The Synthesis and Reactions of 2-Substituted Ethyl N-Alkylmalonylhydroxamic Acids

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Supplementary Material TOC

S1- General Experimental Details

S2- ^{13}C NMR spectrum of 4ab

S3- ^{13}C NMR spectrum of 4ac

S4- ^{13}C NMR spectrum of 4ad

S5- ^{13}C NMR spectrum of 4bb

S6- ^1H NMR spectrum of 4ca

S7- ^1H NMR spectrum of 4ec

S8- ^1H NMR spectrum of 4fa

S9- ^1H NMR spectrum of 6a

S10- ^1H NMR spectrum of 6b

S11- ^1H NMR spectrum of 6c

S12- ^1H NMR spectrum of 6f

S13- ^1H NMR spectrum of 14

S14- ^1H NMR spectrum of 17

S15- ^1H NMR spectrum of 19a

S16- ^1H NMR spectrum of 19b

S17- ^1H NMR spectrum of 19c

S18- ^1H NMR spectrum of 19d

S19- ^1H NMR spectrum of 19e

S20- ^1H NMR spectrum of 20

S21- ^{13}C NMR spectrum of 21bh

S22- ^{13}C NMR spectrum of 21bj

S23- ^{13}C NMR spectrum of 21eh

S24- ^{13}C NMR spectrum of 21ei

S25- ^{13}C NMR spectrum of 21ej

S26- ^{13}C NMR spectrum of 25

S27- ^{13}C NMR spectrum of 26

S28- ^{13}C NMR spectrum of 27

Experimental Section

General Methods. All reactions were carried out under a nitrogen atmosphere in oven dried or flame dried glassware. THF was distilled from benzophenone ketyl. Other solvents were distilled before use. ^1H and ^{13}C spectra were recorded at 200 MHz and 50 MHz, respectively. Both the ^1H and ^{13}C NMR spectra of hydroxamic acids **4** and unsaturated amides **6** were sometimes complicated due to the presence of rotamers. IR spectra were recorded as KBr pellets or neat liquids. Elemental analysis was done by M-H-W Laboratories, Phoenix, AZ. Melting points are uncorrected. Flash column chromatography was carried out using silica gel 60 (230–400 mesh). Thin-layer chromatography was performed using silica gel 60 F254 plates and visualized by UV irradiation or iodine. N-Methylhydroxylamine hydrochloride, N-cyclohexylhydroxylamine hydrochloride, N-(tert-butyl)hydroxylamine hydrochloride, hydrocinnamoyl chloride, valeryl chloride, isovaleryl chloride, N-methylhydroxylamine hydrochloride, N-isopropylhydroxylamine hydrochloride, tert-butylchlorodiphenylsilane and ethylmalonyl chloride and all the amines used in this study were commercially available and used as received.

54

SM014-f
S01888
Solvent: CDCl3
Temp: 39.0 C / 312.1 K
UNITY-400 "fatman"
PULSE SEQUENCE
Relax: delay 2.000 sec
Pulse 60.0 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
1024 repetitions
OBSERVE C13 100.5622433 MHz
DECUPLE H1 399.3804768 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
SFO16 Precision data
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 54 minutes

16 Apr 2002

SM031 P

S7

31 May 2002

SM035 P

5 Jun 2002

58

SM006 III P

21 Mar 2002

59

SIM012 P

8 Apr 2002

S10

SM011 P

S11

10 Apr 2002

SM034 P

6 Nov 2002

S12

SM080 P

31 Aug 2002

S/3

SM094 P

S14

-5.0808
4.2217
4.2107
4.1851
4.1759
-3.6813
-3.4908
-3.1831
-2.6464
1.6885
1.6408
1.3056
1.2690
1.2342

SM087 P

SM073 CP

S17

4.2568
4.2293
4.2220
4.1927
4.1506

3.8282
3.7915
3.7567
3.7219

3.4948

3.2054

SM069 P

1.4616
1.4250
1.3151
1.2785
1.2437

S19

SM098 P

S20

Total time 8 hr, 54 min, 58 sec

13C Observe - New Parameters
 Archive directory: /home/vmr1/vmr/sys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: e2pu1
 Solvent: CDCl₃
 Ambient temperature
 UNITY-400 "Gatman"
 Relax. delay 2.000 sec
 Pulse 6.5 usec
 Acq. time 1.19 sec
 Width 25000.0 Hz
 1632 Repetitions
 OBSERVE C13, 100.562257 MHz
 DECOUPLE H1, 399.8304768 MHz
 Power 45 dB
 continuously on
 VOLT-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FI size 65536
 Total time 4 hr, 27 min, 28 sec

13C OBSERVE

Archive directory: /home/sachin/vnmr/sys/data
Sample directory:
File: CARBON

Pulse Sequence: e2pul
Solvent: CDCl3
Ambient temperature
UNITY-400 "gamma"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.19 sec
Width 25000.0 Hz
1664 repetitions
OBSERVE C13, 100.5622752 MHz
DECOUPLE H1, 500.1304799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Ft size 65536
Total time 4 hr, 27 min, 31 sec

S 24

13C OBSERVE

Archive directory: /home/sachin/vnmr/sv/data
 Sample directory:
 File: CARBON

Pulse Sequence: zgpg30
 Solvent: CDCl3
 Ambient temperature
 UNITY-400 "Jainman"

Relax. delay 2.000 sec
 Pulse 45.0 degrees
 Acq. time 1.189 sec
 Width 25000.0 Hz
 2728 repetitions
 OBSERVE C13, 100.5822688 MHz
 DECOUPLE H1, 399.9304739 MHz
 Power 40 dB
 Continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FI size 65536
 Total time 2 hr, 40 min, 30 sec

S25

13C OBSERVE

Archive directory: /home/eachin/vnmr/sy/data
Sample directory:
File: CARBON

Pulse Sequence: zgpg30
Solvent: CDCl3
Ambient temperature
UNITY-400 "fatman"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.188 sec
Width 25000.0 Hz
15168 repetitions
OBSERVE C13, 100.5822982 MHz
DECOUPLE H1, 399.8304788 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 17 hr, 50 min, 4 sec

13C OBSERVE

Archive directory: /home/sachin/vnmr/s/data
Sample directory:
File: CARBON

Pulse Sequence: szput
Solvent: CDCl3
Ambient temperature
UNITY-400 "fatman"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.19 sec
Width 25000.0 Hz
2032 repetitions
OBSERVE C13, 100.562476 MHz
DECOUPLE H1, 399.8304793 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 85536
Total time 4 hr, 27 min, 31 sec

S28