Supplemental Information

EFFICIENT SYNTHESIS OF SOME NEW 2,3-DIMETHYL- QUINOXALINE-1-OXIDES USING BIS(ACETOXY)PHENYL- λ^3 - IODANE AS AN OXIDANT

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EXPERIMENTAL

Melting points were determined in open capillaries in electrical apparatus and are uncorrected. Infrared spectra were recorded on IR M-500 spectrophotometer (Buck Scientific Inc, Norwalk, CT) in KBr pellets ($\nu^{\max}$ in cm^{-1}). ^1H (300 MHz) and ^{13}C NMR (75 MHz) spectra were measured on a Bruker instrument (Billerica, MA) by using CDCl_3 as solvent and tetramethylsilane as an internal standard. Chemical shifts are expressed as δ in ppm. Coupling constants (J) are given in Hertz (Hz). Elemental analysis was performed at CDRI and the compounds gave satisfactory results (within ± 0.05 of the calculated values).

Commercially available variously substituted anilines (**1**) and biacetyl monoxime (**2**) were used without further purification.

General Procedure for the Synthesis of 2-(Arylimino)-3-(hydroxyimino)butane 3a-g

A solution of biacetyl monoxime **1** (10.1 g, 0.1 mol) and an appropriately substituted aniline **2a-g** (0.1 mol) in toluene (20ml) was refluxed for 4-5 h. The progress of the reaction was monitored by TLC. On completion of the reaction, excess solvent was distilled off in vacuum. Solid thus obtained was recrystallized from ethanol to give **3a-g**.

2-(Phenylimino)-3-(hydroxyimino)butane (3a)

Yield 84%; m.p. 135-138 °C; ¹H NMR, ¹³C NMR and elemental analysis. ^[1]

2-(4-Methylphenylimino)-3-(hydroxyimino)butane (3b)

Yield 88%; m.p. 142-145 °C; ¹H NMR, ¹³C NMR and elemental analysis. ^[1]

2-(4-Methoxyphenylimino)-3-(hydroxyimino)butane (3c)

Yield 82%; m.p. 143-144 °C; IR (cm⁻¹): 3117 (OH), 3086 (C-H), 1612, 1504; ¹H NMR (CDCl₃) δ: 2.05 (s, 3H, -CH₃), 2.21 (s, 3H, -CH₃), 3.83 (s, 3H, -OCH₃), 6.72 (d, 2H, *J*=8.7 Hz, Ph-3, 5-H), 6.94 (d, 2H, *J*=8.7 Hz, Ph-2, 6-H), 8.13 (s, 1H, OH); ¹³C NMR (CDCl₃) δ: 9.30, 15.54, 55.48, 114.25, 120.62, 143.68, 156.34, 159.04, 164.45; Anal. Calcd. for C₁₁H₁₄N₂O₂: C, 64.06; H, 6.84; N, 13.58. Found: C, 64.11; H, 6.86; N, 13.55.

2-(4-Chlorophenylimino)-3-(hydroxyimino)butane (3d)

Yield 74%; m.p. 126-130 °C; IR (cm⁻¹): 3132 (OH), 3001 (C-H), 1612, 1481; ¹H NMR (CDCl₃) δ: 2.02 (s, 3H, -CH₃), 2.19 (s, 3H, -CH₃), 6.69 (d, 2H, *J*=8.4 Hz, Ph-3, 5-H), 7.31 (d, 2H, *J*=8.7 Hz, Ph-2, 6-H), 8.15 (s, 1H, OH); ¹³C NMR (CDCl₃) δ: 9.10, 15.58, 116.14, 120.45, 128.86, 149.52, 157.28, 165.94; Anal. Calcd. for C₁₀H₁₁ClN₂O: C, 57.01; H, 5.26; N, 13.30. Found: C, 57.06; H, 5.24; N, 13.33.

2-(4-Bromophenylimino)-3-(hydroxyimino)butane (3e)

Yield 72%; m.p. 133-134 °C; IR (cm⁻¹): 3132 (OH), 3055 (C-H), 1612, 1481; ¹H NMR (CDCl₃) δ: 2.02 (s, 3H, -CH₃), 2.19 (s, 3H, -CH₃), 6.63 (d, 2H, *J*=8.4 Hz, Ph-3, 5-H), 7.47 (d, 2H, *J*=8.7 Hz, Ph-2, 6-H), 8.13 (s, 1H, OH); ¹³C NMR (CDCl₃) δ: 9.27, 15.64, 116.75, 120.86, 131.98, 149.55, 158.69, 165.09; Anal. Calcd. for C₁₀H₁₁BrN₂O: C, 47.08; H, 4.35; N, 10.98. Found: C, 47.03; H, 4.32; N, 10.92.

2-(4-Fluorophenylimino)-3-(hydroxyimino)butane (3f)

Yield 76%; m.p. 122-125 °C; IR (cm⁻¹): 3140 (OH), 3063 (C-H), 1612, 1497; ¹H NMR (CDCl₃) δ: 2.03 (s, 3H, -CH₃), 2.20 (s, 3H, -CH₃), 6.69-6.73 (m, 2H, Ph-3, 5-H), 7.06 (t, 2H, *J*=8.7 Hz, Ph-2, 6-H), 8.25 (s, 1H, OH); ¹³C NMR (CDCl₃) δ: 9.40, 15.72, 116.82, 122.55, 146.26, **153.47**, 158.49, 166.88; Anal. Calcd. for C₁₀H₁₁FN₂O: C, 61.85; H, 5.71; N, 14.42. Found: C, 61.89; H, 5.67; N, 14.47.

2-(3-Chloro-4-fluorophenylimino)-3-(hydroxyimino)butane (3g)

Yield 76%; m.p. 116-119 °C; IR (cm⁻¹): 3142 (OH), 3058 (C-H), 1615, 1492; ¹H NMR (CDCl₃) δ: 2.04 (s, 3H, -CH₃), 2.18 (s, 3H, -CH₃), 6.58-6.63 (m, 1H, Ph-6-H), 6.81 (dd, 1H, *J*=6.6 Hz, 2.4 Hz, Ph-5-H), 7.13 (t, *J*=8.7 Hz, Ph-2-H), 7.95 (s, 1H, OH); ¹³C NMR (CDCl₃) δ: 9.32, 15.81, 118.90, 120.76, 121.01, 121.22, 147.04, 156.90, 158.24, 166.93; Anal. Calcd. for C₁₀H₁₀ClFN₂O: C, 52.53; H, 4.41; N, 12.25. Found: C, 52.48 ; H, 4.46; N, 12.22.

General Procedure for the Synthesis of 2-(Arylimino)-3-(hydroxyimino)butane 4 a-g

To a solution of **3a-g** (0.001 mol) in dichloromethane (20 ml) was added bis(acetoxy)phenyl-λ³-iodane (0.354g, 0.0011 mol) in small portions over a period of five minutes at room temperature. The reaction mixture was allowed to stir for 5 h at room temperature. The progress of the reaction was monitored by TLC. When all the starting material had been consumed, excess solvent was distilled off *in vacuo* to give a gummy residue containing the

product and iodobenzene. The residue was triturated with petroleum ether to remove iodobenzene, a solid was obtained which was recrystallized from aqueous ethanol to give **4a-g**.

2,3-Dimethylquinoxaline-1-oxide (4a)

Yield 62%; m.p. 84-88 °C; IR (cm⁻¹): 3043 (C-H), 1603, 1487; ¹H NMR (CDCl₃) δ: 2.71 (s, 3H, 3-CH₃), 2.77 (s, 3H, 2-CH₃), 7.66-7.78 (m, 2H, 6-H, 7-H), 8.00-8.03 (m, 1H, 5-H), 8.55-8.58 (m, 1H, 8-H); ¹³C NMR (CDCl₃) δ: 13.90, 23.85, 118.81, 129.14, 130.54, 139.58, 142.77, 154.99; Anal. Calcd. for C₁₀H₁₀N₂O: C, 68.95; H, 5.79; N, 16.08. Found: C, 68.92; H, 5.82; N, 16.05.

2,3,7-Trimethylquinoxaline-1-oxide (4b)

Yield 75 %; m.p. 78-82 °C; IR (cm⁻¹): 3058 (C-H), 1612, 1505; ¹H NMR (CDCl₃) δ: 2.60 (s, 3H, 7-CH₃), 2.70 (s, 3H, 3-CH₃), 2.75 (s, 3H, 2-CH₃), 7.57 (dd, 1H, *J*_o=8.7 Hz, *J*_m=1.5 Hz, 6-H), 7.90 (d, 1H, *J*_o=8.7 Hz, 5-H), 8.36 (d, 1H, 8-H); ¹³C NMR (CDCl₃) δ: 13.90, 21.87, 23.68, 117.76, 128.75, 135.20, 139.50, 140.17, 141.23, 153.78; Anal. Calcd. for C₁₁H₁₂N₂O: C, 70.19; H, 6.43; N, 14.88. Found: C, 70.15; H, 6.48; N, 14.82.

7-Methoxy-2,3-dimethylquinoxaline-1-oxide (4c)

Yield 72 %; m.p. 128-132 °C; IR (cm⁻¹): 3048 (C-H), 1612, 1498; ¹H NMR (CDCl₃) δ: 2.71 (s, 3H, 3-CH₃), 2.74 (s, 3H, 2-CH₃), 4.00 (s, 3H, 7-OCH₃), 7.38 (dd, 1H, *J*_o=9.0 Hz, *J*_m=2.4 Hz, 6-H), 7.89 (s, 1H, 5-H), 7.92 (s, 1H, 8-H); ¹³C NMR (CDCl₃) δ: 13.99, 23.43, 56.10, 97.27, 123.24, 130.37, 136.31, 139.55, 151.72, 160.75; Anal. Calcd. for C₁₁H₁₂N₂O₂: C, 64.69; H, 5.92; N, 13.72. Found: C, 64.74; H, 5.94; N, 13.70.

7-Chloro-2,3-dimethylquinoxaline-1-oxide (4d)

Yield 58 %; m.p. 112-115 °C; IR (cm⁻¹): 3052 (C-H), 1608, 1502; ¹H NMR (CDCl₃) δ: 2.70 (s, 3H, 3-CH₃), 2.76 (s, 3H, 2-CH₃), 7.69 (dd, 1H, *J*_o=9.0 Hz, *J*_m=2.1 Hz, 6-H), 7.96 (d, 1H, *J*_o=9.0 Hz, 5-H), 8.57(d, 1H, *J*_m=2.1Hz, 8-H); ¹³C NMR (CDCl₃) δ: 13.95, 23.81, 118.40, 130.56, 131.60, 135.51,135.70, 140.28, 141.29, 155.25; Anal. Calcd. for C₁₀H₉ClN₂O: C, 57.57; H, 4.35; N, 16.99. Found: C, 57.52; H, 4.38; N, 16.97.

7-Bromo-2,3-dimethylquinoxaline-1-oxide (4e)

Yield 67%; m.p. 130-134°C; IR (cm⁻¹): 3078 (C-H), 1611, 1508; ¹H NMR (CDCl₃) δ: 2.70 (s, 3H, 3-CH₃), 2.76 (s, 3H, 2-CH₃), 7.84 (dd, 1H, *J*_o=9.0 Hz, *J*_m=2.1 Hz, 6-H), 7.90 (d, 1H, *J*_o=8.7 Hz, 5-H), 8.75 (d, 1H, *J*_m=1.8 Hz, 8-H); ¹³C NMR (CDCl₃) δ: 13.93, 23.86, 121.63, 123.35, 130.62, 134.20, 135.96, 140.19, 141.57, 155.40; Anal. Calcd. for C₁₀H₉BrN₂O: C, 47.46; H, 3.58; N, 11.07. Found: C, 47.49; H, 3.56; N, 11.05.

7-Fluoro-2,3-dimethylquinoxaline-1-oxide (4f)

Yield 61%; m.p. 75-78 °C; IR (cm⁻¹): 3086 (C-H), 1620, 1497; ¹H NMR (CDCl₃) δ: 2.71 (s, 3H, 3-CH₃), 2.77 (s, 3H, 2-CH₃), 7.49-7.55 (m, 1H, 6-H), 8.03 (dd, 1H, *J*_{H-F}=9.0 Hz, *J*_o=5.1 Hz, 5-H), 8.23 (dd, 1H, *J*_{H-F}=9.0 Hz, *J*_m=2.7 Hz, 8H); ¹³C NMR (CDCl₃) δ: 13.94, 23.67, 115.72, 121.65, 136.08, 139.85, 140.16, 154.26, 160.81; Anal. Calcd. for C₁₀H₉FN₂O: C, 62.49; H, 4.72; N, 14.58. Found: C, 62.47; H, 4.73; N, 14.57.

6-Chloro-7-fluoro-2,3-dimethylquinoxaline-1-oxide (4g)

Yield 55%; m.p. 66-68 °C; IR (cm⁻¹): 3063 (C-H), 1605, 1474; ¹H NMR (CDCl₃) δ: 2.69 (s, 3H, 3-CH₃), 2.75 (s, 3H, 2-CH₃), 8.10 (d, 1H, *J*=6.9 Hz, 5-H), 8.30 (d, 1H, *J*=9.0 Hz, 8H); ¹³C NMR (CDCl₃) δ: 13.90, 23.76, 105.13, 105.49, 130.80, 139.80, 140.31, 155.65, 159.52; Anal. Calcd. for C₁₀H₈ClFN₂O: C, 53.00; H, 3.56; N, 12.36. Found: C, 53.04; H, 3.55; N, 12.34.

3c

Current Data Parameters
 NAME July 11 2011
 EXPNO 8
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20110712
 Time 12.30
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 8
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.094190 Hz
 AQ 5.3084660 sec
 RG 322.5
 DW 81.000 usec
 DE 6.00 usec
 TE 1273.1 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 7.25 usec
 PL1 0.00 dB
 SFO1 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

3c: ¹³C

Current Data Parameters
NAME July 26 2012
EXPNO 19
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120730
Time_ 15.56
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 4096
DW 27.800 usec
DE 6.00 usec
TE 1273.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 9.10 usec
PL1 1.00 dB
SFO1 75.4752953 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 22.79 dB
PL13 22.79 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

3d

Current Data Parameters
 NAME July 14 2011
 EXPNO 6
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20110715
 Time 12.18
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.094190 Hz
 AQ 5.3084660 sec
 RG 256
 DW 81.000 usec
 DE 6.00 usec
 TE 1273.1 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.25 usec
 PL1 0.00 dB
 SFO1 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

3e

Current Data Parameters
 NAME June 22 2011
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters

Date 20110627
 Time 13.17
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 FWH 6172.839 Hz
 FIDRES 0.094190 Hz
 AQ 5.3084660 sec
 RG 322.5
 DW 81.000 usec
 DE 6.00 usec
 TE 1273.1 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 7.25 usec
 PL1 0.00 dB
 SFO1 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Current Data Parameters
NAME July 30 2012
EXPNO 22
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120806
Time 11.37
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 4096
DW 27.800 usec
DE 6.00 usec
TE 1273.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 ¹³C
P1 9.10 usec
PL1 1.00 dB
SFO1 75.4752953 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 ¹H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 22.79 dB
PL13 22.79 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

3f

Current Data Parameters
 NAME Mar 20 2012
 EXPNO 5
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120321
 Time 9.49
 INSTRUM spect
 PROBUID 5 mm Multinucl
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 FWH 6172.839 Hz
 FIDRES 0.094190 Hz
 AQ 5.3084660 sec
 RG 362
 DW 81.000 usec
 DE 6.00 usec
 TE 0.0 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

----- CHANNEL f1 -----
 NUC1 1H
 P1 7.25 usec
 PL1 0.00 dB
 SFO1 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

3f: ^{13}C

3g

Current Data Parameters
 NAME Mar 30 2012
 EXPNO 17
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120402
 Time 14.40
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.094190 Hz
 AQ 5.3084660 sec
 RG 362
 DW 81.000 usec
 DE 6.00 usec
 TE 0.0 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 7.25 usec
 PL1 0.00 dB
 SFO1 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

3g: ^{13}C

4a

Current Data Parameters
 NAME Jan 9 2012
 EXPNO 9
 PROCNO 1

F2 - Acquisition Parameters
 Date 20120109
 Time 14.42
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.094190 Hz
 AQ 5.3084660 sec
 RG 228.1
 DW 81.000 used
 DE 6.00 used
 TE 0.0 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRE 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.25 used
 PL1 0.00 dB
 SFO1 500.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

4a: ¹³C

Current Data Parameters
NAME Jan 10 2012
EXPNO 32
PROCNO 1

F2 - Acquisition Parameters
Date 20120112
Time 11.19
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1523
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 4096
DW 27.800 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRR 0.01500000 sec

***** CHANNEL f1 *****
NUC1 ¹³C
P1 9.10 usec
PL1 1.00 dB
SFO1 76.4752953 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 ¹H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 22.79 dB
PL13 22.79 dB
SFO2 300.1350005 MHz

F2 - Processing parameters
SI 32768
SF 400.1490 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 0.40

4b

Current Data Parameters
 NAME May 24 2011
 EXPNO 24
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20110525
 Time 12.36
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.094190 Hz
 AQ 5.3084660 sec
 RG 406.4
 DW 81.000 usec
 DE 6.00 usec
 TE 0.0 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 7.25 usec
 PL1 0.00 dB
 SFO1 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

4b: ^{13}C

Current Data Parameters
NAME April 2 2012
EXPNO 22
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120404
Time 10.24
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 4096
DW 27.800 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 ^{13}C
P1 9.10 usec
PL1 1.00 dB
SFO1 75.4752953 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 22.79 dB
PL13 22.79 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

4d

Current Data Parameters
 NAME Jan 9 2012
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date 20120109
 Time 14.46
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 BWH 6172.839 Hz
 FIDRES 0.094190 Hz
 AQ 5.3084660 sec
 RG 228.1
 DW 81.000 usec
 DE 6.00 usec
 TE 0.0 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.25 usec
 PL1 0.00 dB
 SFO1 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300000 MHz
 WDW EM
 SSR 0
 LB 0.30 Hz
 GB 0
 PC 1.00

4d: ¹³C

Current Data Parameters
 NAME Mar 1 2012
 EXPNO 33
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120314
 Time 11.49
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 4096
 DW 27.800 usec
 DE 6.00 usec
 TE 0.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 ¹³C
 P1 9.10 usec
 PL1 1.00 dB
 SFO1 75.4752953 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 ¹H
 PCPD2 100.00 usec
 PL2 0.00 dB
 PL12 22.79 dB
 PL13 22.79 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677490 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

4e

4e: ^{13}C

Current Data Parameters
NAME Mar 30 2012
EXPNO 24
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120403
Time_ 14.18
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 4096
DW 27.800 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWKK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 ^{13}C
P1 9.10 usec
PL1 1.00 dB
SFO1 75.4752953 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 22.79 dB
PL13 22.79 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

4f

Current Data Parameters
 NAME Mar 26 2012
 EXPNO 7
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120327
 Time 15.21
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.094190 Hz
 AQ 5.3084660 sec
 RG 362
 DW 81.000 usec
 DE 6.000 usec
 TE 0.0 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 7.25 usec
 PL1 0.00 dB
 SFO1 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300000 MHz
 HDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

4f: ¹³C

Current Data Parameters
NAME Mar 28 2012
EXPNO 22
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120330
Time_ 14.14
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 4096
DW 27.800 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 9.10 usec
PL1 1.00 dB
SFO1 75.4752953 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 22.79 dB
PL13 22.79 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

4g

4g: ^{13}C

Current Data Parameters
NAME June 18 2012
EXPNO 27
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120621
Time 12.54
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 4096
DW 27.800 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 ^{13}C
P1 9.10 usec
PL1 1.00 dB
SFO1 75.4752953 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 22.79 dB
PL13 22.79 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

References

1. Zangrando, E.; Trani, M.; Stabon, E.; Carfagna, C.; Milani, B.; Mestroni, G. Synthesis and molecular structures of nickel (II) and cobalt (II) complexes with 2-(arylimino)-3-(hydroxyimino)butane. *Eur. J. Inorg. Chem.* **2003**, *14*, 2683-2692.