

Supplementary Material

Synthesis and antimicrobial activity of some new thienopyrimidine derivatives

Mahmoud S. Tolba,^a Adel M. Kamal El-Dean,^b Mostafa Ahmed,^{a*} Reda Hassaniien,^a and Mahmoud Farouk ^a^a Chemistry department, New Valley Faculty of Science, Assiut University, Assiut 72511, Egypt^b Chemistry department, Faculty of Science, Assiut University, Assiut 71516, EgyptE-mail: drmostafa@scinv.au.edu.eg

Mahmoud Tolba-MS2-DMSO-1H

Archive directory: /export/home/vnmr1/vnmr sys/data
Sample directory: DDSmm_test_12Mar2016-21: 34: 40

Pulse Sequence: s2pu1

Solvent: DMSO

Temp.: 30.0 C / 303.1 K

File: Mahmoud Tolba-MS2-DMSO-1H

Mercury-300BB "NMR300"

Relax. delay: 1.000 sec

Pulse: 45.0 degrees

Acq. time: 4.853 sec

width: 6600.7 Hz

48 repetitions

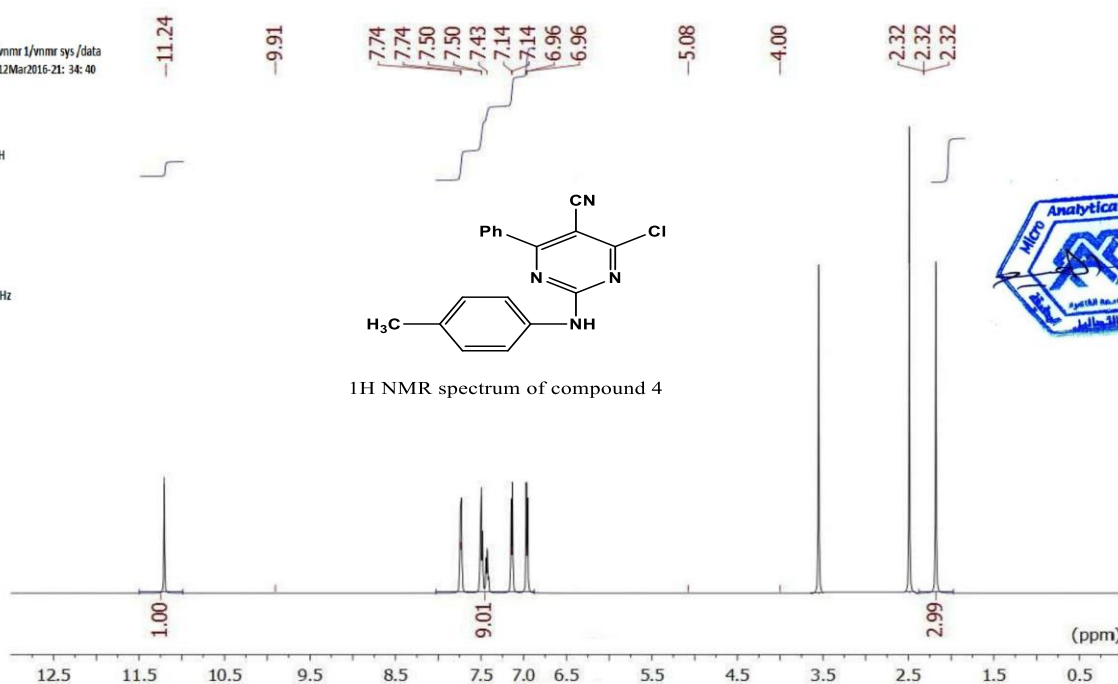
OBSERVE: 1H, 300.0687871 MHz

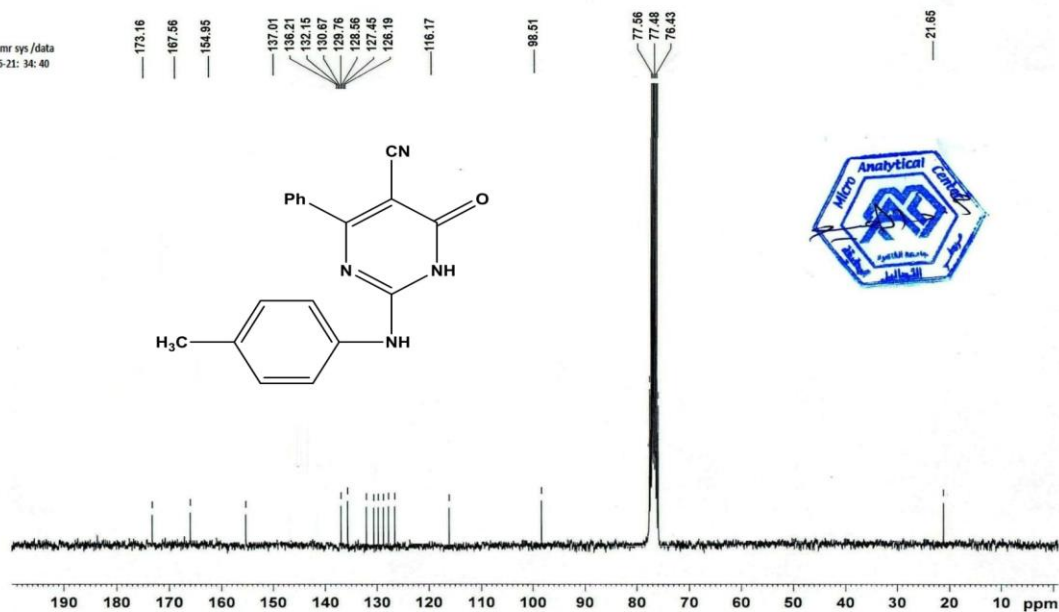
Data PROCESSING

FT Size: 65536

Total time: 58 min., 21 sec

Date: Jan 23 2017



Mahmoud Tolba-MS25-CDCl₃-13CArchive directory: /export/home/vnmr1/vnmr sys/data
Sample directory: D05mm_test_12Mar2016-21: 34: 40Pulse Sequence: s2pu1
Solvent: CDCl₃
Temp. 30.0 C / 303.1 K
File Mahmoud Tolba-MS25-CDCl₃-13C
Mercury-300BB "NMR300"Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 4.853 sec
width 6600.7 HZ
48 repetitions
OBSERVE 13C, 75.4.0687871 MHz
Data PROCESSING
FT Size 65536
Total time 1 h, 48 sec
Date: August. 9 2017

13C NMR of compound 3

Mahmoud Tolba-MS1-DMSO-13C

Archive directory: /export/home/vnmr1/vnmr sys/data
 Sample directory: D05mm_test_12Mar2016-21: 34: 40

Pulse Sequence: s2pu1

Solvent: DMSO

Temp. 30.0 C / 303.1 K

File Mahmoud Tolba-MS20-DMSO-13C

Mercury-300BB "NMR300"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 4.853 sec

width 6600.7 Hz

48 repetitions

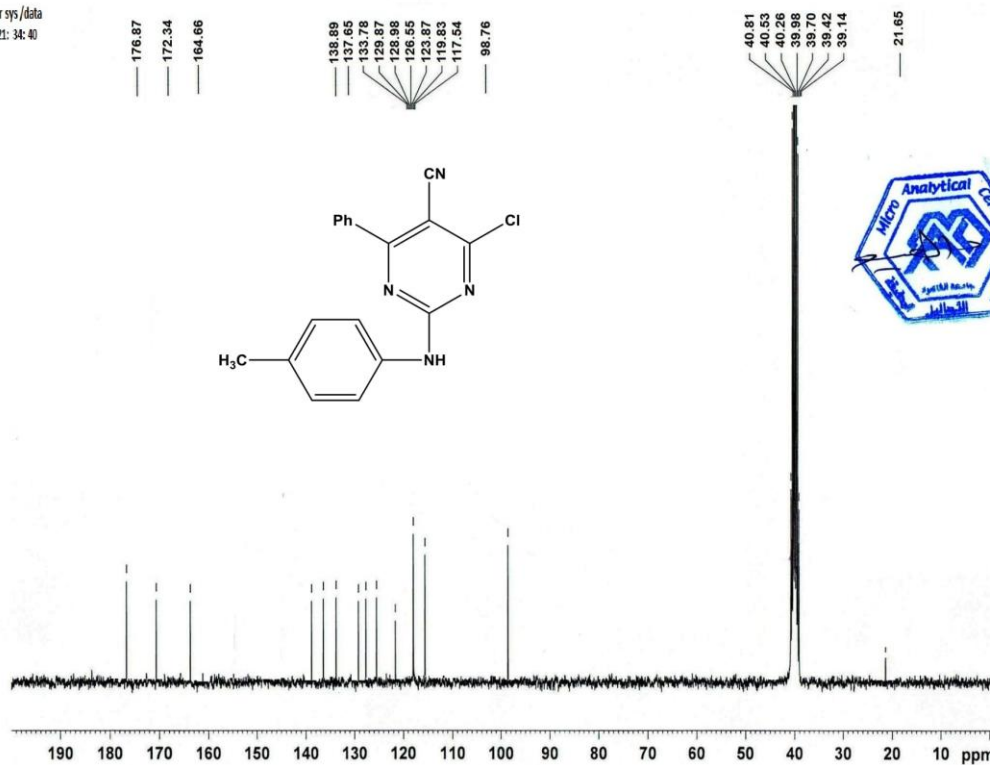
OBSERVE 13C, 75.40687871 MHz

Data PROCESSING

FT Size 65536

Total time 2 h, 21 sec

Date August 9 2017



13C NMR of compound 4

Mahmoud Tolba-MS85-DMSO-13C

Archive directory: /export/home/vnmr1/vnmr sys/data
 Sample directory: D05mm_test_12Mar2016-21: 34: 40

Pulse Sequence: s2pu1

Solvent: DMSO

Temp. 30.0 C / 303.1 K

File Mahmoud Tolba-MS20-DMSO-13C

Mercury-300BB "NMR300"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 4.853 sec

width 6600.7 Hz

48 repetitions

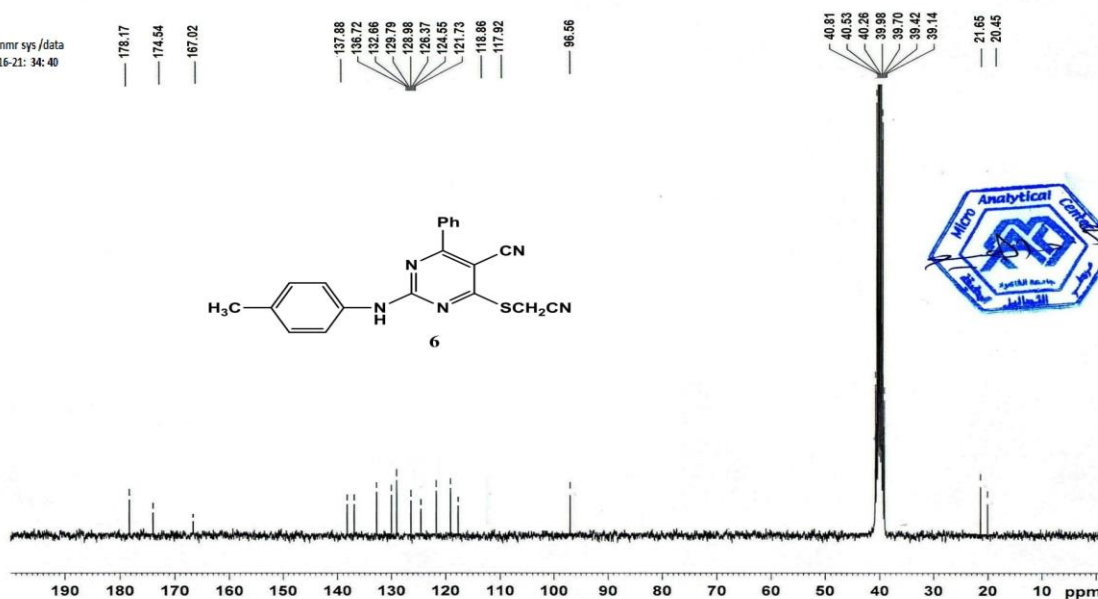
OBSERVE 13C, 75.40687871 MHz

Data PROCESSING

FT Size 65536

Total time 2 h, 21 sec

Date Jan 23 2017



13C NMR of compound 6

Mahmoud Tolba-MS48-CDCl₃-13CArchive directory: /export/home/vnmr1/vnmr sys/data
Sample directory: DD5mm_test_12Mar2016-21:34:40

Pulse Sequence: s2pu1

Solvent: CDCl₃

Temp: 30.0 C / 303.1 K

File Mahmoud Tolba-MS31-CDCl₃-13C

Mercury-3000B "NMR300"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 4.853 sec

width 6600.7 HZ

48 repetitions

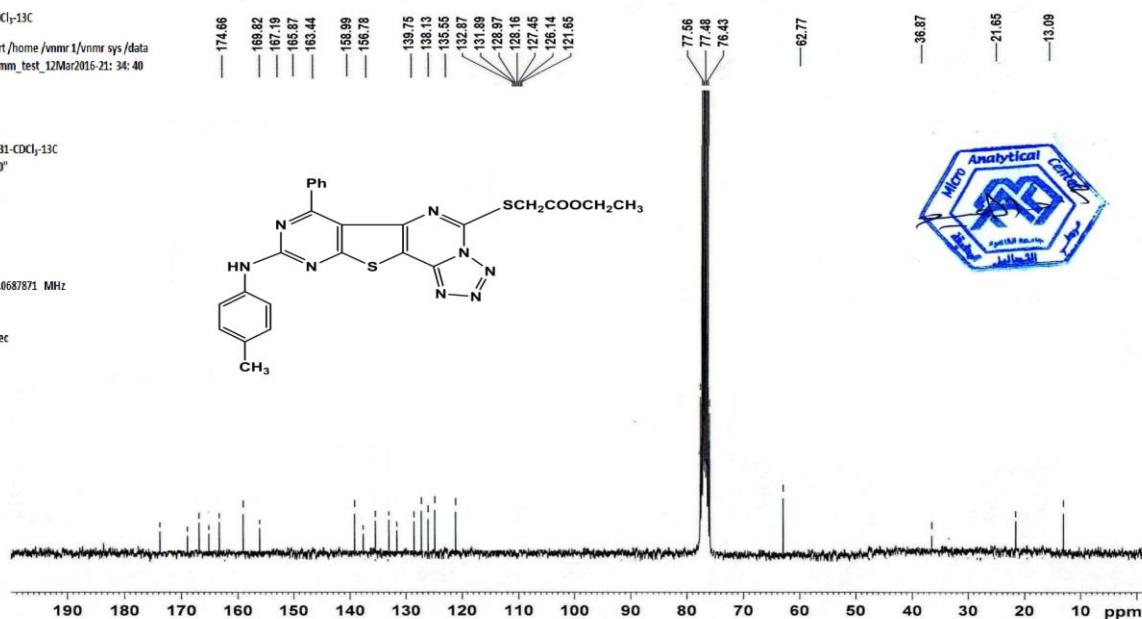
OBSERVE 13C, 75.4.0687871 MHz

Data PROCESSING

FT Size 65536

Total time 1h, 48 sec

Date: Jan 23 2017



13C NMR of compound 12 a

Mahmoud Tolba-MS88-DMSO-13C

Archive directory: /export/home/vnmr1/vnmr sys/data
Sample directory: DD5mm_test_12Mar2016-21:34:40

Pulse Sequence: s2pu1

Solvent: DMSO

Temp: 30.0 C / 303.1 K

File Mahmoud Tolba-MS20-DMSO-13C

Mercury-3000B "NMR300"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 4.853 sec

width 6600.7 HZ

48 repetitions

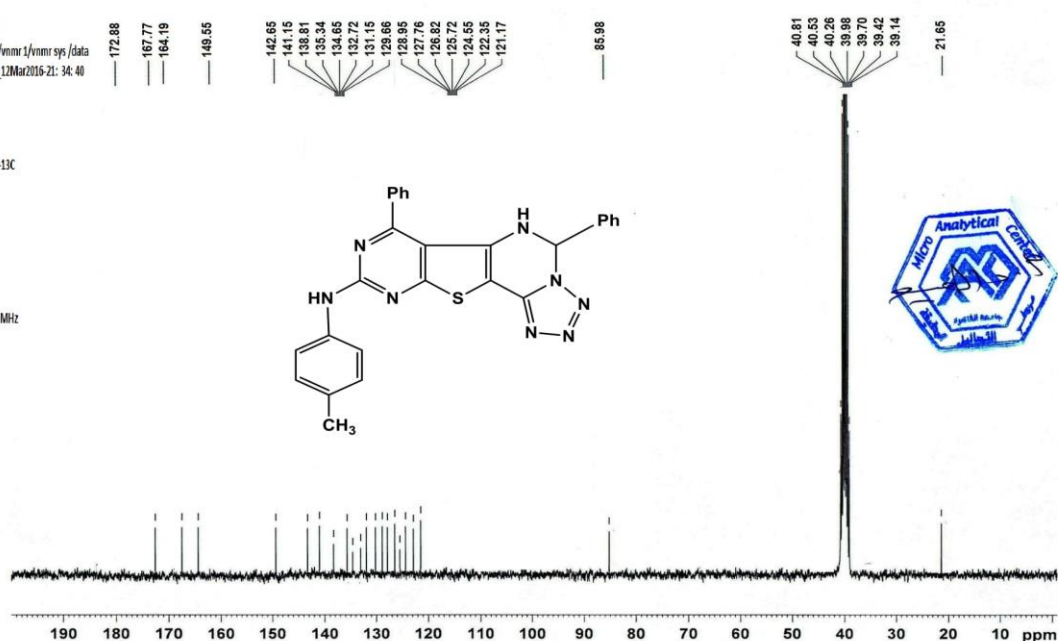
OBSERVE 13C, 75.4.0687871 MHz

Data PROCESSING

FT Size 65536

Total time 2h, 21 sec

Date Jan 23 2017



13C NMR of compound 10

Mahmoud Tolba-MS20-DMSO-13C

Archive directory: /export/home/vnmr1/vnmr sys/data
Sample directory: D05mm_test_12Mar2016-21: 34: 40

Pulse Sequence: s2pu1

Solvent: DMSO

Temp.: 30.0 C / 303.1 K

File: Mahmoud Tolba-MS20-DMSO-13C

Mercury-300BB "NMR300"

Relax. delay: 1.000 sec

Pulse: 45.0 degrees

Acq. time: 4.853 sec

width: 6600.7 HZ

48 repetitions

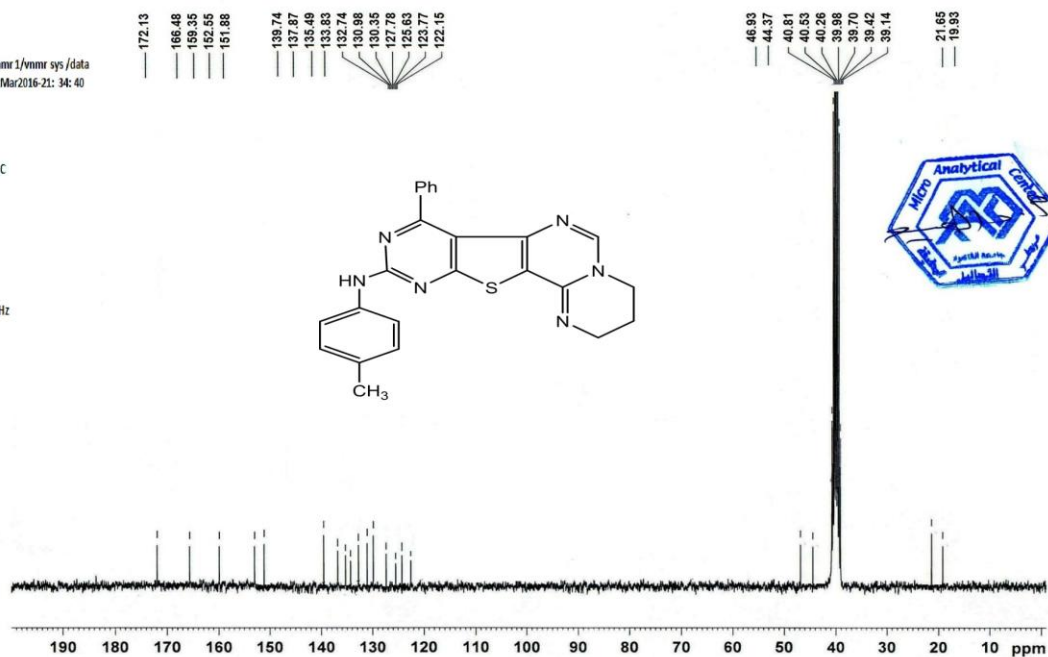
OBSERVE: 13C, 75.4.0687871 MHz

Data PROCESSING

FT Size: 65536

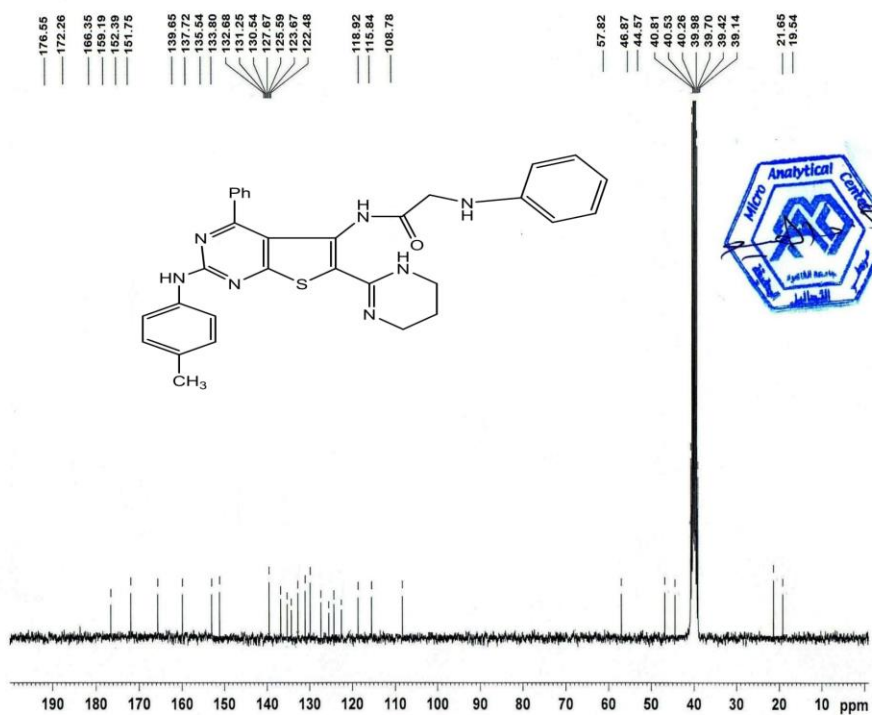
Total time: 2 h, 21 sec

Date: August 23 2017

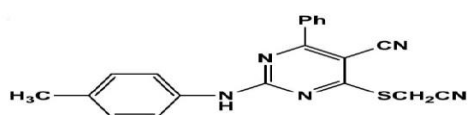
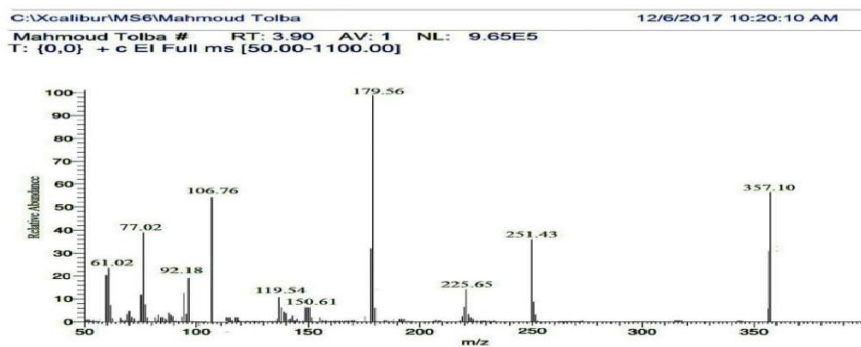


¹³C NMR of compound 14

Mahmoud Tolba-M556-DMSO-13C

Archive directory: /export/home/vnmr1/vnmr/sys/data
Sample directory: D05mm_test_12Mar2016-21: 34:40Pulse Sequence: s2pu1
Solvent: DMSO
Temp: 30.0 C / 303.1 K
File: Mahmoud Tolba-M556-DMSO-13C
Mercury-3000B "NMR300"Relax. delay: 1.000 sec
Pulse: 45.0 degrees
Acq. time: 4.853 sec
width: 6000.7 Hz
48 repetitions
OBSERVE: 13C, 75.40607871 MHz
Data PROCESSING
FT Size: 65536
Total time: 3 h., 21 sec
Date: August 22, 2017

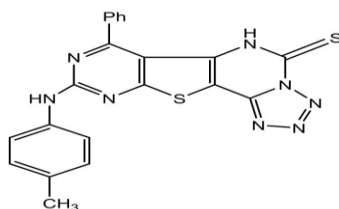
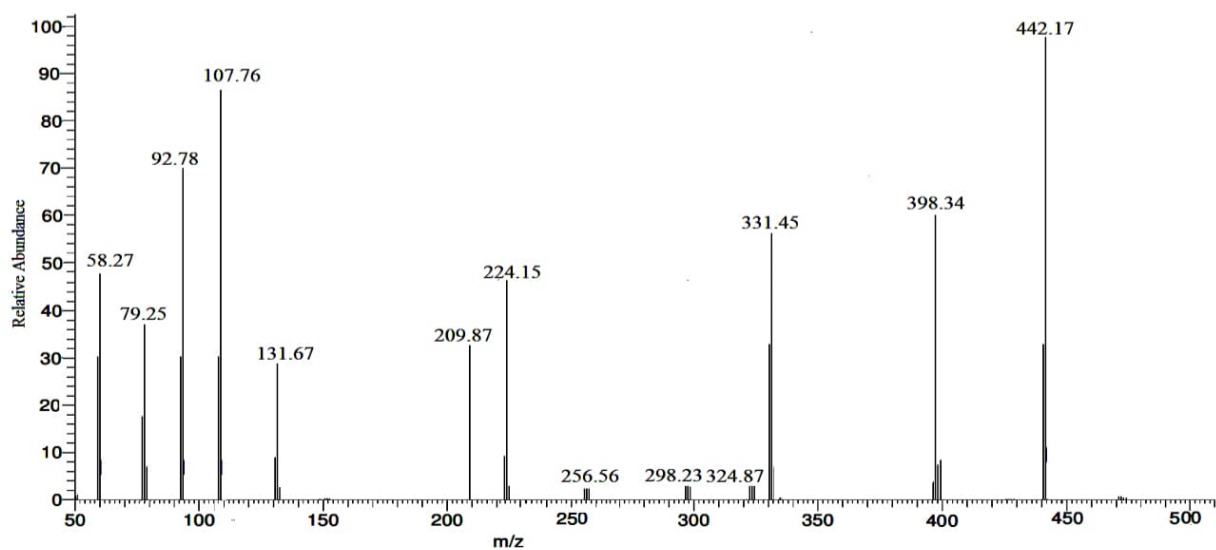
13C NMR of compound 18



Mass spectrum of compound 6

C:\Xcalibur\MS34\Mahmoud Tolba

26/6/2017 12:50:30 AM

Mahmoud Tolba # RT: 3.90 AV: 1 NL: 9.65E5
T: {0,0} + c EI Full ms [50.00-1100.00]**Compound 11**

Antimicrobial screening

The antibacterial activity of all synthesized compounds was tested *in vitro* against *Staphylococcus aureus* and *Bacillus cereus* as Gram-positive bacteria and *Escherichia coli* and *Pseudomonas aeruginosa* as Gram-negative bacteria. The inhibition zone (mm) was compared with a series of antibiotics according to the sensitivity of each type of bacteria to the most effective antibiotic for it as a standard. The compounds were also screened for their antifungal activities against four antifungal species, *Candida albicans*, *Geotrichum candidum*, *Aspergillus flavus*, *Trichophyton rubrum*. The inhibition zone (mm) was compared with Clotrimazole as a standard. These strains are common contaminants of the environment in Egypt and all microbial strains were kindly provided by the Biotechnology Laboratory, Assiut Sugar Technology Research, Assiut University. To prepare inocula for the bioassay, bacterial strains were individually cultured for 48 h in 100 mL conical flasks containing 30 mL nutrient broth medium. Fungi were grown for 7 d in 100 mL conicals containing 30 mL Sabouraud's dextrose broth. Bioassay was done in 10 cm sterile plastic Petri plates in which microbial suspension (1 mL/ plate) and 15 mL appropriate agar medium (15 mL/plate) were poured. Nutrient agar and Sabouraud's dextrose agar were respectively used for bacteria and fungi. After solidification of the media, 5 mm diameter cavities were cut in the solidified agar (4 cavities/plate) using a sterile cork borer. Chemical compounds dissolved in DMSO at 2% weight per volume (w/v) (=20 mg/mL) were pipetted in the cavities (20 μ L/cavity). Cultures were then incubated at 28 °C for 48 h in case of bacteria and up to 7 d in case of fungi. Results were determined as the diameter (in mm) of the inhibition zone around cavities. To determine the MICs, chemical compounds giving positive results were diluted with DMSO to prepare a series of descending concentrations down to 0.02 mg/mL. Diluted chemicals were similarly assayed as mentioned before and the least concentration (below which no activity) was recorded as the MIC. The screening tests were carried out in triplicate and the results were expressed as a mean of three determinations.