Kowalski Ester Homologation. Application to the Synthesis of β -Amino Esters.

Diane Gray, Carmen Concellón and Timothy Gallagher

School of Chemistry, University of Bristol, Bristol BS8 1TS, UK.

Supporting Information

Purity Documentation (^1H NMR spectra)

dg8459H.2
DG3/065-com D. Gray

File Name = dg8459H.2
Author = cg/dg8459
Sample ID = DG3/065-com D. Gray
Content = 3-DEC-2003 13:51:47
Creation Date = 3-DEC-2003 13:49:56
Revision Date = 3-DEC-2003 13:49:56
Spec Site = GX400
Spec Type = DELTA_NMR
Data Format = 1D COMPLEX
Dimensions = 1H
Dim Title = 1H
Dim Size = 16384
Dim Units = [ppm]
X domain = 1H
X offset = 51[ppm]
X freq = 399.7841973 [MHz]
X sweep = 81[Hz]
X pulse = 7.85[us]
X resolution = 2.048[Hz]
X relaxation = 4.8831105[Hz]
Relaxation delay = 4[us]
Scans = 16
Recvr gain = 22
Solvent = CHLOROFORM-D
Spin get = 12[Hz]
Temp get = TEMP OFF
Lock get = 22.4[dc]
Lock state = LOCK ON
Lock gain = 26
Lock level = 180
Lock strength = 859
Changer_sample = 56
Irr_width = 34[us]
Experiment = single_pulse.exp

dg2828H.2
DG-N-Boc pp D.Gray

File Name = dg2828H.2
Author = tg/dg2828
Sample ID = DG-N-Boc pp D.Gray
Content = 22-MAR-2004 17:45:44
Creation Date = 22-MAR-2004 17:12:16
Revision Date = 22-MAR-2004 17:12:16
Spec Site = Eclipset+ 400H
Spec Type = DELTA_NMR
Data Format = 1D COMPLEX
Dimensions = X
Dim Title = 1H
Dim Size = 32768
Dim Units = [ppm]
Autolock Level = 180
Autoshim mode = 21 Z2
Changer sample = 4
Experiment = single_pulse-exp
Lock gain = 19
Lock level = 180
Lock state = LOCK ON
Lock strength = 529
Lock status = IDLE
Recvr gain = 20
Relaxation_delay = 2[s]
Scans = 16
Solvent = CHLOROFORM-D
Spin get = 15[Hz]
Spin state = SPIN ON
Temp get = 22.5[°C]
X acq duration = 4.092723[s]
X domain = 1H
X freq = 400.1787931[MHz]
X offset = 51ppm
X points = 32768
X pulse = 2.333333[us]
X resolution = 0.2443609[Hz]
X_sweep = 8.00640512[kHz]

JEOL

dg0286H.2
DG2058(2B-3A) D. Gray

X : parts per Million : 1H

JEOL

```

File Name      = dg0286H.2
Author         = 
Sample ID      = cg/4g0286
Content        = DG2/058 (2B-3A) D. Gray
Creation Date  = 18-JUN-2003 16:21:44
Revision Date  = 18-JUN-2003 16:23:12

Spec Site      = GX400
Spec Type      = DELTA NMR

Data Format     = ID COMPLEX
Dimensions     = X
Dim Title      = 1H
Dim Size       = 16384
Dim Units      = [ppm]
X domain       = 1H
X offset       = 5 [ppm]
X freq         = 399.7841973 [MHz]
X sweep        = 8 [Hz]
X pulse        = 7.75 [us]
X resolution   = 2.048 [s]
X relaxation   = 0.48831105 [Hz]
Relaxation delay = 4 [s]
Scans          = 16
Recvr gain     = 13
Solvent        = CHLOROFORM-D
Spin get       = 13 [Hz]
Temp state     = TEMP OFF
Temp get       = 22.4 [C]
Lock state     = LOCK ON
Lock gain      = 26
Lock level     = 180
Lock strength  = 657
Charger sample = 58
Irr pwldth     = 40 [us]
Experiment     = single_pulse.exp
  
```

dg3599H.2
DG2/056 D. Gray

JEOL

File Name = dg3599H.2
Author = tg/dg3599
Sample ID = DG2/056 D. Gray
Content = 31-AUG-2003 19:46:27
Creation Date = 31-AUG-2003 19:01:46
Revision Date = 31-AUG-2003 19:01:46

Spec Site = GX400
Spec Type = DELTA_NMR

Data Format = ID COMPLEX
Dimensions = X
Dim Title = 1H
Dim Size = 16384
Dim Units = [ppm]
X domain = 1H
X offset = 5 [ppm]
X freq = 399.7841973 [MHz]
X sweep = 5.99880024 [kHz]
X pulse = 7.85 [us]
X resolution = 2.7312128 [s]
X resolution = 0.3661006 [Hz]
Relaxation delay = 4 [s]
Scans = 16
Recvr gain = 12
Solvent = CHLOROFORM-D
Spin get = 12 [Hz]
Temp state = TEMP OFF
Temp get = 23 [dc]
Lock state = LOCK ON
Lock gain = 26
Lock level = 180
Lock strength = 582
Changer sample = 44
Irr pwidth = 34 [us]
Experiment = single_pulse.exp

dg3001H.2
DG2081 D. Gray

File Name
dg3001H.2

Author
Eg/dg3001

Sample ID
DG2081 D. Gray

Content
12-AUG-2003 10:38:52

Creation Date
12-AUG-2003 10:06:04

Revision Date

Spec file
GX400

Spec type
DEL

Data Format
X

Dimensions
1H

Dim title
16384

Dim size
16384

Dim units
1ppm

X domain
1H

X offset
511ppm

X freq
399.7841973

X sweep
81kHz

X pulse
7.35[us]

X pulse duration
2.048[us]

X resolution
0.48631105 [Hz]

Relaxation delay
4[s]

Scans
16

Solvent
1H

Solvent gain
12

Spin proc
1H [Hz]

Temp set
22.8 [C]

Temp get
22.8 [C]

Lock state
LOCK ON

Lock gain
26

Lock level
180

Lock strength
612

Charger sample
30

1H width
34 [us]

Experiment
single_pulse.exp

JEOL

1801e20

cc3924H.2
cc-N-BnProline

X : parts per Million : 1H

JEOL

File Name = cc3924H.2
 Author = tg/cc3924
 Sample ID = cc-N-BnProline
 Content = 5-SEP-2003 14:21:49
 Creation Date = 5-SEP-2003 13:35:49
 Revision Date =
 Spec Site = GX400
 Spec Type = DELTA_NMR
 Data Format = ID COMPLEX
 Dimensions = X
 Dim Title = 1H
 Dim Size = 16384
 Dim Units = [ppm]
 X domain = 1H
 X offset = 5[ppm]
 X freq = 399.7841973[MHz]
 X sweep = 8[Hz]
 X pulse = 7.85[us]
 X pulse duration = 2.048[us]
 X resolution = 0.4883105[Hz]
 Relaxation delay = 4[s]
 Scans = 16
 Recvr gain = 11
 Solvent = CHLOROFORM-D
 Spin get = 12[Hz]
 Temp state = TEMP OFF
 Temp get = 22.8[degC]
 Lock state = LOCK ON
 Lock gain = 26
 Lock level = 180
 Lock strength = 442
 Changer sample = 2
 lrf_pidich = 34[us]
 Experiment = single_pulse.exp

dg7961H.2
DG3/059(2F-3E) D. Gray

JEOL

File Name = dg7961H.2
 Author = tg/dg7961
 Sample ID = DG3/059(2F-3E) D. Gray
 Content = 26-NOV-2003 02:38:57
 Creation Date = 26-NOV-2003 01:18:13
 Revision Date = 26-NOV-2003 01:18:13
 Spec Site = GX400
 Spec Type = DELTA_NMR
 Data Format = 1D COMPLEX
 Dimensions = X
 Dim Title = 1H
 Dim Size = 16384
 Dim Units = [ppm]
 X domain = 1H
 X offset = 5[ppm]
 X freq = 399.7841973[MHz]
 X sweep = 5.99880024[kHz]
 X pulse = 7.83[us]
 X resolution = 2.7312128[Hz]
 X resolution = 0.3616006[Hz]
 Relaxation delay = 4[s]
 Scans = 16
 Recvr gain = 11
 Solvent = CHLOROFORM-D
 Spin get = 13[Hz]
 Temp state = TEMP OFF
 Temp get = 22.6[degC]
 Lock state = LOCK ON
 Lock gain = 26
 Lock level = 180
 Lock strength = 605
 Changer sample = 47
 Irr pwldth = 34[us]
 Experiment = single_pulse.exp

```

File Name      = cc5110H.2
Author         =
Sample ID      = tg/cc5110
Content        = C.conce11
Creation Date   = 6-0CC-2003 13:05:52
Revision Date  = 6-0CC-2003 12:03:10

Spec Site
Spec Type      = GA100
               = DELTA_NMR

Data Format     = 1D COMPLEX
Dimensions     =
Dim 1 title    = 1H
Dim 2 title    = 16384
Dim 3 title    = [ppm]
Dim 4 title    = 1H
Dim 5 title    = 5 [ppm]
X offset       = 399.7841973 [kHz]
X freq         = 81 [kHz]
X sweep        = 7.85 [us]
X pulse       = 2.048 [us]
X acq duration = 0.48831105 [Hz]
X resolution   = 4 [s]
Relaxation_delay = 16
Scans          = 13
Recur gain     =
Solvent        = CHLOROFORM-D
Spin get       = 11 [Hz]
Temp get       = TEMP OFF
Temp set       = 22.4 [dC]
lock state     = LOCK ON
lock gain      = 26
lock state     = 180
lock level     = 403
Changer sample = 5
T1r1_pvt[1]    = 34 [us]
Experiment     = single pulse.exp

```

JEOL

File Name = cc5231H.2
 Author = tg/cc5231
 Sample ID = cc-homolaf.26-45 C.
 Content = 8-OCT-2003 15:17:09
 Creation Date = 8-OCT-2003 14:12:54
 Revision Date =
 Spec Site = GX400
 Spec Type = DELTA_NMR
 Data Format = ID COMPLEX
 Dimensions = X
 Dim Title = 1H
 Dim Size = 16384
 Dim Units = [ppm]
 X domain = 1H
 X offset = 5 [ppm]
 X freq = 399.784193 [MHz]
 X sweep = 8 [kHz]
 X pulse = 7.85 [us]
 X acq duration = 2.048 [s]
 X resolution = 0.4883105 [Hz]
 Relaxation delay = 4 [s]
 Scans = 16
 Recvr gain = 10
 Solvent = CHLOROFORM-D
 Spin get = 13 [Hz]
 Temp state = TEMP OFF
 Temp get = 22 [C]
 Lock state = LOCK ON
 Lock gain = 26
 Lock level = 180
 Lock strength = 332
 Changer sample = 58
 Irr pwidth = 34 [us]
 Experiment = single_pulse.exp

cc5012H.2
cc-homolpheracemic C. Concelton

File Name = cc5012H.2
 Author = tg/cc5012
 Sample ID = cc-homolpheracemic C.C
 Content = 2-OCT-2003 15:05:48
 Creation Date = 2-OCT-2003 14:06:54
 Revision Date =
 Spec Site = GX400
 Spec Type = DELTA_NMR
 Data Format = ID COMPLEX
 Dimensions = X
 Dim 1 Size = 1H
 Dim 2 Size = 16384
 Dim 3 Size = [ppm]
 X domain = 1H
 X offset = 51ppm
 X freq = 399.7841973[MHz]
 X sweep = 8[KHz]
 X pulse = 7.85[us]
 X acq duration = 2.048[us]
 X resolution = 0.4883105[Hz]
 Relaxation delay = 4[us]
 Scans = 16
 Recvr gain = 10
 Solvent = CHLOROFORM-D
 Spin get = 11[Hz]
 Temp state = TEMP OFF
 Temp get = 22.5[dc]
 Lock state = LOCK ON
 Lock gain = 26
 Lock level = 180
 Lock strength = 348
 Changer sample = 59
 Irr pwidth = 34[us]
 Experiment = single_pulse.exp

cc3668H.2
cc-dibnValOMe F2.21-23

X : parts per Million : 1H

JEOL

File Name = cc3668H.2
 Author = tg/cc3668
 Sample ID = cc-dibnValOMe F2.21-23
 Content = 2-SEP-2003 02:28:09
 Creation Date = 2-SEP-2003 01:42:34
 Revision Date =
 Spec Site = GX400
 Spec Type = DELTA_NMR
 Data Format = ID COMPLEX
 Dimensions = 1H
 Dim Title = 1H
 Dim Size = 16384
 Dim Units = [ppm]
 X Domain = 1H
 X Offset = 5 [ppm]
 X Freq = 399.7841973 [MHz]
 X Sweep = 8 [Hz]
 X Pulse = 7.85 [us]
 X Acq Duration = 2.048 [s]
 X Resolution = 0.4881105 [Hz]
 Relaxation Delay = 4 [s]
 Scans = 16
 Recvr Gain = 9
 Solvent = CHLOROFORM-D
 Spin Get = 12 [Hz]
 Temp Get = TEMP OFF
 Lock Get = 22.6 [dC]
 Lock State = LOCK ON
 Lock Gain = 26
 Lock Level = 180
 Lock Strength = 327
 Change Sample = 26 [us]
 Iir Width = 34 [us]
 Experiment = single_pulse.exp

cc4978H.2
cchomolval2F.35-40

JEOL

File Name = cc4978H.2
Author =
Sample ID = tg/cc4978
Content = cchomolval2F.35-40
Creation Date = 1-OCT-2003 12:00:38
Revision Date = 1-OCT-2003 11:02:20

Spec Name = G3400
Spec Type = DELTA_NMR

Data Format = 1D
Dimensions = 1H
Dim 1 Size = 16384
Dim 2 Size = 1H
Dim 3 Size = 1H
X_domain = 51ppm
X_offset = 399.7841973 [MHz]
X_freq = 8 [kHz]
X_sweep = 7.85 [us]
X_pulse = 2.048 [us]
X_acq_duration = 0.48831105 [Hz]
X_resolution = 41s
Relaxation_delay = 16
Scans = 15
Recvr_gain = 17 [Hz]
Solvant = CHLOROFORM-D
Spin_get = TEMP OFF
Temp_get = 22.3 [C]
Lock_get = LOCK ON
Lock_gain = 26
Lock_level = 180
Lock_strength = 470
Changer_sample = 31
Irr_width = 34 [us]
Experiment = single_pulse.exp

dg8233H.2
DG3062 D. Gray

X : parts per Million : 1H

File Name dg8233H.2
Anchor cg/dg8233
Sample ID DG3/062 D. Gray
Content 29-NOV-2003 02:03:07
Creation Date 29-NOV-2003 00:40:55
Revision Date

Spec Site
Spec Type DEPTA_NMR

Data Format
Dimensions 1D
D1m Title 1H
D1m Size 16384
D1m Units [ppm]
X domain 1H
X offset 5 [ppm]
X freq 399.7841973 [MHz]
X sweep 5.99880024 [Hz]
X pulse 7.85 [us]
X acq duration 2.7312128 [s]
X resolution 0.36616006 [Hz]
Relaxation delay 4 [s]
Scans 16
Acq gain 8
SOLVENT CHLOROFORM-D
SOLN_GCT 11 [Hz]
TEMP_OVF 23.2 [C]
LOCK ON 26
Lock gain 180
Lock strength 352
Changer sample 21
Irr width 34 [us]
Experiment single_pulse.exp

JEOL

dg7253H.2
DG3/053(2E-3F) D. Gray

JEOL

File Name	dg7253H.2
Author	
Sample ID	cg/dg7253
Content	DG3/053(2E-3F) D. Gray
Creation Date	14-NOV-2003 12:49:32
Revision Date	14-NOV-2003 11:34:52
Spec Site	GX400
Spec Type	DELTA_NMR
Data Format	1D COMPLEX
Dimensions	1H
Dim Title	1H
Dim Size	16384
Dim Units	[ppm]
X domain	1H
X offset	51ppm
X freq	399.7641973 [MHz]
X sweep	81kHz
X pulses	7.85[us]
X acq_duration	2.048[us]
X resolution	0.4881105[Hz]
Relaxation_delay	4[us]
Scan	16
Receiver_gain	15
Solvent	CHLOROFORM-D
Spin_get	10[Hz]
Temp_get	TEMP OFF
Temp_set	22.9[°C]
Lock_state	LOCK ON
Lock_gain	26
Lock_level	180
Lock_strength	774
Change_sample	22
irr_width	1[us]
Experiment	single_pulse.exp