

Supplementary Information

Direct chemoselective synthesis of N-3 substituted pyrimidinones in a microwave-assisted method

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Graphical abstract

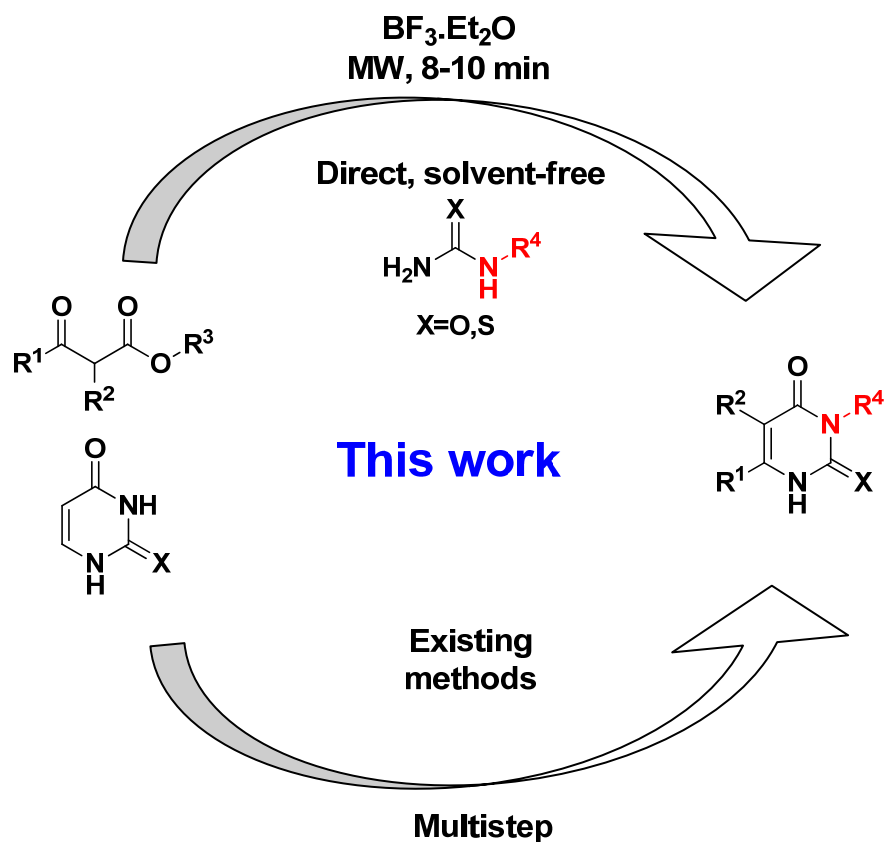


Table of Contents

1. General Information
2. Procedure for the products
3. Characterization Data
4. ^1H , ^{13}C NMR and NOESY spectra
5. HRMS and XRD data
6. References for melting point data of the already reported compounds

1. General Information: All chemicals were purchased from reputed pharmaceuticals and were used without further purification. All microwave-directed reactions were carried out in a closed vessel *CEM Discover LabMate* microwave reactor at about 145°C for variable durations. The temperature of the reaction mixtures were all measured by an internal built-in IR sensor. ^1H -NMR (400 MHz) and ^{13}C -NMR (100MHz) were all recorded from a *DRX-400 Varian spectrometer* using CDCl_3 and DMSO-D_6 as solvents. Chemical shifts are reported in parts per million (ppm). Melting points were determined using *Büchi B-545* apparatus and are uncorrected. High resolution mass spectrometry was analyzed from *Agilent Q-TOF 6500 LC/MS* system and *Micromass Q-TOF ESI-MS* instrument (model HAB 273). X-Ray data were collected from a *Bruker SMART APEX* equipped with a CCD area detector using Mo. The structures were solved by direct method using *SHELLX-97* (Göttingen, Germany). The melting points, characterization and relevant literature of the reported compounds are given.

2. Procedure for the products

A). General Procedure (compound 1-19): A β -ketoester (2 mmol), taken in a reactor vessel was mixed thoroughly for 1 min with urea derivative (2.6 mmol). The vessel was closed immediately and was subjected to microwave irradiation at about 145°C. Reactions were also performed at 130°C and 140°C, however, best results were obtained at 145°C. The compound (**1-18**) was further purified by column chromatography (50-65% ethyl acetate in hexane). The time of irradiation and observed yield of the compounds are listed in Table 1.

Synthesis using $\text{BF}_3 \cdot \text{Et}_2\text{O}$ (1-19): A β -ketoester (2 mmol), taken in a reactor vessel with $\text{BF}_3 \cdot \text{Et}_2\text{O}$ (339 mg, 2.4 mmol) was mixed thoroughly for 1 min with urea derivatives (2.6 mmol). The vessel was closed immediately and was subjected to microwave irradiation at 145°C. The compound (**1-18**) was further purified by column chromatography. The time of irradiation and observed yield of the compounds are listed in Table 1.

B). Procedure for synthesis of compound 4: Ethyl 3-oxo-3-phenylpropanoate (**4a**, 2mmol), taken in a reactor vessel, was mixed thoroughly for 1 min with Methyl urea (2.6mmol). The vessel was closed immediately and was subjected to microwave irradiation for 12 min at about 145°C. The completion of reaction was monitored by checking TLC at regular time interval. Compound (**4**) was further purified by column chromatography (Silica gel 60-120 mesh, 60% ethyl acetate in hexane).

C). Procedure for synthesis of compound 4 with Lewis acid: Ethyl 3-oxo-3-phenylpropanoate (**4a**, 2mmol), taken in a reactor vessel with $\text{BF}_3 \cdot \text{Et}_2\text{O}$ (339mg, 2.4mmol) was mixed thoroughly for 1 min with urea (2.6mmol). The vessel was closed immediately and was subjected to microwave irradiation at 145°C for about 8 min. Reaction was complete within 8 min irradiation, which was verified by TLC. Compound (**4**) was further purified by column chromatography (Silica gel 60-120 mesh, 60% ethyl acetate in hexane).

3. Characterization Data

3,6-dimethylpyrimidine-2,4(1H,3H)-dione (1):

Yield: 85%, white solid, m.p: 260-265°C (lit ^{1, 2}), ¹H NMR (400 MHz, DMSO-d₆) δ 11.13 (s, 1H), 5.47 (s, 1H), 3.22 (s, 3H), 2.20 (s, 3H). ¹³C NMR (100 MHz, DMSO-d₆) δ 167.8, 153.6, 152.3, 99.6, 27.8, 19.1. HRMS (ESI) m/z [M+H]⁺ calculated (C₆H₈N₂O₂): 141.0659; observed: 141.0659.

6-ethyl-3-methylpyrimidine-2,4(1H,3H)-dione (2):

Yield: 80%, white solid, m.p: 240-243°C, ¹H NMR (400 MHz, DMSO-d₆) δ 11.08 (s, 1H), 5.44 (s, 1H), 3.08 (s, 3H), 2.33 (t, 2H, *J* = 7.8 Hz), 1.14 (t, 3H, *J* = 7.2 Hz). ¹³C NMR (100 MHz, DMSO-d₆) δ 157.3, 152.5, 150.9, 97.2, 27.9, 25.5, 12.1. HRMS (ESI) m/z [M+H]⁺ calculated (C₇H₁₀N₂O₂): 155.0815; observed: 155.0811.

3-methyl-6-propylpyrimidine-2,4(1H,3H)-dione (3):

Yield: 78%, white solid, m.p: 245-248°C, ¹H NMR (400 MHz, DMSO-d₆) δ 11.07 (s, 1H), 5.45 (s, 1H), 3.08 (s, 3H), 2.28 (t, 2H, *J* = 7.2 Hz), 1.58-1.54 (m, 2H), 0.89 (t, 3H, *J* = 7.6 Hz). ¹³C NMR (100 MHz, DMSO-d₆) δ 160.0, 156.4, 153.0, 98.5, 31.1, 27.5, 21.3, 14.2. HRMS (ESI) m/z [M+H]⁺ calculated (C₈H₁₂N₂O₂): 169.0972; observed: 169.0969.

Crystal data: CCDC# 991094; C₈ H₁₂ N₂ O₂; M = 168.20, m.p. = 245-248°C, monoclinic; P2₁/c, a = 4.7152(8)Å; b = 21.823(3)Å, c = 8.8290(15)Å, α = 90°, β = 94.553(11)°, γ = 90°, V = 905.6(3) Å³, Z = 4, μ = 0.090 m^{m-1}, ρ = 1.234 g.c^{m-3}, Mo-Kα radiation, R1 = 0.1520, wR2 = 0.1299, S = 0.922.

3-methyl-6-phenylpyrimidine-2,4(1H,3H)-dione (4):

Yield: 90%, white solid, m.p: 230-232°C (lit ¹), ¹H NMR (400 MHz, DMSO-d₆) δ 11.41 (s, 1H), 7.74 (d, 2H, *J* = 6.8 Hz), 7.56-7.49 (m, 3H), 5.96 (s, 1H), 3.17 (s, 3H). ¹³C NMR (100 MHz, DMSO-d₆) δ 164.3, 152.6, 151.7, 132.0, 129.6, 128.4, 127.5, 97.5, 27.3. HRMS (ESI) m/z [M+H]⁺ calculated (C₁₁H₁₀N₂O₂): 203.0815; observed: 203.0817.

Crystal data: CCDC# 991093; C₁₁ H₁₀ N₂ O₂; M = 202.21, m.p. = 230-232°C, monoclinic; P2₁/n, a = 5.8924(19)Å; b = 21.161(6)Å, c = 8.054(3)Å, α = 90°, β = 103.67(2)°, γ = 90°, V =

975.8(5)Å, $Z = 4$, $\mu = 0.097 \text{ m}^{\text{m-1}}$, $\rho = 1.376 \text{ g} \cdot \text{cm}^{-3}$, Mo-K α radiation, $R1 = 0.0621$, $wR2 = 0.1032$, $S = 0.968$.

6-isopropyl-3-methylpyrimidine-2,4(1H,3H)-dione (5):

Yield: 75%, white solid, m.p: 235-238°C, ^1H NMR (400 MHz, CDCl_3) δ 10.52 (s, 1H), 5.53 (s, 1H), 3.24 (s, 3H), 2.59-2.55 (m, 1H), 1.19 (d, 2H, $J = 7.2 \text{ Hz}$). ^{13}C NMR (100 MHz, DMSO-d_6) δ 164.2, 159.3, 153.9, 97.2, 31.9, 27.1, 20.4. HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calculated ($\text{C}_8\text{H}_{12}\text{N}_2\text{O}_2$): 169.0972; observed: 169.0978.

6,7-dihydro-3-methyl-1H-cyclopenta[d]pyrimidine-2,4(3H,5H)-dione (6):

Yield: 73%, white solid, m.p: 225-228°C, ^1H NMR (400 MHz, DMSO-d_6) δ 11.38 (s, 1H), 3.09 (s, 3H), 2.67 (t, 2H, $J = 7.2 \text{ Hz}$), 2.50-2.47 (m, 2H), 1.97 (t, 2H, $J = 7.2 \text{ Hz}$). HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calculated ($\text{C}_8\text{H}_{10}\text{N}_2\text{O}_2$): 167.0815; observed: 167.0814.

3-benzyl-6-methylpyrimidine-2,4(1H,3H)-dione (7):

Yield: 72%, white solid, m.p: 194-198°C (lit ^{3, 4}), ^1H NMR (400 MHz, DMSO-d_6) δ 10.43 (s, 1H), 7.40 (d, 2H, $J = 6.8 \text{ Hz}$), 7.27-7.22 (m, 3H), 5.50 (s, 1H), 5.03 (s, 3H), 2.04 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 163.5, 158.6, 152.0, 137.0, 128.2, 128.1, 127.1, 99.1, 42.9, 18.4. HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calculated ($\text{C}_{12}\text{H}_{12}\text{N}_2\text{O}_2$): 217.0972; observed: 217.0973.

3-benzyl-6-propylpyrimidine-2,4(1H,3H)-dione (8):

Yield: 70%, white solid, m.p: 200-202°C ^1H NMR (400 MHz, CDCl_3) δ 10.43 (s, 1H), 7.42 (d, 2H, $J = 6.4 \text{ Hz}$), 7.28-7.23 (m, 3H), 5.55 (s, 1H), 5.05 (s, 3H) 2.31 (t, 2H, $J = 6.4 \text{ Hz}$), 1.66-1.60 (m, 2H), 0.98 (t, 3H, $J = 6.4 \text{ Hz}$). ^{13}C NMR (100 MHz, CDCl_3) δ 163.1, 158.1, 154.3, 139.6, 127.6, 126.6, 126.1, 97.5, 42.5, 34.4, 19.8, 12.7. HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calculated ($\text{C}_{14}\text{H}_{17}\text{N}_2\text{O}_2$): 245.1285; observed: 245.1286.

3-benzyl-6-isopropylpyrimidine-2,4(1H,3H)-dione (9):

Yield: 65%, white solid, m.p: 215-220°C, NMR (400 MHz, CDCl_3) δ 10.36 (s, 1H), 7.45 (d, 2H, $J = 6.8 \text{ Hz}$), 7.30-7.26 (m, 3H), 5.59 (s, 1H), 5.06 (s, 2H) 2.59-2.55 (m, 1H), 1.23 (d, 6H, $J = 7.2 \text{ Hz}$). ^{13}C NMR (100 MHz, CDCl_3) δ 163.9, 159.6, 153.8, 136.9, 129.1, 128.4, 127.3, 97.5, 44.6, 31.9, 20.3. HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calculated ($\text{C}_{14}\text{H}_{17}\text{N}_2\text{O}_2$): 245.1285; observed: 245.1292.

3-benzyl-6-phenylpyrimidine-2,4(1H,3H)-dione (10):

Yield: 70%, white solid, m.p: 195-198°C ¹H NMR (400MHz, CDCl₃) δ 9.56 (s, 1H), 7.62 (d, 2H, *J*= 8.0 Hz), 7.42-7.35 (m, 8H), 6.01 (s, 1H), 5.12 (s, 2H), ¹³C NMR (100 MHz, CDCl₃) δ 158.9, 155.7, 153.3, 139.3, 138.5, 128.7, 128.6, 128.5, 127.4, 127.3, 126.2, 98.9, 44.5. HRMS (ESI) *m/z* [M+H]⁺ calculated (C₁₇H₁₄N₂O₂): 279.1128; observed: 279.1128.

3-benzyl-5-isopropyl-6-methylpyrimidine-2,4(1H,3H)-dione (11):

Yield: 40%, white solid, m.p: 180-183°C ¹H NMR (400 MHz, CDCl₃) δ 9.34 (s, 1H), 7.27-7.20 (m, 5H), 4.96 (s, 2H), 2.45 (m, 1H), 1.15 (d, 6H, *J*= 6.8 Hz). ¹³C NMR (100 MHz, CDCl₃) δ 163.8, 159.4, 153.8, 139.2, 137.0, 129.1, 128.8, 127.5, 107.6, 43.7, 32.0, 20.4. HRMS (ESI) *m/z* [M+H]⁺ calculated (C₁₅H₁₉N₂O₂): 259.1441; observed: 259.1444.

3-ethyl-6-phenylpyrimidine-2,4(1H,3H)-dione (12):

Yield: 64%, white solid, m.p: 220-223°C ¹H NMR (400 MHz, CDCl₃) δ 8.69 (s, 1H), 7.62-7.45 (m, 5H), 5.84 (s, 1H), 3.24 (q, 2H, *J*= 6.0 Hz), 1.15 (t, 3H, *J*= 7.2 Hz). ¹³C NMR (100 MHz, CDCl₃) δ 163.5, 152.8, 150.5, 136.2, 131.4, 126.5, 125.6, 98.7, 35.6, 14.5. HRMS (ESI) *m/z* [M+H]⁺ calculated (C₁₂H₁₃N₂O₂): 217.0972; observed: 217.0971.

3,6-diphenylpyrimidine-2,4(1H,3H)-dione (13):

Yield: 40%, white solid, m.p: 286-290°C (lit⁵) ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.42 (s, 1H), 7.82 (d, 2H, *J*= 8.0 Hz), 7.82-7.45 (m, 8H), 6.01 (s, 1H).

2,3-dihydro-3-methyl-6-phenyl-2-thioxopyrimidin-4(1H)-one (14):

Yield: 85%, white solid, m.p: 240-245°C ¹H NMR (400 MHz, CDCl₃) δ 10.48 (s, 1H), 7.53 (d, 2H, *J*= 7.2 Hz), 7.40-7.33 (m, 3H), 5.91 (s, 1H), 3.54 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 177.8, 159.7, 157.6, 133.5, 130.7, 129.4, 127.7, 108.4, 41.0

6-isopropyl-3-methyl-2-thioxo-2,3-dihydropyrimidin-4(1H)-one(15):

Yield: 78%, white solid, m.p: 260-263°C ¹H NMR (400 MHz, CDCl₃) δ 10.48 (s, 1H), 5.95 (s, 1H), 4.82 (s, 3H), 3.86 (m, 1H), 1.24 (d, 6H, *J*= 7.6 Hz). ¹³C NMR (100 MHz, CDCl₃) δ 178.1, 164.0, 103.7, 37.3, 31.0, 21.0

Crystal data: CCDC# 991092; C₈ H₁₂ N₂ O S; M = 184.26, m.p. = 260-263°C, monoclinic; C2/c, a = 21.5926(10)Å; b = 6.8375(3)Å, c = 14.9348(8)Å, $\alpha = 90^\circ$, $\beta = 122.333(4)^\circ$, $\gamma = 90^\circ$, V = 1863.09(16)Å³, Z = 8, $\mu = 0.302 \text{ m}^{-1}$, $\rho = 1.314 \text{ g}\cdot\text{cm}^{-3}$, Mo-K α radiation, R1 = 0.0424, wR2 = 0.0766, S = 1.083

3-methyl-2-thioxo-2,3,6,7-tetrahydro-1H-cyclopenta[d]pyrimidin-4(5H)-one(16):

Yield: 75%, white solid, m.p: 296-300°C (lit ⁶), ¹H NMR (400 MHz, DMSO-d₆) δ 12.89 (s, 1H), 3.35 (s, 1H), 2.74 (t, $J = 7.2 \text{ Hz}$), 2.55 (m, 2H), 1.98 (t, $J = 7.2 \text{ Hz}$).

3-methyl-5-(naphthalen-1-ylmethyl)-6-phenyl-2-thioxo-2,3-dihydropyrimidin-4(1H)-one(17):

Yield: 59%, white solid, m.p: 305-308°C, ¹H NMR (400 MHz, CDCl₃) δ 9.82 (s, 1H), 7.86-7.68 (m, 4H), 7.40-7.25 (m, 6H), 6.92-6.85 (m, 2H), 3.90 (s, 3H), 3.79 (s, 2H) ¹³C NMR (100 MHz, CDCl₃) δ 176.1, 160.5, 155.0, 133.8, 132.3, 131.7, 131.1, 130.2, 129.6, 129.3, 128.8, 128.7, 127.6, 127.4, 125.4, 124.6, 124.4, 117.7, 41.2, 29.9. HRMS (ESI) m/z [M+H]⁺ calculated (C₂₂H₁₉N₂OS): 359.1213; observed: 359.1213.

3-ethyl-2,3-dihydro-6-phenyl-2-thioxopyrimidin-4(1H)-one (18):

Yield: 62%, white solid, m.p: 206-208°C, ¹H NMR (400 MHz, DMSO-d₆) δ 9.25 (s, 1H), 7.82 (d, 2H, $J = 7.4 \text{ Hz}$), 7.58 (t, 2H, $J = 7.6 \text{ Hz}$), 5.97 (s, 1H), 3.35 (q, 2H, $J = 6.8 \text{ Hz}$), 1.21 (t, 3H, $J = 7.2 \text{ Hz}$). ¹³C NMR (100 MHz, DMSO-d₆) δ 174.2, 161.5, 149.5, 139.4, 128.0, 127.6, 126.9, 113.8, 43.0, 16.0. HRMS (ESI) m/z [M+H]⁺ calculated (C₁₂H₁₃N₂OS): 233.0743; observed: 233.0749.

Intermediates:

(Z)-Ethyl 3-(3-methylureido) but-2-enoate (Intermediate for 1):

LRMS (ES) [M+Na]⁺ calculated (C₈H₁₄N₂O₃Na): 209.090; found:209.8277.

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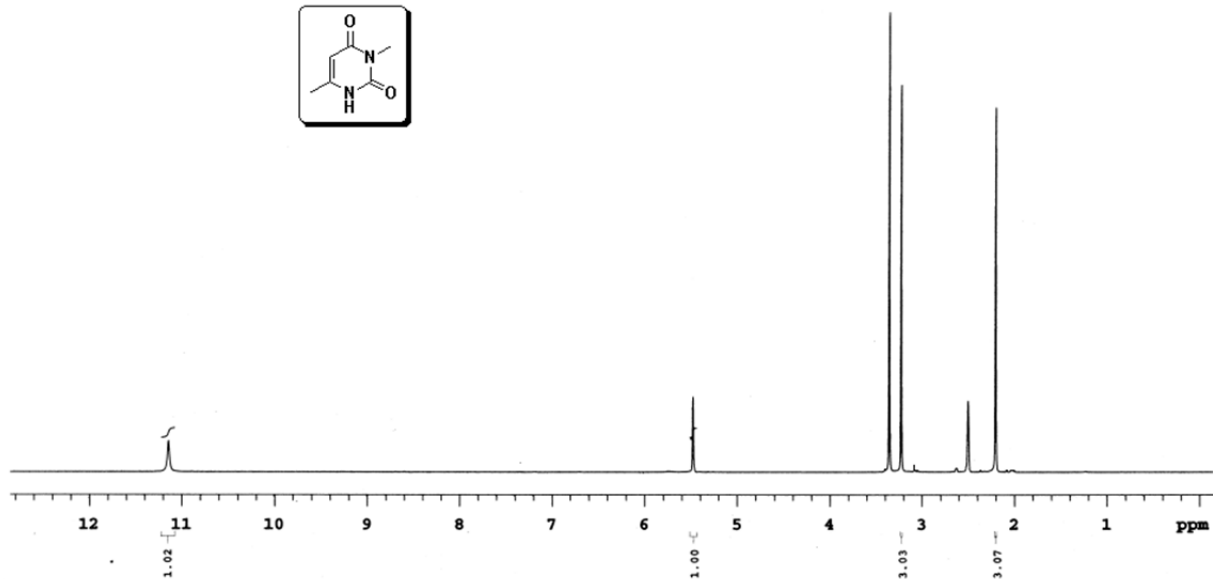
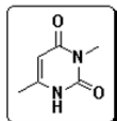
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LRMS (ES) [M+Na]⁺ calculated (C₁₀H₁₆N₂O₂SNa): 251.0830; found:251.5941

4. ^1H , ^{13}C NMR and NOESY spectra:

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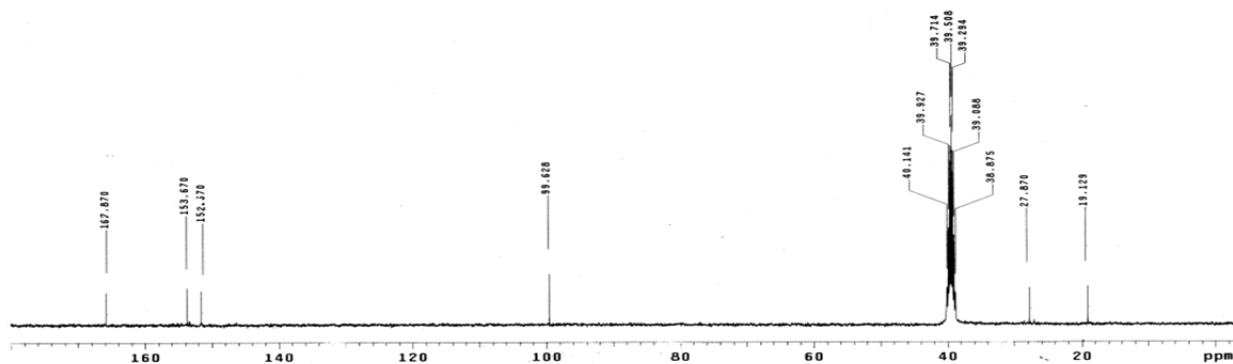
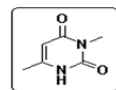


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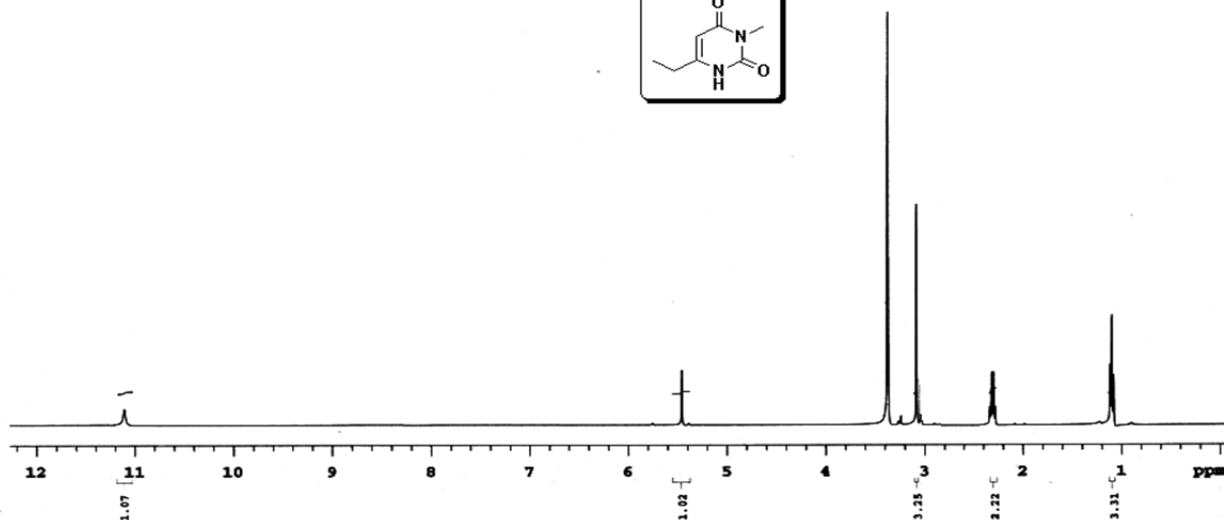
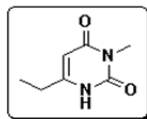
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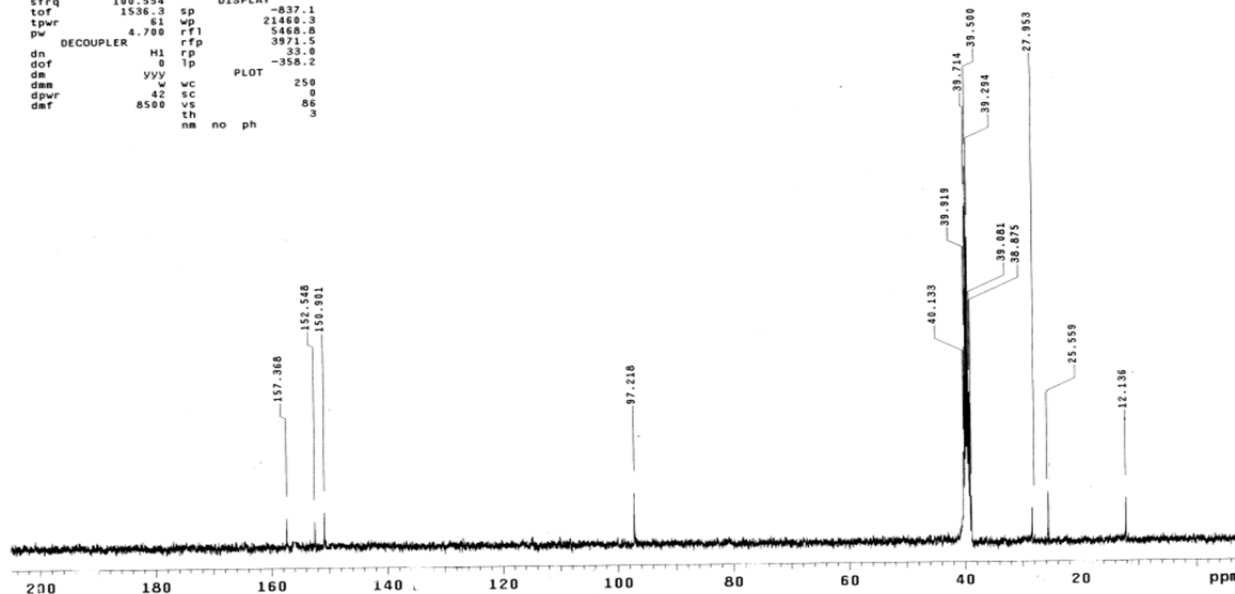
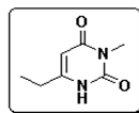
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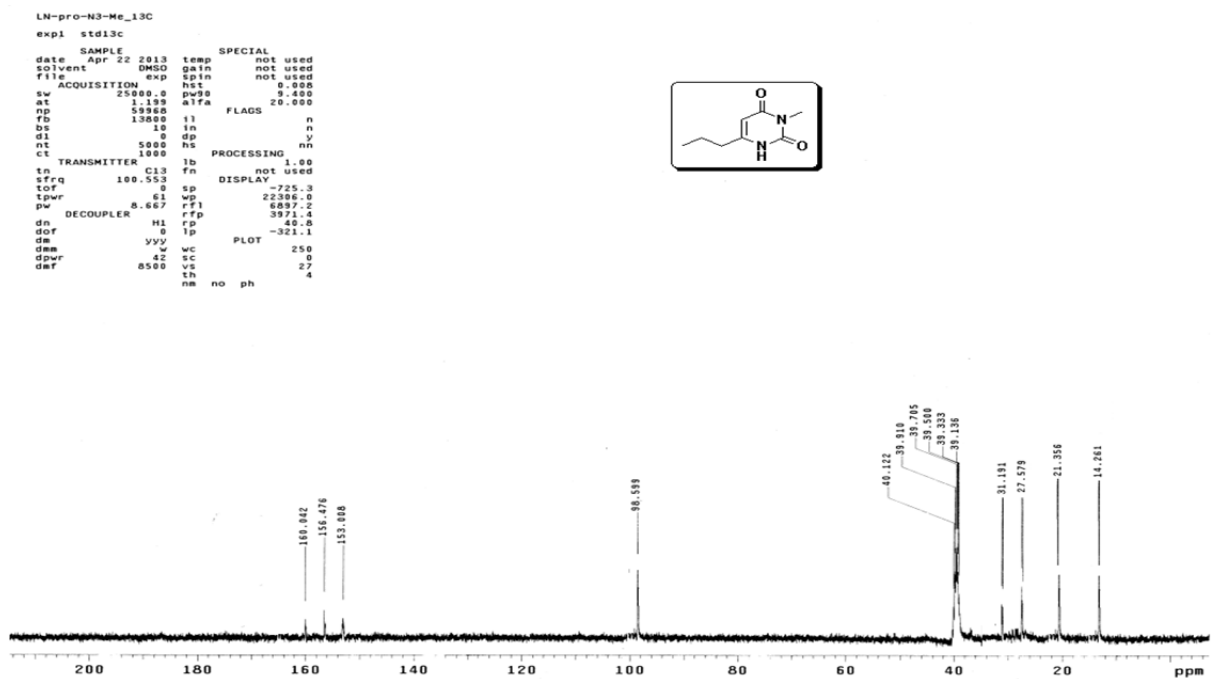
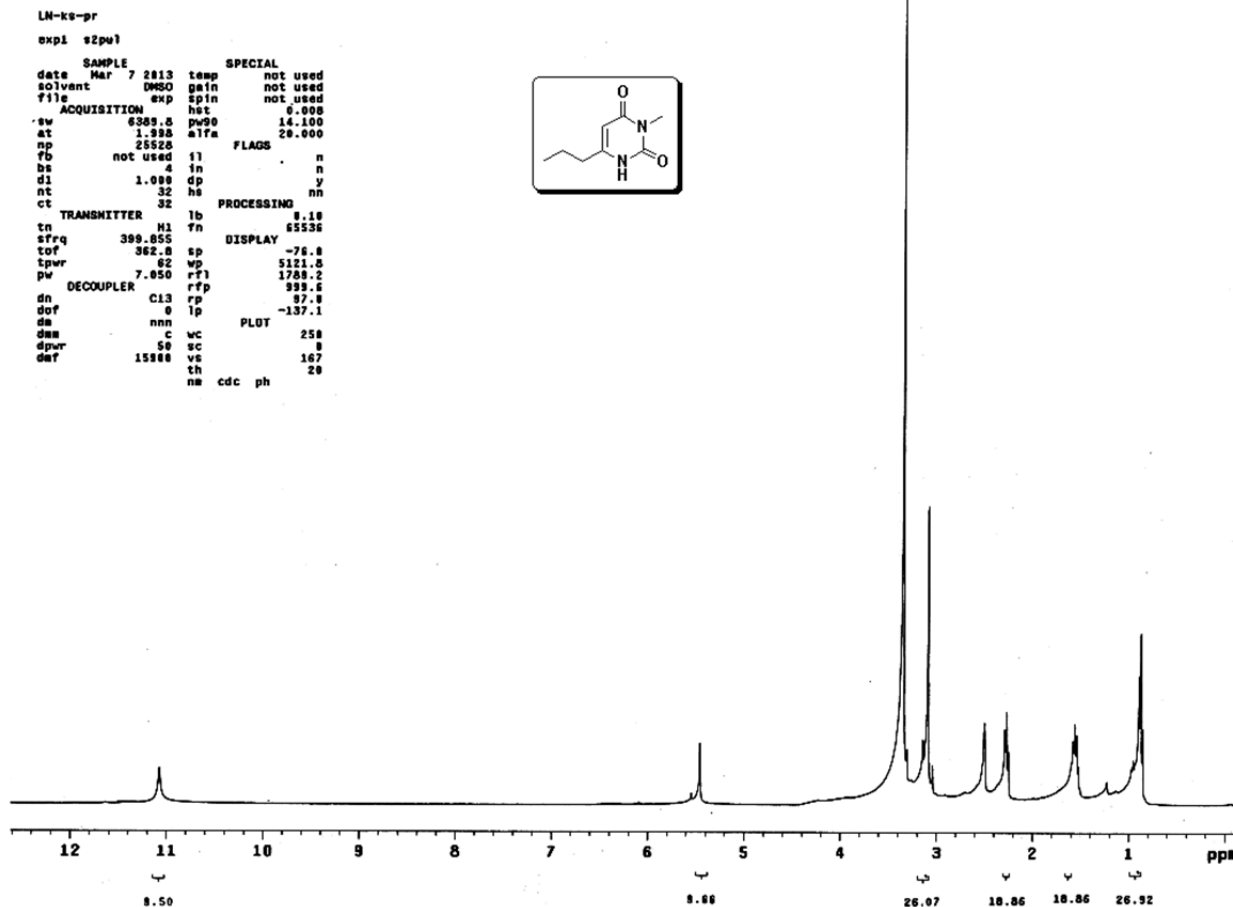
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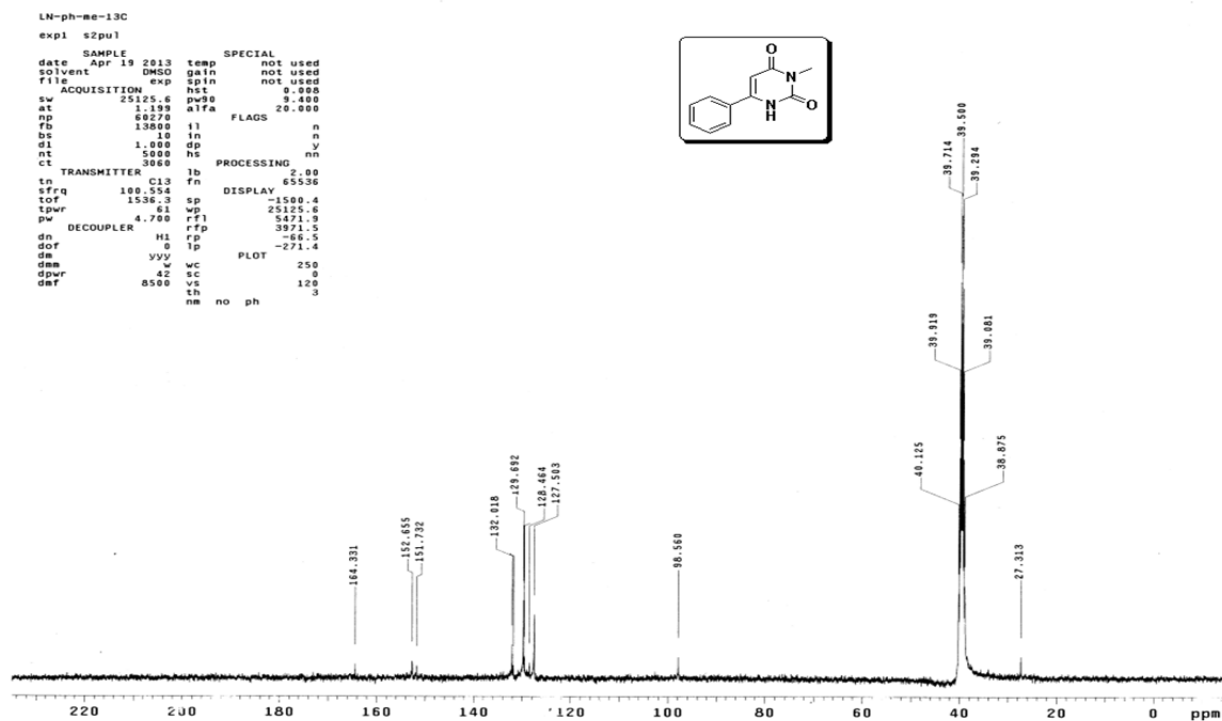
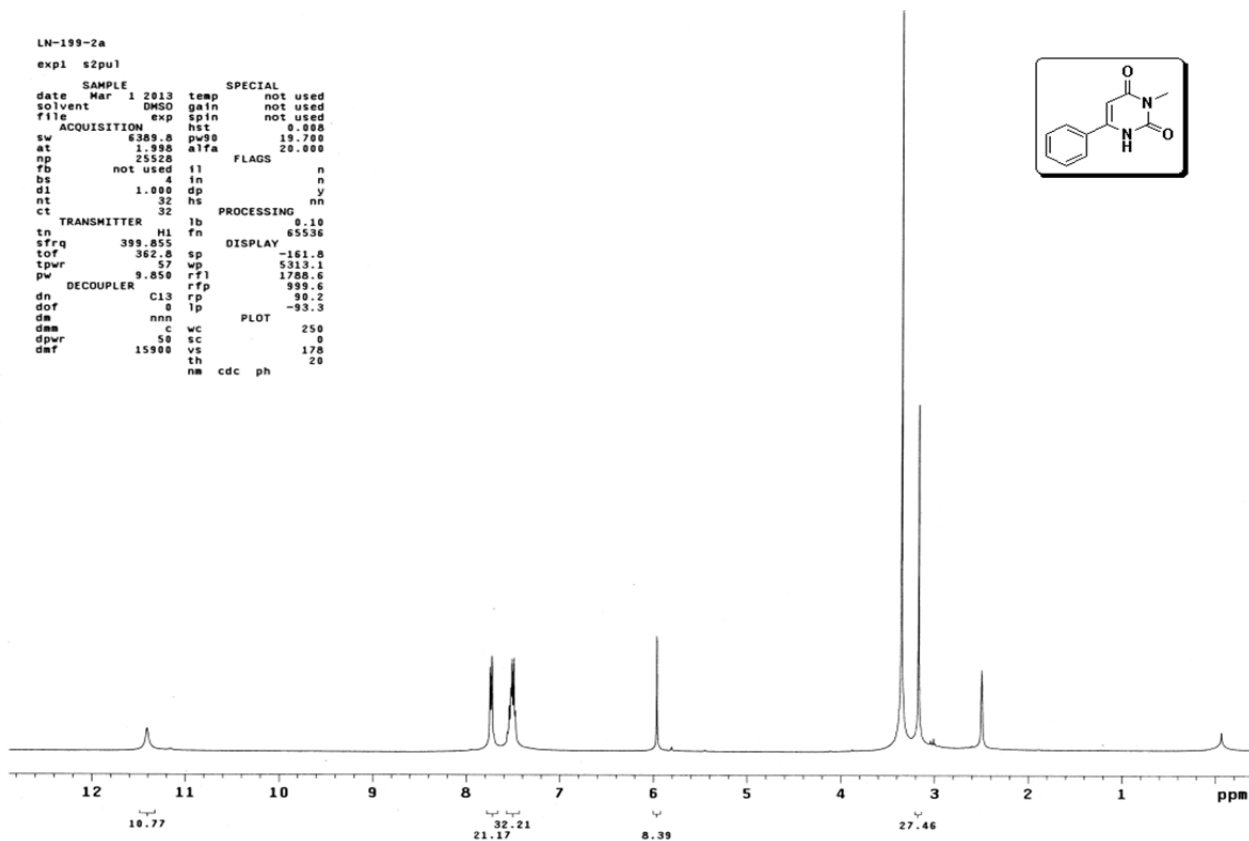
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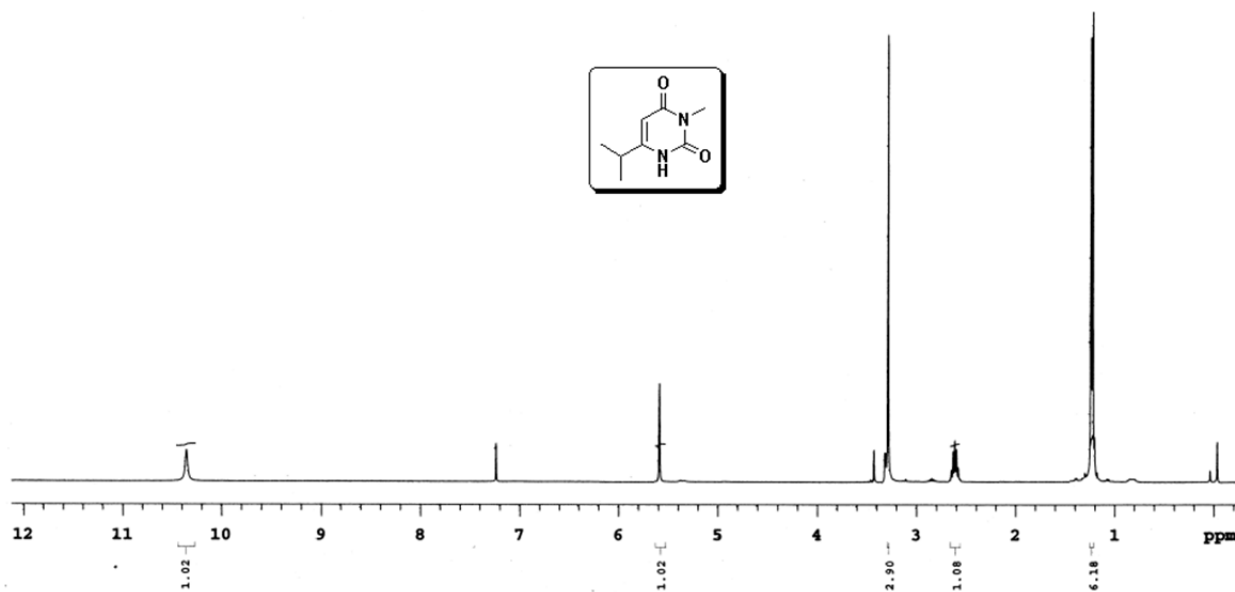
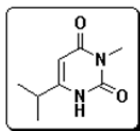
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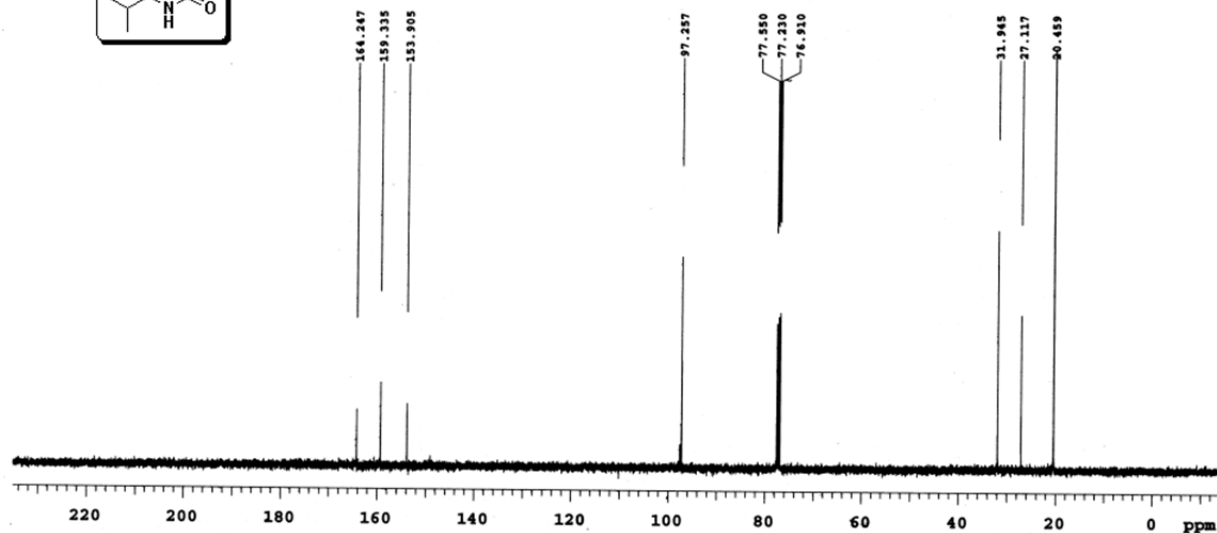
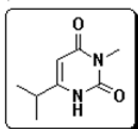
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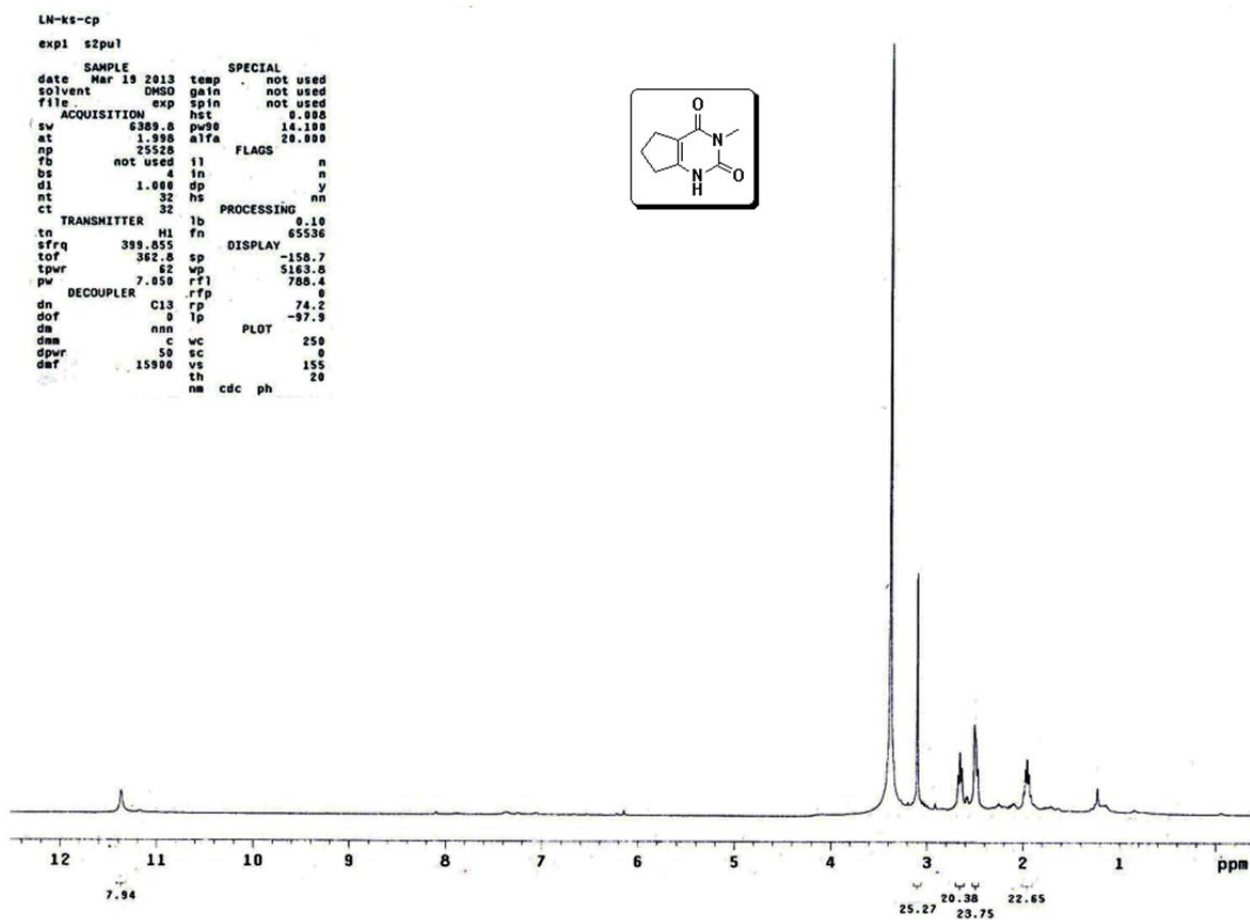


PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 2.561 sec Width 6398.0 Hz 32 repetitions	OBSERVE ¹ H, 399.8509721	DATA PROCESSING FT size 32768 Total time 1 minutes	LN-ISO_NMe-thiourea Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chem Mercury-400 *IITG-MMR*
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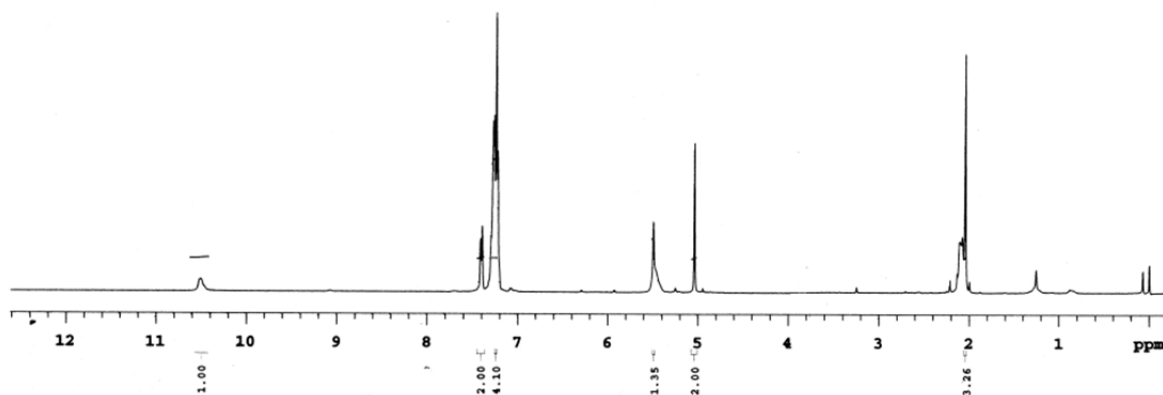
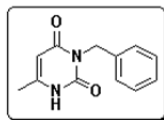


PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 1.304 sec Width 20125.6 Hz 330 repetitions	OBSERVE C13, 100.5425870 DECOUPLE ¹ H, 399.8529994 Power 42 dB continuously on WALTZ-16 modulated	DATA PROCESSING Line broadening 0.5 Hz FT size 65536 Total time 12 minutes	LN-6-Iso-NMe-13C Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chem File: LN-6-Iso-NMe-13C Mercury-400 *IITG-MMR*
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Compound(6):



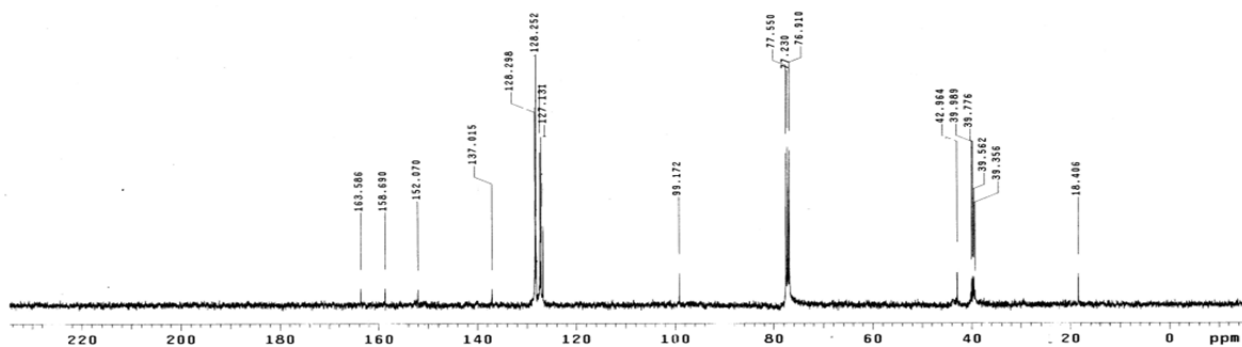
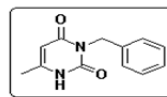
Compound (7):



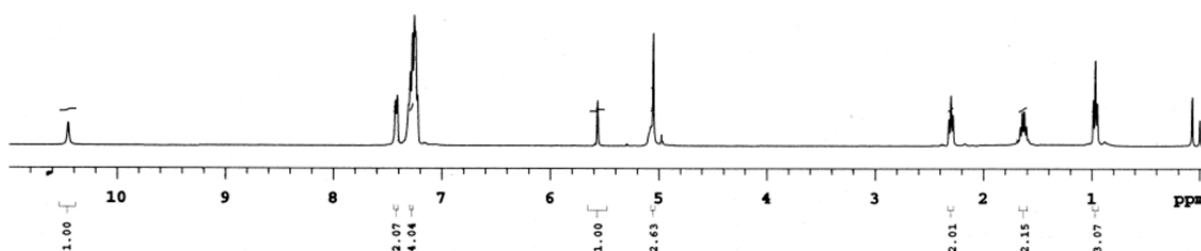
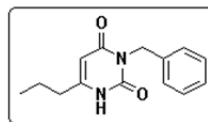
PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degree Acq. time 2.561 sec Width 6398.0 Hz 32 repetitions	OBSERVE H1, 399.8509590	DATA PROCESSING FT size 32768 Total time 1 minutes	LN-6me-M3-Bz Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chm File: LN-6me-M3-Bz Mercury-400 *HITG-MMR*
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LN-N3-bz-MeU
expt1 szpul

SAMPLE		SPECIAL	
date	Apr 19 2013	temp	not used
solvent	CDCl3	gain	not used
file		spin	not used
ACQUISITION		FLAGS	
sw	25125.6	pv90	0.000
at	1.139	alpha	20.000
pp	68270		
fb	13000	il	n
bs	10	in	n
dl	1.000	dp	v
nt	5000	hs	nn
ct	1000		
TRANSMITTER		PROCESSING	
in	CL3	lb	2.00
sfreq	100.554	fn	65536
tof	1536.3	sp	-1533.3
tpwr	61	wp	25125.6
pw	4.700	rfl	3296.2
dn	DECOUPLER	rff	7764.9
dof	H1	rp	-80.1
dm	0	lp	-271.4
dmm	vuv		
dpr	w	wc	250
dof	42	sc	0
	8500	vs	43
		th	4
	nm	no	ph



Compound (8):

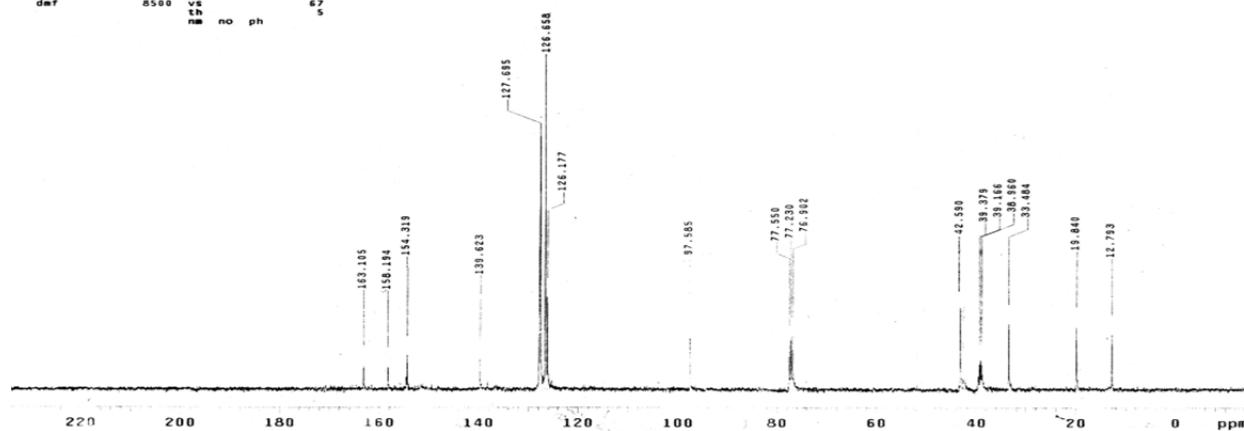
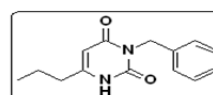


PULSE SEQUENCE	OBSERVE	EX1, 399.8509644	DATA PROCESSING	LN-pro-NBS_MOR_1H
Relax. delay 1.000 sec			PT size 32768	Solvent: cdcl3
Pulse 45.0 degrees			Total time 1 minutes	Temp. 25.0 C / 298.1 K
Acq. time 2.561 sec				Operator: cham
Width 6398.0 Hz				File: LN-pro-NBS_MOR_1H
				Mercury-400 *IITG-MS*

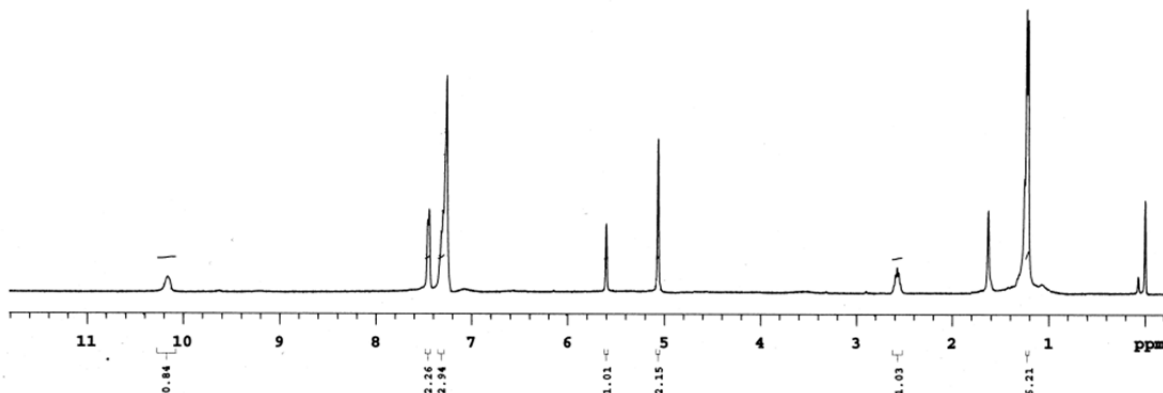
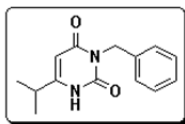
```

LN-ks-pro13C
exp1 s2pu1
SAMPLE
date Apr 15 2013 temp not used
solvent CDCl3 gain not used
file exp spin not used
ACQUISITION exp hst 8.400
sw 25125.6 pv90 9.400
at 1.199 a1fa 28.000
np 68270 il FLAGS
fb 13800 in n
bs 18 in n
dl 1.000 dp 25125.6
nt 5000 hs n
ct 410
TRANSMITTER lb fn PROCESSING 2.88
tn C13 fn 65536
sfq 180.554 DISPLAY -1570.8
tof 1536.3 sp -1570.8
tpwr 61 vp 25125.6
pw 4.700 rf1 3335.8
DECOUPLER rf1 7764.9
dn H1 rf 18.6
dof 0 lp -300.6
dm yyv PLOT 250
dme v wc 250
dpr 42 sc 0
dmf 8500 vs 67
lh ne no ph 5

```



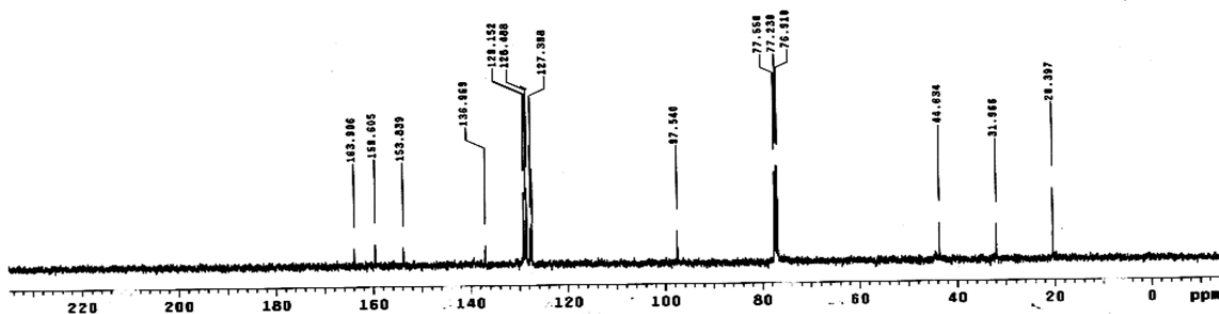
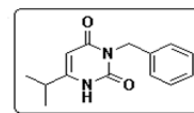
Compound (9):



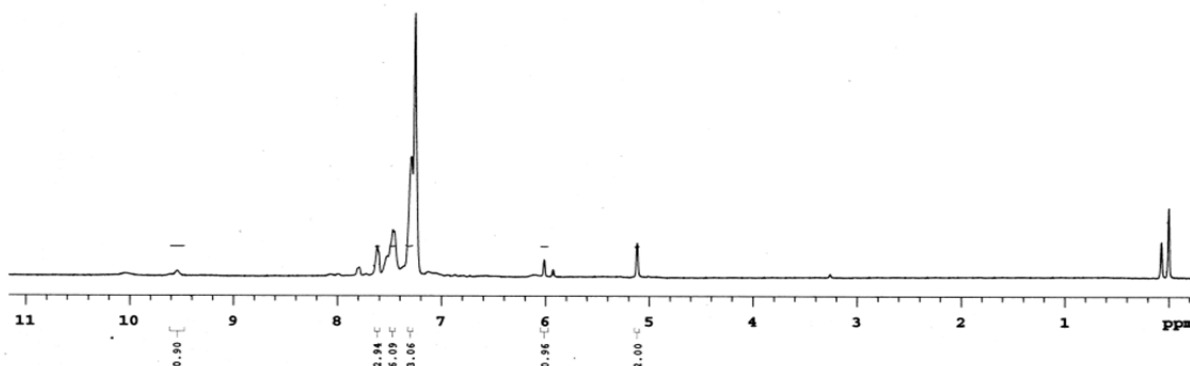
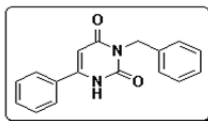
PULSE SEQUENCE	OBSERVE H1, 399.8509636	DATA PROCESSING	LN-ISO-MBZ-181
Relax. delay 1.000 sec		FT size 32768	Solvent: cdcl3
Pulse 45.0 degrees		Total time 1 minutes	Temp. 25.0 C / 298.1 K
Acq. time 2.561 sec			Operator: cham
Width 6398.0 Hz			Mercury-400 "IITG-MBZ"
32 repetitions			

```

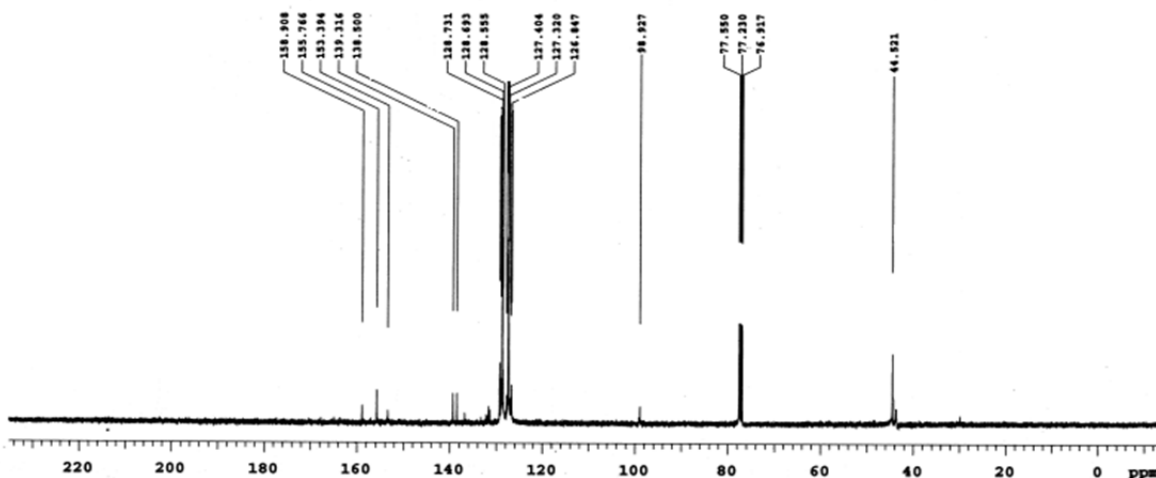
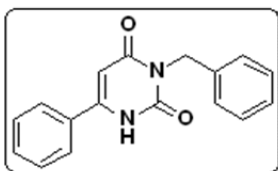
LN-hs-iso-BZ-13C
expt szpul
date: Apr 17 2013      temp not used
solvent CDC13         gain not used
Title exp             spin not used
ACQUISITION          hst 8.085
sw 25125.6            pps 5.408
at 1.189             A17a 20.808
np 88278             flags
fb 13080             n
bs 18                n
dl 1.808             y
nc 5808              nm
ct 560
TRANSMITTER          C13  fn 2.00
tn 180.554           pr 85536
sfrq 180.554         ep 1518.3
tof 1536.3          wp 25125.6
tpwr 61             rfi 8275.2
pw 4.768            rfs 7764.9
DECOUPLER            H1  rp -81.4
dn 0                lp -271.4
dof 0               PLOT
dm 0                wc 254
dms 42              sc 6
dpr 4580            vs 23
dat 0               th 4
                      nm no ph
  
```



Compound (10):



PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degree Acq. time 2.561 sec Width 6398.0 Hz 32 repetitions	OBSERVE H1, 399.8509634	DATA PROCESSING FT size 32768 Total time 1 minutes	LN-ph-NBz-1H_1 Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chem Mercury-400 "1H NMR"
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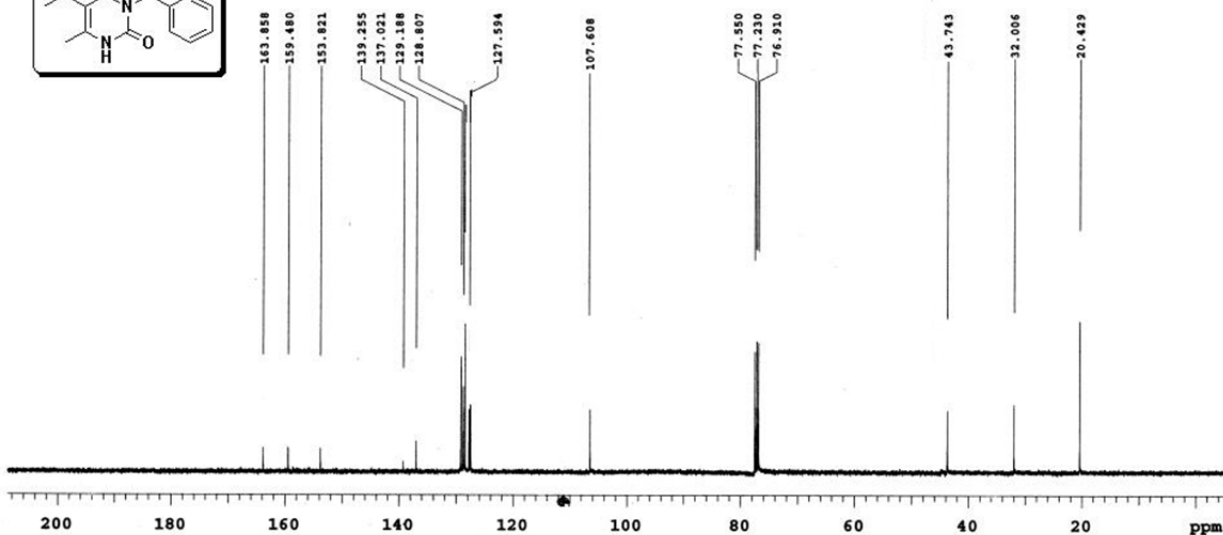
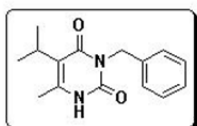
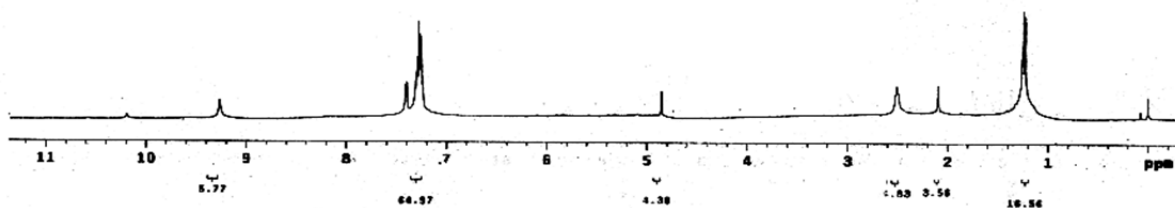
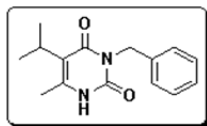
PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degree Acq. time 1.304 sec Width 25125.6 Hz 1440 repetitions	OBSERVE C13, 100.5425909 DECOUPLE H1, 399.8525994 Power 42 dB continuously on WALTZ-16 modulated	DATA PROCESSING Line broadening 0.5 Hz FT size 65536 Total time 55 minutes	LN-ph-NBz-C13 Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chem Mercury-400 "13C NMR"
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Compound (11):

```

LN-001-iso-ma
exp1 s1p1
SAMPLE
date Apr 12 2012 temp not used
solvent CDCl3 gain not used
F100 400.130 spin not used
ACQUISITION exp h1 1.000
sv 0200.0 pvs0 15.100
st 1.000 a1fa 15.000
sp 15528
7h not used 11 n
8h 4 1a n
d1 1.000 sp y
st 1.000 hs mh
ct 0
TRANSMITTER 1b 8.10
1a 1b 65538
sfrs 399.8513 DISPLAY
tof 362.6 sp -120.3
1pwr 85 vp 4866.3
pw 7.550 r71 789.3
DECOUPLE C13 r7p 0
dn 0 142.1
dof 0 1p -68.9
dm new vc 250
dpcr 44 sc 0
dof 17100 va 49
nm cdc gb 12

```



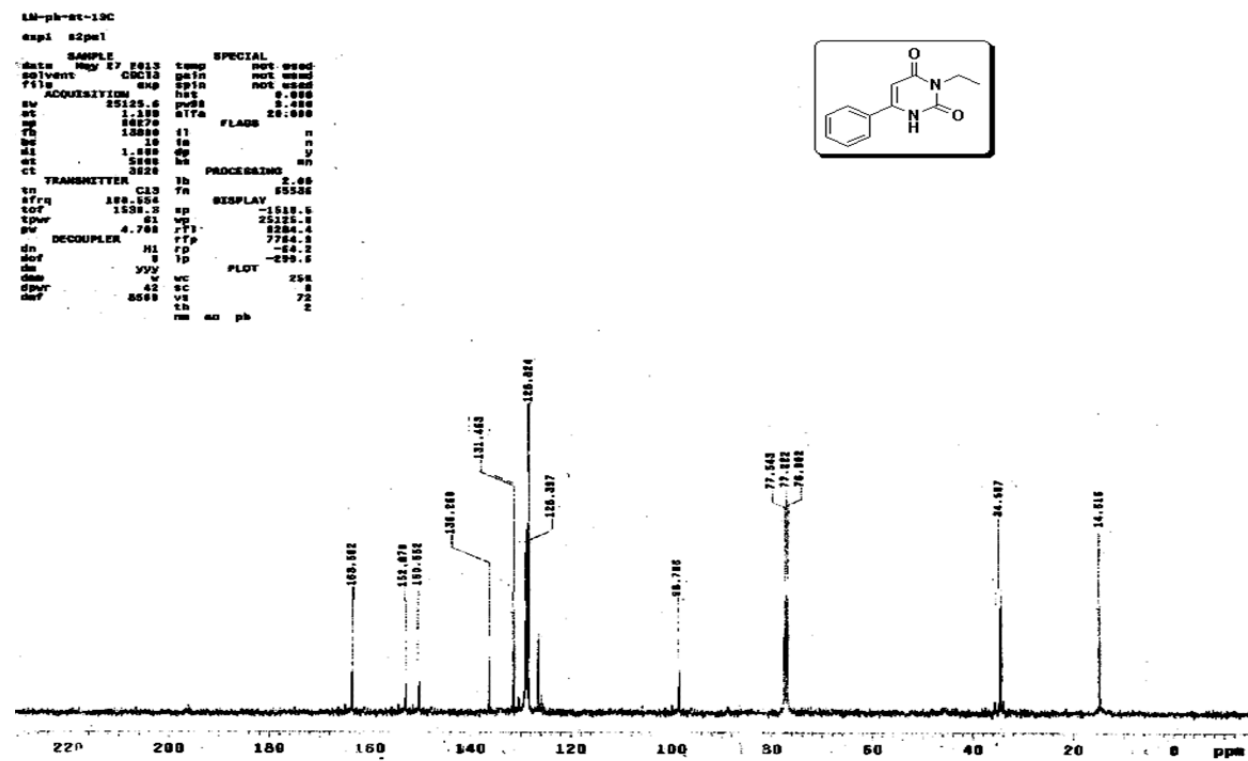
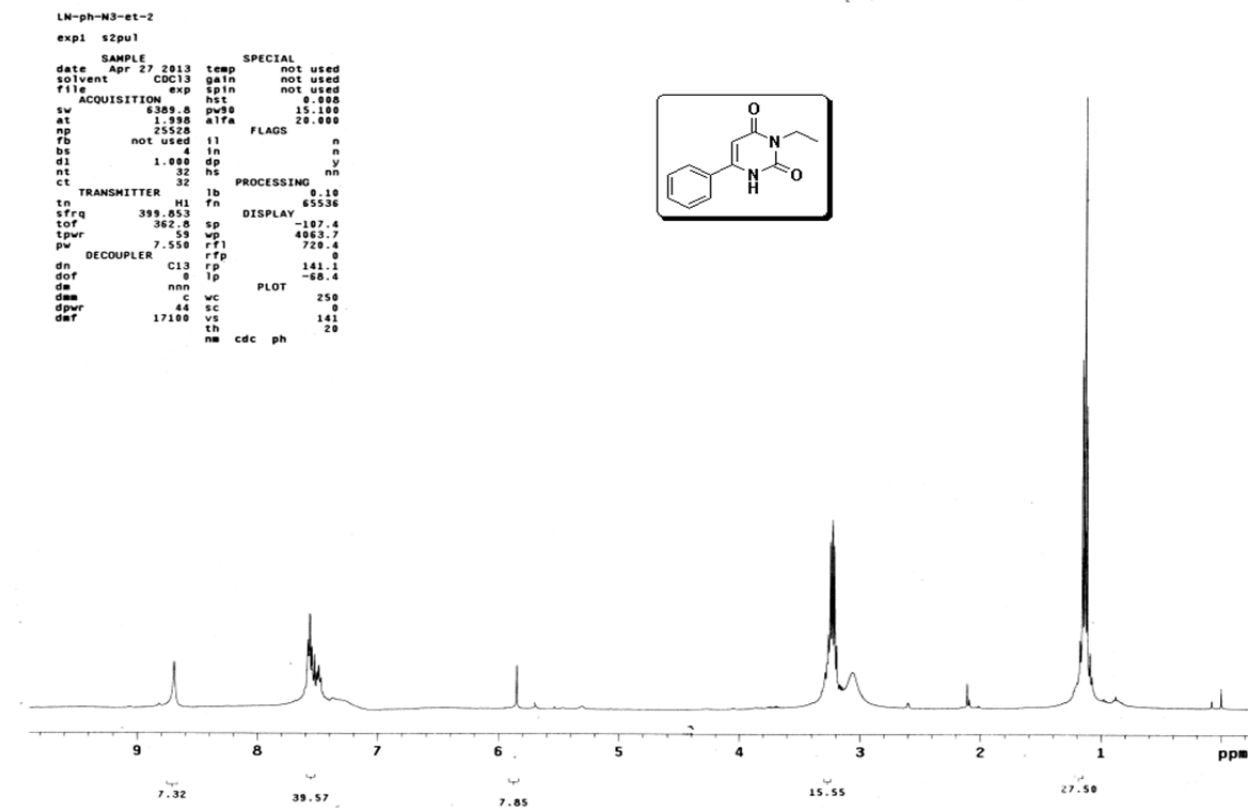
SEQUENCE
 Relax delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.104 sec
 Width 25125.6 Hz
 930 repetitions

OBSERVE C13-100W5425848
 DECOUPLE H1, 399.8529994
 Power 42 dB
 continuously on
 WALTZ-16 modulated

DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 65536
 Total time 35 minutes

LN-001-iso-ma13C
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
 Operator: chem
 Mercury-400 *IITG-NMR*

Compound (12):



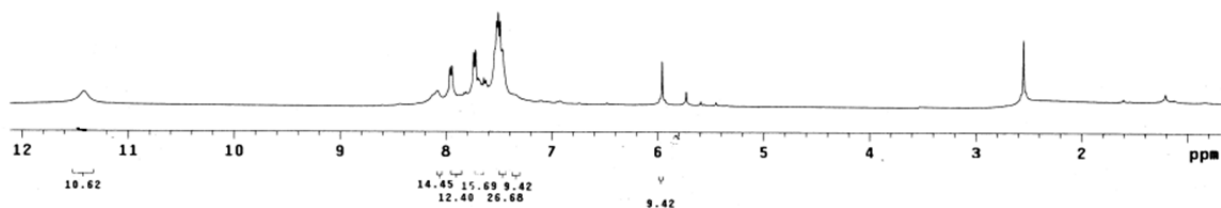
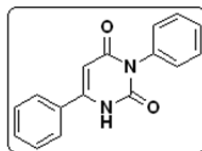
Compound (13):

```

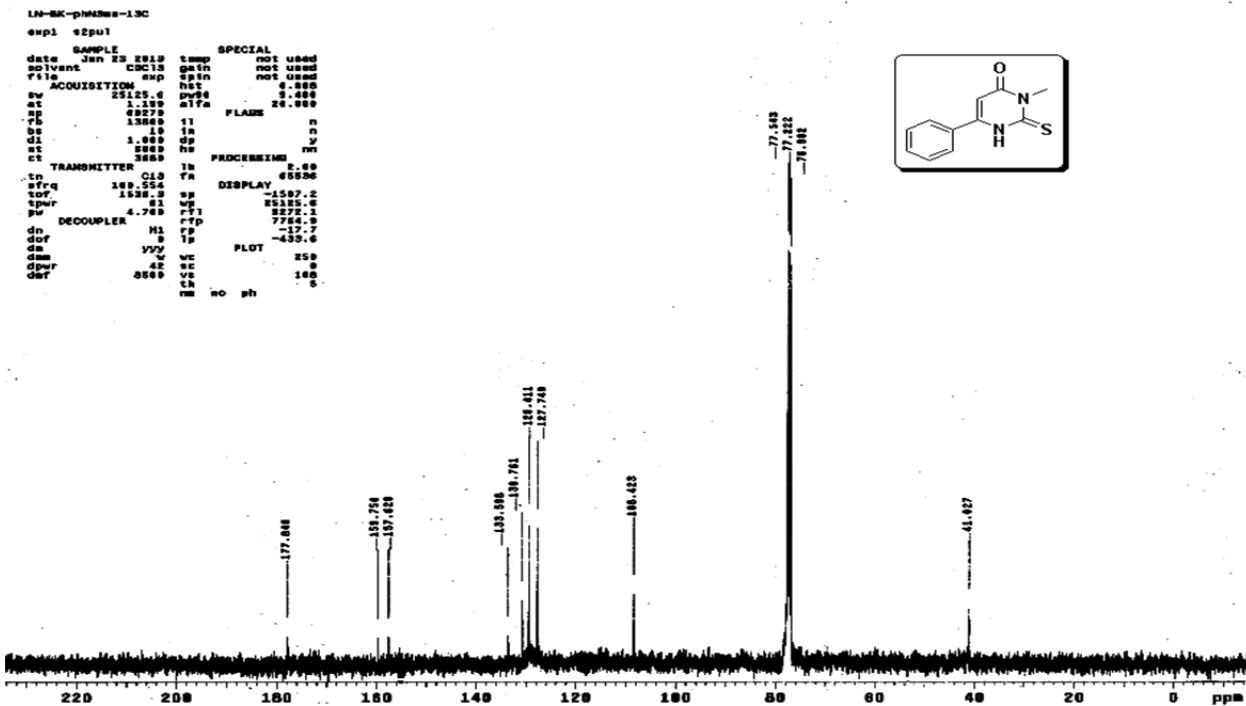
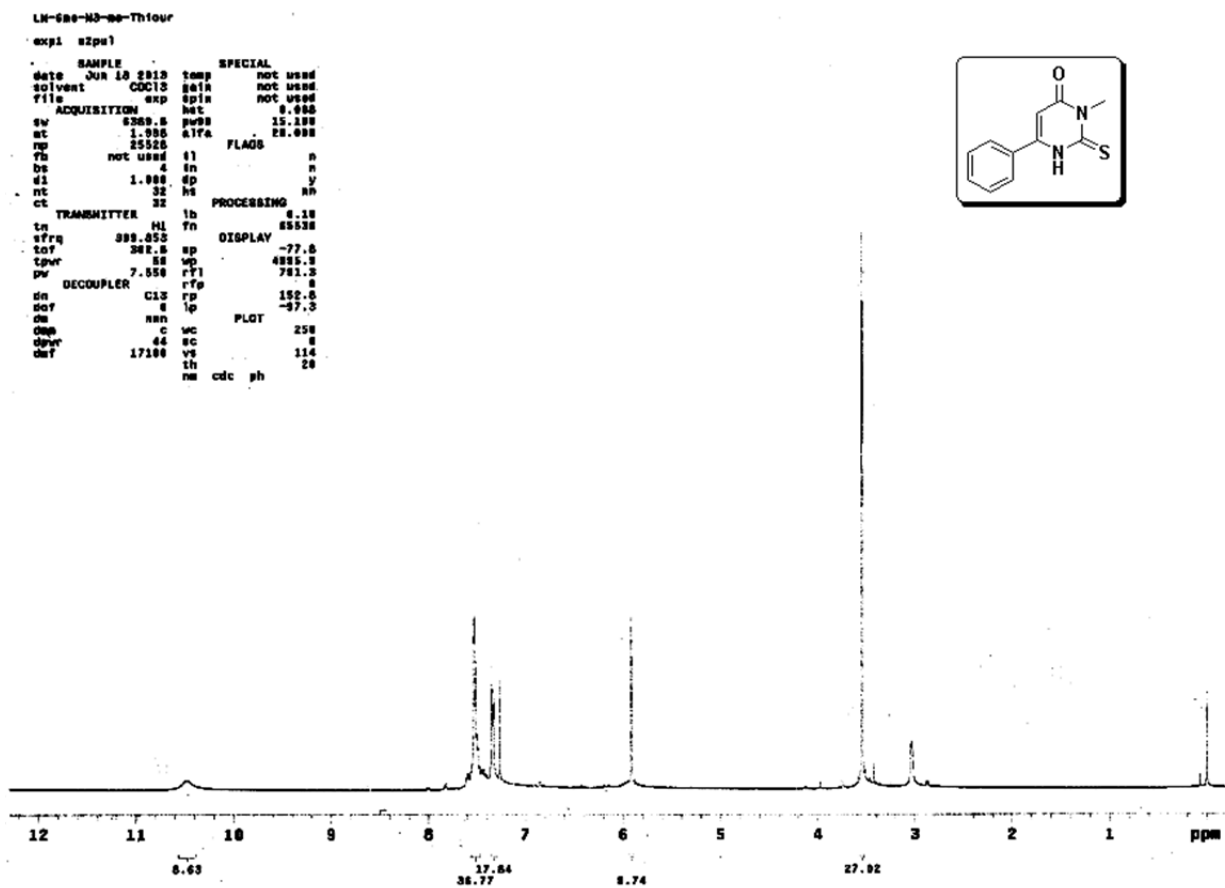
LN-199-1
expt s2pu1

SAMPLE
date Feb 21 2013 temp not used
solvent DMSO gain not used
file exp spin not used
ACQUISITION exp hst 0.000
sw 6389.8 pv90 19.700
at 1.998 alfa 20.000
np 25520
fb not used il FLAGS n
bs 4 in n
dl 1.000 dp y
nt 32 hs nn
ct 32
TRANSMITTER lb 0.10
tn H1 fn 65536
sfrq 399.855 DISPLAY
tof 362.8 sp 260.2
tpwr 57 wp 4584.4
pv 9.850 rf1 1700.0
DECOUPLER rfp 999.6
dn C13 rp 69.8
dof 0 lp -74.4
dm nnn PLOT
dnn c wc 250
dpwr 50 sc 0
daf 15900 vs 65
nm th 30
nm cdc ph

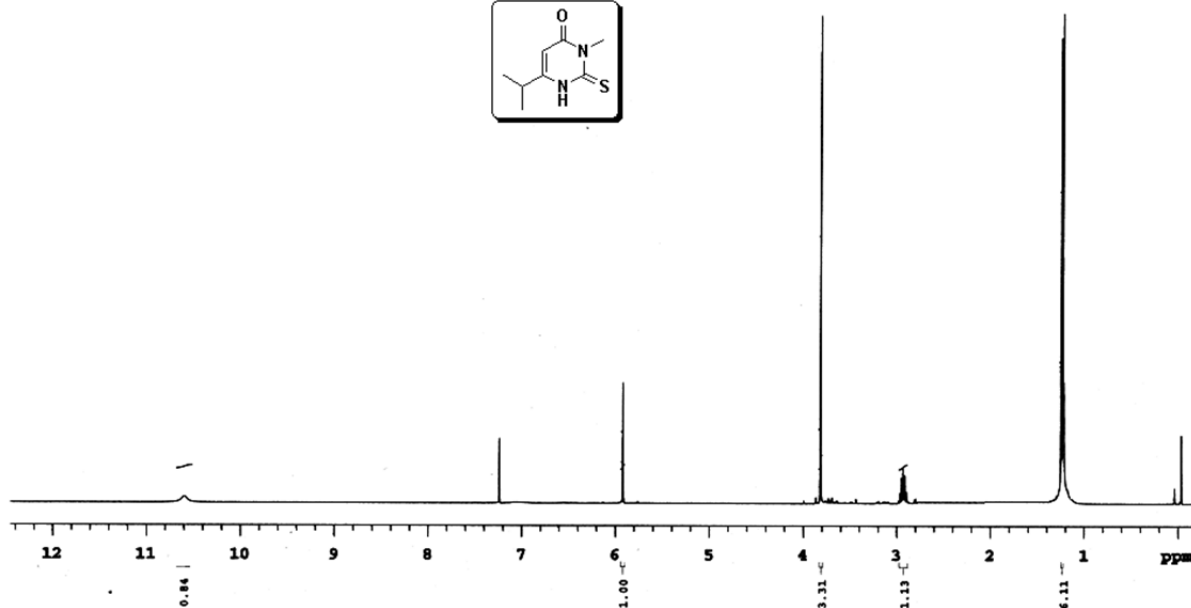
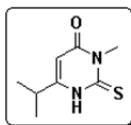
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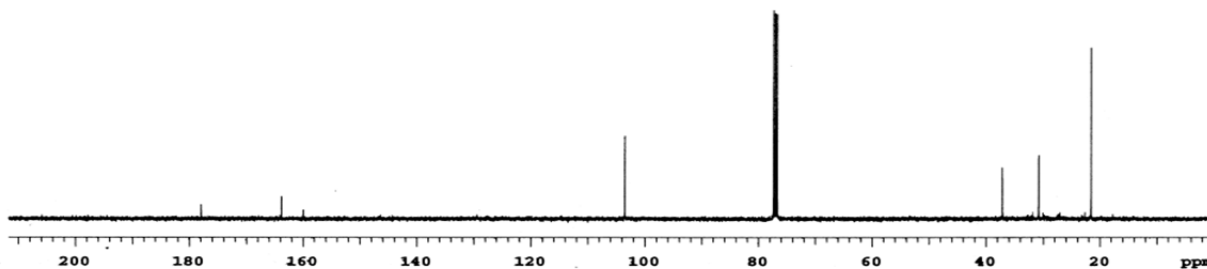
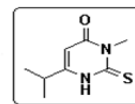
Compound (14):



Compound (15):

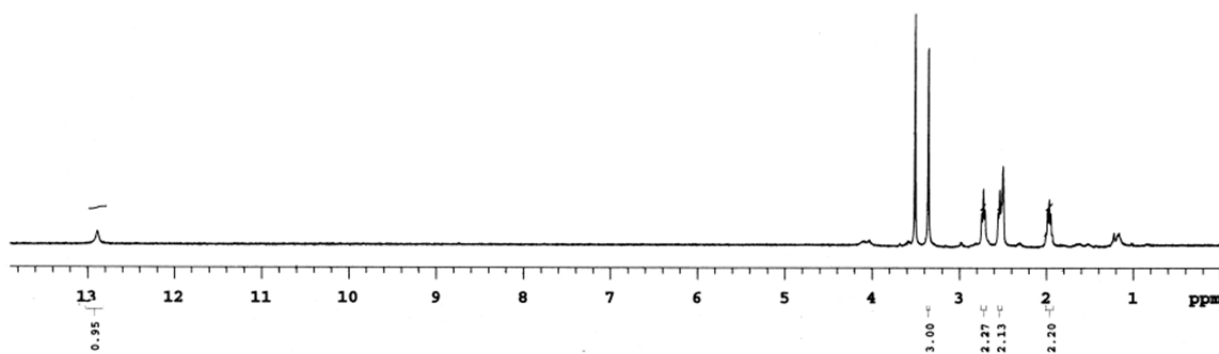
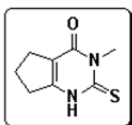


PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 2.561 sec Width 6398.0 Hz 32 repetitions	OBSERVE H1, 399.8509725	DATA PROCESSING 172- F1 size 32768 Total time 1 minutes	LN-249-noc Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chem File: LN-249-noc Mercury-400 "IITG-MMR"
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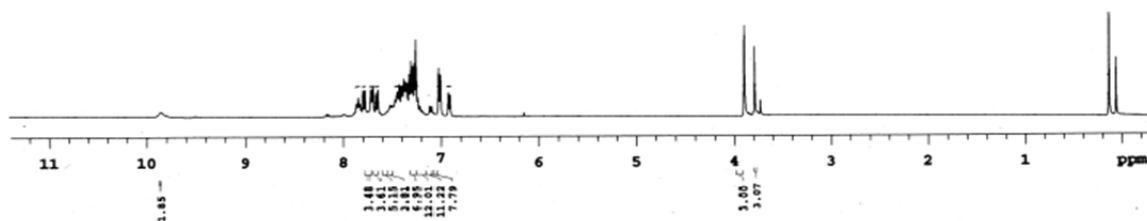
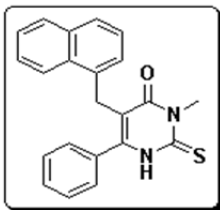
PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 1.304 sec Width 25125.6 Hz 2360 repetitions	OBSERVE C13, 100.5426047 DECOUPLE H1, 399.8529994 Power 42 dB continuously on WALTZ-16 modulated	DATA PROCESSING 165-10- Line broadening 0.5 Hz F1 size 65536 Total time 90 minutes	LN-249-13C Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chem File: LN-249-13C Mercury-400 "IITG-MMR"
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Compound (16):

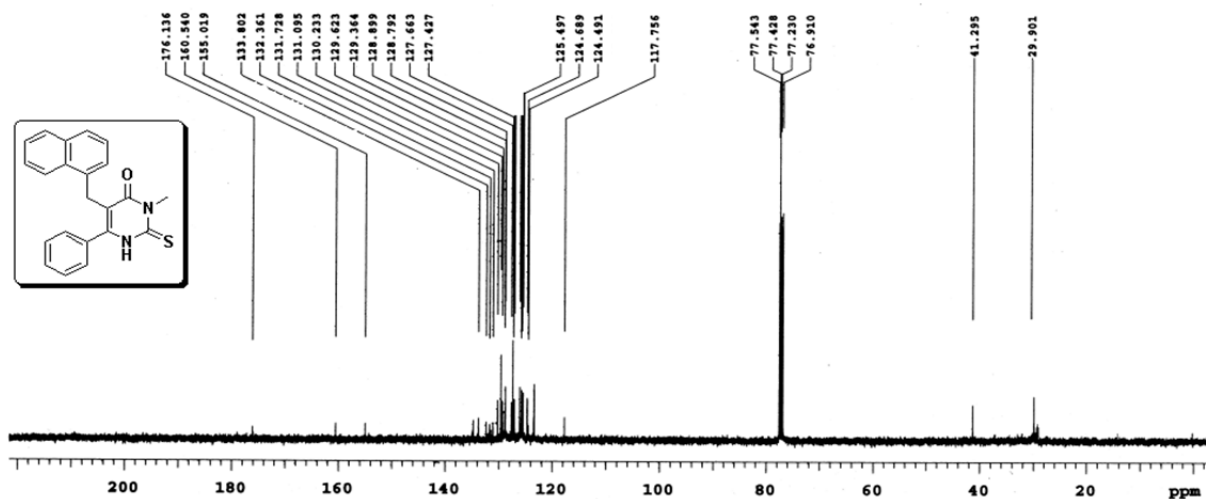


PULSE SEQUENCE	OBSERVE M1, 399.8528575	DATA PROCESSING	LM-6-cpro-MOE
Relax. delay 1.000 sec		FT size 32768	Solvent: dmsc
Pulse 22.6 degrees		Total time 1 minutes	Temp. 25.0 C / 298.1 K
Acq. time 2.561 sec			Operator: cham
Width 6398.0 Hz			File: LM-6-cpro-MOE
32 repetitions			Mercury-400 *IITG-MNR*

Compound (17):



PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 2.561 sec Width 6398.0 Hz 32 repetitions	OBSERVE H1, 399.8509656	DATA PROCESSING FT size 32768 Total time 1 minutes	LN-251 Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chem Mercury-400 "IITG-MMR"
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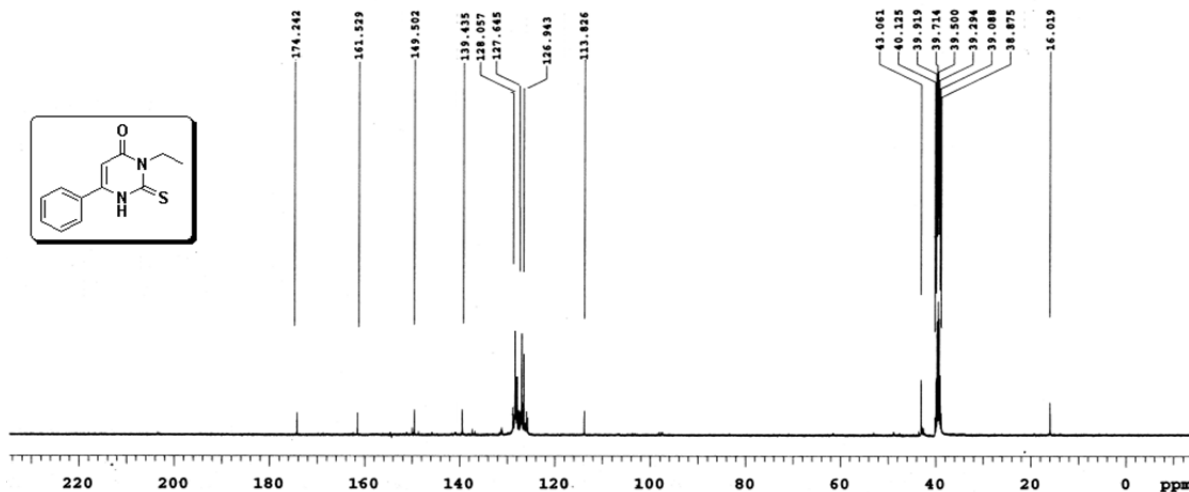
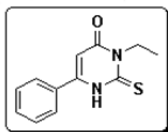
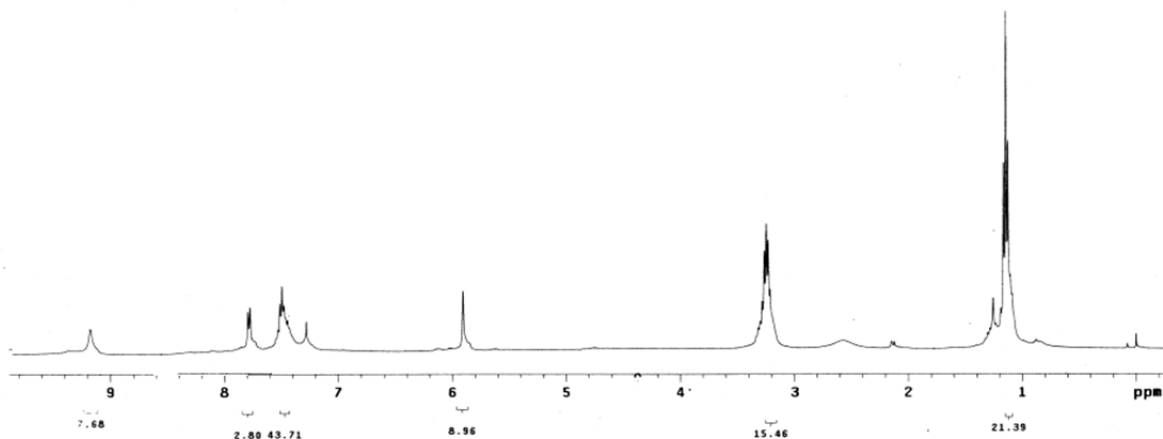
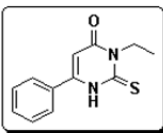
PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 1.304 sec Width 25125.6 Hz 1170 repetitions	OBSERVE C13, 100.5425847 DECOUPLE H1, 399.8529994 Power 42 dB continuously on WALTZ-16 modulated	DATA PROCESSING Line broadening 0.5 Hz FT size 65536 Total time 44 minutes	LN-251_13C Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chem File: LN-251_13C Mercury-400 "IITG-MMR"
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Compound (18):

LN-ph-M3-et

exp1 s2pu1

SAMPLE		SPECIAL	
date	Apr 24 2013	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	not used
ACQUISITION		nst	8.000
sw	6389.8	pw90	15.100
at	1.998	alpha	20.000
np	25528	FLAGS	
fb	not used	il	n
bs	4	in	n
dl	1.000	dp	y
nt	32	hs	nn
ct	32	PROCESSING	
tn	TRANSMITTER	lb	8.10
tfreq	M1	fn	65536
toif	399.853	DISPLAY	-187.4
tpwr	59	wp	4062.2
pw	7.550	rfl	785.9
DECOUPLER		rfg	0
dn	C13	rp	135.6
dof	0	lp	-69.3
dm	nmn	PLOT	
dpm	c	wc	250
dpwr	44	sc	8
daf	17100	vs	77
	th	26	
	nm	cdc	ph



OPUS2 SEQUENCE

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.304 sec
Width 25125.6 Hz
2562 repetitions

OBSERVE CHANNEL 000-5034955

DECOUPLE M1, 399.8540987
Power 42 dB
continuously on
WALTZ-16 modulated

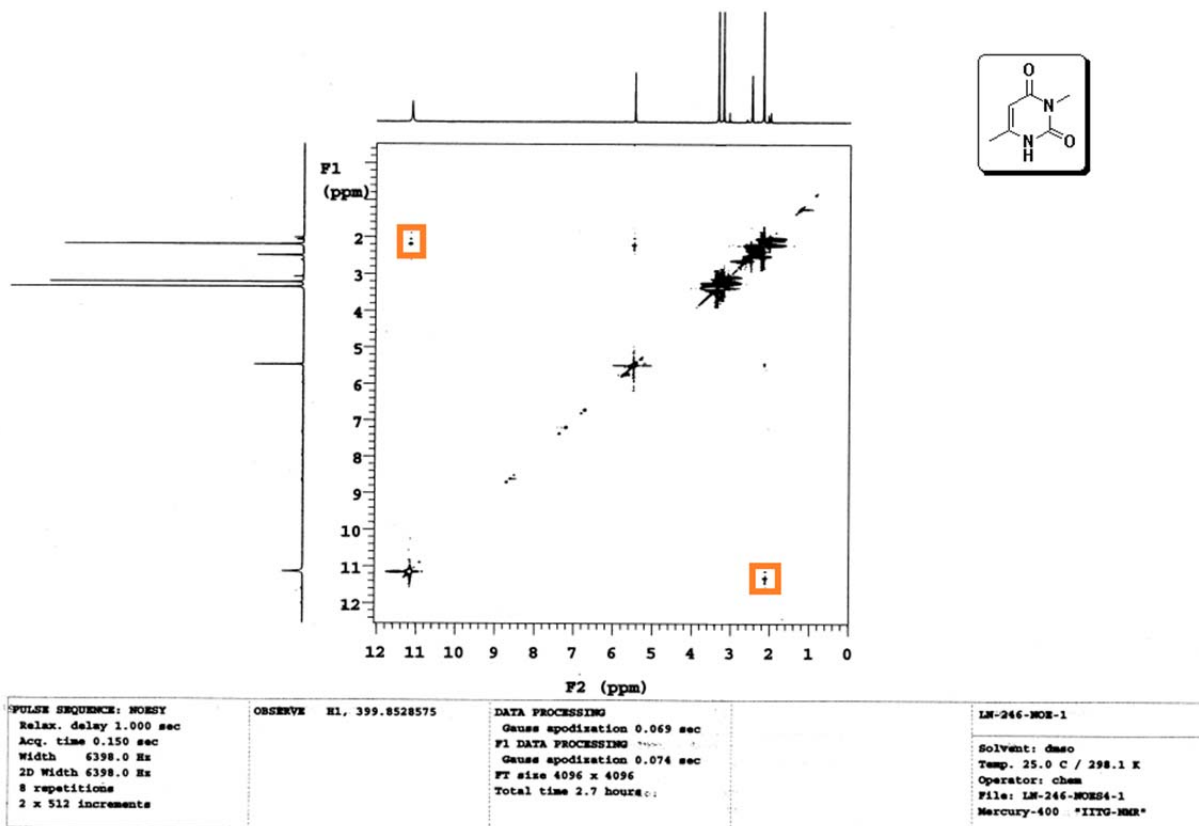
DATA PROCESSING

Line broadening 0.5 Hz
FT size 65536
Total time 98 minutes

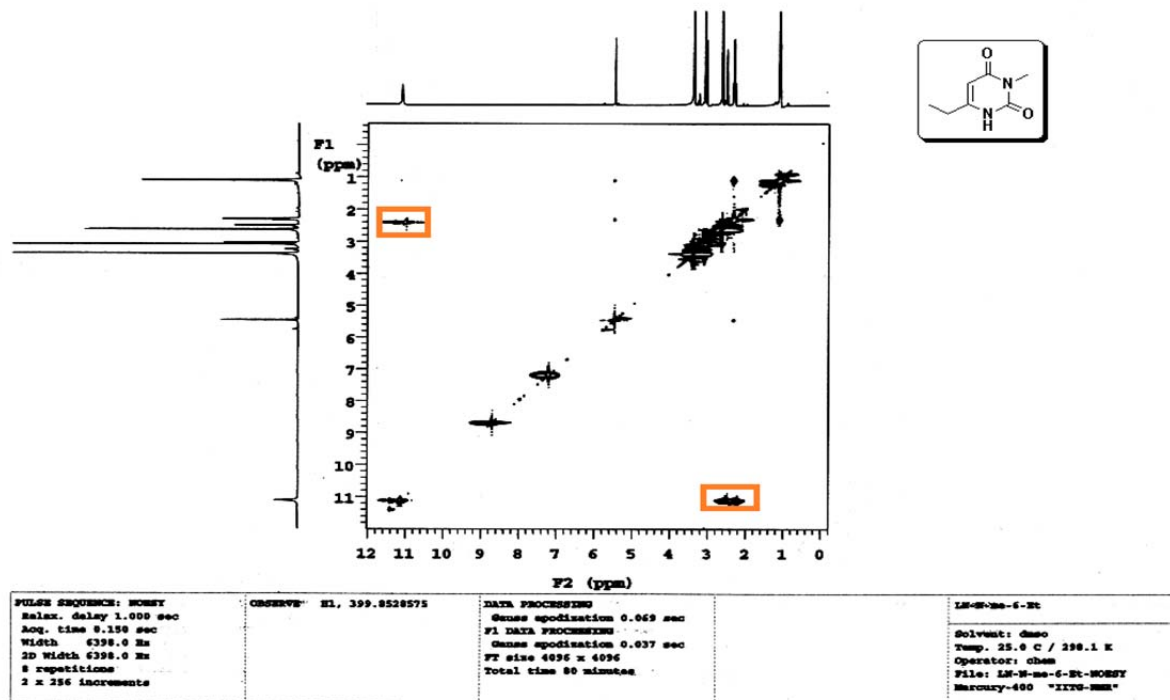
LN-M3-Et-6Ph-ThioU-13C

Solvent: dmsc
Temp. 25.0 C / 298.1 K
Operator: chem
File: LN-M3-Et-6Ph-ThioU-13C
Mercury-400 "IITG-NMR"

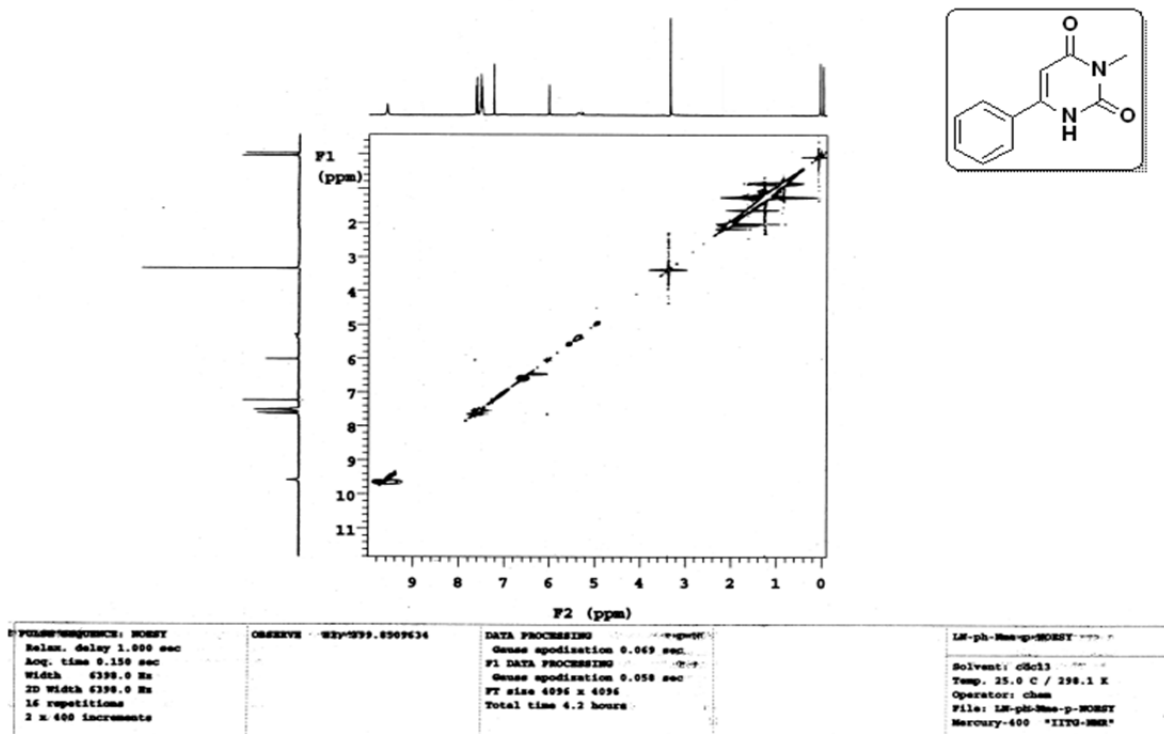
Compound (1):



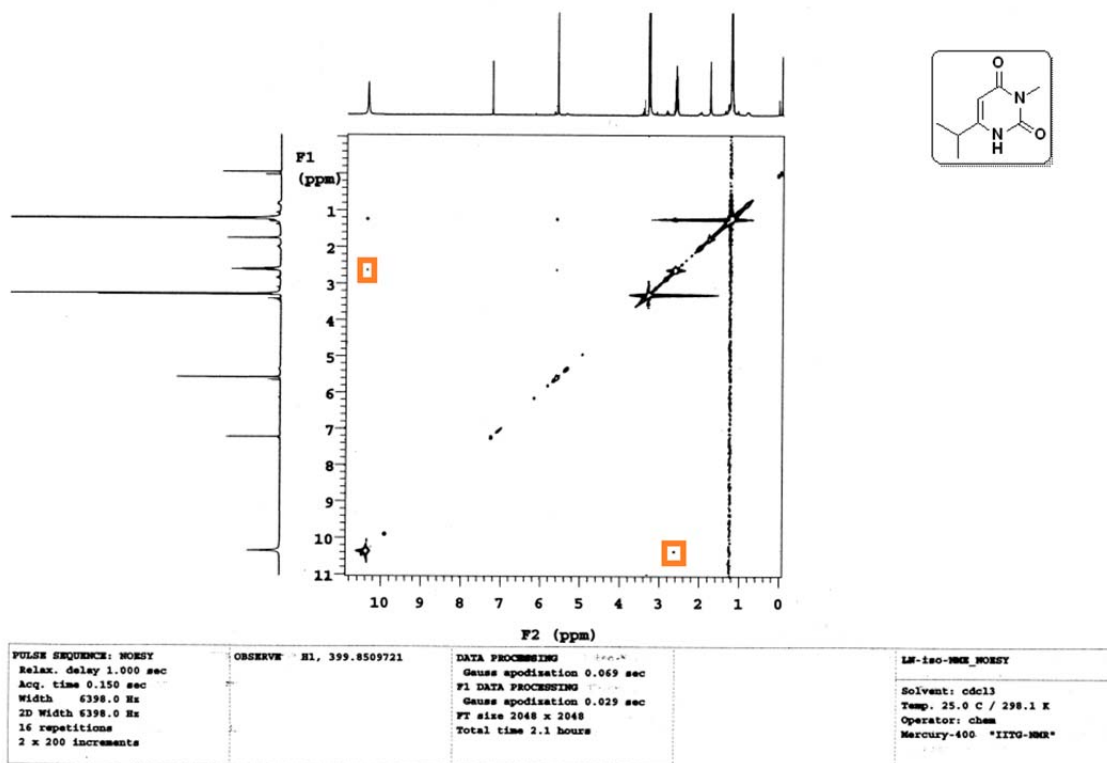
Compound (2):



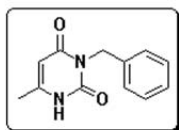
Compound (4):



Compound (5):



Compound (7):



LN_N_BZ_6ME NOESY



Current Data Parameters
NAME LN_N_BZ_6ME NOESY
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140121
Time 13.28
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG noesypphpgp
TD 2048
SOLVENT CDCl3
NS 4
DS 32
SMH 12019.230 Hz
FIDRES 5.868765 Hz
AQ 0.0851968 sec
RG 54.94
DM 41.600 usec
DE 6.50 usec
TE 298.2 K
DO 0.0002632 sec
D1 2.00000000 sec
D8 0.30000001 sec
D11 0.03000000 sec
D12 0.00020000 sec
D16 0.00020000 sec
IN0 0.0008320 sec

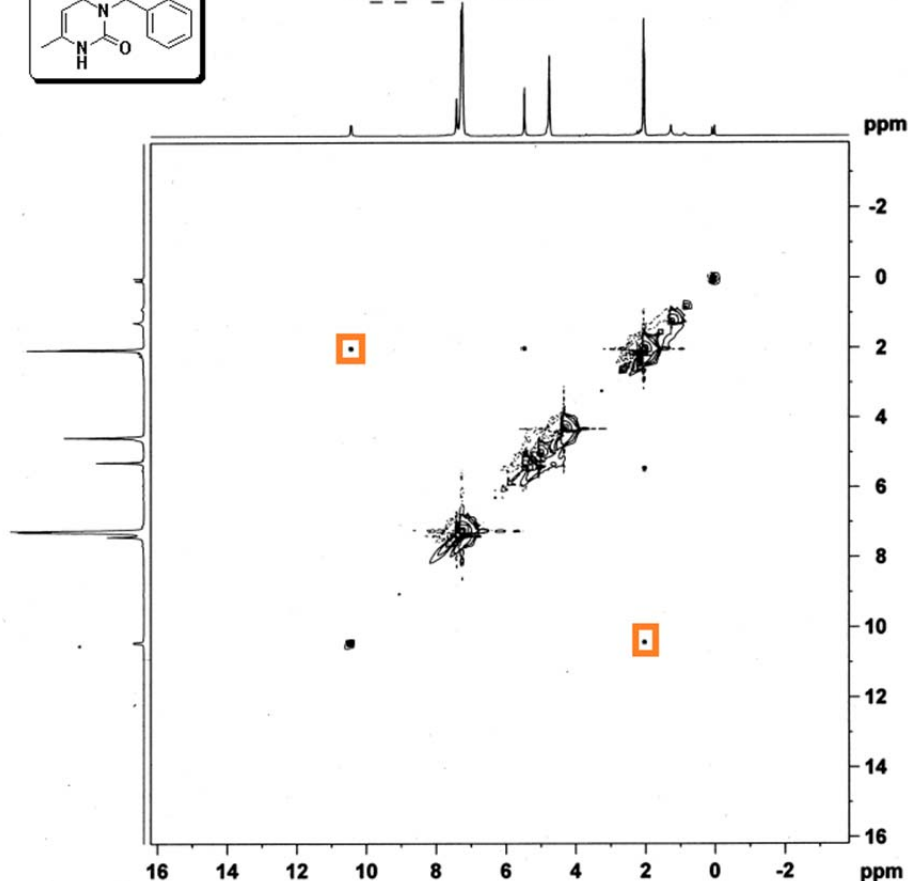
----- CHANNEL f1 -----
SFO1 600.1737063 MHz
NUC1 1H
P1 12.00 usec
P2 24.00 usec
P17 2500.00 usec
PLW1 21.00000000 W
PLW0 4.47340012 W

----- GRADIENT CHANNEL -----
GPRAM[1] SMSQ10.100
GP21 40.00 %
P16 1000.00 usec

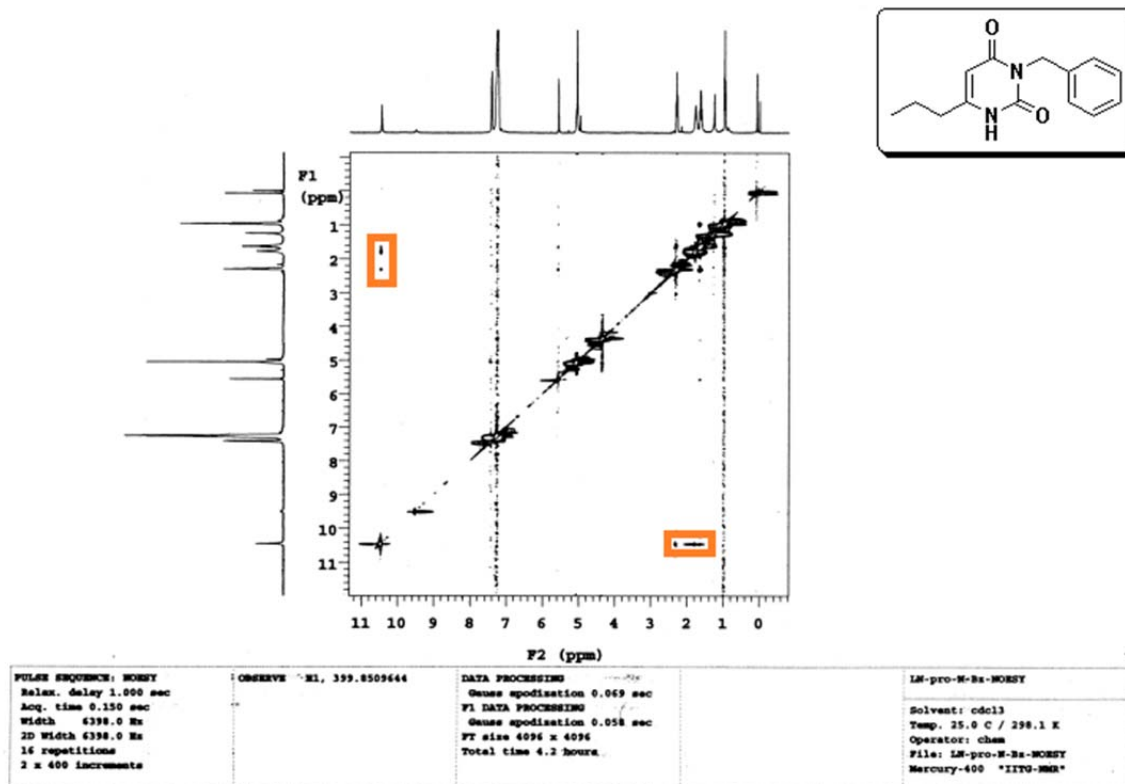
F1 - Acquisition parameters
TD 64
SFO1 600.1737 MHz
FIDRES 143.086075 Hz
SW 20.026 ppm
F0MODE States-TFPI

F2 - Processing parameters
SI 1024
SF 600.1700000 MHz
WDW QSI
SSB 2
LB 0 Hz
GB 0
PC 1.00

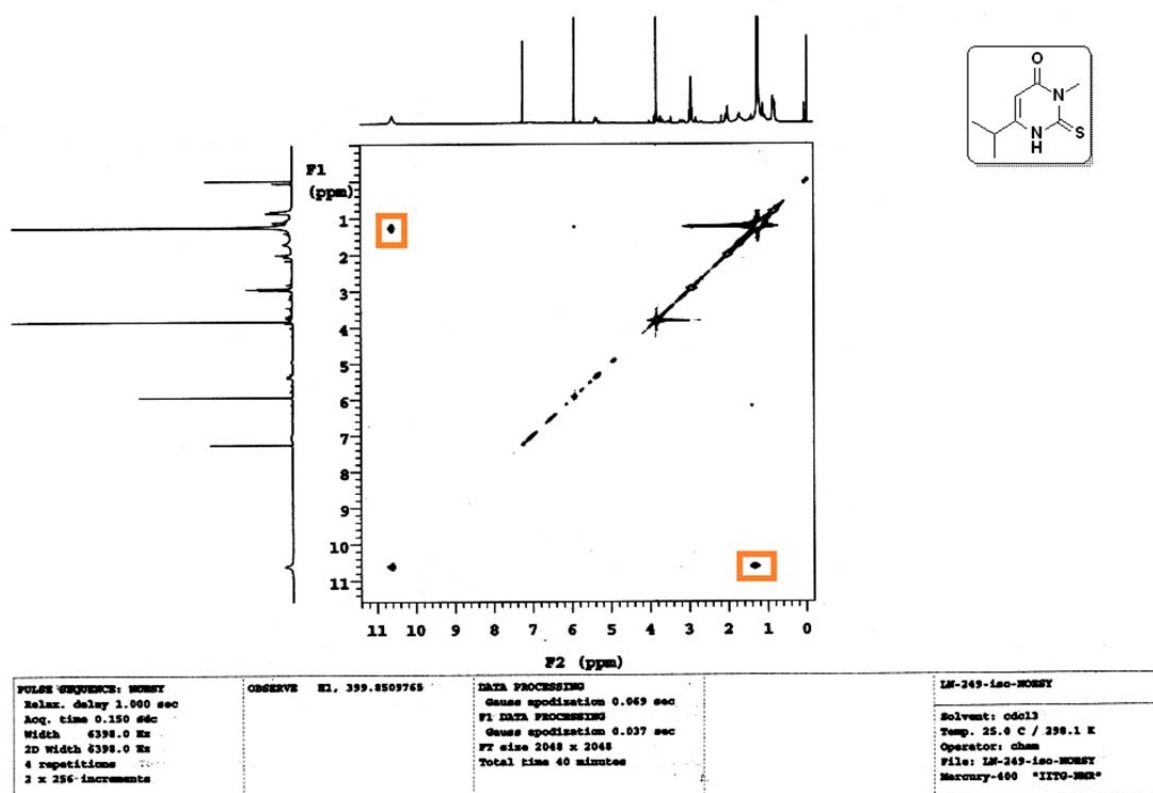
F1 - Processing parameters
SI 1024
MC2 States-TFPI
SF 600.1700000 MHz
WDW QSI
SSB 2
LB 0 Hz
GB 0



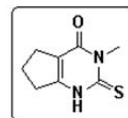
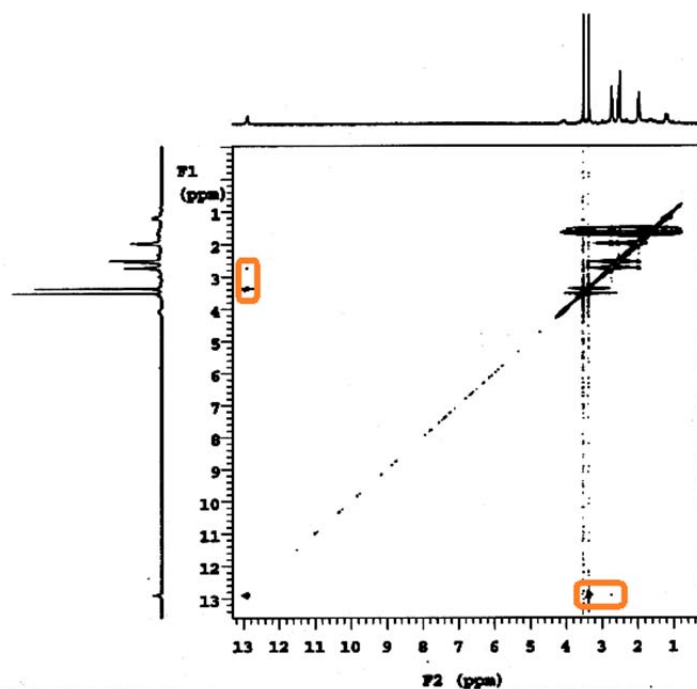
Compound (8):



Compound (15):



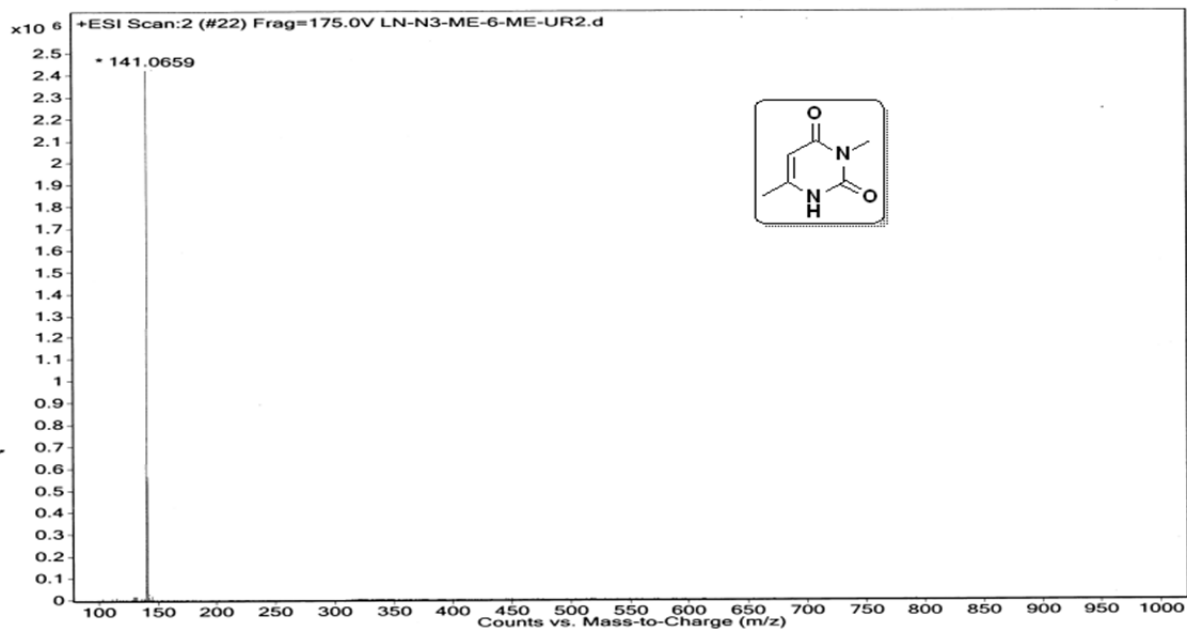
Compound (16):



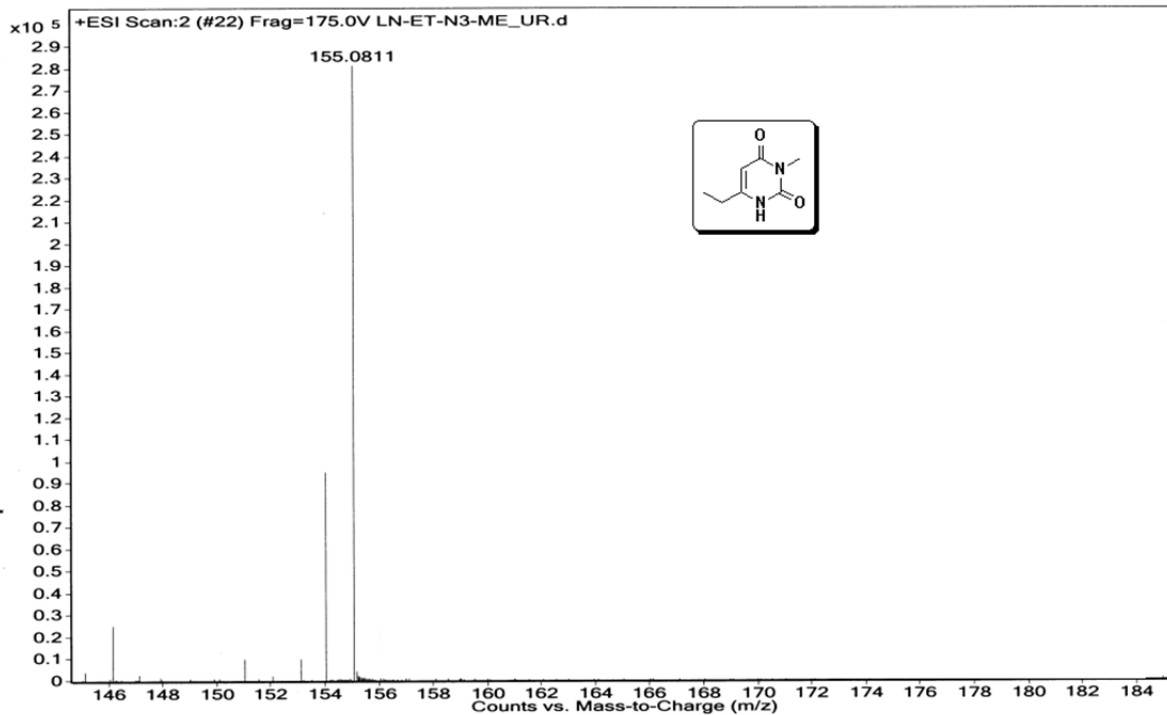
PULPRO SEQUENCE: M0001 Relax. delay 1.000 sec Acq. time 0.150 sec Width 6398.0 Hz X0 width 6398.0 Hz 0 repetitions 2 x 400 increments	OBSERVE E1, 359.8528575	DATA PROCESSING Gamma apodisation 0.059 sec F1 DATA PROCESSING Gamma apodisation 0.058 sec FT size 4096 x 4096 Total time 2.1 hours	LN-6-cpro-M0001 Solvent: dmsc Temp. 25.0 C / 298.1 K Operator: chm File: LN-6-cpro-M0001 Mercury-400 "XTC-M00"
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5. HRMS and XRD data:

Sample Name	Position	Instrument Name	User Name
Inj Vol	InjPosition	SampleType	IRM Calibration Status
Data Filename	ACQ Method	Comment	Acquired Time



Sample Name	Position	Instrument Name	User Name
Inj Vol	InjPosition	SampleType	IRM Calibration Status
Data Filename	ACQ Method	Comment	Acquired Time

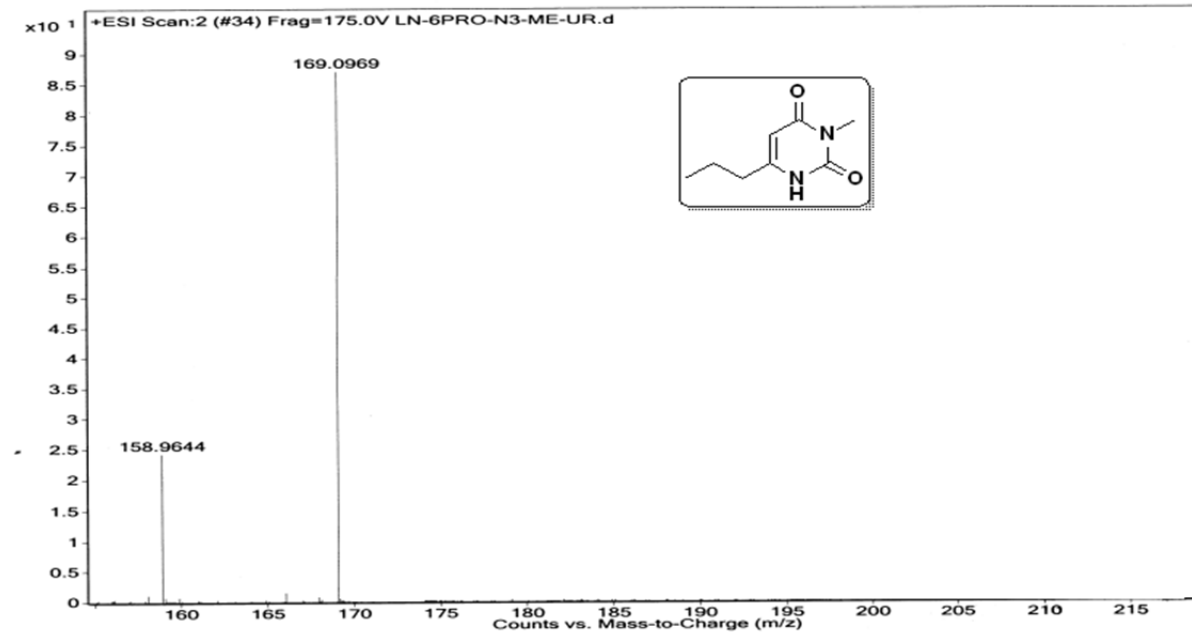


Sample Name
Inj Vol
Data Filename

Position
InjPosition
ACQ Method

Instrument Name
SampleType
Comment

User Name
IRM Calibration Status
Acquired Time

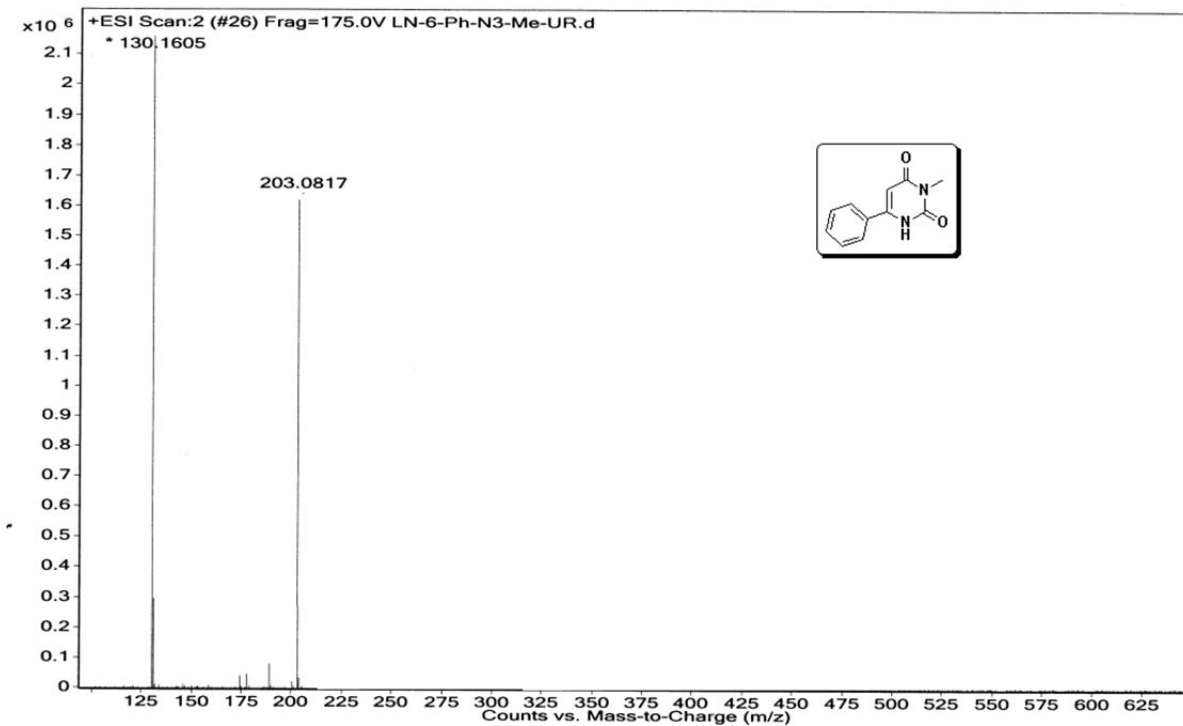


Sample Name
Inj Vol
Data Filename

Position
InjPosition
ACQ Method

Instrument Name
SampleType
Comment

User Name
IRM Calibration Status
Acquired Time

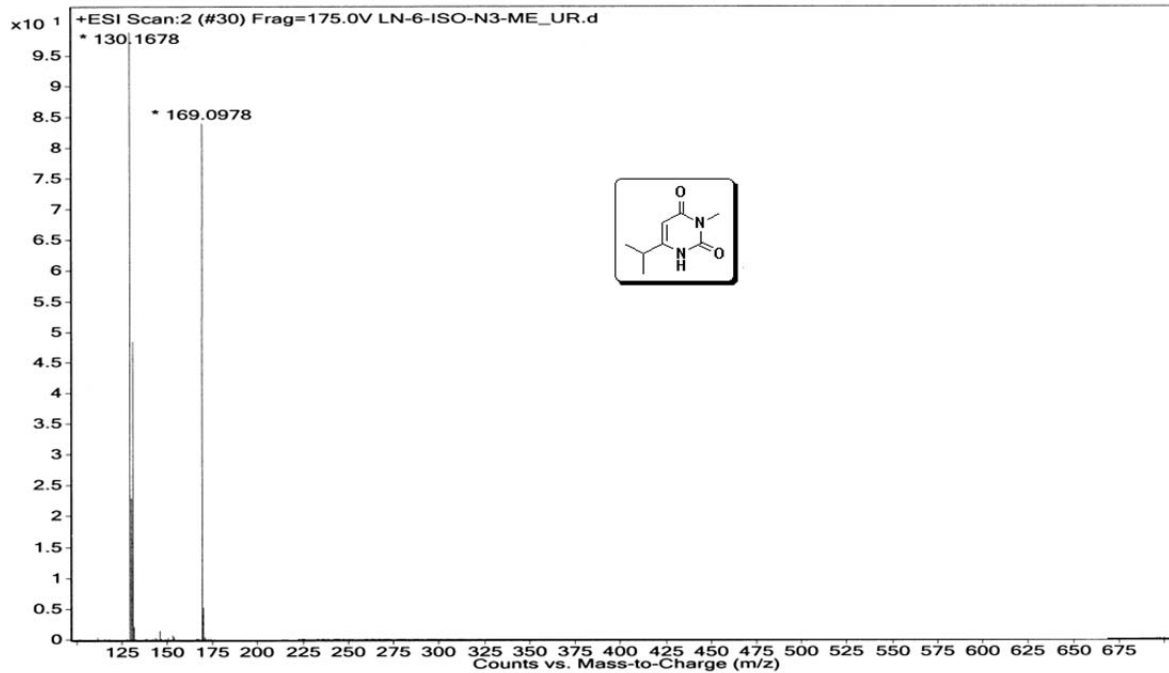


Sample Name
Inj Vol
Data Filename

Position
InjPosition
ACQ Method

Instrument Name
SampleType
Comment

User Name
IRM Calibration Status
Acquired Time

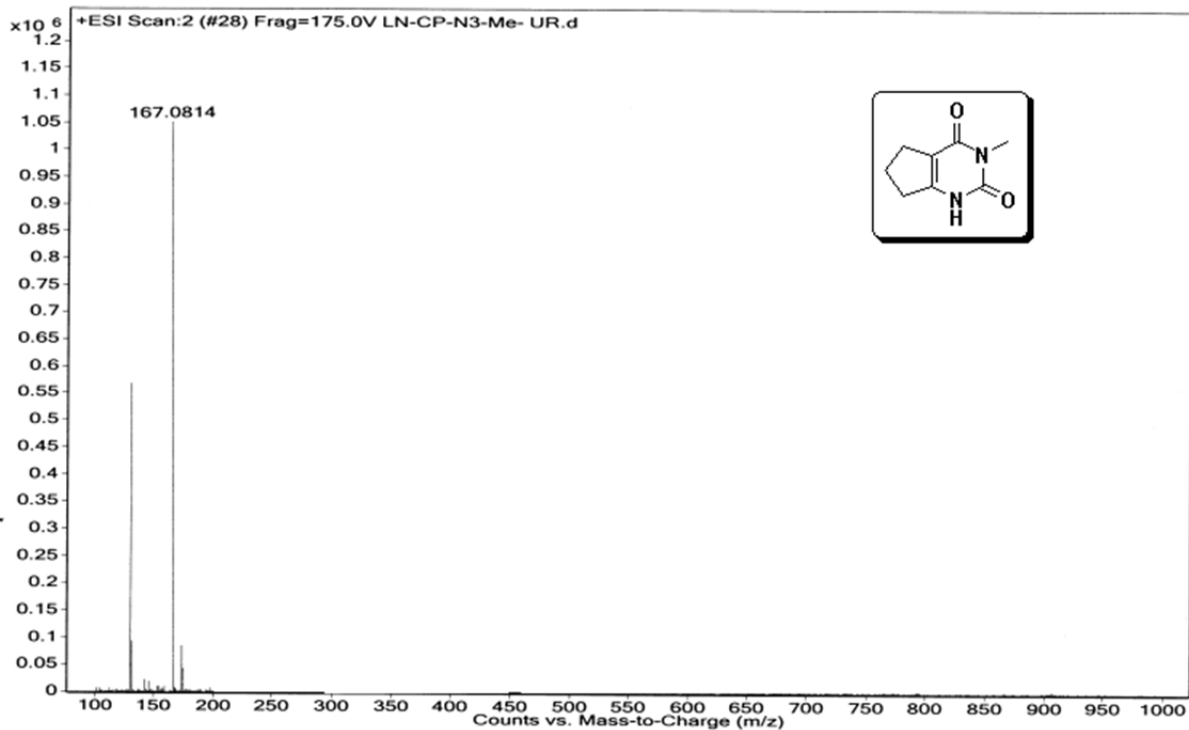


Sample Name
Inj Vol
Data Filename

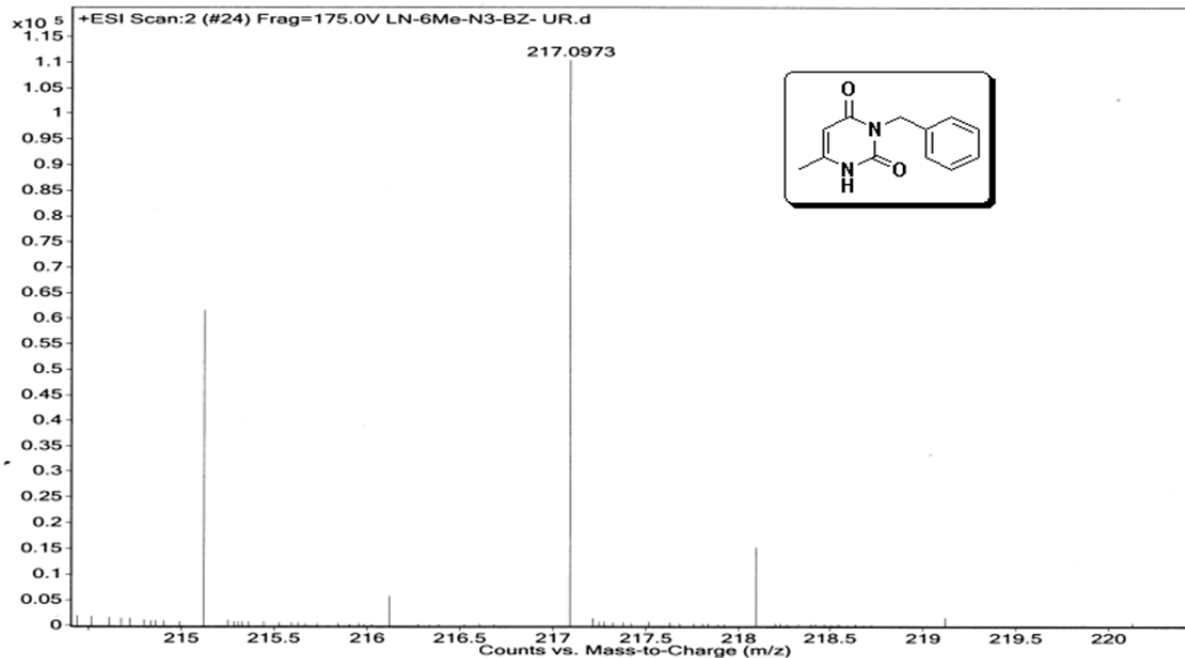
Position
InjPosition
ACQ Method

Instrument Name
SampleType
Comment

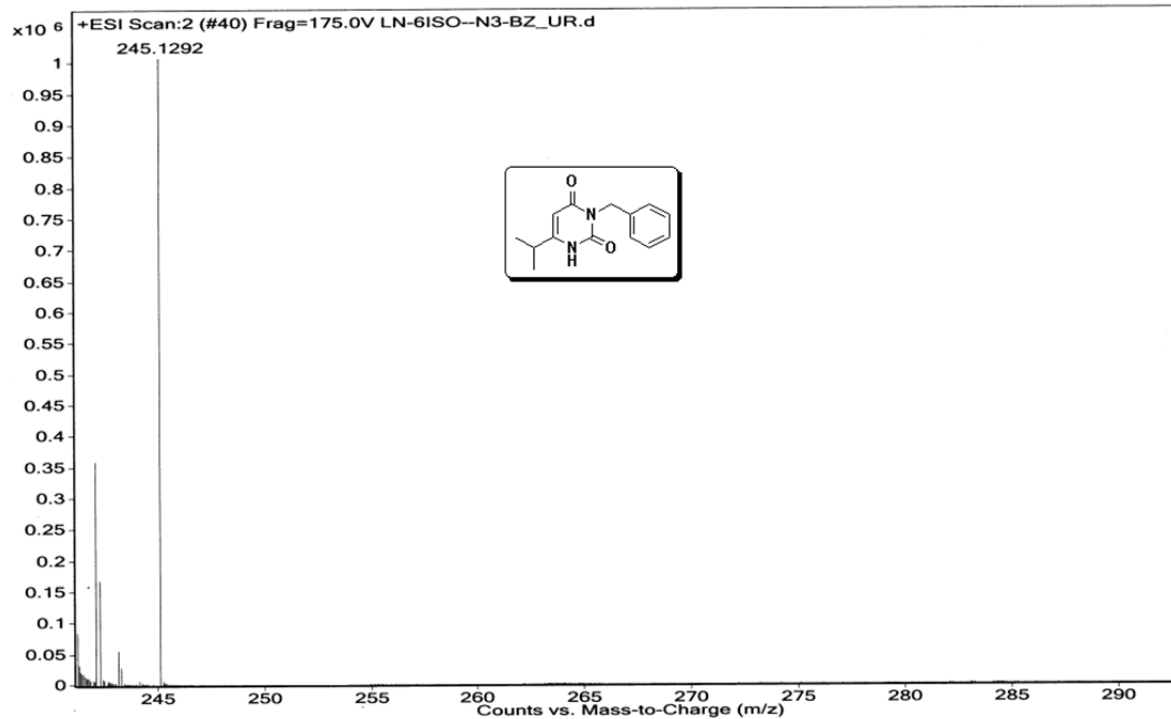
User Name
IRM Calibration Status
Acquired Time



Sample Name	Position	Instrument Name	User Name
Inj Vol	InjPosition	SampleType	IRM Calibration Status
Data Filename	ACQ Method	Comment	Acquired Time



Sample Name	Position	Instrument Name	User Name
Inj Vol	InjPosition	SampleType	IRM Calibration Status
Data Filename	ACQ Method	Comment	Acquired Time

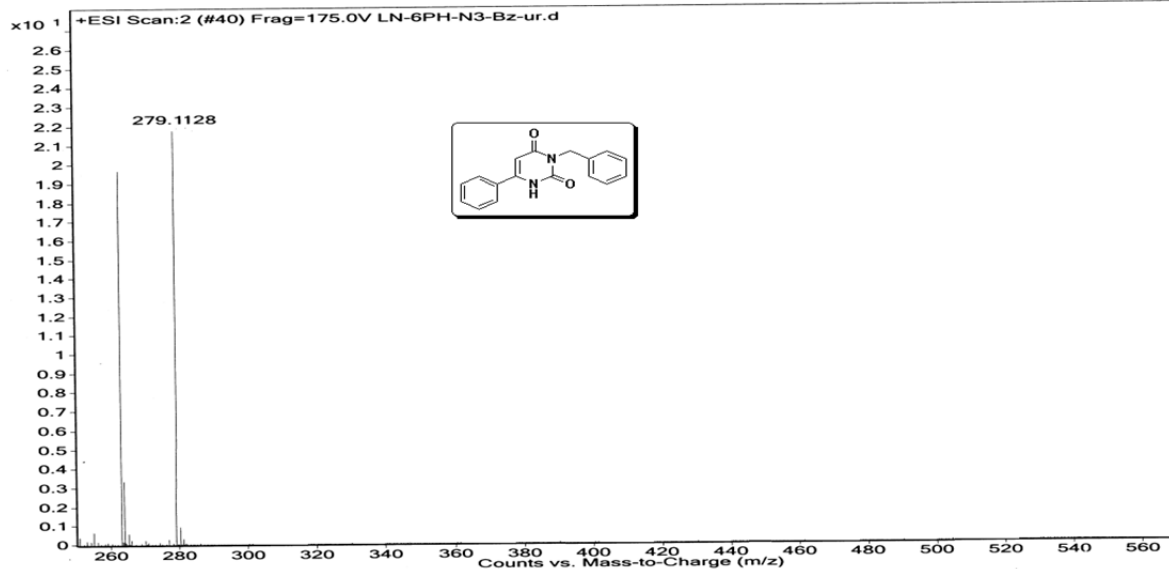


Sample Name
Inj Vol
Data Filename

Position
InjPosition
ACQ Method

Instrument Name
SampleType
Comment

User Name
IRM Calibration Status
Acquired Time

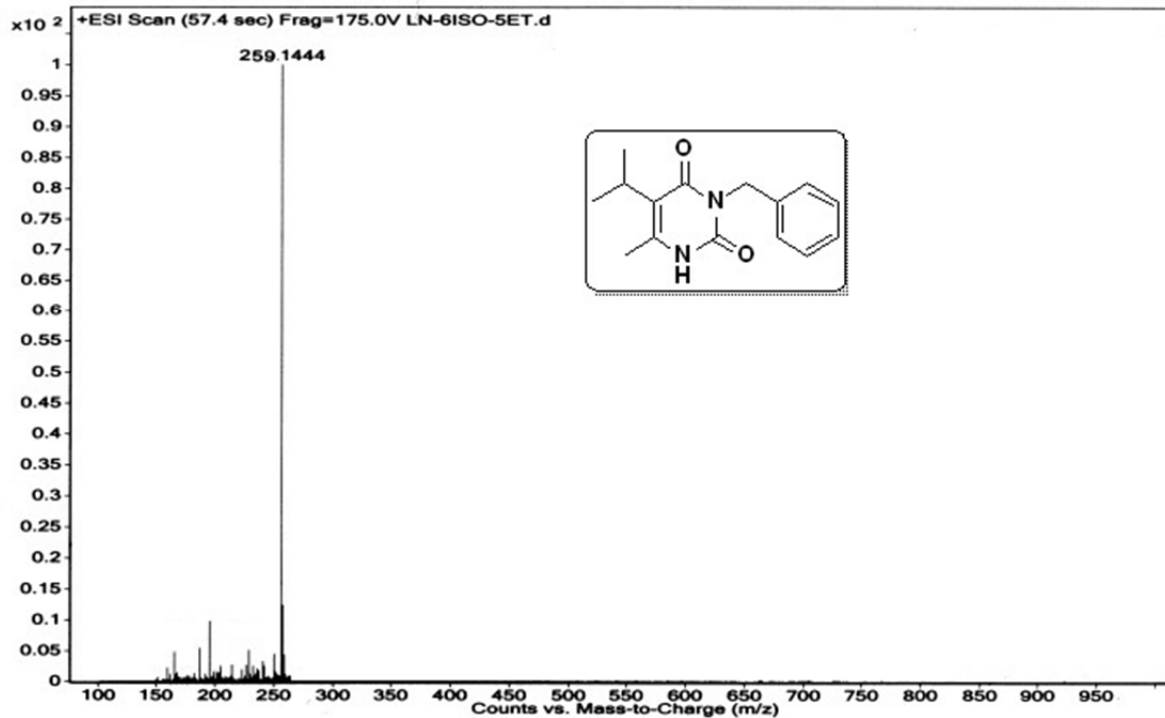


Sample Name LN-6ISO-SET
Inj Vol -10
Data Filename LN-6ISO-SET.d

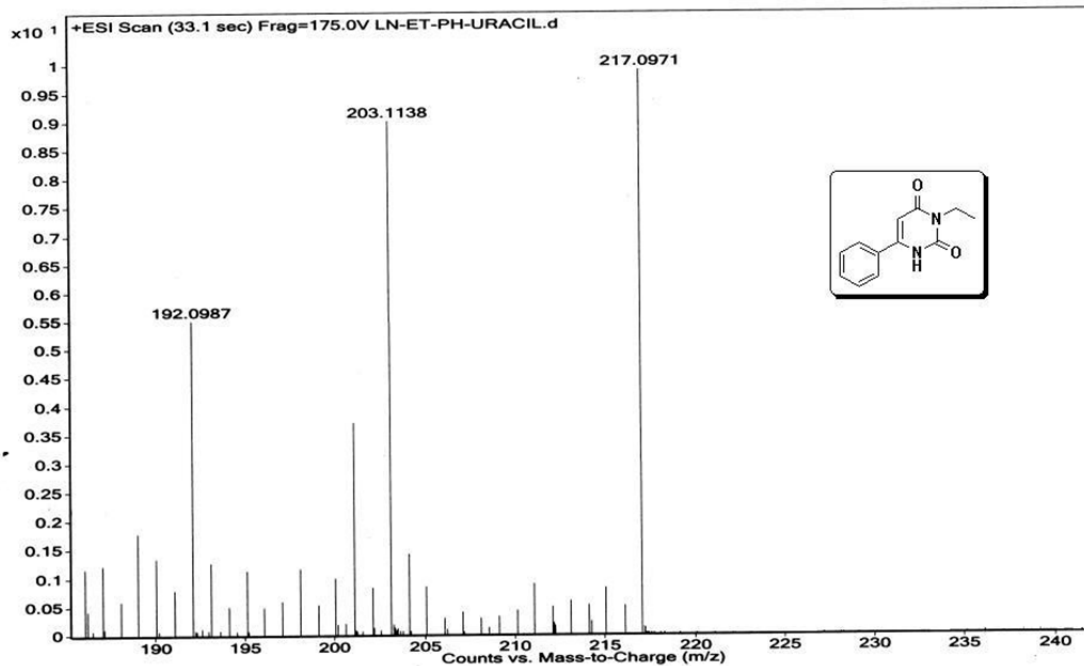
Position -1
InjPosition
ACQ Method

Instrument Name Instrument 1
SampleType Sample
Comment

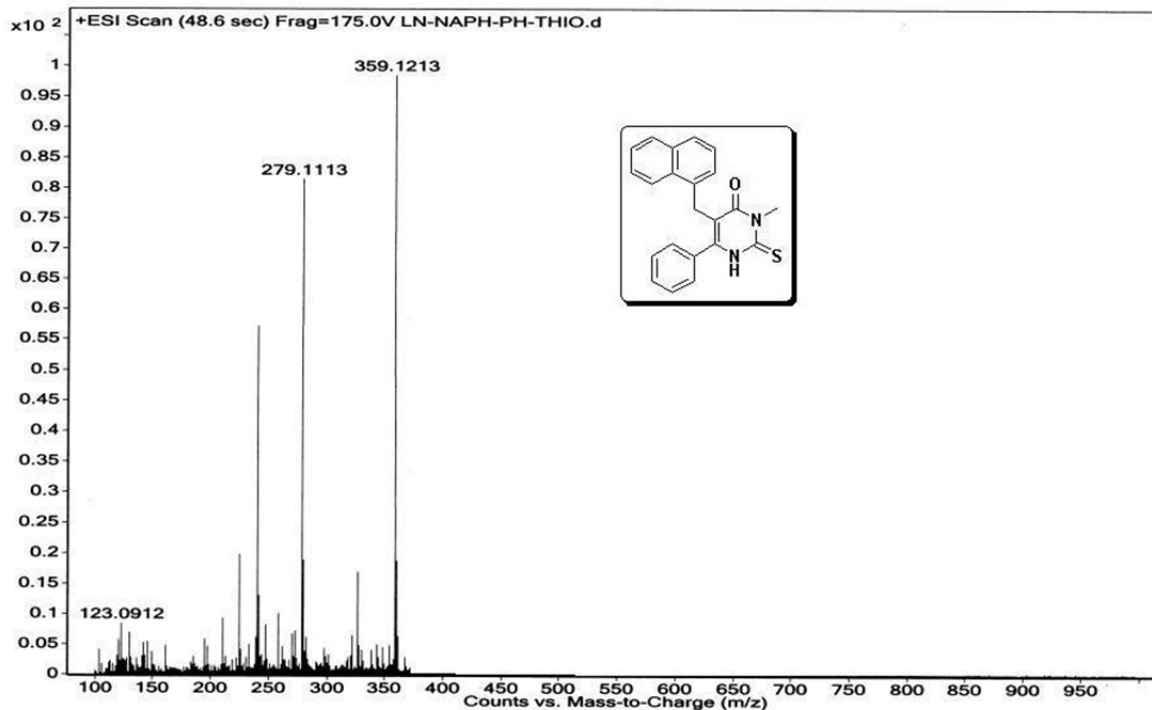
User Name
IRM Calibration Status Success
Acquired Time



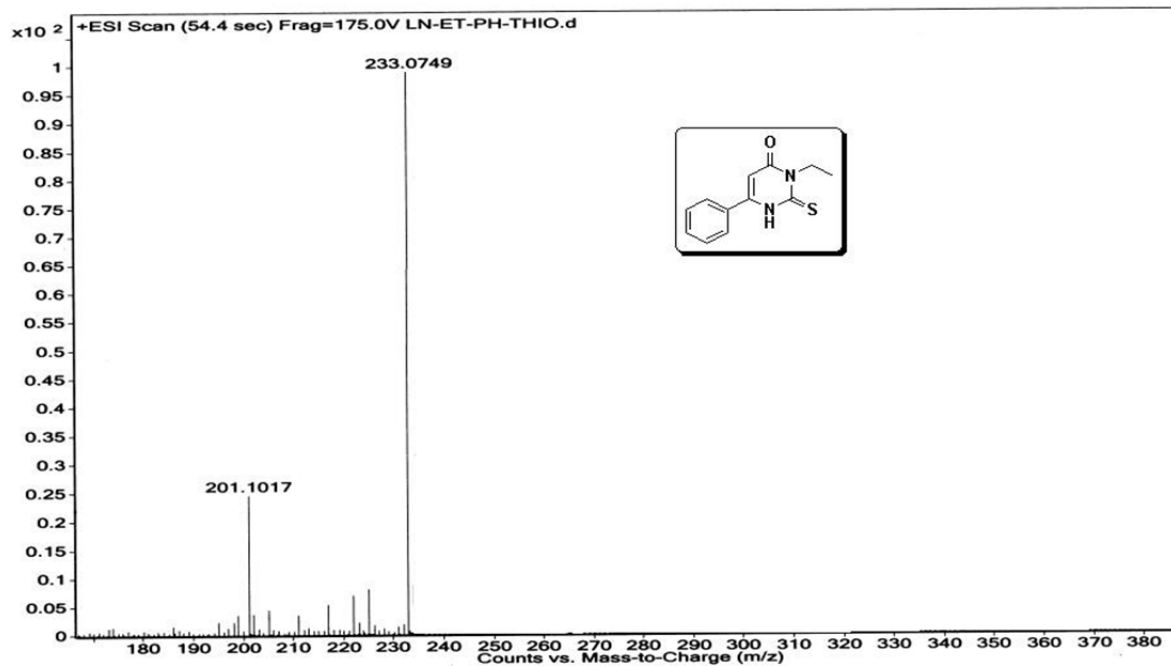
Sample Name	LN-ET-PH-URACIL	Position	-1	Instrument Name	Instrument 1	User Name	
Inj Vol	-10	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	LN-ET-PH-URACIL.d	ACQ Method		Comment		Acquired Time	3/24/2014 11:11:16 AM



Sample Name	LN-NAPH-PH-THIO	Position	-1	Instrument Name	Instrument 1	User Name	
Inj Vol	-10	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	LN-NAPH-PH-THIO.d	ACQ Method		Comment		Acquired Time	3/24/2014 11:04:01 AM



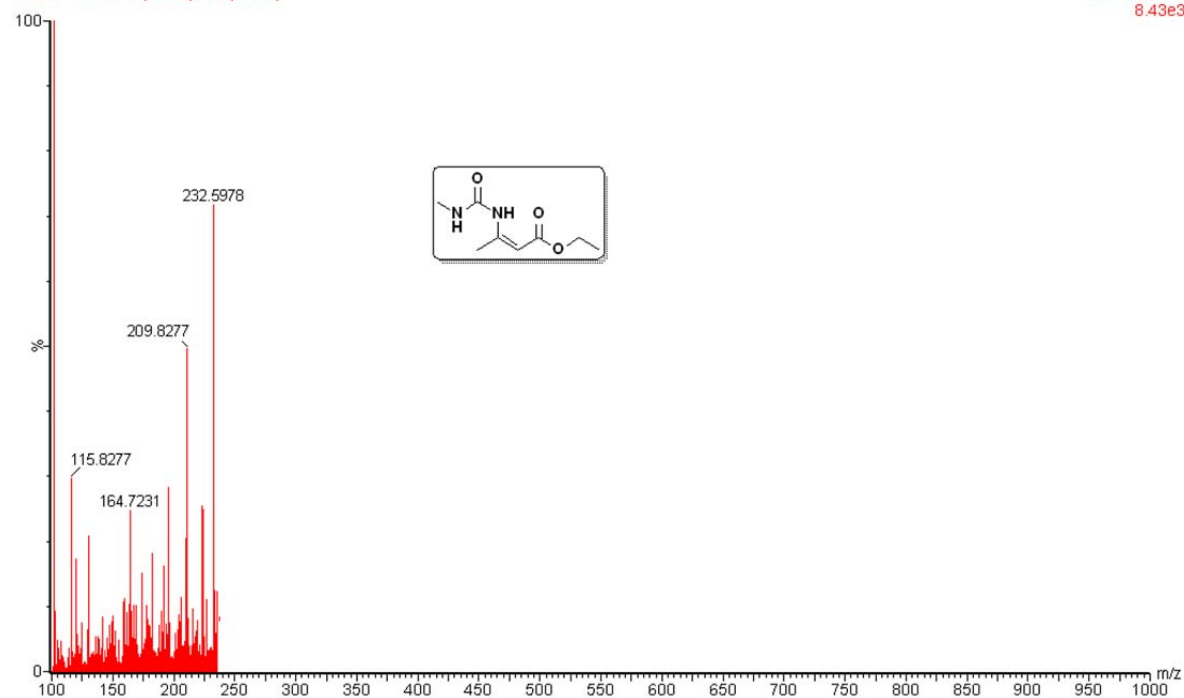
Sample Name	LN-ET-PH-THIO	Position	-1	Instrument Name	Instrument 1	User Name	
Inj Vol	-10	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	LN-ET-PH-THIO.d	ACQ Method		Comment		Acquired Time	3/24/2014 11:05:38 AM



Intermediates:

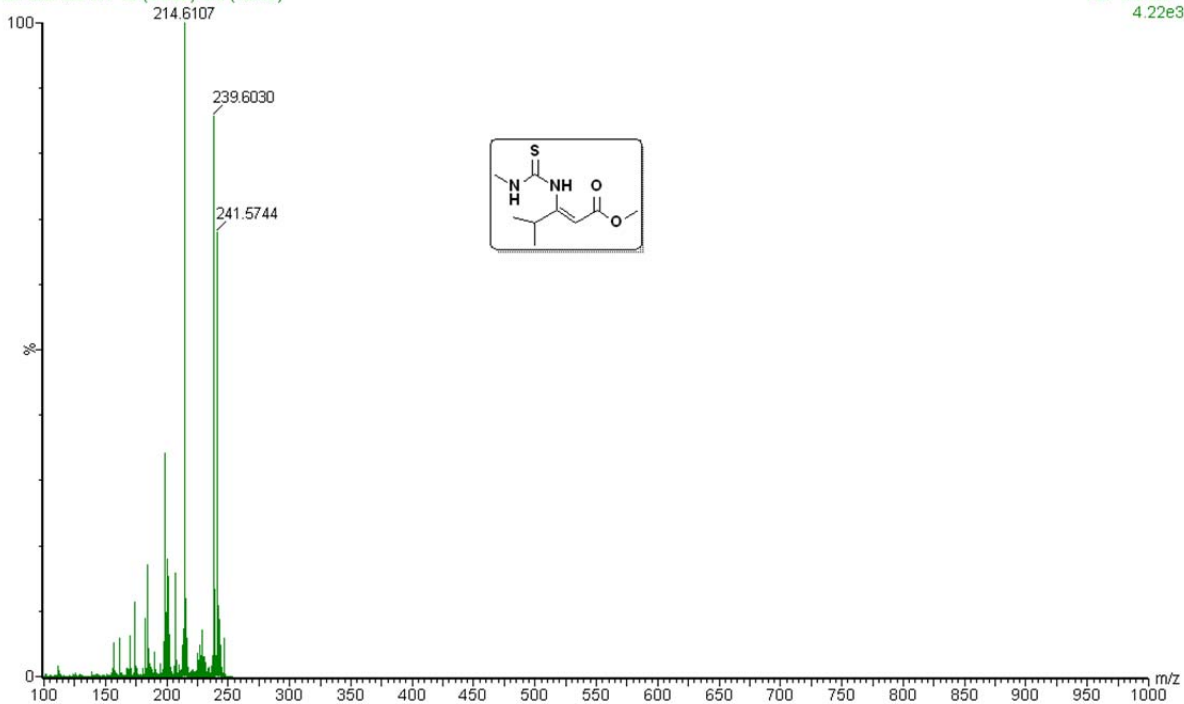
LN-THIO-INTE 30 (0.545) Cm (24.41)

TOF MS ES+
8.43e3



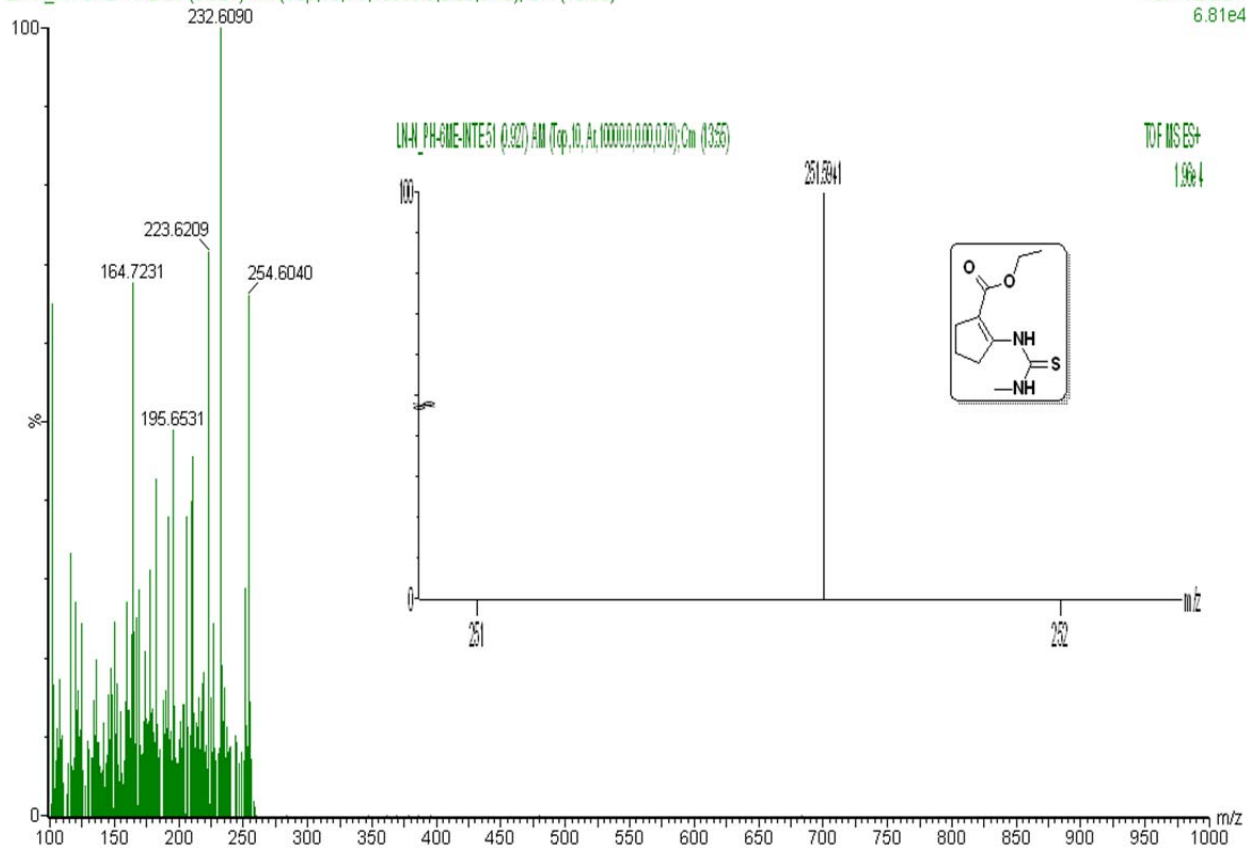
LN-ISO-ME-INT 13 (0.238) Cm (10.69)

TOF MS ES+
4.22e3



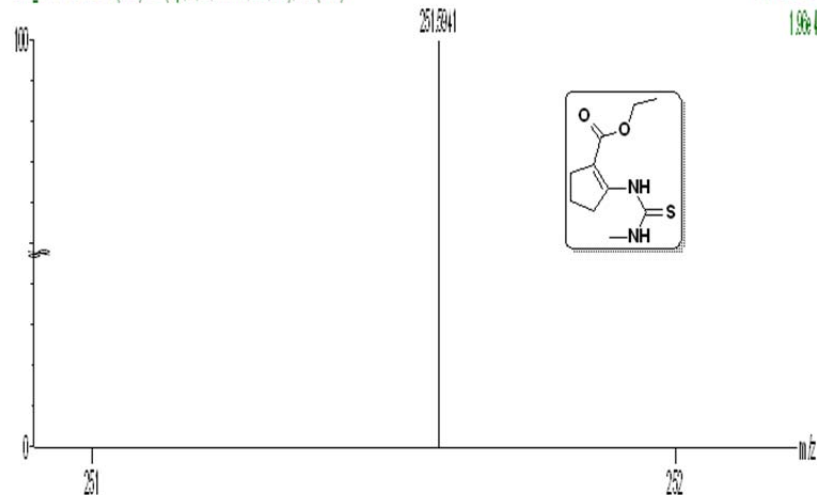
LN-N_PH-6ME-INTE 51 (0.927) AM (Top,10, Ar,10000.0,0.00,0.70); Cm (13:55)

TOF MS ES+
6.81e4



LN-N_PH-6ME-INTE 51 (0.927) AM (Top,10, Ar,10000.0,0.00,0.70); Cm (13:55)

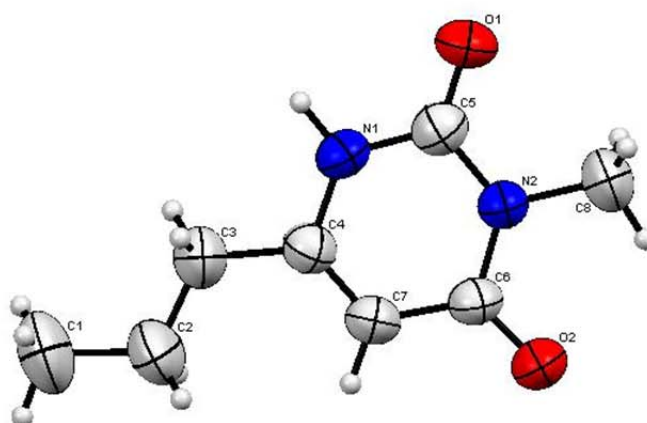
TOF MS ES+
1.90e4



3-methyl-6-propylpyrimidine-2,4(1H,3H)-dione (3):

The crystal structure of **compound 3** was obtained from DMSO-d₆ solution

Table 2: Crystallographic data of compound 3	CCDC# 991094
Chemical formula	C ₈ H ₁₂ N ₂ O ₂
Formula Mass	168.20
Temperature/K	296 K
Crystal system	Monoclinic
Space group	P2 ₁ /c
a/Å	4.7152(8)
b/Å	21.823(3)
c/Å	8.8290(15)
α /°	90
β /°	94.553(11)
γ /°	90
Unit cell volume/Å ³	905.6(3)
Z	4
μ (mm ⁻¹)	0.090
ρ _{calcd} (g cm ⁻³)	1.234
No. of reflections measured	1627
No. of independent reflections	904
Final R ₁ values ($I > 2\sigma(I)$)	0.0553
Final wR(F ₂) values ($I > 2\sigma(I)$)	0.1193
Final R ₁ values (all data)	0.1520
Final wR(F ₂) values (all data)	0.1299
Goodness of fit (F^2)	0.922

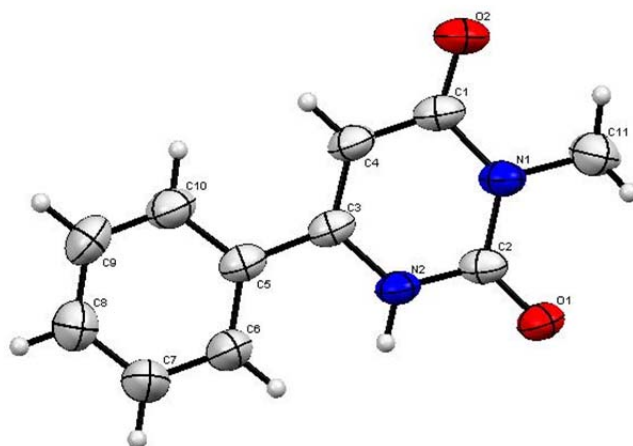


ORTEP diagram of **compound 3**: The ellipsoid countour probability level is 50%

3-methyl-6-phenylpyrimidine-2,4(1H,3H)-dione (4):

The crystal structure of **compound 4** was obtained from methanol/ethanol solution

Table 2: Crystallographic data of compound 4	CCDC# 991093
Chemical formula	C11 H10 N2 O2
Formula Mass	202.21
Temperature/K	296 K
Crystal system	Monoclinic
Space group	P21/n
a/Å	5.8924(19)
b/Å	21.161(6)
c/Å	8.054(3)
$\alpha/^\circ$	90
$\beta/^\circ$	103.67(2)
$\gamma/^\circ$	90
Unit cell volume/Å ³	975.8(5)
Z	4
μ (mm ⁻¹)	0.097
ρ_{calcd} (g cm ⁻³)	1.376
No. of reflections measured	1732
No. of independent reflections	1271
Final R1 values ($I > 2\sigma(I)$)	0.0462
Final wR(F2) values ($I > 2\sigma(I)$)	0.0952
Final R1 values (all data)	0.0621
Final wR(F2) values (all data)	0.1032
Goodness of fit (F^2)	0.968

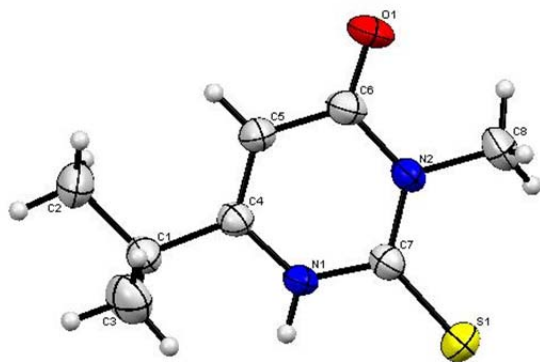


ORTEP diagram of **compound 4**: The ellipsoid countour probablity level is 50%

6-isopropyl-3-methyl-2-thioxo-2,3-dihydropyrimidin-4(1H)-one(15):

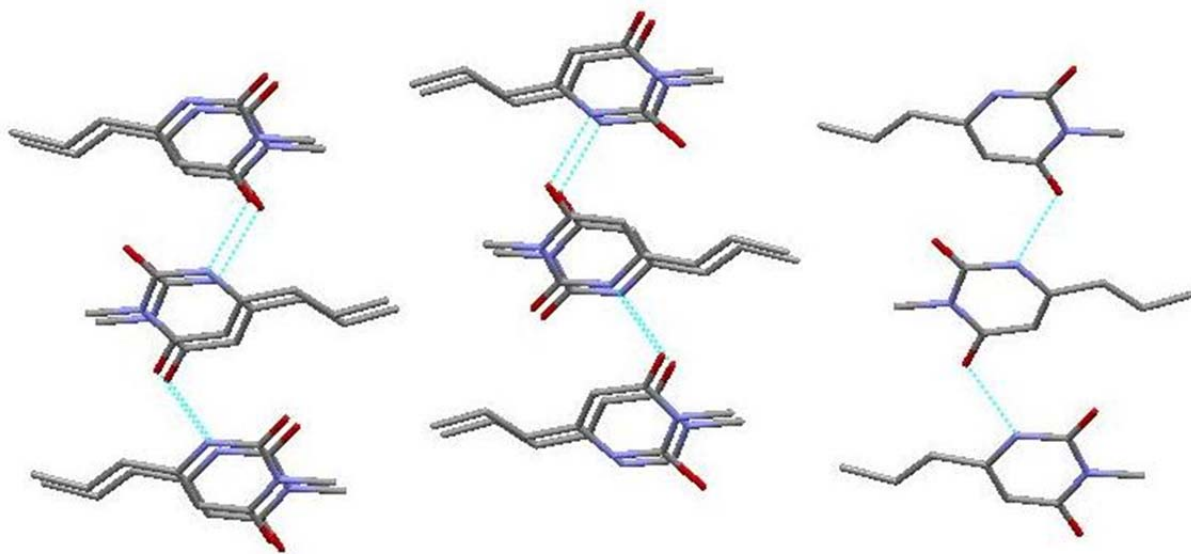
The crystal structure of **compound 15** was obtained from methanol/ethyl acetate solution

Table 2: Crystallographic data of compound 15	CCDC# 991092
Chemical formula	C ₈ H ₁₂ N ₂ O S
Formula Mass	184.26
Temperature/K	296 K
Crystal system	Monoclinic
Space group	C2/c
a/Å	21.5926(10)
b/Å	6.8375(3)
c/Å	14.9348(8)
$\alpha/^\circ$	90
$\beta/^\circ$	122.333(4)
$\gamma/^\circ$	90
Unit cell volume/Å ³	1863.09(16)
Z	8
μ (mm ⁻¹)	0.302
ρ_{calcd} (g cm ⁻³)	1.314
No. of reflections measured	1680
No. of independent reflections	1293
Final R1 values ($I > 2\sigma(I)$)	0.0343
Final wR(F2) values ($I > 2\sigma(I)$)	0.0739
Final R1 values (all data)	0.0424
Final wR(F2) values (all data)	0.0766
Goodness of fit (F^2)	1.083

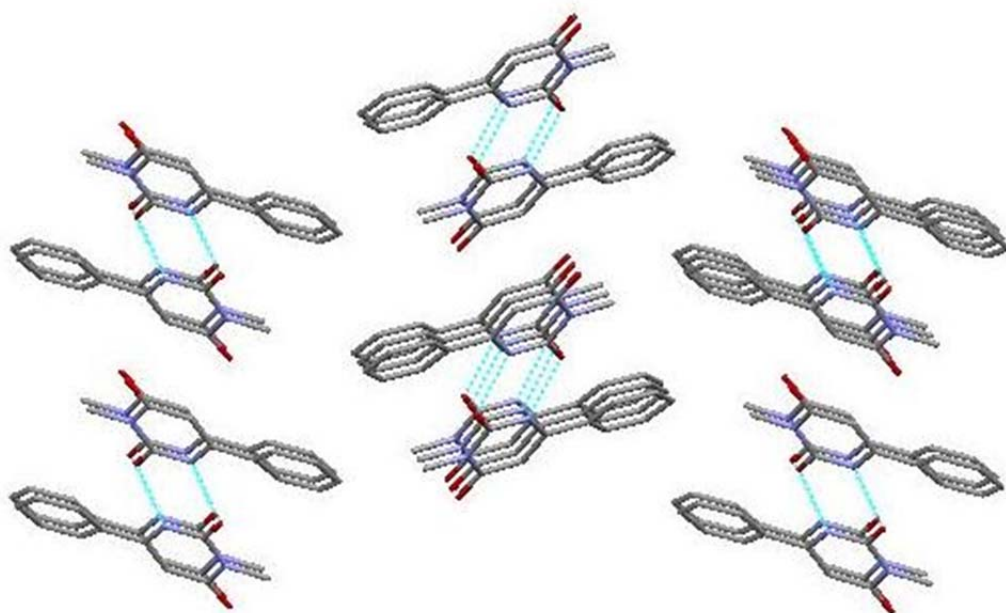


ORTEP diagram of **compound 15**: The ellipsoid countour probablity level is 50%

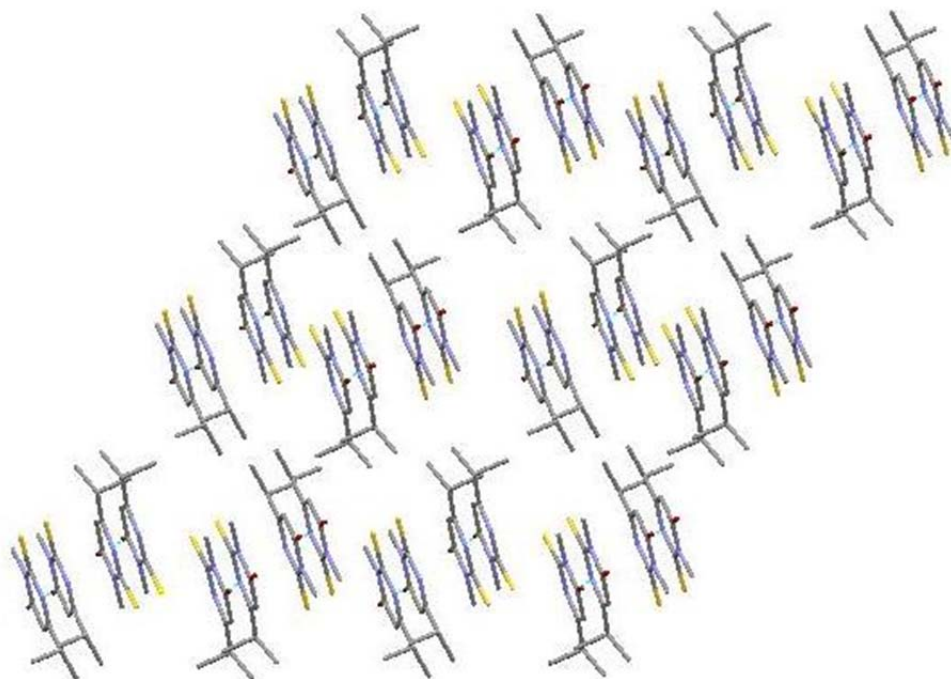
Compound (3) supramolecular architecture diagram:



Compound (4) supramolecular architecture diagram:



Compound (15) supramolecular architecture diagram:



6. References:

- 1). Ahmed, S.; Lofthouse, R.; Shaw, G. *J. Chem. Soc. Perkin Trans. 1*, **1976**, 18, 1969-1975.
- 2). Botta, M.; Cavalieri, M.; Ceci, D.; De Angelis, F.; Finizia, G.; Nicoletti, R. *Tetrahedron*. **1984**, 17, 3313-3320.
- 3). Lacey, R. N.; *J. Chem. Soc.* **1954**, 845-849.
- 4). Skulnick, H. I.; Ludens, J. H.; Wendling, M, G.; Glenn, E. M ; Rohloff, N. A.; Smith, R. J.; Wierenga, W.; *J. Med. Chem.* **1986**, 29(8), 1499-504.
- 5). Robinson, R.; Tomlinson, M. L. *J. Chem. Soc.* **1935**, 1283-284
- 6). De Stevens, G.; Halamandaris, A.; Wenk, P.; Mull, R. A.; Schlittler, E. *Archives of Biochemistry and Biophysics*. **1959**. 83. 141-51.