Supporting Information

On Formation of Thiohydantoins From Amino Acids Under Acylation Conditions

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NMR (^1H , ^{13}C and COSY) of Compounds **1**, **2a-c**, **4a-c** and **5**

NMR of 1 -----	S2
NMR of 2a -----	S3
NMR of 2b -----	S5
NMR of 2c -----	S7
NMR of 4a -----	S8
NMR of 4b -----	S9
NMR of 4c -----	S11
NMR of 5 -----	S12

1
 ^1H NMR (500 MHz, $\text{DMSO}-d_6$)

^{13}C NMR (125 MHz, $\text{DMSO}-d_6$)

C13-3-Bn-5-Me-2-thiohydantoin
Pulse Sequence: zgpg30
Solvent: dms
Ambient temperature
User: 1-14-87
File: C13-3-Bn-5-Me-2-thiohydantoin
INNOVA-500 "nmrsum1"

Pulse 61.4 degrees
Acq. time 1.360 sec
Width 25000.0 Hz
387 repetitions
OBSERVE C13, 125.7242675 MHz
DECOUPLE H1, 499.9990248 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 21 min, 49 sec

COSY of **1** **2a**¹H NMR (500 MHz, DMSO-*d*₆)

^{13}C NMR (75 MHz, $\text{DMSO-}d_6$)

COSY of **2a**

The NH peak around 12.6 shows that no cross peak is observed with the C-alpha H at ~4.4 ppm.

2b¹H NMR (500 MHz, DMSO-*d*₆) ¹³C NMR (125 MHz, DMSO-*d*₆)

Solvent: dmsc
Ambient temperature
User: 1-14-87
File: C13_1-acetyl-2-thiohydantoin_of_alanine_Ac20method
INOVA-500 "nmrsun1"

Pulse 67.5 degrees
Acq. time 1.300 sec
Width 25000.0 Hz
323 repetitions
OBSERVE C13, 125.7427953 MHz
DECOUPLE H1, 500.0726397 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 21 min, 49 sec

COSY of **2b**

The NH peak around 12.6 shows that no cross peak is observed with the C-alpha H at ~4.7 ppm

2c

¹H NMR (500 MHz, DMSO-*d*₆)

^{13}C NMR (125 MHz, DMSO- d_6) COSY of **2c**

The NH peak around 12.4 shows that no cross peak is observed with the C-alpha H at ~5 ppm

4a¹H NMR (500 MHz, DMSO-*d*₆) ¹³C NMR (125 MHz, DMSO-*d*₆)

COSY of **4a**

After deacylation, the N1 is now protonated and resonates ~9.9 ppm. A cross peak is observed with the C-alpha H ~4 ppm.

4b

^1H NMR (500 MHz, DMSO- d_6)

```
-thiohydantoin_of_alanine
Archive directory: /home/burgess/sreyes/nmrays/data
Sample directory: 2-thiohydantoin_of_alanine_29Jul2004

size Sequence: s2pul
Solvent: dmsc
Ambient temperature
file: PROTON
NOVA-500 "nmrsun1"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.892 sec
Width 6783.1 Hz
# repetitions
SSERVE H1, 500.0460374 MHz
ATA PROCESSING
F size 65536
otal time 0 min, 31 sec
```

^{13}C NMR (125 MHz, DMSO- d_6)

Solvent: dmsc
Ambient temperature
User: 1-14-87
File: C13_5-Me-2-thiohydantoin
INNOVA-500 "nursun1"

Pulse 61.4 degrees
Acq. time 1.300 sec
Width 25000.0 Hz
194 repetitions
OBSERVE C13, 125.7242836 MHz
DECOUPLE H1, 499.9990268 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 21 min, 49 sec

COSY of **4b**

After deacylation, the N1 is now protonated and resonates ~10 ppm. A cross peak is observed with the C-alpha H ~4.2 ppm

NMR of **4c** ^1H NMR (500 MHz, DMSO- d_6) ^{13}C NMR (125 MHz, DMSO- d_6)

C13_5-Bn-2-thiohydantoin

Pulse Sequence: s2pul

Solvent: dmsc

Ambient temperature

User: 1-14-87

File: C13_5-Bn-2-thiohydantoin

INOVA-500 "nmrsun1"

Pulse 61.4 degrees

Acq. time 1.300 sec

Width 25000.0 Hz

194 repetitions

OBSERVE C13, 125.7242691 MHz

DECOUPLE H1, 499.9990268 MHz

Power 42 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 65536

Total time 21 min, 49 sec

COSY of **4c**

After deacylation, the N1 is now protonated and resonates ~10 ppm. A cross peak is observed with the C-alpha H ~4.5 ppm

NMR of **5**

¹H NMR (500 MHz, DMSO-*d*₆)

^{13}C NMR (125 MHz, DMSO- d_6)

COSY of **5**