Supplementary Information

A simple and efficient synthesis of 5-substituted-3-phenyl-2-thioxoimidazolidin-4-one derivatives from S-amino acids and phenylisothiocyanate in Et₃N/DMF-H₂O

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1. Experimental

1.1 General Comments

All *L*-amino acids were purchased from Himedia Limited and Sd fine Chemicals limited. Phenylisothiocyanate was purchased from Sigma Aldrich India and used without further purification. Melting points were measured in open capillary tubes and are uncorrected. IR spectra were recorded on Shimadzu IR-affinity spectrometer (KBr Pelletes). ¹H NMR and ¹³C NMR spectra were recorded on a Bruker ADVANCE II 400 MHz with TMS as an internal standard. Mass spectra were under ESI mode on WATERS, Q-TOF MICROMASS (LC-MS) mass spectrometer.

1.2 Synthesis of PTH-amino acid

Experimental Section

Typical experimental procedure for the preparation of amino acid thiohydantoin

To a solution of amino acid (02 mmol) with TEA in (8:2) DMF and water (10 mL), phenylisothiocyanate (02 mmol) was added. The reaction mixture was stirred at room temperature. The progress of reaction was monitored by TLC (30% EtOAc: Hexane or 5-10% MeOH: CHCl₃). The solid product was obtained after acidification with 1N HCl. Filter the solid and recrystallize with aq. EtOH to afford pure products.

2. Spectroscopic characterization data

3-phenyl-2-thioxoimidazolidin-4-one (Entry 1) 95%, mp 246-248 °C (lit.¹⁵ 245-248 °C); IR ν_{max} (KBr): 3151, 3003, 2912, 1766, 1529, 1417, 769 cm⁻¹; ¹H NMR (400 MHz, DMSO): δ_{H} = 10.32 (*brs*, 1H), 7.27-7.46 (m, 5H), 4.25 (s, 2H) *ppm*; ¹³C NMR (100 MHz, DMSO): δ_{C} = 183.40, 171.82, 133.34, 128.59, 128.45, 128.28, 48.99 *ppm*; MS (ESI): *m/z* 193 [M+H]⁺.

5-methyl-3-phenyl-2-thioxoimidazolidin-4-one (Entry 2) 92%, mp 181 °C (lit.¹⁵ 185 °C); IR $\nu_{\max}$ (KBr): 3190, 3003, 2916, 1755, 1521, 1409, 738 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ_{H} = 10.46 (*brs*, 1H), 7.27-7.48 (m, 5H), 4.37 (q, 2H, J = 7.02 Hz), 1.45 (d, J = 7.04 Hz) *ppm*; ^{13}C NMR (100 MHz, CDCl_3): δ_{C} = 182.21, 172.75, 132.84, 128.39, 128.31, 128.26, 55.00, 16.23 *ppm*; MS (ESI): m/z 207 $[\text{M}+\text{H}]^+$.

5-isopropyl-3-phenyl-2-thioxoimidazolidin-4-one (Entry 3) 97%, mp 206-208 °C (lit.¹⁵ 206-208 °C); IR $\nu_{\max}$ (KBr): 3163, 3057, 2970, 1759, 1508, 1406, 742 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ_{H} = 10.44 (*brs*, 1H), 7.22-7.48 (m, 5H), 4.15 (d, 1H, J = 3.64 Hz), 2.24-2.32 (m, 1H), 1.11 (d, 3H, J = 7 Hz), 0.99 (d, 3H, J = 6.8 Hz) *ppm*; ^{13}C NMR (100 MHz, CDCl_3): δ_{C} = 183.02, 173.27, 132.98, 128.44, 128.32, 128.23, 64.45, 30.63, 18.05, 15.96 *ppm*; MS (ESI): m/z 234 $[\text{M}+\text{H}]^+$.

5-isobutyl-3-phenyl-2-thioxoimidazolidin-4-one (Entry 4) 97%, mp 177 °C (lit.¹⁵ 176-179 °C); IR $\nu_{\max}$ (KBr): 3169, 3060, 2933, 1759, 1519, 1408, 731 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ_{H} = 10.56 (*brs*, 1H), 7.25-7.48 (m, 5H), 4.33 (t, 1H, J = 6.72 Hz), 1.91-2.01 (m, 1H), 1.63-1.74 (m, 2H), 0.97 (d, 3H, J = 1.92 Hz), 0.96 (d, 3H, J = 2.00 Hz) *ppm*; ^{13}C NMR (100 MHz, CDCl_3): δ_{C} = 182.36, 174.27, 133.20, 128.41, 128.27, 57.90, 23.89, 22.84, 21.50 *ppm*; MS (ESI): m/z 248 $[\text{M}+\text{H}]^+$.

3,5-diphenyl-2-thioxoimidazolidin-4-one (Entry 5) 98%, mp 230-232 °C (lit.³⁰ 232-233 °C); IR $\nu_{\max}$ (KBr): 3145, 3062, 2985, 1759, 1519, 1404, 744 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ_{H} = 10.95 (*brs*, 1H), 7.28-7.52 (m, 10H), 5.51 (d, 1H) *ppm*; ^{13}C NMR (100 MHz, CDCl_3): δ_{C} = 182.77, 172.37, 139.31, 134.26, 133.19, 128.77, 128.60, 128.54, 128.46, 128.17, 126.86, 124.24, 123.53, 62.65 *ppm*; MS (ESI): m/z 267 $[\text{M}-\text{H}]^+$.

5-benzyl-3-phenyl-2-thioxoimidazolidin-4-one (Entry 6) 96%, mp 188 °C (lit.¹⁵ 187 °C); IR $\nu_{\max}$ (KBr): 3159, 3064, 2904, 1749, 1519, 1406, 690 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ_{H} = 8.20 (*brs*, 1H), 6.99-7.45 (m, 10H), 4.48 (dd, 1H, J = 4.14 Hz, 6.66 Hz), 3.26 (dd, 1H, J = 4.02, 14 Hz), 3.10 (dd, 1H, J = 6.84 Hz, 14 Hz) *ppm*; ^{13}C NMR (100 MHz, CDCl_3): δ_{C} = 183.62, 172.76, 133.96, 132.43, 129.56, 129.29, 129.12, 128.89, 128.16, 127.78, 60.86, 37.42 *ppm*; MS (ESI): m/z 283 $[\text{M}+\text{H}]^+$.

5-(hydroxymethyl)-3-phenyl-2-thioxoimidazolidin-4-one (Entry 7) 98%, mp 255 °C (lit.^a 250-253 °C); IR $\nu_{\max}$ (KBr): 3332, 3251, 3097, 2945, 1714, 1502, 1398, 688 cm^{-1} ; ^1H NMR (400 MHz, DMSO): δ_{H} = 7.94 (*brs*, 1H), 7.26-7.53 (m, 5H), 5.35 (d, 1H, J = 1.68 Hz), 5.17 (d, 1H, J = 1.72 Hz), 2.89 (s, 1H), 2.74 (s, 1H) *ppm*; ^{13}C NMR (100 MHz, DMSO): δ_{C} = 178.03, 162.31, 135.23, 132.92, 128.69, 128.59, 97.54, 35.80 *ppm*; MS (ESI): m/z 223 $[\text{M}+\text{H}]^+$.

5-(mercaptomethyl)-3-phenyl-2-thioxoimidazolidin-4-one (Entry 8) 88%, mp 155 °C (lit.¹⁶ 155-160 °C); IR $\nu_{\max}$ (KBr): 3147, 3055, 2980, 2451, 1755, 1502, 1409, 690 cm^{-1} ; ^1H NMR (400 MHz, DMSO): δ_{H} = 10.57 (*brs*, 1H), 7.24-7.50 (m, 5H), 4.76-4.80 (m, 1H), 2.47-2.70 (m, 2H), 1.38 (d, 1H) *ppm*; ^{13}C NMR (100 MHz, DMSO): δ_{C} = 182.35, 162.30, 133.33, 133.00, 128.86, 128.78, 74.77, 34.22 *ppm*; MS (ESI): m/z 239 $[\text{M}+\text{H}]^+$.

2-(5-oxo-1-phenyl-2-thioxoimidazolidin-4-yl)acetic acid (Entry 9) 95%, mp 230 °C (lit.¹⁵ 229 °C); IR $\nu_{\max}$ (KBr): 3207, 2987, 2924, 1759, 1697, 1521, 1404, 750 cm^{-1} ; ^1H NMR (400 MHz, DMSO): δ_{H} = 13.05 (*brs*, 1H), 10.38 (*brs*, 1H), 7.27-7.49 (m, 5H), 4.55 (t, 1H, J = 4.28 Hz), 2.94 (dd, 1H, J = 4.48 Hz, 12.88 Hz), 2.83 (dd, 1H, J = 4.26 Hz, 13.21 Hz) *ppm*; ^{13}C NMR (100 MHz, DMSO): δ_{C} = 183.10, 173.84, 170.54, 133.70, 128.54, 128.47, 128.32, 55.68, 34.72 *ppm*; MS (ESI): m/z 251 $[\text{M}+\text{H}]^+$.

2-(5-oxo-1-phenyl-2-thioxoimidazolidin-4-yl)acetamide (Entry 10) 91%, mp 234 °C (lit.¹⁵ 234 °C); IR $\nu_{\max}$ (KBr): 3398, 3184, 3097, 2962, 1747, 1658, 1531, 1409, 688 cm^{-1} ; ^1H NMR (400 MHz, DMSO): δ_{H} = 10.31 (*brs*, 1H), 7.49 (*brs*, 1H), 7.29-7.48 (m, 5H), 6.97 (*brs*, 1H), 4.48 (t, 1H, J = 4.38 Hz), 2.77 (d, 2H, J = 4.52 Hz) *ppm*; ^{13}C NMR (100 MHz, DMSO): δ_{C} = 183.01, 174.34, 169.80, 133.94, 128.65, 128.38, 128.19, 55.88, 35.67 *ppm*; MS (ESI): m/z 250 $[\text{M}+\text{H}]^+$.

3-(5-oxo-1-phenyl-2-thioxoimidazolidin-4-yl)propanoic acid (Entry 11) 96%, mp 169 °C (lit.¹⁵ 166-167 °C); IR $\nu_{\max}$ (KBr): 3201, 3061, 2980, 1766, 1712, 1508, 1411, 742 cm^{-1} ; ^1H NMR (400 MHz, DMSO): δ_{H} = 12.18 (*brs*, 1H), 10.56 (*brs*, 1H), 7.27-7.49 (m, 5H), 4.42 (dd, 1H, J = 5.56 Hz, 7.26 Hz), 2.41-2.49 (m, 2H), 2.09-2.18 (m, 1H), 1.95-2.04 (m, 1H) *ppm*; ^{13}C NMR (100 MHz, DMSO): δ_{C} = 182.50, 173.73, 173.47, 133.19, 128.55, 128.43, 128.34, 58.29, 28.74, 26.31 *ppm*; MS (ESI): m/z 265 $[\text{M}+\text{H}]^+$.

3-(5-oxo-1-phenyl-2-thioxoimidazolidin-4-yl)propanamide (Entry 12) 95%, mp 202-206 °C (lit.^b 205 °C); IR $\nu_{\max}$ (KBr): 3371, 3219, 3062, 2935, 1732, 1633, 1504, 1404, 736 cm^{-1} ; ^1H NMR (400 MHz, DMSO): δ_{H} = 10.52 (*brs*, 1H), 7.35 (*brs*, 1H), 7.28-7.48 (m, 5H), 6.80 (*brs*, 1H), 4.42 (t, 1H, J = 6.06 Hz), 2.28-2.33 (m, 2H), 2.07-2.16 (m, 1H), 1.97-2.04 (m, 1H) *ppm*; ^{13}C NMR (100 MHz, DMSO): δ_{C} = 182.48, 173.87, 173.18, 133.26, 128.63, 128.50, 128.40, 58.62, 29.65, 26.75 *ppm*; MS (ESI): m/z 264 $[\text{M}+\text{H}]^+$.

hexahydro-2-phenyl-3-thioxopyrrolo[1,2-*e*]imidazol-1-one (Entry 13) 92%, mp 177 °C (lit.¹⁵ 179 °C); IR $\nu_{\max}$ (KBr): 1176 cm^{-1} ; ^1H NMR (400 MHz, DMSO): δ_{H} = 7.50-7.30 (m, 5H), 4.61 (dd, 1H, J = 6.64 Hz, 10.2 Hz), 3.87-3.80 (m, 1H), 3.52-3.58 (m, 1H), 2.25-2.15 (m, 2H), 1.88-1.97 (m, 2H) *ppm*; ^{13}C NMR (100 MHz, DMSO): δ_{C} = 185.39, 173.02, 133.72, 128.63, 128.49, 65.20, 48.14, 26.28, 25.95 *ppm*; MS (ESI): m/z 233 $[\text{M}+\text{H}]^+$.

5-(1-hydroxyethyl)-3-phenyl-2-thioxoimidazolidin-4-one (Entry 14) 97%, mp 187 °C (lit.¹⁵ 194 °C); IR $\nu_{\max}$ (KBr): 3278, 3010, 2899, 1737, 1516, 1415, 750 cm^{-1} ; ^1H NMR (400 MHz, DMSO): δ_{H} = 10.46 (*brs*, 1H), 7.24-7.47 (m, 5H), 5.19 (d, 1H, J = 5.52 Hz), 4.15 (t, 1H, J = 5.78 Hz), 4.13 (*brs*, 1H), 1.32 (d, 3H, J = 6.36 Hz) *ppm*; ^{13}C NMR (100 MHz, DMSO): δ_{C} = 183.38, 172.81, 133.32, 128.48, 128.34, 128.20, 65.04, 65.53, 19.96 *ppm*; MS (ESI): m/z 237 $[\text{M}+\text{H}]^+$.

5-(2-(methylthio)ethyl)-3-phenyl-2-thioxoimidazolidin-4-one (Entry 15) 95%, mp 130-132 °C (lit.¹⁵ 132 °C); IR $\nu_{\max}$ (KBr): 3169, 2989, 2912, 1747, 1517, 1408, 707 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ_{H} = 8.34 (*brs*, 1H), 7.31-7.53 (m, 5H), 4.43 (dd, 1H, J = 4.68 Hz, 7.44 Hz), 2.71 (t, 2H, J = 6.79 Hz), 2.28-2.34 (m, 1H), 2.12-2.18 (m, 1H), 2.11 (s, 3H) *ppm*; ^{13}C NMR (100 MHz, CDCl_3): δ_{C} = 183.84, 173.43, 132.60, 129.34, 129.19, 128.23, 58.83, 30.08, 30.00, 15.20 *ppm*; MS (ESI): m/z 267 $[\text{M}+\text{H}]^+$.

5-(4-aminobutyl)-3-phenyl-2-thioxoimidazolidin-4-one (Entry 16) 93%, mp 160 °C (lit.¹⁵ 162-164 °C); IR $\nu_{\max}$ (KBr): 3211, 3035, 2904, 1749, 1554, 1450, 690 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ_{H} = 10.53 (*brs*, 1H), 9.37 (*brs*, 1H), 7.59 (*brs*, 1H), 7.29-7.48 (m, 5H), 4.35 (t, 1H, J = 5.76 Hz), 3.54 (*brs*, 2H), 1.78-1.97 (m, 2H), 1.63-1.66 (m, 2H), 1.48-1.61 (m, 2H) *ppm*; ^{13}C NMR (100 MHz, CDCl_3): δ_{C} = 182.48, 173.91, 133.13, 128.45, 128.43, 128.31, 59.17, 43.51, 30.59, 28.11, 21.36 *ppm*; MS (ESI): m/z 301 $[\text{M}+\text{K}]^+$.

3. Selected copies of IR, ^1H , ^{13}C NMR and Mass Spectra

1. IR, Mass, ^1H NMR and ^{13}C NMR spectra of (Entry 1)

GLY

10.3209

7.4696
7.4526
7.4270
7.4112
7.2909
7.2731

4.2557

3.3223

2.5181

0.0000

BRUKER
AVANCE II 400 NMR
Spectrometer
SAIF
Panjab University
Chandigarh

Current Data Parameters
NAME 05062014
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140506
Time_ 20.13
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PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 256
DW 41.600 usec
DE 6.00 usec
TE 299.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.90 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1299970 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

avtar_saifpu@yahoo.co.in

2. IR, Mass, ^1H NMR and ^{13}C NMR spectra of (Entry 2)

Molecular Weight: 206.26

ALA

10.4663

7.4864
7.4688
7.4496
7.4402
7.4280
7.4102
7.3920
7.2967
7.2775
7.2550

4.4019
4.3844
4.3667
4.3492

3.3160

2.5318

1.4632
1.4456

0.0000

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Spectrometer
SAIF
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Chandigarh

Current Data Parameters
NAME 05062014
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140506
Time 20.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 512
DW 41.600 usec
DE 6.00 usec
TE 299.3 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.90 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1299914 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

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3. IR, Mass, ^1H NMR and ^{13}C NMR spectra of (Entry 3)

WATERS, Q-TOF MICROMASS (LC-MS)

ASHA Val 19 (0.213) Cm (10:38)

SAIF/CIL,PANJAB UNIVERSITY,CHANDIGARH

TOF MS ES+
8.90e3

4. IR, Mass, ^1H NMR and ^{13}C NMR spectra of (Entry 4)

WATERS, Q-TOF MICROMASS (LC-MS)

ASHA Leu 21 (0.235) Cm (11:37)

SAIF/CIL, PANJAB UNIVERSITY, CHANDIGARH

TOF MS ES+
1.72e4

Molecular Weight: 248.34

LEU

10.5665
7.4859
7.4827
7.4782
7.4652
7.4611
7.4494
7.4461
7.4401
7.4260
7.4230
7.4197
7.4108
7.4051
7.3977
7.3895
7.3865
7.2804
7.2768
7.2715
7.2591
7.2501
4.3509
4.3329
4.3173
3.3286
2.5320
2.5276
2.5232
2.0124
1.9958
1.9792
1.9622
1.9438
1.9272
1.9107
1.7481
1.7343
1.7287
1.7135
1.7062
1.6996
1.6938
1.6897
1.6867
1.6796
1.6707
1.6517
1.6359
1.2474
1.0095
0.9810
0.9762
0.9646
0.9596
0.0000

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AVANCE II 400 NMR
Spectrometer
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Current Data Parameters
NAME 05062014
EXPNO 40
PROCNO 1

F2 - Acquisition Parameters
Date 20140506
Time 21.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 228
DW 41.600 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.90 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1299929 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

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5. IR, Mass, ^1H NMR and ^{13}C NMR spectra of (Entry 5)

DNN-808 #1479 RT: 23.43 AV: 1 NL: 1.96E5

T: + c Full ms [50.00-1000.00]

Molecular Weight: 268.334

PHG

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Current Data Parameters
NAME 05062014
EXPNO 50
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140506
Time_ 22.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 256
DW 41.600 usec
DE 6.00 usec
TE 297.8 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.90 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1299961 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

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6. IR, Mass, ^1H NMR and ^{13}C NMR spectra of (Entry 6)

WATERS, Q-TOF MICROMASS (LC-MS)

ASHA Phe 21 (0.235) Cm (9:43)

SAIF/CIL, PANJAB UNIVERSITY, CHANDIGARH

TOF MS ES+
7.29e3

Molecular Weight: 282.36

PHE

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Spectrometer
SAIF
Panjab University
Chandigarh

Current Data Parameters
NAME 05062014
EXPNO 60
PROCNO 1

F2 - Acquisition Parameters
Date 20140506
Time 22.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 144
DW 41.600 usec
DE 6.00 usec
TE 297.5 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.90 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300180 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

avtar saifpu@yahoo.co.in

7. IR, Mass, ^1H NMR and ^{13}C NMR spectra of (Entry 7)

WATERS, Q-TOF MICROMASS (LC-MS)

ASHA Ser 5 (0.056) Cm (2:39)

SAIF/CIL, PANJAB UNIVERSITY, CHANDIGARH

TOF MS ES+
1.79e3

Molecular Weight: 222.264

8. IR, Mass, ^1H NMR and ^{13}C NMR spectra of (Entry 8)

WATERS, Q-TOF MICROMASS (LC-MS)

ASHA CYS 2 (0.023) Cm (1:37)

SAIF/CIL,PANJAB UNIVERSITY,CHANDIGARH

TOF MS ES+
512

9. IR, Mass, ^1H NMR and ^{13}C NMR spectra of (Entry 9)

WATERS, Q-TOF MICROMASS (LC-MS)

ASHA Asp 51 (0.572) Cm (14:51)

SAIF/CIL,PANJAB UNIVERSITY,CHANDIGARH

TOF MS ES+
2.81e3

Molecular Weight: 250.27

ASP

13.0511

10.3802

7.4924
7.4884
7.4749
7.4557
7.4311
7.4184
7.4132
7.4064
7.3947
7.2936
7.2904
7.2726

4.5696
4.5590
4.5482
2.9721
2.9608
2.9286
2.9175
2.8618
2.8512
2.8184
2.8077
2.5213
2.5170
2.5128

0.0000

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Spectrometer
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Current Data Parameters
NAME 05062014
EXPNO 90
PROCNO 1

F2 - Acquisition Parameters
Date 20140506
Time 23.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 456
DW 41.600 usec
DE 6.00 usec
TE 296.5 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.90 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1299967 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

avtar_saifpu@yahoo.co.in

10. IR, Mass, ^1H NMR and ^{13}C NMR spectra of (Entry 10)

WATERS, Q-TOF MICROMASS (LC-MS)

ASHA Asn 14 (0.156) Cm (11:46)

SAIF/CIL,PANJAB UNIVERSITY,CHANDIGARH

TOF MS ES+
5.80e3

ASN

10.3131
7.4983
7.4836
7.4805
7.4763
7.4629
7.4590
7.4439
7.4211
7.4180
7.4149
7.4057
7.4001
7.3930
7.3847
7.3816
7.3206
7.3170
7.3120
7.2993
6.9707
4.5000
4.4891
4.4781
3.3740
2.7800
2.7687
2.5196
2.5151
2.5107
0.0000

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Spectrometer
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Panjab University
Chandigarh

Current Data Parameters
NAME 05062014
EXPNO 100
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140507
Time_ 0.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 181
DW 41.600 usec
DE 6.00 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.90 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1299974 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

avtar saifpu@yahoo.co.in

11. IR, Mass, ^1H NMR and ^{13}C NMR spectra of (Entry 11)

WATERS, Q-TOF MICROMASS (LC-MS)

ASHA Glu 17 (0.190) Cm (11:44)

SAIF/CIL,PANJAB UNIVERSITY,CHANDIGARH

TOF MS ES+
6.09e3

Molecular Weight: 264.300

GLU

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SAIF
Panjab University
Chandigarh

Current Data Parameters
NAME 05062014
EXPNO 110
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140507
Time_ 0.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 512
DW 41.600 usec
DE 6.00 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.90 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1299904 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

avtar saifpu@yahoo.co.in

12. IR, Mass, ^1H NMR and ^{13}C NMR spectra of (Entry 12)

Spectrum Plot - 6/26/2013 4:20 PM

1 A Scan 19 from c:\lilt\2013-2014\lcms-24\d9-264-msms.xms

Spectrum 1A
BP: 247.6 (13027=100%), d9-264-msms.xms

0.376 min, Scan: 19, 264.0>91:274 [0.60V], Ion: 197639 us, RIC: 42476

kCounts

12.5

10.0

7.5

5.0

2.5

0.0

Molecular Weight: 263.316

247.6
13027

263.6
11196

264.5
8880

248.5
828

264.0
1195

m/z

100

150

200

GLN

10.5202

7.4844
7.4804
7.4670
7.4478
7.4265
7.4141
7.4087
7.4014
7.3902
7.3528
7.3027
7.2995
7.2816
6.8067
4.4370
4.4217
4.4067
3.3986
2.5131
2.3306
2.3220
2.3099
2.3065
2.2905
2.2859
2.1604
2.1409
2.1264
2.1070
2.0919
2.0733
2.0440
2.0275
2.0098
1.9914
1.9898
1.9748
1.9716

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Current Data Parameters
NAME Oct03-2013
EXPNO 140
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131003
Time 17.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 64
DW 41.600 usec
DE 6.00 usec
TE 297.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.90 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1299985 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

avtar saifpu@yahoo.co.in

13. IR, Mass, ^1H NMR and ^{13}C NMR spectra of (Entry 13)

WATERS, Q-TOF MICROMASS (LC-MS)

ASHA PRO 94 (1.052) Cm (1:98)

SAIF/CIL, PANJAB UNIVERSITY, CHANDIGARH

TOF MS ES+

4.53e3

Molecular Weight: 232.301

299.1
4526

Pro 1

7.5028
7.4852
7.4657
7.4422
7.4299
7.4243
7.4059
7.3280
7.3247
7.3060
4.7759
4.6369
4.6210
4.6121
4.5948
3.8710
3.8503
3.8420
3.8292
3.8220
3.8020
3.5852
3.5782
3.5635
3.5559
3.5490
3.5351
3.5278
3.3399
2.5109
2.5072
2.5032
2.2589
2.2374
2.2284
2.2246
2.2131
2.2033
2.1981
2.1840
2.1664
2.1524
1.9791
1.9621
1.9349
1.9073
1.8881
1.7720
1.2376
0.0000

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Current Data Parameters
NAME Sep15-2014
EXPNO 490
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140915
Time_ 22.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 645
DW 41.600 usec
DE 6.00 usec
TE 294.5 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.90 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300005 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

avtar_saifpu@yahoo.co.in

14. IR, Mass, ^1H NMR and ^{13}C NMR spectra of (Entry 14)

WATERS, Q-TOF MICROMASS (LC-MS)

ASHA Thr 33 (0.370) Cm (13:45)

SAIF/CIL,PANJAB UNIVERSITY,CHANDIGARH

TOF MS ES+
5.97e3

Molecular Weight: 236.290

15. IR, Mass, ^1H NMR and ^{13}C NMR spectra of (Entry 15)

WATERS, Q-TOF MICROMASS (LC-MS)

ASHA Met 23 (0.258) Cm (13:39)

SAIF/CIL,PANJAB UNIVERSITY,CHANDIGARH

TOF MS ES+
4.28e3

Molecular Weight: 266.38

MET

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Current Data Parameters
NAME 05062014
EXPNO 130
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140507
Time 1.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 256
DW 41.600 usec
DE 6.00 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.90 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

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16. IR, Mass, ^1H NMR and ^{13}C NMR spectra of (Entry 16)

ASHA Lys 25 (0.280) Cm (12:51)

TOF MS ES+
3.44e3

Molecular Weight: 263.36

LYS

10.5395
9.3748
7.5907
7.4801
7.4629
7.4438
7.4266
7.4081
7.3917
7.3107
7.2908
7.2707
4.3671
4.3519
4.3383
3.5487
3.3732
2.5345
1.9725
1.9499
1.9383
1.9261
1.9131
1.9001
1.8762
1.8611
1.8378
1.8216
1.8036
1.7873
1.6678
1.6505
1.6331
1.6162
1.5992
1.5766
1.5602

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Current Data Parameters
NAME 05062014
EXPNO 140
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140507
Time 2.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 256
DW 41.600 usec
DE 6.00 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.90 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1299902 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

avtar saifpu@yahoo.co.in

References:

(a) Ramachandran, L. K.; Epp, A.; McConnell, W. B. Infrared Spectra of 3-Phenyl-2-thiohydantoins of amino acids and their application to identification of N-terminal groups in peptides, *Anal. Chem.* **1955**, 27, 1734.

(b) Phenylthiohydantoin-glutamine, CAS No. 10567-86-9

website: http://www.chemicalbook.com/ChemicalProductProperty_EN_CB0695950.htm