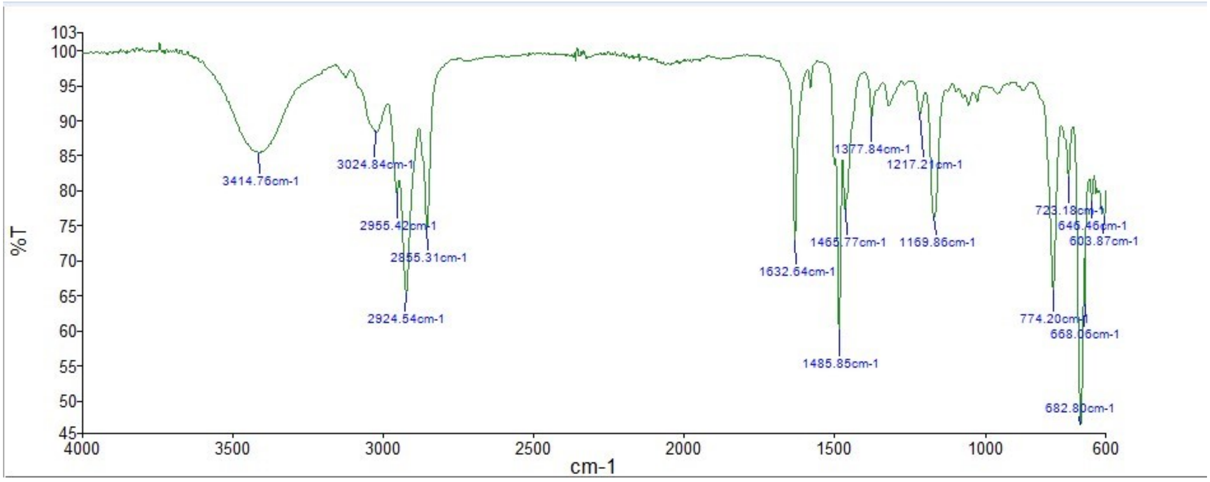
^{13}C NMR spectrum (101 MHz, DMSO-*d*6) of **48**



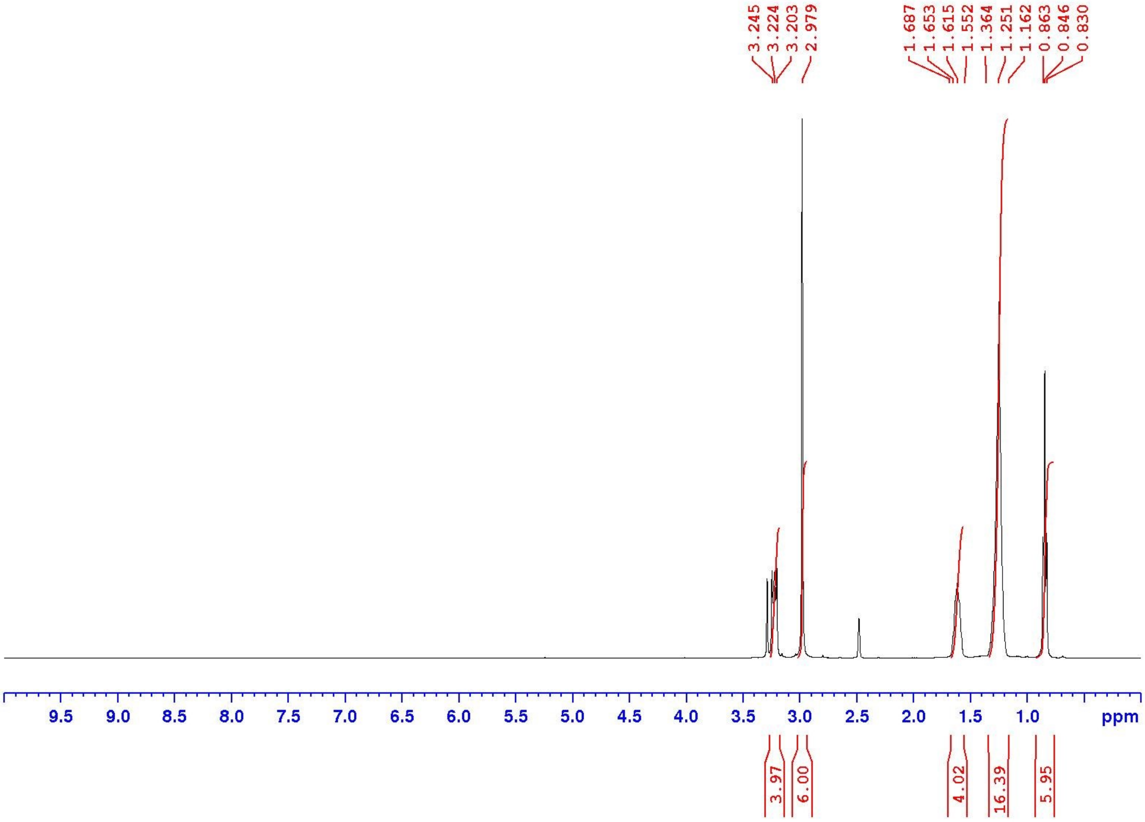
^{1}H NMR spectrum (400 MHz, DMSO-*d*6) of **49**



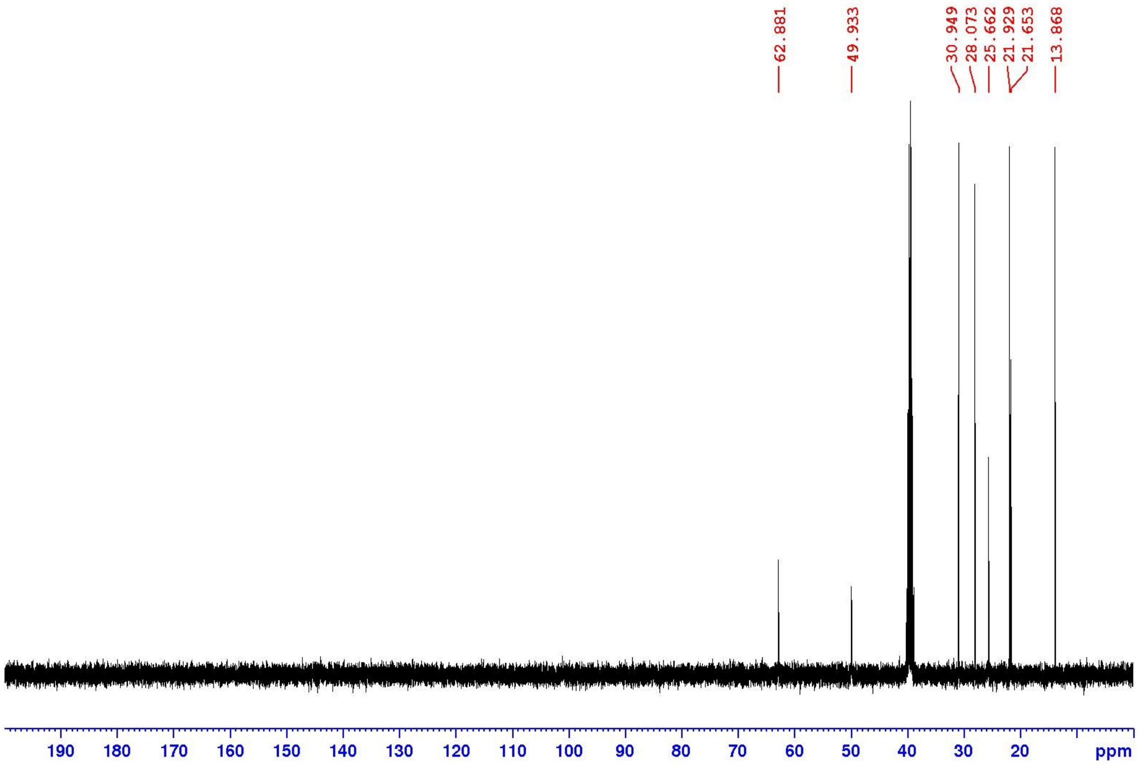
^{13}C NMR spectrum (101 MHz, DMSO-*d*6) of **49**



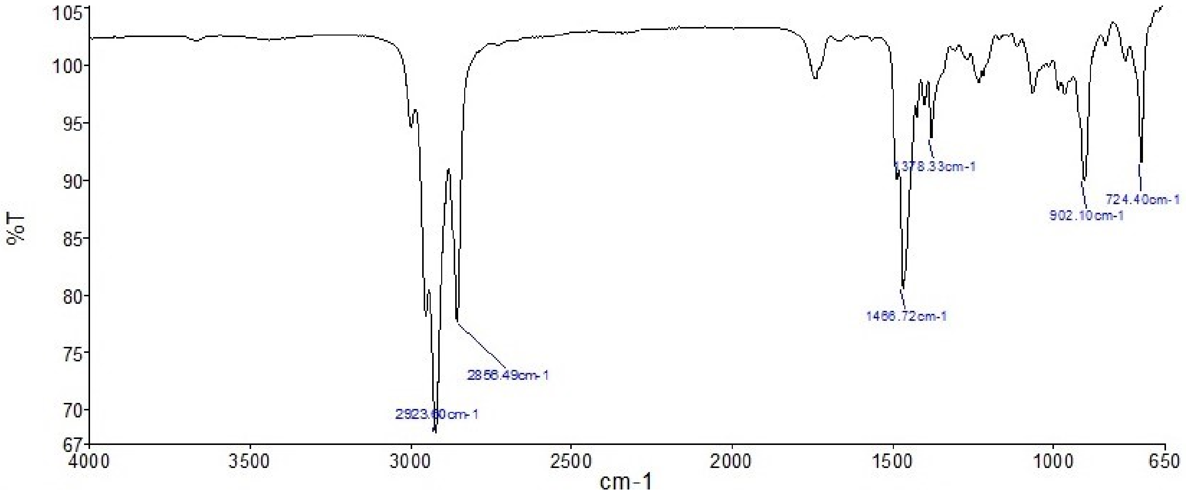
FT-IR spectrum of **49**



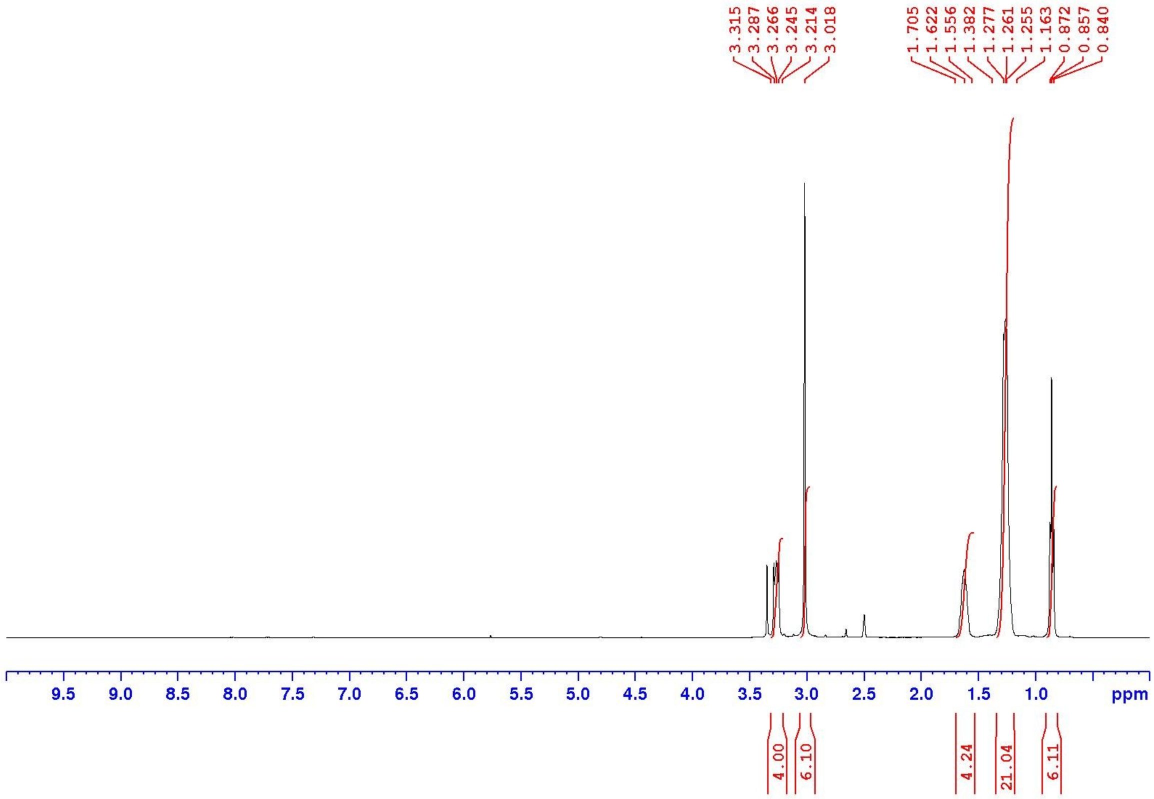
^{1}H NMR spectrum (400 MHz, DMSO-*d*6) of **52**



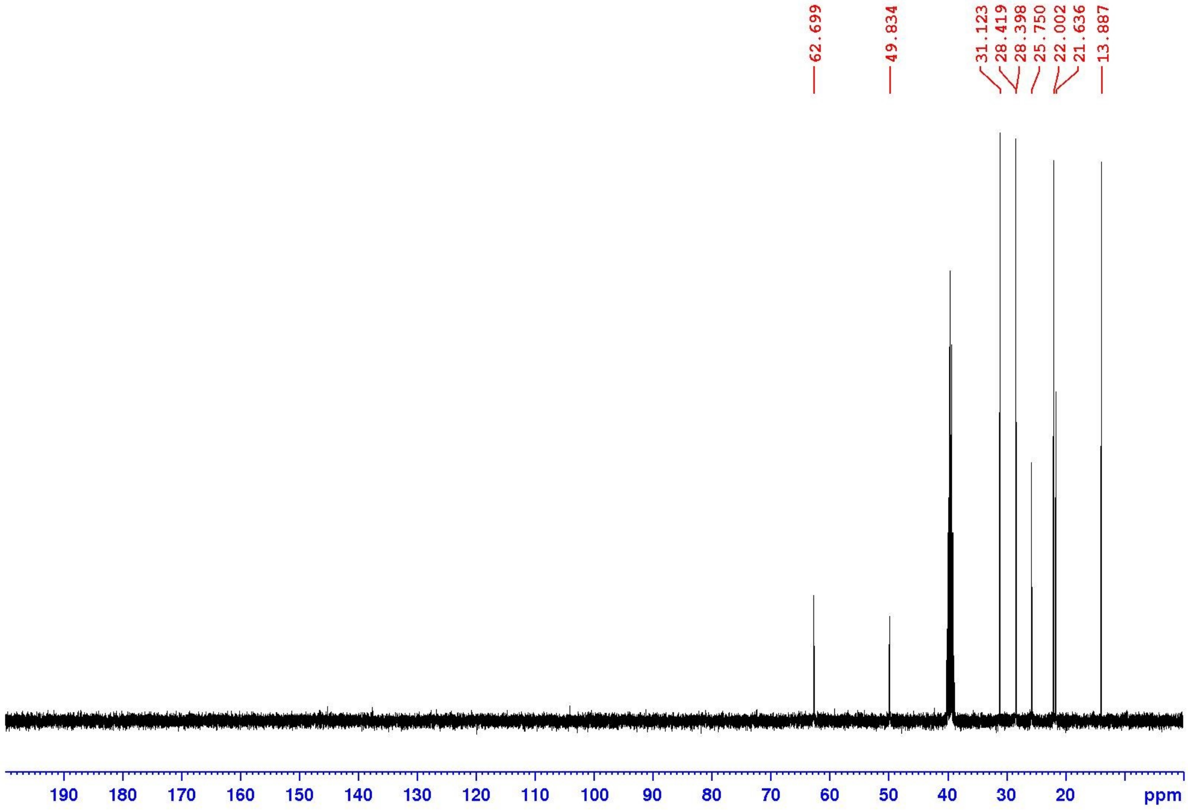
^{13}C NMR spectrum (101 MHz, DMSO-*d*6) of **52**



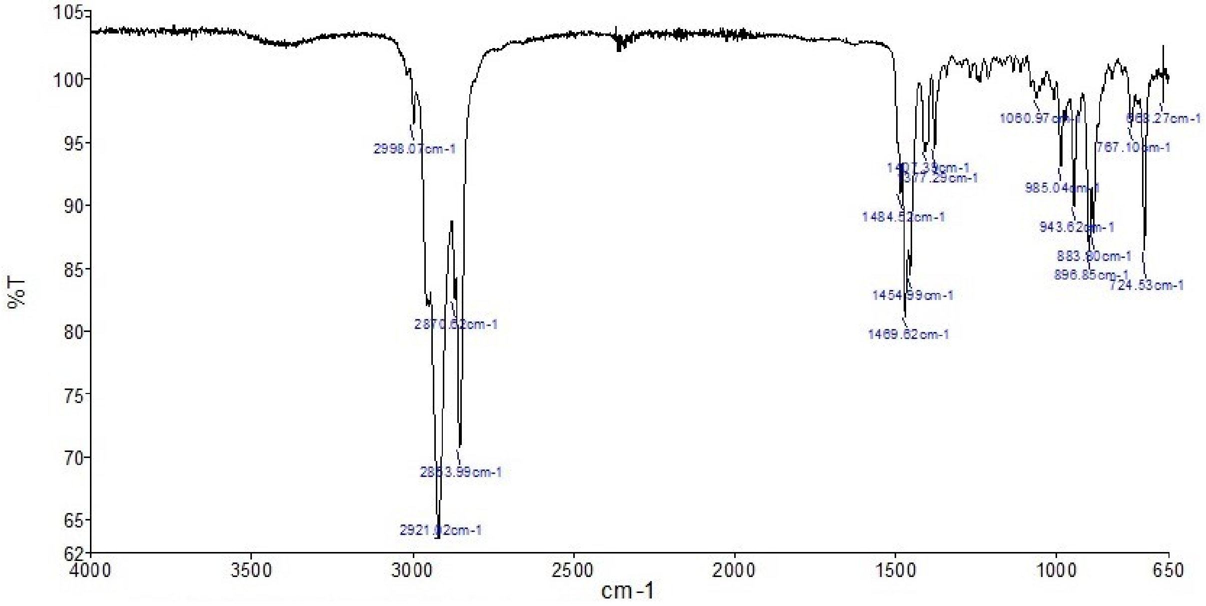
FT-IR spectrum of **52**



^{1}H NMR spectrum (400 MHz, DMSO-*d6*) of **53**



^{13}C NMR spectrum (101 MHz, DMSO-*d*6) of **53**



FT-IR spectrum of **53**

5.0 References

1. X. Wang, C. Wang, Y. Liu and J. Xiao, *Green Chem.*, 2016, **18**, 4605-4610.

2. M. Selva, M. Fabris, V. Lucchini, A. Perosa and M. Noè, *Org. Biomol. Chem.*, 2010, **8**, 5187-5198.

3. J. Cvengros, S. Toma, S. Marque and A. Loupy, *Can. J. Chem.*, 2004, **82**, 1365-1371

4. N. Lichtin, B. Wasserman, E. Clougherty, J. Wasserman and J. F. Reardon, *J. Phys. Chem.*, 1980, **84**, 2946–2952.