Highly atom efficient aluminium triflate catalysed acetal formation

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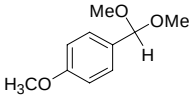
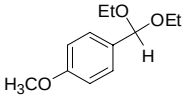
Supplementary data

The majority of the acetals prepared in this study have previously been prepared and characterised. The relevant references are given below for these compounds. In instances where the compounds have not previously been synthesised or where the characterisation data are inadequate or incomplete, additional data have been provided here.

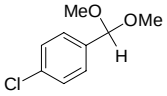
4-Methoxybenzaldehyde dimethyl acetal[1]



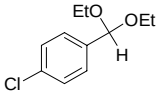
4-Methoxybenzaldehyde diethyl acetal[2]



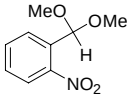
1-Chloro-4-dimethoxymethyl benzene[3]



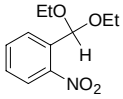
1-Chloro-4-diethoxymethyl benzene[2]



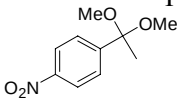
o-Nitrobenzaldehyde dimethyl acetal[4]



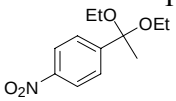
o-Nitrobenzaldehyde diethyl acetal[4]



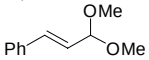
4-Nitroacetophenone dimethyl acetal[5]



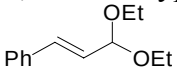
4-Nitroacetophenone dimethyl acetal[6]



3,3-Dimethoxypropenyl benzene[3]



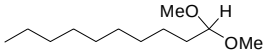
3,3-Diethoxypropenyl benzene[7]



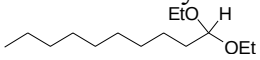
IR ν_{max} (KBr) cm^{-1} 2976, 1138, 1123, 1052, 734; ^1H NMR: (300 MHz, $CDCl_3$,) δ_H 7.40 (d, 2H, *J* = 7.8 Hz), 7.28 (m, 3H), 6.70 (d, 1H, *J* = 16.2 Hz), 6.20 (dd, 1H, *J* = 16.2 and

5.1 Hz), 5.05 (dd, 1H, *J* = 5.3 and 1.0 Hz), 3.70 (m, 2H), 3.55 (m, 2H), 1.24 (t, 6H, *J* = 7.0 Hz); ^{13}C NMR: (75 MHz, $CDCl_3$) δ_C 136.1, 132.9, 128.5 (2C), 128.0, 126.6 (2C), 126.5, 101.4, 61.0 (2C), 15.2 (2C); EIMS (*m/z*): 206 ($[M]^+$, 10%), 161 ($[M–OCH_2CH_3]^+$, 100%), 133 ($[M–C_4H_9O]^+$, 90%).

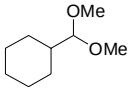
Decanal dimethal acetal[8]



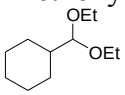
Decanal diethyl acetal[8]



Dimethoxymethyl-cyclohexane[9]

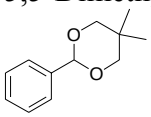


Diethoxymethyl-cyclohexane[10]

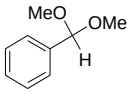


IR: ν_{max} ($CHCl_3$) cm^{-1} 2925, 2854, 1131, 1081, 1056; ^{1}H NMR: (300 MHz, $CDCl_3$,) δ_H 4.07 (d, 1H, *J* = 7.2 Hz), 3.60 (m, 2H), 3.44 (m, 2H), 1.70 (m, 4H), 1.53 (m, 2H), 1.14 (m, 9H), 0.96 (m, 2H); ^{13}C NMR: (75 MHz, $CDCl_3$) δ_C 106.7, 61.5 (2C), 40.7, 28.1 (2C), 26.4, 25.7 (2C), 15.2 (2C); EIMS (*m/z*): 141 ($[M–OCH_2CH_3]^+$, 20%), 95 ($[M–C_4H_{11}O_2]^+$, 40%), 103 ($[C_5H_{11}O_2]^+$, 100%).

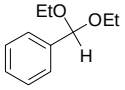
5,5-Dimethyl-2-phenyl-[1,3]dioxane[11]



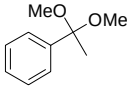
Dimethoxymethyl-benzene[3]



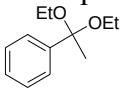
Dimethoxymethyl-benzene[2]



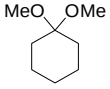
Acetophenone dimethyl acetal[4]



Acetophenone diethyl acetal[4]



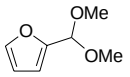
1,1-Dimethoxy cyclohexane[12]



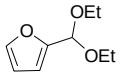
1,1-Diethoxy cyclohexane[13]

IR: ν_{max} (KBr) cm^{-1} 2975, 2937, 1090, 1054, 732; ^1H NMR: (300 MHz, $CDCl_3$,) δ_H 3.42 (q, 4H, *J* = 7.1 Hz), 1.62 (m, 4H), 1.45 (m, 4H), 1.40 (m, 2H), 1.13 (t, 6H, *J* = 7.1 Hz); ^{13}C NMR: (75 MHz, $CDCl_3$) δ_C 100.0, 54.7 (2C), 33.8 (2C), 25.6, 23.0 (2C), 15.5; EIMS (*m/z*): 172 ($[M]^+$, 10%), 143 ($[M–CH_2CH_3]^+$, 5%), 129 ($[M–C_3H_7]^+$, 90%), 127 ($[M–OCH_2CH_3]^+$, 90%), 99 ($[M–C_4H_9O]^+$, 85%), 81 ($[M–C_4H_{11}O_2]^+$, 100%).

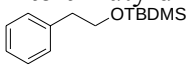
2-Furaldehyde dimethyl acetal[4]



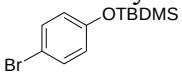
2-Furaldehyde diethyl acetal[4]



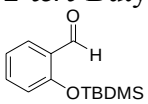
1-*tert*-Butyldimethylsilyloxy-3-phenylpropane[14]



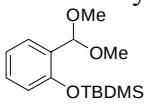
1-*tert*-Butyldimethylsilyloxy-4-bromophenyl[15]



2-*tert*-Butyldimethylsilyloxybenzaldehyde[16]

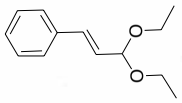


2-*tert*-Butyldimethylsilyloxy dimethoxy methyl benzene.



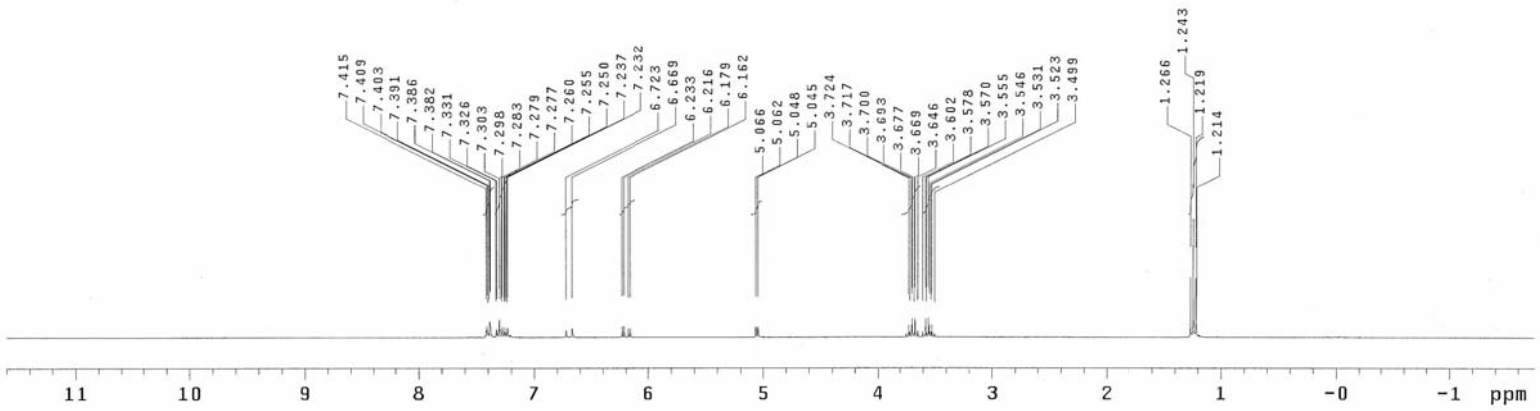
IR: ν_{max} (KBr) cm^{-1} 2931, 1257, 1092, 1055, 926; ^1H NMR: (300 MHz, CD_3OD,) δ_H 7.34 (dd, 1H, *J* = 7.8 and 1.5 Hz), 7.10 (dt, 1H, *J* = 7.6 and 1.6 Hz), 6.85 (t, 1H, *J* = 7.5 Hz), 6.74 (d, 1H, *J* = 8.1, Hz), 5.50 (s, 1H), 3.20 (s, 6H), 0.93 (s, 9H), 0.16 (s, 6H); ^{13}C NMR: (75 MHz, CD_3OD) δ_C 154.7, 130.5, 129.5, 128.8, 121.8, 120.0, 100.7, 53.8 (2C), 26.3 (3C), 19.2, -4.03 (2C); EIMS (*m/z*): 251 ($[M–OCH_3]^+$, 10%), 225 ($[M–C_4H_9]^+$, 70%), 179 ($[M–C_4H_{15}O]^+$, 50%), 165 ($[M–C_6H_{17}Si]^+$, 100%).

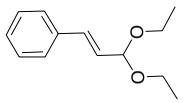
transcinnamaldehyde diethyl acetal



Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-300 "nmr"

Pulse 34.6 degrees
Acq. time 3.744 sec
Width 4001.6 Hz
Single scan
OBSERVE H1, 299.9464487 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 3 sec

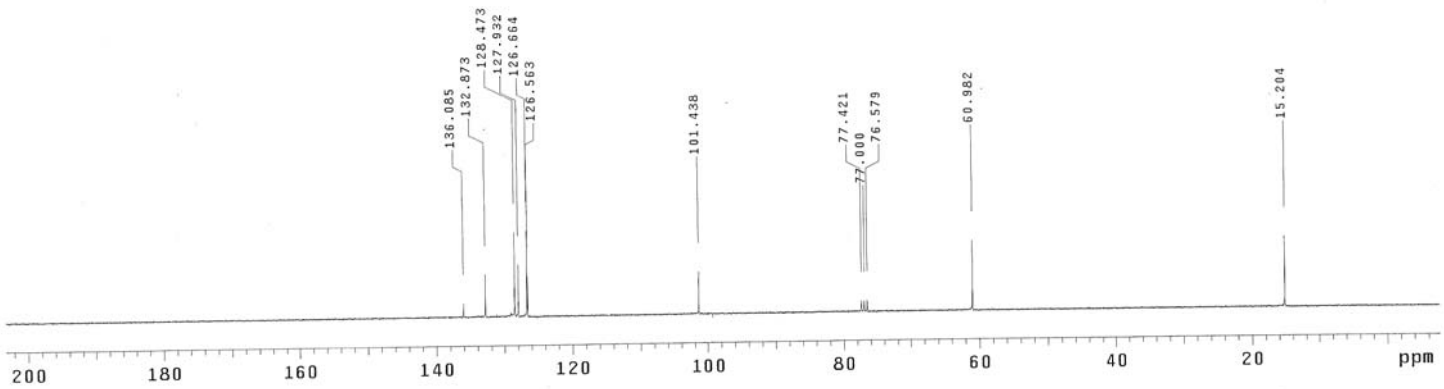




transcinnamaldehyde diethyl acetal

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-300 "nmr"

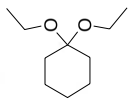
Relax. delay 1.000 sec
Pulse 83.9 degrees
Acq. time 1.815 sec
Width 16501.7 Hz
76 repetitions
OBSERVE C13, 75.4215997 MHz
DECOUPLE H1, 299.9479281 MHz
Power 30 dB
continucusly on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 48 min, 11 sec

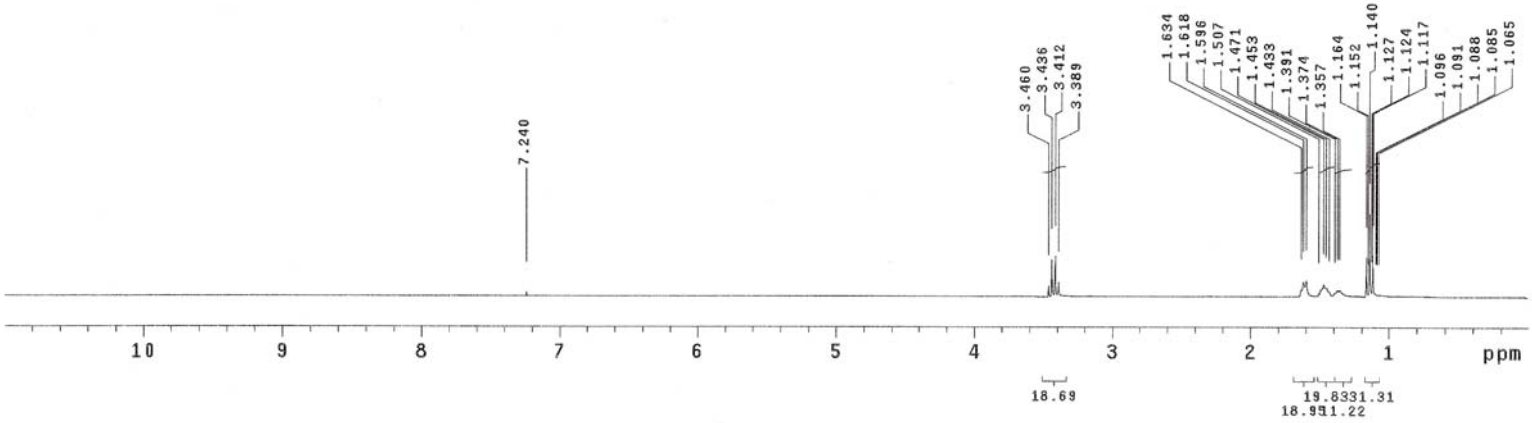


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "kmr300"

Relax. delay 1.000 sec
Pulse 32.1 degrees
Acq. time 2.000 sec
Width 4800.0 Hz
4 repetitions
OBSERVE H1, 300.0575478 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 13 sec

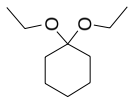


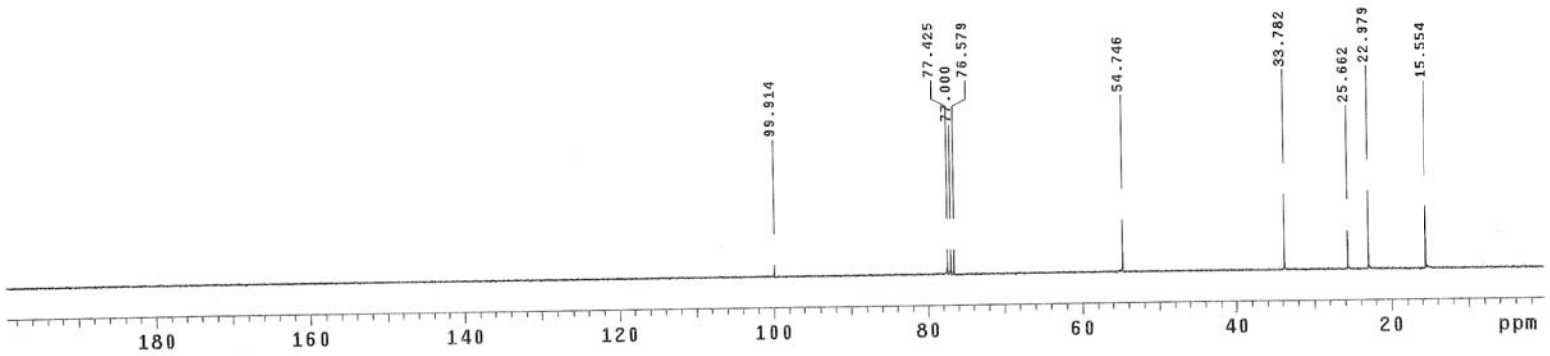


13C OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
GEMINI-300BB "kmr300"

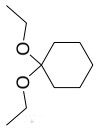
 Pulse 46.0 degrees
 Acq. time 1.815 sec
 Width 18761.7 Hz
 512 repetitions
OBSERVE C13, 75.4495295 MHz
DECOUPLE H1, 300.0590418 MHz
 Power 39 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 131072
Total time 24 min, 50 sec

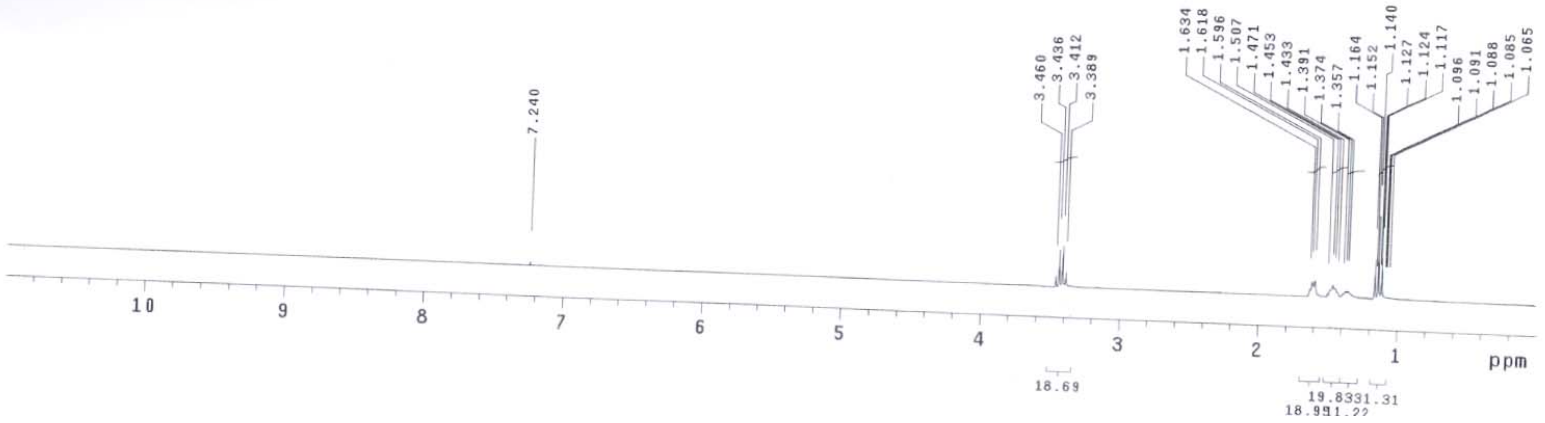




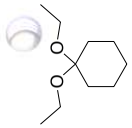
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "kmr300"

Relax. delay 1.000 sec
Pulse 32.1 degrees
Acq. time 2.000 sec
Width 4800.0 Hz
4 repetitions
OBSERVE H1, 300.0575478 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 13 sec



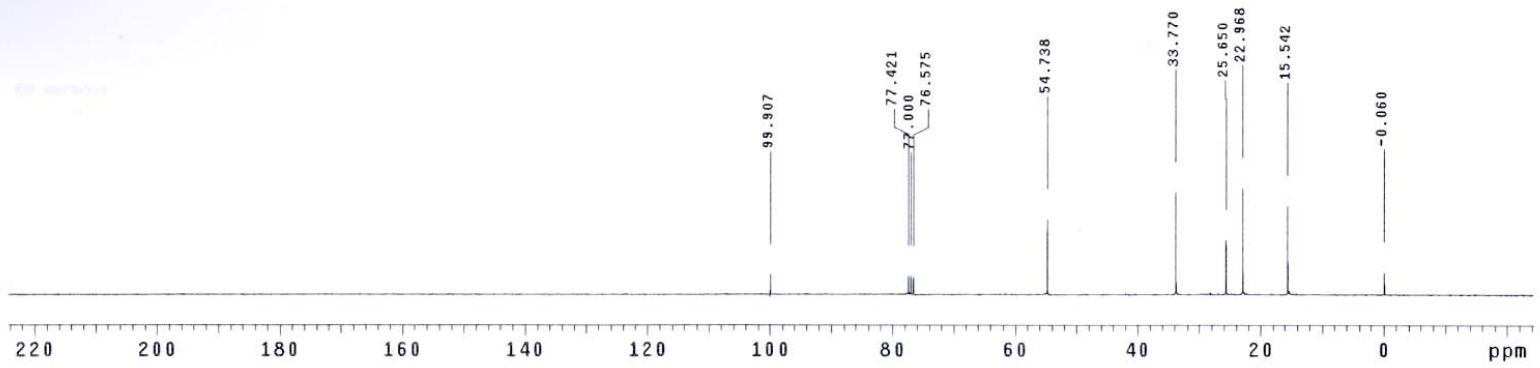


cyclohexanone diethyl acetal



Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "kmr300"

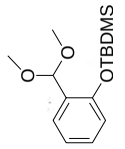
Pulse 46.0 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
5764 repetitions
OBSERVE C13, 75.4495298 MHz
DECOUPLE H1, 300.0590418 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 16 hr, 10 min, 33 sec

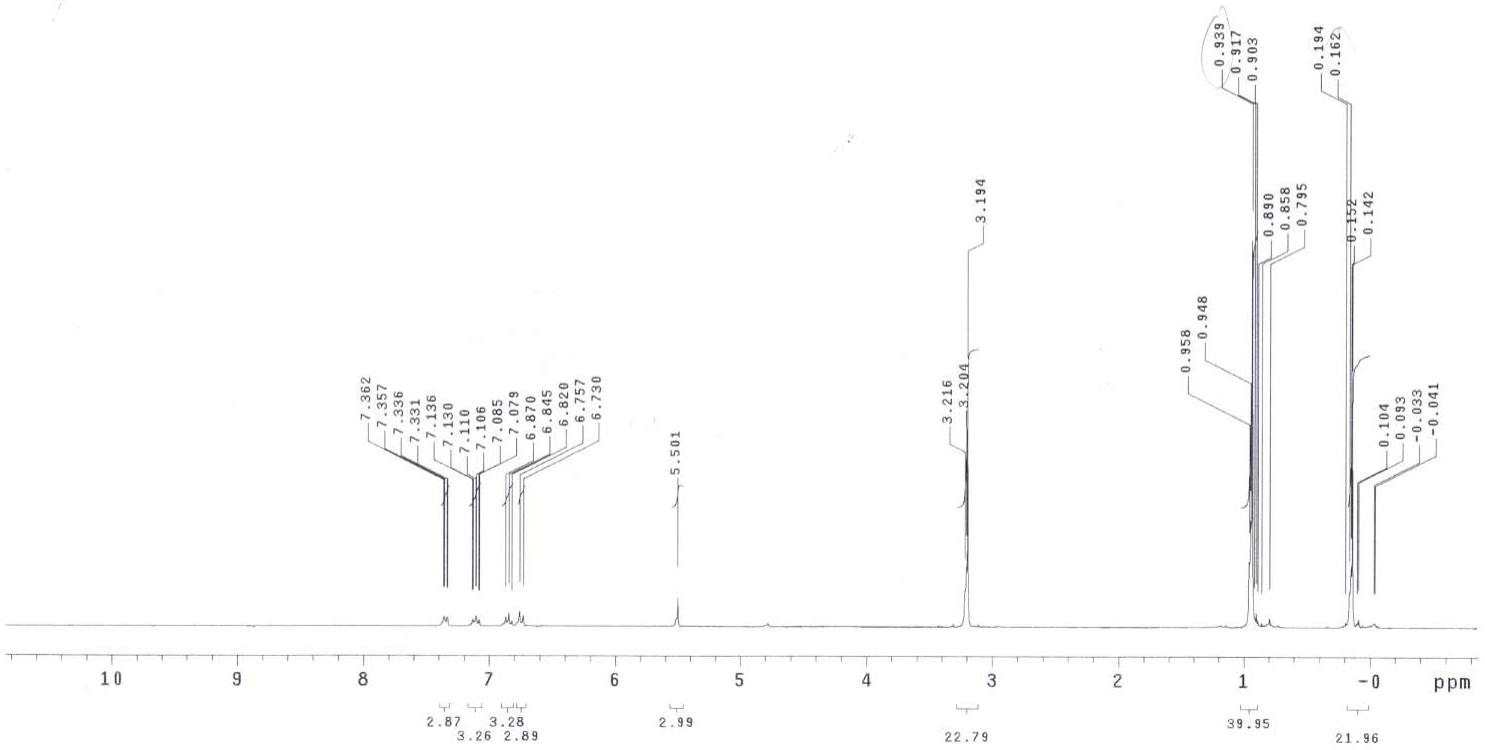


salicylaldehyde dimethyl acetal TBDMs protected

Pulse Sequence: s2pul
Solvent: CD3OD
Ambient temperature
GEMINI-300BB "kmr300"

Relax. delay 1.000 sec
Pulse 90.6 degrees
Acq. time 2.000 sec
Width 4800.0 Hz
Single scan
OBSERVE H1, 300.0587530 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 4 sec

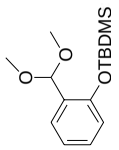


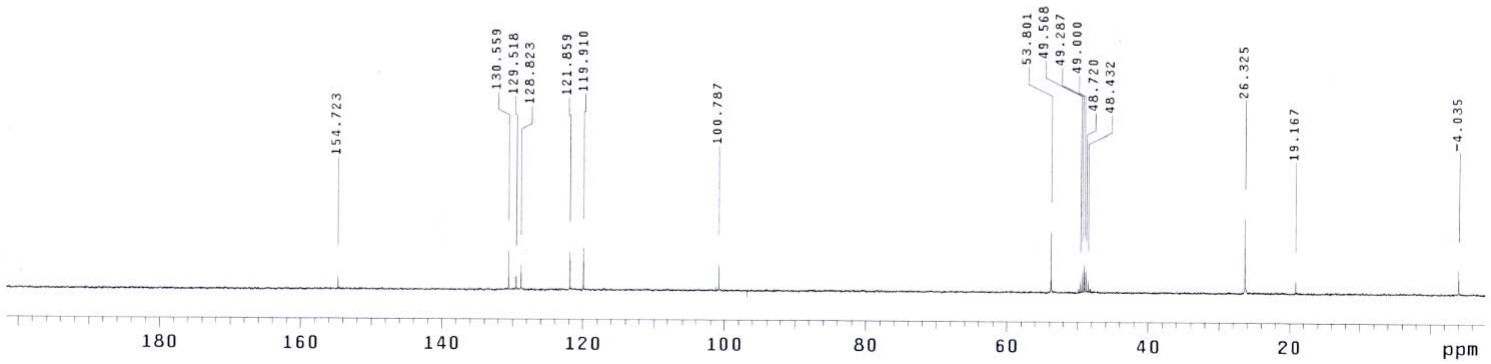


Salicylaldehyd dimethyl acetal TBDMS protected

Pulse Sequence: s2pul
Solvent: CD3OD
Ambient temperature
INOVA-300 "nmr"

Relax. delay 1.000 sec
Pulse 83.9 degrees
Acq. time 1.815 sec
Width 16501.7 Hz
80 repetitions
OBSERVE C13, 75.4217889 MHz
DECOUPLE H1, 299.9491099 MHz
Power 30 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 47 min, 3 sec





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