SUPPLEMENTARY INFORMATION

Conversion of Cycloalk-2-enones into 2-Methylocycloalkane-1,3-diones—Assessment of Various Tamao-Fleming Procedures and Mechanistic Insight into the Use of the Me₃SiMe₂Si Unit

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NMR spectra of 11
NMR spectra of 12
NMR spectra of 13
NMR spectra of 16a
NMR spectra of 16b
NMR spectra of 16c
NMR spectra of 17a
NMR spectra of 17b
NMR spectra of 17c
NMR spectra of trans-18a

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Guojun Compd 11 Carbon APT Env GY0155
128.690 MHz C13[H1] APT_ad in ccdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, colidual probe
C & CH2 same, CH & CH3 opposite side of solvent signal

File: /mnt/6D00/Florida/chem/data/GY0155/Guojun014.06/GY0155-Frac-05-07-APT
Guojun Compd 11 Hydrogen Env GY0155
499.806 MHz H1 PResAT in cdc3 (ref. to CDC3 @ 7.26 ppm), temp 27.7 C -> actual temp = 27.0 C, colddual probe
Guojun Crmpd 12 Hydrogen Env GY01-15
498.118 MHz H1 1D in cdc13 (ref. to CDC13 @ 7.26 ppm), temp 26.4 C -> actual temp = 27.0 C, autoXdb probe

- Chemical structure with peaks indicated on the spectrum.
Guojun Compd 13 Carbon APT Env GY91-111
128.690 MHz C13[H1] APT_ad in cdc13 (ref. to CDC13 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, colidual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
Guanjun Crmpd 13 Hydrogen Env GY01-111
499.806 MHz H1 PRESAT in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.7 C => actual temp = 27.0 C, colidual probe
Guojun Compd 16 Carbon APT Env GY03-35
128.960 MHz C13[H1] APT_seq in cdcl3 (ref. to CDC13 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, colidual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
Gujun Crmpd 26 Carbon APT Env GY03-33
128.690 MHz C13[H1] APT_sd in cdcl3 (ref. to CDC13 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, colloidual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
Gaojun Compd 36 Hydrogen Env GY03-33
499.806 MHz H1 PRESAT in cdcl3 (ref. to CDC13 δ 7.26 ppm), temp 27.7 °C → actual temp = 27.0 °C, colddual probe

File: /mnt/d0/Data/12gionsml/102ml/D1/CLIVE/Gaojun2015.05/GY03-33-DP
Guanjun Compd 33 Carbon APT Env GY02-51
128.690 MHz C13[H1] APT_ad in cdc13 (ref. to CDC13 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, colloidal probe
C & CH2 same, CH & CH3 opposite side of solvent signal
Gaojun Compd 33 Hydrogen Env GY02-51
498.118 MHz H1 1D in cdcl3 (ref. to CDC13 @ 7.26 ppm), temp 26.4 °C -> actual temp = 27.0 °C, autoxdrb probe

File: /mnt/b000/hom/v12/homework/nmrdata/CLIVE/Gaojun/2015.02/GY02-109-DP
Guojun Crmpd 17 Carbon APT Env GY02-23
128.690 MHz C13[H1] APT_9d in ccd3 (ref. to CDC13 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.6 C, colldual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
Guojun Crmpd 17 Hydrogen Env GY02-23
499.806 MHz H1 PRESAT in cdc13 (ref. to CDCl3 @ 7.26 ppm), temp 27.7 C -> actual temp = 27.0 C, colloidal probe
Guojun Crmpd 27 Carbon APT Env GY03-41
128.690 MHz C13[H1] APT sd in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.6 C, colloidal probe
C & CH2 same, CH & CH3 opposite side of solvent signal

File: hvntd000/home12/gilmore/nmrdata/CL/FVE/Guojun2015.06/GY03-41-DC-APT
Gaojun Compd 27 Hydrogen Env GY03-41
498.118 MHz H1 1D in ccd3 (ref. to CDCl3 @ 7.26 ppm), temp 26.4 °C -> actual temp = 27.0 °C, autoexdb probe
Guanid Compd 34 Carbon APT EnV GY02-53
128.690 MHz C13[H1] APT_sd in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, colloidal probe
C & CH2 same, CH & CH3 opposite side of solvent signal
Guojun Compd 9' Carbon APT Env GY02-07
128.690 MHz C13[1] APT_sd in cdc3 (ref. to CDC13 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, coliddual probe
C & CH2 same, CH & CH3 opposite side of solvent signal

Guojun Compd 9' Hydrogen Env GY03-07
499.06 MHz H1 PRESAT in cdcl3 (ref. to CDC13 0 ppm), temp 27.7 C -> actual temp = 27.0 C, colidual probe
Guan Jun Crmpd 28 Carbon APT Env GV03-43
128.690 MHz C13[H1] APT ud in ccd/t3 (ref. to CDCl3 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, colidual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
Guojun Crmpd 38 Hydrogen Env GY03-43
400.118 MHz H1 1D in cdc13 (ref. to CDC13 @ 7.26 ppm), temp 26.4 C -> actual temp = 27.0 C, autoexdb probe
Guojun Crmpd 35 Carbon APT Env GY02-67
128.690 MHz C13[H1] APT_sd in cddt3 (ref. to CDC13 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, coliddual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
Gaojun Compd 35 Hydrogen Env GY02-67
499.006 MHz H1 PRESAT in cdc3 (ref. to CDCl3 @ 7.26 ppm), temp 27.7 °C -> actual temp = 27.0 °C, cold dual probe

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Guojun Compd 33 trans (major isomer) Carbon APT Env GV02-193
128.690 MHz C13[1H] APT ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.6 C, colidual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
Guojun Compd 33 trans (major isomer) Hydrogen Env GY03-193
499.806 MHz H1 PRESAT in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.7°C – actual temp = 27.0°C, coiled probe

File: /mnt/d/00/00/12/gemnswin/data/CLVPS/Guojun/Paper-1_Compounds/Guojun_Compd_34_trans_major_isomer_Hydrogen_Env_GY03-193
Compound 29 Carbon APT Env GY03-117
128.690 MHz C13{H1} APT_ad in CDCl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.7°C => actual temp = 27.0°C, oloidual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
Guojun Compd 29 Hydrogen Env GY03-117
499.806 MHz H1 PRESAT in cdCl3 (ref. to CDC13 @ 7.26 ppm), temp 27.7 C -> actual temp = 27.0 C, cold dual probe

[Diagram of molecular structure]

File: http://example.com/GY03-117-NMR.png
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Agilent Technologies

Recorded on: 5/00, Apr 11 2015
Pulse Sequence: APT_6d
Sweep Width(Hz): 33783.8
Acquisition Time(s): 2
Relaxation Delay(s): 0.5
Digital Res.(Hz/pt): 0.26
Hz per mm(Hz/mm): 140.75
Completed scans: 1000

Gaojun, Crmpd 34 Carbon APT Env GY02-189
125.690 MHz C13[1H] APT_6d in cdCl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.7 C => actual temp = 27.6 C, colidual probe
C & CH2 same, CH & CH3 opposite side of solvent signal

20a

240 220 200 180 160 140 120 100 80 60 40 20 0 ppm

File: /mnt/homed00/homw12genome/mndata/CLIVE/Gaojun/2015.04/GY02-189-OP-APT
Guojun Compd 24 Hydrogen Env GY02-195
408.118 MHz H1 1D in cdc13 (ref. to CDCl3 @ 7.26 ppm), temp 26.4°C -> actual temp = 27.0°C, autoexdb probe
Guzhan Compd 30 Carbon APT Env GY03-111
128.690 MHz C13[H1] APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, colloidal probe C & CH2 same, CH & CH3 opposite side of solvent signal

File: \hms\3000\homer12\genomen\hmsdata\CLIVE\Guzhan2015.08\GY03-67-APT
Guanjun Compd 38 Hydrogen Env GY03-111
408.118 MHz H1 1D in ccd3 (ref. to CDC3 @ 7.26 ppm), temp 26.4 C - actual temp = 27.0 C, autodx probe
Guojun Compd 18 Carbon Normal Env GY2197
125.79 MHz C13[H1] 1D in dmsso (ref. to DMSO @ 39.5 ppm), temp 27.7°C -> actual temp = 27.0°C, cold dual probe.
Guojun Compd 18 Hydron Env GY03-97
499.809 MHz H1 PRESAT in dmso (ref. to DMSO @ 2.49 ppm), temp 27.7 C → actual temp = 27.0 C, cold dual probe
Guojun Crmpd 31 Carbon APT Env GY03-113
128.490 MHz C13[H] APT_Ad in cddct (ref. to CDC13 @ 77.06 ppm), temp 27.7°C -> actual temp = 27.6°C, colindual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
Guojun Cmpd 31 Hydrogen Env GY03-113
499.806 MHz H1 PRESAT in cdc3 (ref. to CDC3 @ 7.26 ppm), temp 27.7 °C → actual temp = 27.0 °C, cold dual probe
Gaojun Compd 51 Carbon Normal Env GY03-199
128.691 MHz C13[1H1] 1D in dmsos (ref. to DMSO @ 39.5 ppm), temp 27.7 °C → actual temp = 27.0 °C, coldiual probe
Guojun Crmpd 51 Hydrogen Env GY03-198
499.809 MHz H1 PRESAT in dimso (ref. to DMSO @ 2.49 ppm), temp 27.7 C = actual temp = 27.0 C, colddual probe
Guojun Crmpd 36 Carbon APT Env GY03-127

128.690 MHz C13[H1] APT sd in dcd3 (ref. to CDC13 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, coliodual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
Guojun Compd 36 Hydrogen Env GY03-137
499.806 MHz H1 PRESAT in cdc3 (ref. to CDCl3 @ 7.26 ppm), temp 27.7 C => actual temp = 27.0 C, colddual probe
Guanjun Compd 43 Carbon APT Env GY04-07
125.690 MHz C13{H} APT sd in ccdt3 (ref. to CDC3 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, cold probe
C & CH2 same, CH & CH3 opposite side of solvent signal
Guojun Compd 43 Hydrogen Env GY04-07
499.808 MHz H1 PRESAT in cdcl3 (ref. to CDC3 @ 7.26 ppm), temp 27.7 C => actual temp = 27.0 C, colddual probe

Guojun Crmpd 47 Carbon APT Env GY03-183
128.690 MHz C13[H1] APT_ad in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, colloidal probe
C & CH2 same, CH & CH3 opposite side of solvent signal
Guojun Compd 37 Hydrogen Env GYO3-171
699.762 MHz H1 PRESAT in cdc3 (ref. to CDC3 = 7.26 ppm), temp 27.5 C -> actual temp = 27.0 C, coldid probe

File: /mnt/hdd/00/homna12/1gcnrnm/nnrdata/CLIVE/Guoju015.09/GYO4-31-CP
Guojun Crmtd 44 Carbon APT Env GY04-11
128.690 MHz C13[H1] APT_se in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, coildual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
Guojun Crmpd 48 Carbon APT Env GY03-185
128.690 MHz C13[H1] APT_ad in cdc13 (ref. to CDC13 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, colidual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
Guojun Compd 48 Hydrogen Env GY03-185
408.118 MHz H1 1D in cdc3 (ref. to CDC3 @ 7.26 ppm), temp 26.4 °C -> actual temp = 27.0 °C, auxorxtb probe
Gaojun Crmpd 3B Carbon APT Env GY04-33
128.690 MHz C13[H1] APT_ad in cdcd3 (ref. to CDCl3 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, colidudal probe
C & CH2 same, CH & CH3 opposite side of solvent signal
Gaojun Crmpd 38 Hydrogen Env GY03-173
408.18 MHz H1 1D in d6d3 (ref. to CDCl3 @ 7.26 ppm), temp 26.4 C -> actual temp = 27.0 C, rfxdb probe
Guojun Compd 45 Carbon APT Env GY04-13
128.690 MHz C13[H1] APT.recycle in cdc13 (ref. to CDC13 @ 77.06 ppm), temp 27.7°C -> actual temp = 27.0°C, colidual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
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Gaojun Crmpd 45 Hydrogen Env GY04-13
499.806 MHz H1 PRESAT in CDCl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.7°C → actual temp = 27.0°C, colidual probe

File: /mnt/d/000/posn12/gnornin/nmmdata/CLIVE/Gaojun0915.09/GY04-13-DP
Guojun Crmpd 48 Carbon APT Env GY03-187
128.690 MHz C13[H1] APT_sd in cdcl3 (ref. to CDC13 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, colidual probe
C & CH2 same, CH & CH3 opposite side of solvent signal

![Chemical Structure Image]
Gaojun Compd 21 Carbon APT Env GY01-101
128.690 MHz C13[H1] APT_od in CDCl3 (ref. to CDC13 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, colpidual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
File: /mnt/home/12tgromov/minidata/CLIVE/Guojuun2014.09/GYO1-147-OP
Guojun Crmpd 46 Carbon APT Env GY04-15
128.690 MHz C13{H1} APT_ad in cdc13 (ref. to CDC13 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, colidual probe
C & CH2 same, CH & CH3 opposite side of solvent signal
Guojun Crmpd 46 Hydrogen Env GYO4-15
499.806 MHz H1 PRESAT in cdcl3 (ref. to CDC13 @ 7.26 ppm), temp 27.7 C => actual temp = 27.0 C, cold dual probe

File: /mnt/d/00/home/12/geom/remdata/UV/UV/Guojun02015.09/GYO4-15-DP
Goujun Crmpd 58 Hydrogen Enr GY03-197
499.006 MHz H1 PRESAT in cdcl3 (ref. to CDC13 @ 7.26 ppm), temp 27.7 C -> actual temp = 27.0 C, colddual probe

File: /mnt/d/000/Joseph/spectra/./Goujun/GY03-197-1D-DP