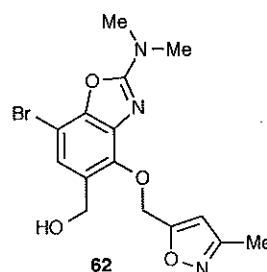
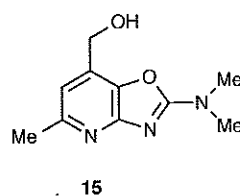


The synthesis of a series of adenosine A₃ receptor agonists

Kenneth J. Broadley, Erica Burnell, Robin H. Davies, Alan T. L. Lee, Stephen Snee and Eric J. Thomas

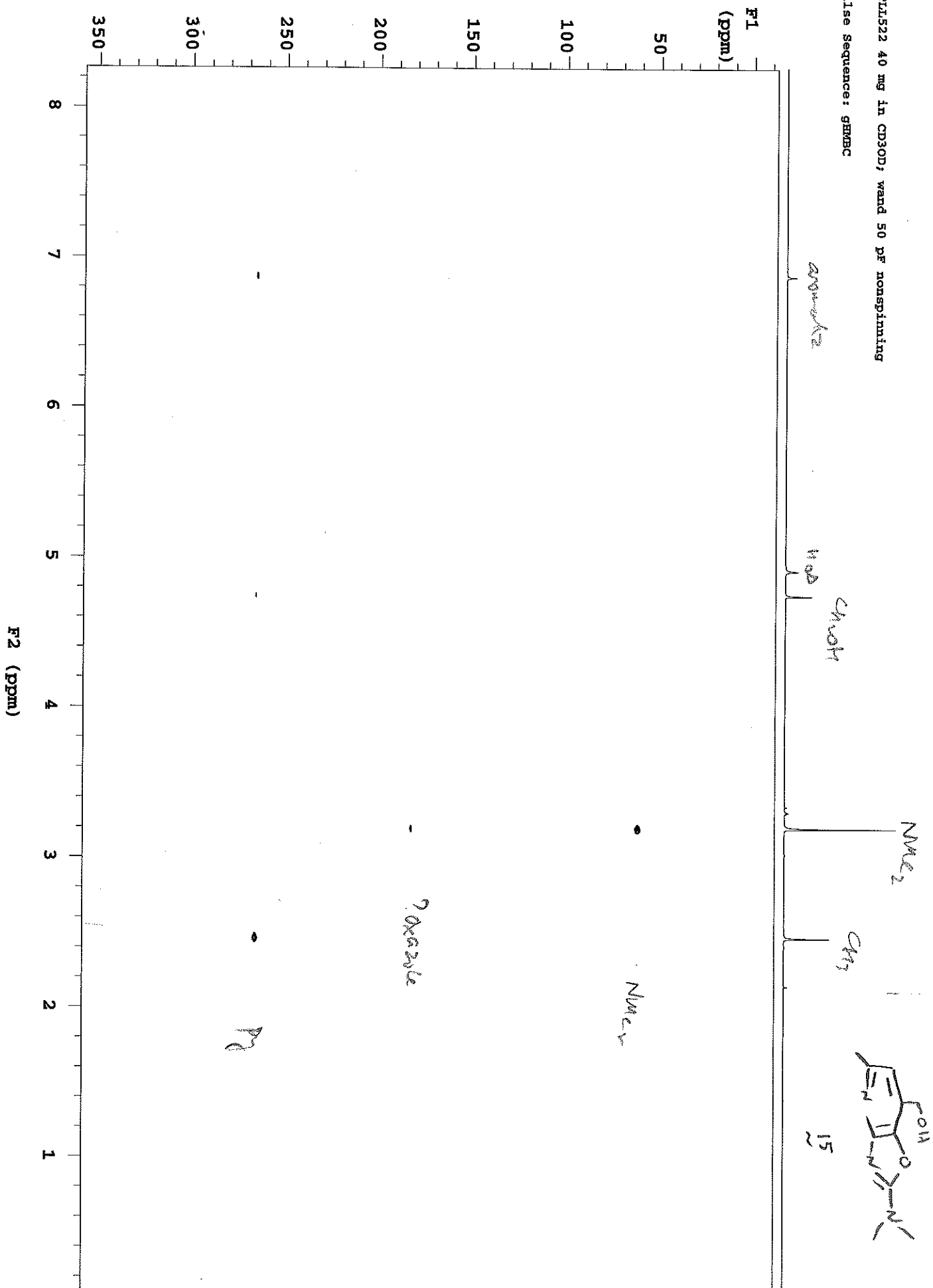
Supplementary Data

Correlation NMR spectra for compounds **15** and **62**



ATL1522 40 mg in CD3OD, wand 50 pf nonspinning

Pulse Sequence: ghmrc



ATL522 40 mg in CD3OD; wand 50 pf nonsp
inning

exp2 ghmhc

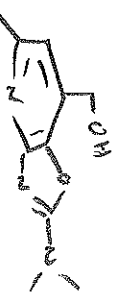
SAMPLE		FLAGS	
date	Dec 8 2006	hs	n
solvent	cd3od	sspul	n
sample	undefined	prcflg	y
ACQUISITION	hsq1v1		10193
sw	3265.0	SPECIAL	
at	1.255	temp	not used
np	8192	gain	50
fb	2000	spin	0
ss	8	GRADIENTS	
dl	2.745	g1v11	10193
nt	32	g1	0.001000
2D ACQUISITION	g21v13		2070
sw1	15000.0	g13	0.001000
n1	1024	gstrab	0.000500
phase	0	F2 PROCESSING	
TRANSMITTER	sb	sb	0.627
tn	H1	sbs	not used
sfrq	399.962	fn	8192
tof	-333.7	F1 PROCESSING	
tpwr	58	sbl	0.034
pw	6.800	sbsl	not used
DECOUPLER	fn1	fn1	2048
dn	N15	DISPLAY	
dof	-6167.0	sp	40.6
dm	nm	wp	3265.0
dmm	ccc	sp1	-509.5
dmf	200	wp1	15000.0
dprz	0	rfl	-40.6
pwrclv1	55	rfd	0
pwr	39.000	rfl1	509.5
HMBC	rfl1		0
j1h	10000.0	PLOT	
jmr	3.0	wc	243.0
		sc	6.8
		wc2	138.0
		sc2	0
		vs	322
		th	2

F2 trace at 50 of pyridine

Intensity ratio
⇒ J_{NH} ~ 50x smaller for
CH₂ than for Me,
consistent with J

CH₂

15



8 7 6 5 4 3 2 1 ppm

ATL522 40 mg in CD3OD; wand 50 pf nonsp
inning

exp2 GRMBC

SAMPLE		FLAGS	
date	Dec 8 2006	hs	n
solvent	cd3od	ssupl	n
sample	undefined	pfefl	y
ACQUISITION	hsplv1		10193
sw	3265.0	SPECIAL	
at	1.255	temp	not used
np	8192	gain	50
fb	2000	spin	0
ss	8	GRADIENTS	
d1	2.745	gzlv1	10193
nt	32	gtl	0.001000
2D ACQUISITION	gzlv13		2070
sw1	15000.0	gt3	0.001000
ni	1024	gstab	0.000500
phase	0	F2 PROCESSING	
TRANSMITTER	sb	sbs	0.627
tn	hl	sbs	not used
sfreq	399.962	fn	8192
tof	-333.7	F1 PROCESSING	
tpwr	58	sbl	0.034
pw	6.800	sbsl	not used
DECOUPLER	fn1		2048
dn	N15	DISPLAY	
dof	-6167.0	sp	40.6
dm	nm	wp	3265.0
dmm	gcc	sp1	-509.5
dmf	200	wp1	15000.0
dpwr	0	rfl	-40.6
pwrlv1	55	rflp	0
pw	39.000	rfl1	509.5
HMBC	rflp1		0
j1kh	10000.0	PLOT	
j1kh	3.0	wc	243.0
		sc	6.8
		wc2	138.0
		sc2	0
		vs	214
		th	2

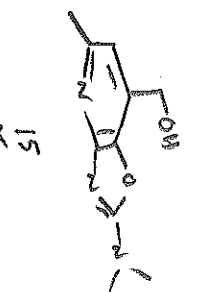
350 300 250 200 150 100 50 ppm



Used oxide from 552 ppm?

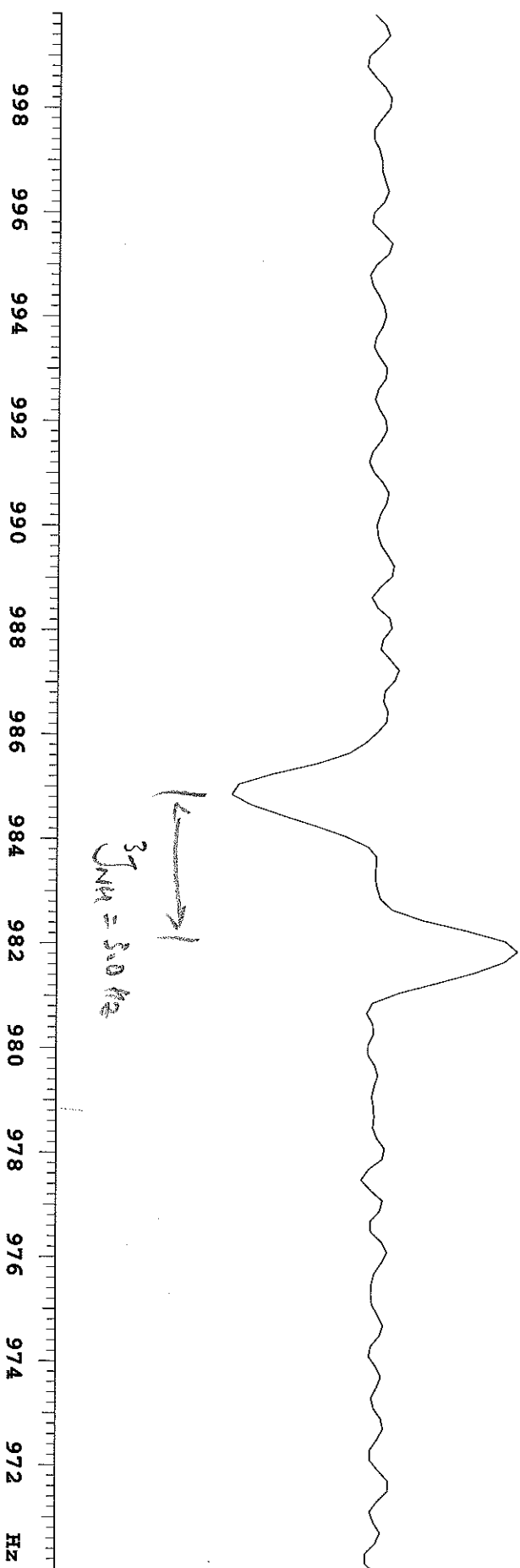
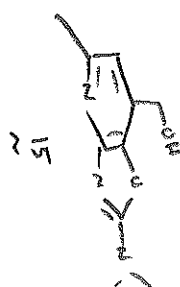
F₁ trace at δ_H 4 MHz

NMe₂

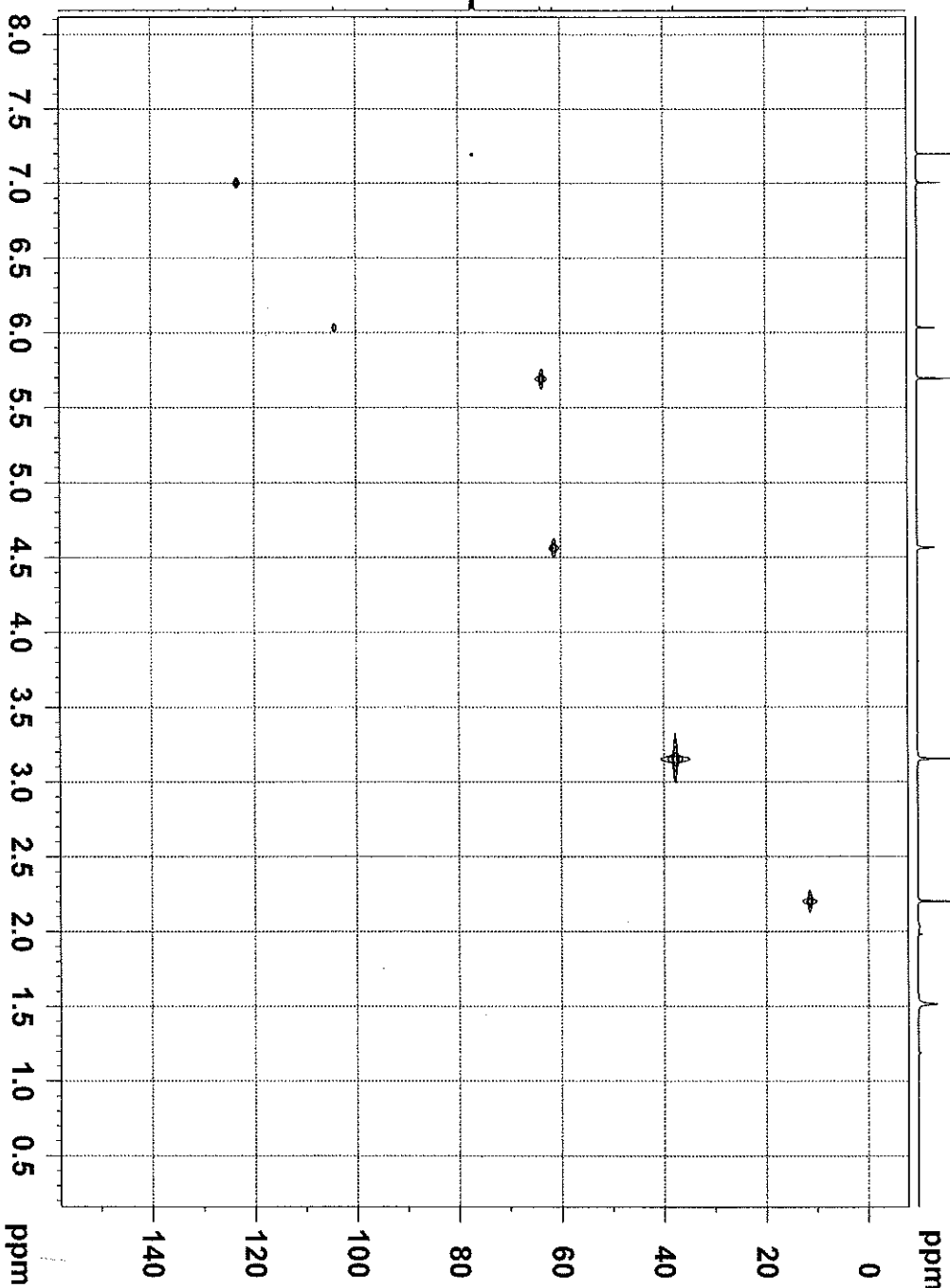
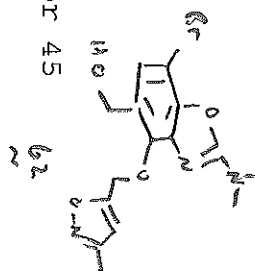


ATLL522 40 mg in CD3OD, wand 50 pf nonspinning
Pulse Sequence: gHMBC

¹³C FID of gHBC, resolution enhanced

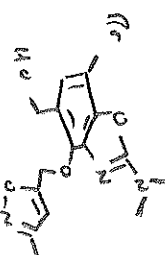


E. Burnell
0910-047
EB111
mhmoc_Bg CDCl3 {E:\bruk400service_data\2010\sep} Administrator 45



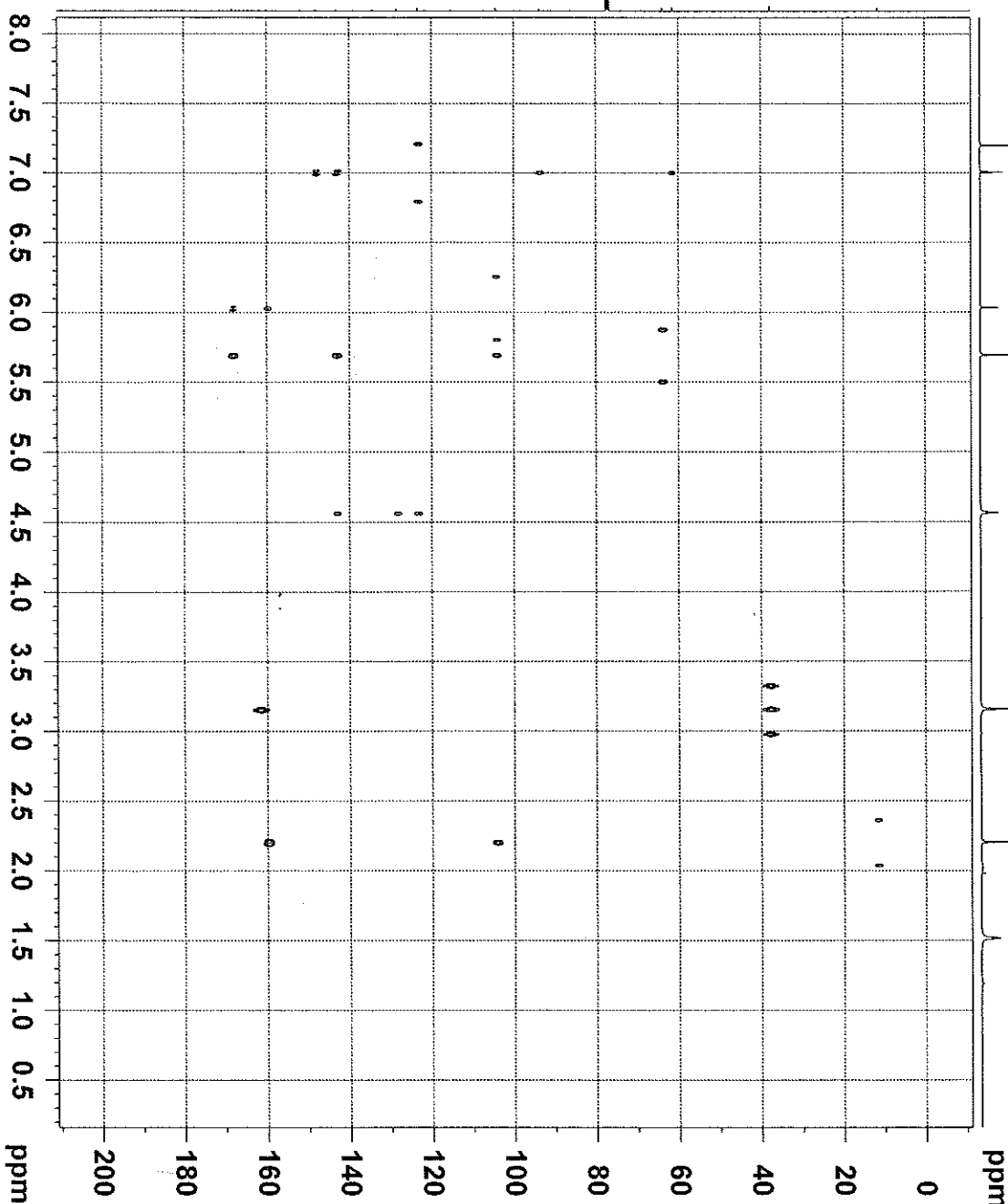
NAME 2010-09-21-Administrator-45
EXPNO 1
PROCNO 17
Date_ 20100922
Time_ 6.49
INSTRUM AV400 S
PROBHD 5 mm PABBO BB-
PULPROG hmcgpgpgf
TD 1024
SOLVENT CDCl3
NS 16
DS 16
SCN 3184.713 Hz
FIDRES 3.110072 Hz
AQ 0.1608180 sec
RG 2050
DE 157.000 usec
TE 295.9 K
CNS12 145.0000000
D0 0.00000300 sec
D1 1.4679859 sec
D2 0.00344828 sec
D12 0.00002000 sec
D13 0.00002000 sec
D16 0.00010000 sec
IN0 0.00003000 sec
===== CHANNEL f1 =====
NUC1 1H
P1 10.00 usec
F2 20.00 usec
PL1 -3.60 dB
PL1W 18.36511721 W
SFO1 400.2316952 MHz
===== CHANNEL f2 =====
CPDPRG2 garp
NUC2 13C
P3 8.40 usec
PCPD2 65.00 usec
PL2 -2.50 dB
PL12 15.27 dB
PL1W 60.30228424 W
PL13W 1.00770569 W
SFO2 100.6454609 MHz
===== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec
ND0 2
TD 256
SFO1 100.6455 MHz
FIDRES 65.10438 Hz
SW 165.639 ppm
FMODE QF
SI 1024
SF 400.2300392 MHz
WDW OSLINE
SSB 2
LB 0.00 Hz
GB 0
SI 1024
MC2 QF
SF 100.6379140 MHz
WDW OSLINE
SSB 2
LB 0.00 Hz
GB 0

E. Burnell
0910-047
EB111
MHMBC CDC13 {E:\bruk400service_data\2010\Sep} Administrator 45



2xCH₃

CH₃



BRUKER
(400MHz)

NAME 2010-09-21-Administrator-45

EXPNO 1

PROCNO 1

Date_ 20100922

Time_ 4.34

INSTRUM AV400

PROBHD 5 mm PABBO BB-

PULPROG hmcgprdgf

TD 3560

SOVENT CDC13

DS 32

SWH 3184.713 Hz

FIDRES 0.894582 Hz

AQ 0.5589700 sec

RG 2050

DW 157.000 usec

DE 6.50 usec

TE 295.7 K

CNSTR13 8.0000000

D0 0.00000300 sec

D1 1.32463396 sec

D6 0.06250000 sec

D16 0.00010000 sec

INO 0.00002235 sec

===== CHANNEL f1 =====

NUC1 1H

P1 10.00 usec

P2 20.00 usec

PL1 -3.60 dB

PL1W 18.98951721 W

SFO1 400.2316952 MHz

===== CHANNEL f2 =====

NUC2 13C

P3 8.40 usec

PL2 -2.50 dB

PL2W 60.30228424 W

SFO2 100.6479613 MHz

===== GRADIENT CHANNEL =====

GENAM1 SINE 100

GENAM2 SINE 100

GENAM3 SINE 100

GFZ1 50.00 %

GFZ2 30.00 %

GFZ3 40.10 %

P16 1000.00 usec

ND0 2

TD 128

SFO1 100.648 MHz

FIDRES 174.636047 Hz

SM 222.092 ppm

FMODE QF

SI 1024

SF 400.2300392 MHz

WDW SINE

SSB 0

GB 0.00 Hz