**[Bis(2-Methoxyethyl)amino]sulfur Trifluoride – The Deoxo-Fluor™ Reagent:
Application Toward One-Flask Transformations of Carboxylic Acids to Amides**

Jonathan M. White, Ashok Rao Tunoori, Brandon J. Turunen, and Gunda I. Georg*

*Department of Medicinal Chemistry, University of Kansas, 1251 Wescoe Hall Drive,
Lawrence, Kansas 60045-7582*

Table of Contents

1. General experimental paragraph and characterization data.....	S2-4
2. Copy of ¹ H-NMR and ¹³ C-NMR compound 3	S5-6
3. Copy of ¹ H-NMR and ¹³ C-NMR compound 4	S7-8
4. Copy of ¹ H-NMR and ¹³ C-NMR compound 8	S9-10
5. Copy of ¹ H-NMR and ¹³ C-NMR compound 14	S11-12
6. Copy of ¹ H-NMR and ¹³ C-NMR compound 16	S13-14
7. Copy of ¹ H-NMR and ¹³ C-NMR compound 17	S15-16
8. Copy of ¹ H-NMR and ¹³ C-NMR compound 23	S17-18
9. Copy of ¹ H-NMR and ¹³ C-NMR compound 24	S19-20

General Methods. All commercially available reagents and solvents were used without further purification, unless otherwise noted. Methylene chloride was dried by distillation from calcium hydride. Flash column chromatography was carried out on silica gel. TLC was conducted on silica gel 250 micron, F₂₅₄ plates. Compounds 5,²⁴ 6,²⁵ 7,²⁶ 9,⁴ 11,²⁷ 12,²⁸ 13,¹⁹ 15,²⁹ and 18-22^{10,19,30,31} have been previously reported. Compound 10 is commercially available. Our spectroscopic data were in agreement with the reported values.

Heptanoic Acid Diethylamide (3). Isolated as an oil. *R_f* 0.6 (hexanes:ethyl acetate 1:1); ¹H NMR (400 MHz, CDCl₃) δ 3.18-3.28 (m, 4H), 2.19 (t, *J* = 7.5 Hz, 2H), 1.52-1.54 (m, 2H), 1.20 (bm, 6H), 1.07 (t, *J* = 7.1 Hz, 3H), 1.00 (t, *J* = 7.1 Hz, 3H), 0.79 (t, *J* = 6.9 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 172.6, 42.3, 40.3, 33.4, 32.0, 29.5, 25.8, 22.8, 14.7, 14.3, 13.4; IR (film) 1639 cm⁻¹; MS HRFAB (pos) *m/z* calc'd for [MH⁺] C₁₁H₂₄NO: 186.1858, found 186.1856.

1-Morpholin-4-yl-heptan-1-one (4). Isolated as an oil. *R_f* 0.4 (hexanes:ethyl acetate 1:1); ¹H NMR (400 MHz, CDCl₃) δ 3.54-3.57 (m, 4H), 3.50 (d, *J* = 5.0 Hz, 2H), 2.21 (t, *J* = 7.5 Hz, 2H), 1.48-1.53 (m, 2H), 1.17-1.25 (m, 6H), 0.76-0.80 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 172.1, 67.2, 67.0, 46.3, 42.1, 33.4, 31.9, 29.4, 25.5, 22.8, 14.4; IR (film) 1644 cm⁻¹; MS HRFAB (pos) *m/z* calc'd for [MH⁺] C₁₁H₂₂NO₂: 200.1651, found 200.1672.

(4-Diethylcarbamoylphenyl)-carbamic Acid *tert*-Butyl Ester (8). Isolated as an off-white solid, mp 160-163 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.39 (d, $J = 8.2$ Hz, 2H), 7.28 (d, $J = 8.1$ Hz, 2H), 7.12 (s, 1H), 3.31-3.45 (bm, 4H), 1.50 (s, 9H), 1.15-1.26 (m, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 171.3, 152.9, 139.7, 131.5, 127.5, 118.2, 80.7, 43.4, 28.4, 14.2, 13.0; IR (film) 1726, 1603 cm^{-1} ; MS HRFAB (pos) m/z calc'd for $[\text{MH}^+]$ $\text{C}_{16}\text{H}_{25}\text{N}_2\text{O}_3$: 293.1865, found 293.1866.

Cyclopentanecarboxylic Acid Methoxymethylamide (14). Isolated as an oil. R_f 0.7 (hexanes:ethyl acetate 1:1); ^1H NMR (400 MHz, CDCl_3) δ 3.69 (s, 3H), 3.20 (s, 3H), 3.00 (bs, 1H), 1.74-1.88 (m, 6H), 1.56-1.61 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 178.0, 61.5, 40.4, 32.5, 26.3; IR (film) 1659 cm^{-1} ; MS HRFAB (pos) m/z calc'd for $[\text{MH}^+]$ $\text{C}_8\text{H}_{16}\text{NO}_2$: 158.1181, found 158.1181.

3,4-Dichloro-*N*-methoxy-*N*-methylbenzamide (16). Isolated as an oil. R_f 0.4 (hexanes:ethyl acetate 1:1); ^1H NMR (400 MHz, CDCl_3) δ 7.83 (s, 1H), 7.58 (d, $J = 8.3$ Hz, 1H), 7.50 (d, $J = 8.3$ Hz, 1H), 3.56 (s, 3H), 3.34 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 167.4, 135.2, 133.8, 132.5, 130.7, 130.3, 127.9, 61.5, 33.6; IR (film) 1644 cm^{-1} ; MS HRFAB (pos) m/z calc'd for $[\text{MH}^+]$ $\text{C}_9\text{H}_{10}\text{NO}_2\text{Cl}_2$: 234.0089, found 234.0082.

Thiophene-2-carboxylic Acid Methoxymethylamide (17). Isolated as an oil. R_f 0.4 (hexanes:ethyl acetate 1:1); ^1H NMR (400 MHz, CDCl_3) δ 7.98 (d, $J = 3.8$ Hz, 1H), 7.57 (d, $J = 4.1$ Hz, 1H), 7.11 (t, $J = 4.1$ Hz, 1H), 3.78 (s, 3H), 3.82 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 162.4, 134.5, 133.4, 132.4, 127.0, 61.7, 33.2; IR (film) 1623 cm^{-1} ; MS HRFAB (pos) m/z calc'd for $[\text{MH}^+]$ $\text{C}_7\text{H}_{10}\text{NO}_2\text{S}$: 172.0432, found 172.0452.

Thiophene-2-carboxylic Acid Bis-(2-methoxyethyl)amide (23). Isolated as an oil. R_f 0.2 (hexanes:ethyl acetate 1:1); ^1H NMR (400 MHz, CDCl_3) δ 7.45 (d, $J = 4.0$ Hz, 1H), 7.42 (d, $J = 5.0$ Hz, 1H), 7.02 (s, 1H), 3.78 (bt, 4H), 3.61 (bt, 4H), 3.34 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 164.9, 138.1, 129.3, 128.8, 126.9, 71.0, 59.0, 48.0, 31.0; IR (film) 1608 cm^{-1} ; MS HRFAB (pos) m/z calc'd for $[\text{MH}^+]$ $\text{C}_{11}\text{H}_{18}\text{NO}_3\text{S}$: 244.1007, found 244.1014.

***N,N*-Bis-(2-methoxyethyl)-benzamide (24).** Isolated as an oil. ^1H NMR (400 MHz, CDCl_3) δ 7.34-7.40 (m, 5H), 3.65-3.74 (m, 4H), 3.23-3.50 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3) δ 172.2, 136.8, 129.9, 129.2, 126.9, 70.7, 58.9, 45.4; IR (film) 1639 cm^{-1} ; MS HRFAB (pos) m/z calc'd for $[\text{MH}^+]$ $\text{C}_{13}\text{H}_{20}\text{NO}_3$ 238.1443, found 238.1447.

ppm

Current Data Parameters
NAME 2251
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010814
Time 9.59

INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TO 32768
SOLVENT CDCl3
NS 16
DS 2
SMH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 16
DM 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SF01 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 10.985 ppm
F1 4395.28 Hz
F2P -0.985 ppm
F2 -393.99 Hz
PPMCM 0.59846 ppm/cm
HZCM 239.46362 Hz/cm

ppm

172.579

77.904
77.584
77.26642.255
40.288
33.441
32.000
29.494
25.766
22.844
14.684
14.336
13.391Current Data Parameters
NAME 2251_c13
EXRNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20010814
Time 10.02
INSTRUM dx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 44
DS 2SMH 23146.148 Hz
FIDRES 0.353213 Hz

AQ 1.4156276 sec

RG 1625.5

DE 21.600 usec

TE 300.0 K

d1 0.05000000 sec

d11 0.03000000 sec

d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C

P1 12.30 usec

PL1 2.00 dB

SFO1 100.6232933 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16

NUC2 1H

PCPD2 100.00 usec

PL2 0.00 dB

PL12 18.00 dB

PL13 18.00 dB

SFO2 400.1316005 MHz

F2 - Processing parameters

SI 32768

SF 100.6127290 MHz

WDW EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40

ID NMR plot parameters

CX 20.00 cm

FLP 215.000 ppm

F1 21631.74 Hz

F2P -5.000 ppm

PPMCM 11.00000 ppm/cm

HZCM 1106.73999 Hz/cm

ppm

3.57178
3.56001
3.54845
3.54229
3.51327
3.50084
3.49140
3.37873
3.36544
3.35492
3.23205
2.22734
2.20861
2.18895
1.53405
1.51760
1.49894
1.47962
1.25393
1.23198
1.20536
1.20012
1.17326
0.79701
0.78026
0.76292

Current Data Parameters

NAME 2253
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20010814
Time 10.05
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TO 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 16
DM 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters

SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10 NMR plot parameters

CX 20.00 cm
CY 12.50 cm
F1P 10.985 ppm
F1 4395.28 Hz
F2P -0.985 ppm
F2 -393.99 Hz
PPMCM 0.59846 ppm/cm
HZCM 239.46362 Hz/cm

ppm

200
175
150
125
100
75
50
25
0

172.138

4

77.954
77.635
77.316
71.150
67.223
66.982
49.048
46.518
46.340
42.132
33.396
31.910
29.422
25.507
22.827
14.353

Current Data Parameters

NAME 2253_c13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20010814
Time 10.08
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 53
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 4096
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters

SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 215.000 ppm
F1 21631.74 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.73999 Hz/cm

8

Current Data Parameters
NAME Boc-aniline-diethan-ol
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030312
Time 9.22
INSTRUM dx400
PROBHD 5 mm Multinuc1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 124
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 512
RG 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127545 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
FLP 215.000 ppm
F1 21631.74 Hz
F2 -5.000 ppm
PC 11.00000 ppm/cm
PCCH 1106.74036 Hz/cm

ppm

7.39929
7.37871
7.29540
7.27514
7.11534

1.7214
1.9781
1.0103

3.45426
3.31067

3.7115

2.10425
2.03298
2.03124
1.49604
1.34723
1.33668
1.26186
1.24622
1.24433
1.22606
1.15070

8.4044
6.0000

Current Data Parameters
NAME boc_aniline_p_diamide
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030312

Time 9.17

INSTRUM drx400

PROBHD 5 mm Multinuc1

PULPROG zg30

TD 32768

SOLVENT CDCl3

NS 16

DS 2

SWH 4789.272 Hz

FIDRES 0.146157 Hz

AQ 3.4210291 sec

RG 64

DW 104.400 usec

DE 4.50 usec

TE 300.0 K

D1 1.00000000 sec

===== CHANNEL f1 =====

NUC1 ¹H

P1 7.70 usec

PL1 -6.00 dB

SFO1 400.1320007 MHz

F2 - Processing parameters

SI 16384

SF 400.1300000 MHz

WDW EM

SSB 0

LB 0.30 Hz

GB 0

PC 1.00

1D NMR plot parameters

CX 20.00 cm

CY 34.30 cm

FLP 11.000 ppm

F1 4401.43 Hz

F2P -1.000 DPM

F2 -400.13 Hz

PPMCM 0.60000 ppm/cm

HZCM 240.07800 Hz/cm

ppm

7.2829

3.8630
3.6852
3.5028
3.3514
3.1793
3.0842
3.0030
1.8776
1.8685
1.8477
1.8290
1.8131
1.8087
1.7873
1.7692
1.7512
1.7372
1.6927
1.6191
1.6023
1.5734
1.5679
1.5552
1.2417
0.0138

3.1068
3.0000
0.9797

6.3221
2.0800

Current Data Parameters
NAME df_cypent_amide
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030314
Time 14.56
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 64
DE 104.400 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====

NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters

SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
CY 12.50 cm
F1P 11.000 ppm
F1 4401.43 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.60000 ppm/cm
HZCM 240.07800 Hz/cm

ppm

14

Current Data Parameters
NAME df_cycpent_amide_c13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030314
Time 14.59
INSTRUM dx400
PROBHD 5 mm Multinuc1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 205
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 812.7
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SF01 100.6232933 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters

SI 32768
SF 100.6127538 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

ID NMR plot parameters

CX 20.00 cm
CY 13.74 cm
FLP 198.980 ppm
F1 20019.90 Hz
F2P -3.268 ppm
F2 -328.83 Hz
PPMCH 10.11240 ppm/cm
HZCM 1017.43640 Hz/cm

ppm

16

7.83452
7.82990
7.58619
7.58149
7.56537
7.56071
7.50288
7.48203

3.55744
3.36971

2.17594
1.76364

0.8831
0.8406
1.1240

2.9497
3.0699

Current Data Parameters
NAME df_ar_cl_top
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030313
Time 7.41
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 101.6
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CV 9.80 cm
F1P 11.000 ppm
F1 4401.43 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.60000 ppm/cm
HZCM 240.07800 Hz/cm

ppm

Current Data Parameters

NAME dl_ar_cl_top_c13

EXPNO 1

PROCNO 1

F2 - Acquisition Parameters

Date_ 20030313

Time 7.44

INSTRUM dtx400

PROBHD 5 mm Multinucl

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 85

DS 2

SWH 23148.148 Hz

FIDRES 0.353213 Hz

AQ 1.4156276 sec

RG 1625.5

DW 21.600 usec

DE 4.50 usec

TE 300.0 K

d11 0.03000000 sec

d12 0.0002000 sec

===== CHANNEL f1 =====

NUC1 13C

P1 12.30 usec

PL1 2.00 dB

SFO1 100.6232933 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 1H

PCPD2 100.00 usec

PL2 0.00 dB

PL12 18.00 dB

PL13 18.00 dB

SFO2 400.1316005 MHz

F2 - Processing parameters

SI 32768

SF 100.6127517 MHz

WDW EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40

1D NMR plot parameters

CX 20.00 cm

CY 12.50 cm

FLP 215.000 ppm

F1 21631.74 Hz

F2 -5.000 ppm

PPMCH 11.00000 ppm/cm

HZCM 1106.74036 Hz/cm

ppm

Integral

Current Data Parameters
NAME df_thio_am_top
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030313
Time 7.33

INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 90.5
DM 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16364
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 47.16 cm
F1P 10.985 ppm
F1 4395.28 Hz
F2P -0.985 ppm
F2 -393.99 Hz
PPMCH 0.59846 ppm/cm
HZCM 239.46362 Hz/cm

ppm

Current Data Parameters
NAME df_thio_am_top.c13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030313
Time 7.37
INSTRUM drx400
PROBHD 5 mm Multinuc1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 84
DS 2
SWH 23148.148 Hz
FIDRES 0.351213 Hz
AQ 1.4156276 sec
RG 2580.3
RW 21.600 usec
DE 4.50 usec
TE 300.0 K
d1 0.05000000 sec
d11 0.03000000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters

SI 32768
SF 100.6127538 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
CY 8.85 cm
FLP 215.000 ppm
F1 21631.74 Hz
F2 -5.000 ppm
F2 -503.06 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.74036 Hz/cm

ppm

23

Current Data Parameters
 NAME df_thio_amide
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030313
 Time 7.22
 INSTRUM drx400
 PROBD 5 mm Multinuc1
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 64
 DW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.70 usec
 PL1 -6.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

ID NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 11.000 ppm
 F1 4401.43 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCH 0.60000 ppm/cm
 HZCM 240.07800 Hz/cm

ppm

23

Current Data Parameters
 NAME df_thioamide_c13
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030313
 Time 7.29

INSTRUM dx400
 PROBD 5 mm Multinuc1
 PULPROG zgpg30

TD 65536
 SOLVENT CDCl3
 NS 154

DS 2
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz

AQ 1.4156276 sec
 RG 1448.2
 DW 21.600 usec

DE 4.50 usec
 TE 300.0 K
 D1 0.05000000 sec

d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 12.30 usec
 PL1 2.00 dB

SFO1 100.6232933 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H

PCPD2 100.00 usec
 PL2 0.00 dB

PL12 18.00 dB
 PL13 18.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127574 MHz

WDW EM
 SSB 0
 LB 1.00 Hz

GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm

F1P 215.000 ppm
 F1 21631.74 Hz

F2P -5.000 ppm
 F2 -503.06 Hz

PPMCM 11.00000 ppm/cm
 HZCM 1106.74036 Hz/cm

ppm 10 8 6 4 2 0

Integral

ppm

24

7.39758
7.39249
7.38798
7.38626
7.38284
7.37798
7.37660
7.37219
7.36773
7.36478
7.35806
7.35022
7.34668
7.34102
7.28224

3.73612
3.64493
3.49221
3.38970
3.35576
3.22616

5.000

4.119

10.230

Current Data Parameters
NAME df_benz_amide
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030319
Time 8.08

INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 40.3
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
FLP 11.000 ppm
F1 4401.43 Hz
F2 -1.000 ppm
PPMCM 0.60000 ppm/cm
HZCM 240.07800 Hz/cm

24

ppm

Current Data Parameters
NAME df_benz_c13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030319
Time 8.11

INSTRUM dxt400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 110
DS 2

SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 1149.4

DW 21.600 usec
DE 4.50 usec
TE 300.0 K

D1 0.05000000 sec
d11 0.03000000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

===== CHANNEL f2 =====

CEPPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters

SI 32768
SF 100.6127630 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

ID NMR plot parameters

CX 20.00 cm
CY 12.50 cm
PLP 215.000 ppm
F1 21631.75 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PCKM 11.00000 ppm/cm
HZCM 1106.74036 Hz/cm