



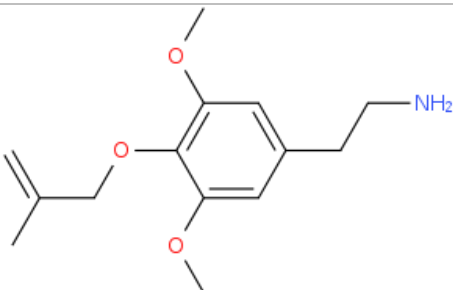
ANALYTICAL REPORT^{1,2}

Methallylescaline (C₁₄H₂₁NO₃)

2-{3,5-dimethoxy-4-[(2-methylprop-2-en-1-yl)oxy]phenyl}ethan-1-amine

Remark – other NPS detected: **none**

Sample ID:	1441-16
Sample description:	crystalinic - white-off
Sample type:	collected /Institute of Forensic medicine, University Freiburg, Germany
Date of sample receipt (M/D/Y):	1/14/2016
Date of entry (M/D/Y) into NFL database:	1/27/2017
Report updates (if any) will be published here:	http://www.policija.si/apps/nfl_response_web/seznam.php

Substance identified - structure ³ (base form)	
Systematic name	2-{3,5-dimethoxy-4-[(2-methylprop-2-en-1-yl)oxy]phenyl}ethan-1-amine
Other names	MAL; 4-methallyloxy-3,5-dimethoxyphenethylamine
Formula (per base form)	C ₁₄ H ₂₁ NO ₃
M _w (g/mol)	251,33
Salt form/anions detected	HCl
StdInChIKey (for base form)	FOXJBFGGULACD-UHFFFAOYSA-N
Other NPS detected	none
Add.info (purity..)	Sample is pure by GC-MS, TOF, and NMR.

¹ This report has been produced with the financial support of the Prevention of and fight against crime Programme of the European Union (grant agreement number JUST/2013/ISEC/DRUGS/AG/6413). The contents of this report are the sole responsibility of the National Forensic Laboratory and can in no way be taken to reflect the views of the European Commission.

² Acknowledgement: Sample (not NMR confirmed) was kindly provided by the Institute of Forensic Medicine, University of Freiburg, Germany (Dr. Verena Angerer).

³ Created by OPSIN free tool: <http://opsin.ch.cam.ac.uk/> DOI: 10.1021/ci100384d

Report updates

date	comments (explanation)

Instrumental methods (if applied) in NFL

1. GC-MS (Agilent): GC-method is RT locked to tetracosane (9.258 min). Injection volume 1 ml and split mode (1:50). Injector temperature: 280 °C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickness 0.25 µm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 °C for 1 min, followed by heating up to 190 °C at rate 8 °C/min, then heating up to 293 °C at a rate of 18 °C/min, hold for 7.1 min, then heating at 50 °C/min up to 325 °C and finally 6.1 min isothermal. MSD source EI = 70 eV. GC-MS transfer line T= 235 °C, source and quadropole temperatures 280 °C and 180 °C, respectively. Scan range m/z scan range: from 50 (30 until 6 min.) to 550 (300 until 6 min) amu.

2. HPLC-TOF (Agilent): 6230B TOF with Agilent 1260 Infinity HPLC with binary pump, column: Zorbax Eclipse XDB-C18, 50 x 4.6 mm, 1.8 micron. Mobile phases (A) 0.1% formic acid and 1mM ammonium formate in water; (B) 0.1% formic acid in methanol (B). Gradient: starting at 5% B, changing to 40% B over 4 min, then to 70% over 2 min and in 5 min to 100%, hold 1 min and back to 5%, equilibration for 1.7 min. The flow rate: 1.0 ml/min; Injection volume 1 µl. MS parameters: 2GHz, Extended Dynamic range mode to a maximum of 1700 amu, acquisition rate 1.30 spectra/sec. Sample ionisation: by Agilent Jet Stream technology (Dual AJS ESI). Ion source: positive ion scan mode with mass scanning from 82 to 1000 amu. Other TOF parameters: drying gas (N₂) and sheath temperature 325 °C; drying gas flow rate 6 l/min; sheath gas flow rate 8 l/min; nebulizer 25 psig; Vcap. 4000 V; nozzle 2000 V; skimmer 65 V; fragmentor 175 V and Octopole RF 750 V.

3. FTIR-ATR (Perkin Elmer): scan range 4000-400 cm⁻¹; resolution 4cm⁻¹

4. GC- (MS)-IR condensed phase (GC-MS (Agilent) & IR (Spectra analyses-Danny)

GC-method: Injection volume 1 ml and split mode (1:5). Injector temperature 280 °C. Chromatographic separation as above (**1**). Split MS : IR = 1: 9.

MSD source EI = 70 eV. GC-MS transfer line T= 235 °C, source and quadropole temperatures 280 °C and 180 °C, respectively. Scan range m/z scan range: from 50 (30 until 6 min.) to 550 (300) amu.

IR (condensed (solid) phase): IR scan range 4000 to 650, resolution 4 cm⁻¹.

5. IC (anions) (Thermo Scientific, Dionex ICS 2100), Column: IonPac AS19, 2 x 250mm; Eluent: 10mM from 0 to 10 min, 10-58 mM from 10 to 40min; Flow rate: 0.25 ml/min; Temperature: 30 °C; Suppressor: AERS 500 2mm, suppressor current 13mA; Inj. Volume: 25 µl

Supporting information

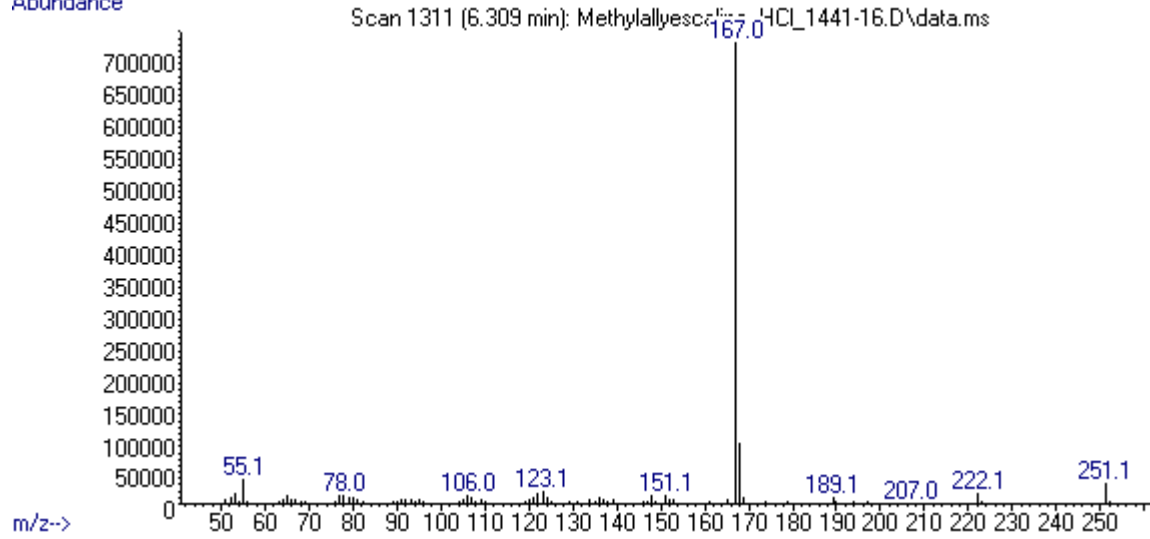
Solubility in	result/remark
CH ₂ Cl ₂	soluble
MeOH	soluble
H ₂ O	soluble

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 6,31 BP(1): 167; BP(2): 168,BP(3) :55,
HPLC-TOF	+	Exact mass (theoretical): 251,1521; measured value Δppm:-1,32; formula:C14H21NO3
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		MS consistent by the one published in ENFSI2015.L
other		

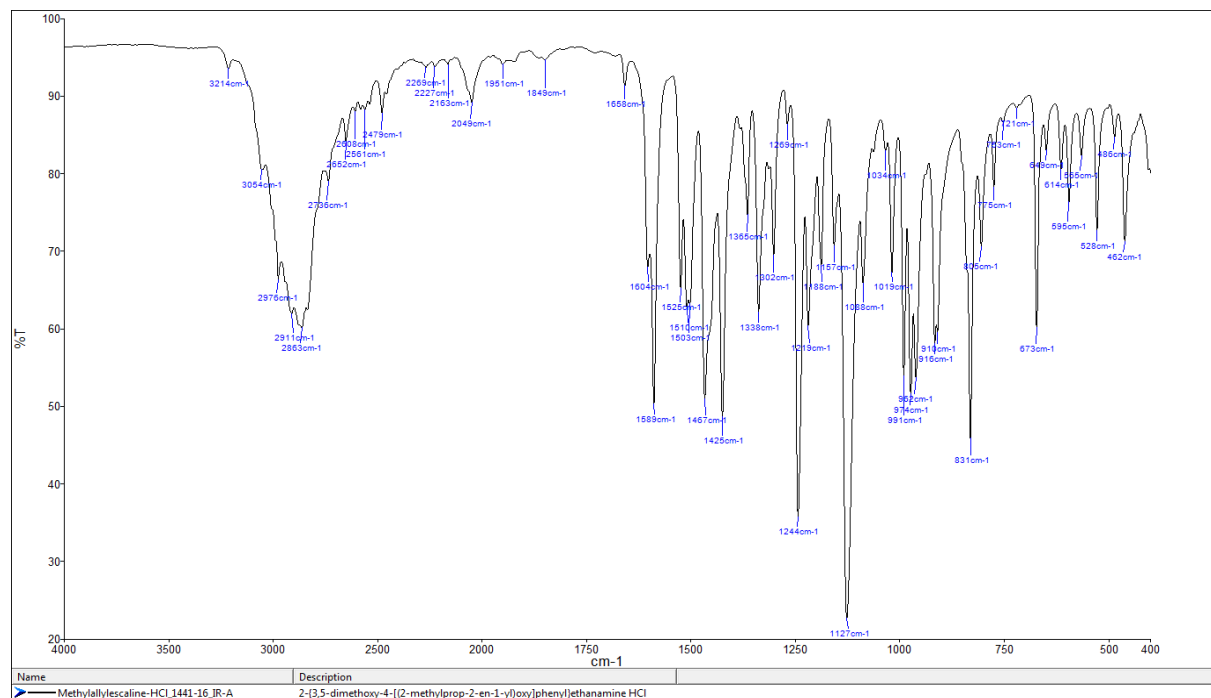
ANALYTICAL RESULTS

MS (EI)

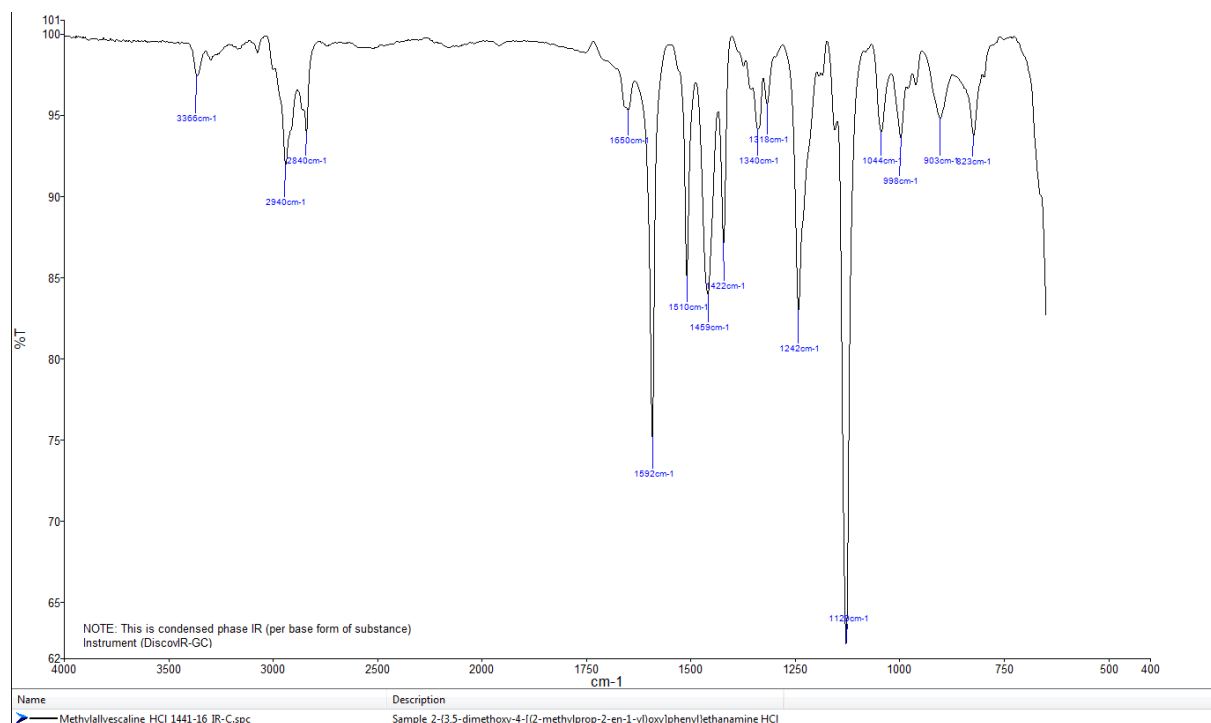
Abundance



FTIR-ATR - direct measurement (sample as received)



IR (condensed phase – after chromatographic separation)



TOF REPORT

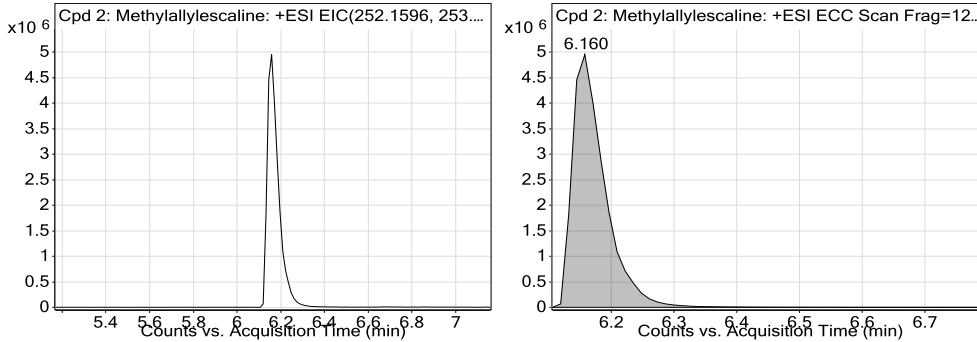
Data File	Methylallylescaline_1441-16_TOF.d	Sample Name	ID_1441-16
Sample Type	Sample	Position	P1-E8
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-1512015-XDB-C18-ESI-poz-pod.m	Acquired Time	2/23/2016 10:06:32 AM
IRM Calibration Status	Success	DA Method	Drugs_NFL.m
Comment	extract in MeOH		

Compound Table

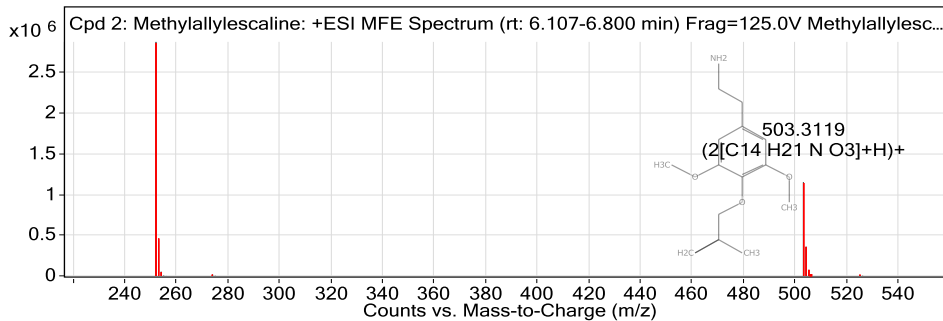
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 2: Methylallylescaline	Methylallylescaline	C14 H21 N O3	6.16	251.1525

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Methylallylescaline	252.1594	6.16	251.1525	6.16	C14 H21 N O3	251.1521	-1.32

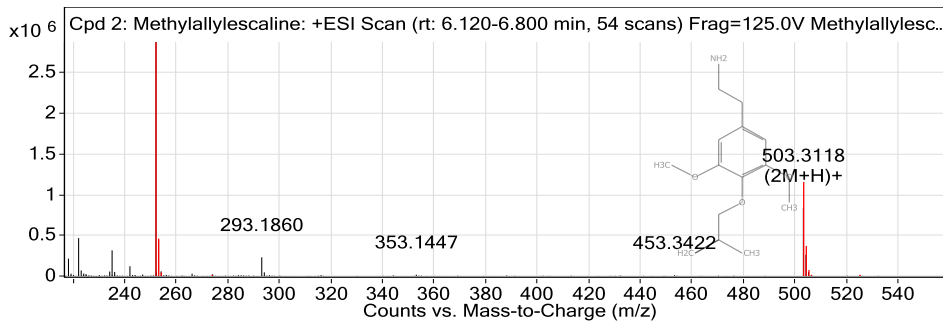
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

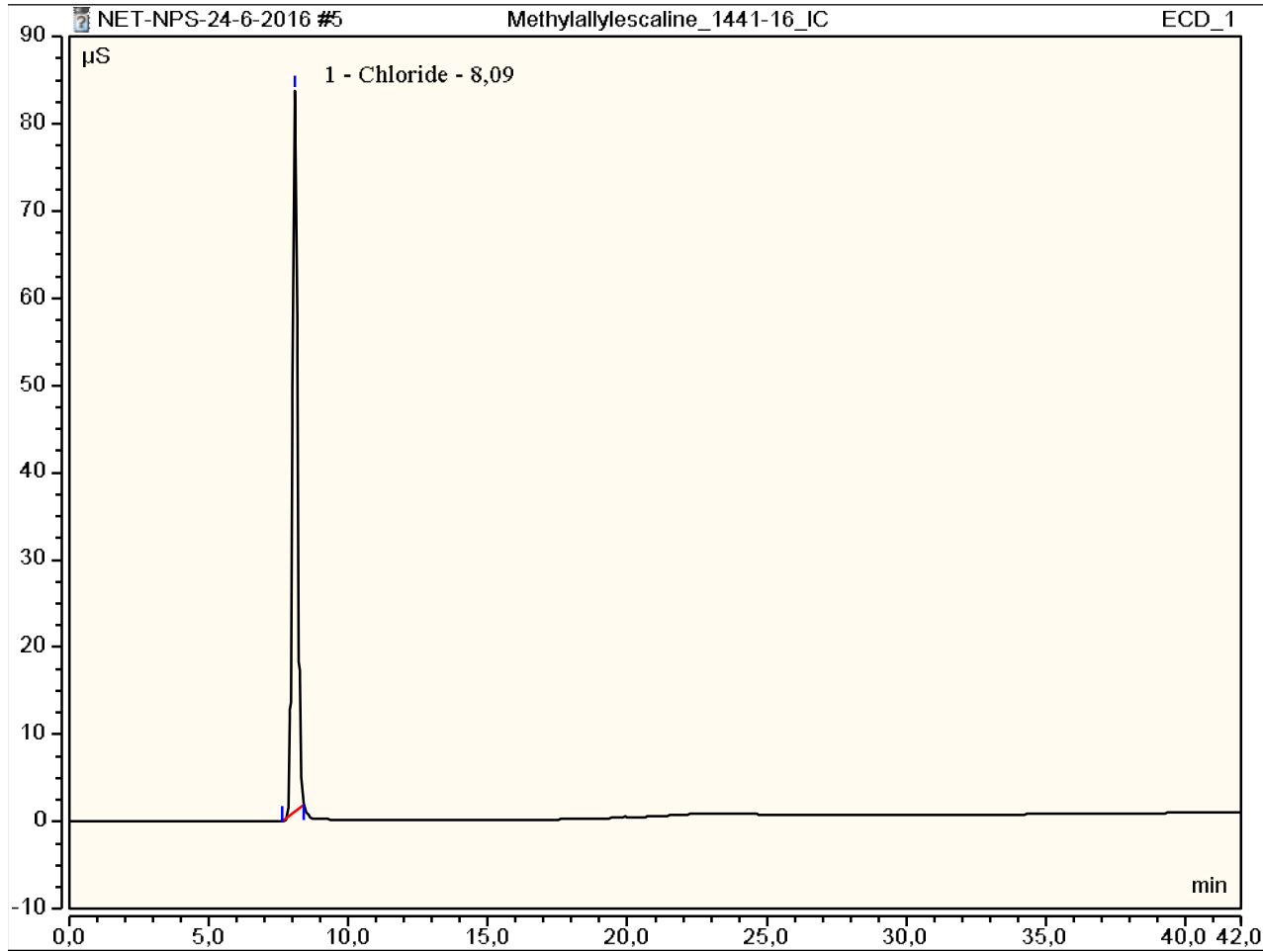
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
252.1594	1	2876514	C14 H21 N O3	(M+H)+
253.1634	1	431380.97	C14 H21 N O3	(M+H)+
254.1651	1	51281.76	C14 H21 N O3	(M+H)+
255.1662	1	4864.79	C14 H21 N O3	(M+H)+
274.1413	1	16358.99	C14 H21 N O3	(M+Na)+
503.3119	1	1150321.88	C14 H21 N O3	(2M+H)+
504.3153	1	358420.26	C14 H21 N O3	(2M+H)+
505.3171	1	65920.84	C14 H21 N O3	(2M+H)+
506.3191	1	9158.12	C14 H21 N O3	(2M+H)+
525.2926	1	7810.33	C14 H21 N O3	(2M+Na)+

--- End Of Report ---

Peak Integration Report

Sample Name:	Methylallylescaline_1441-16_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	24-jun-2016 / 14:23	Run Time:	42,00

No.	Time min	Peak Name	Peak Type	Area $\mu\text{S}\cdot\text{min}$	Height μS	Amount mg/L
1,00	8,09	Chloride	BMB	17,22	82,64	n.a.
TOTAL:				17,22	82,64	0,00





REPORT

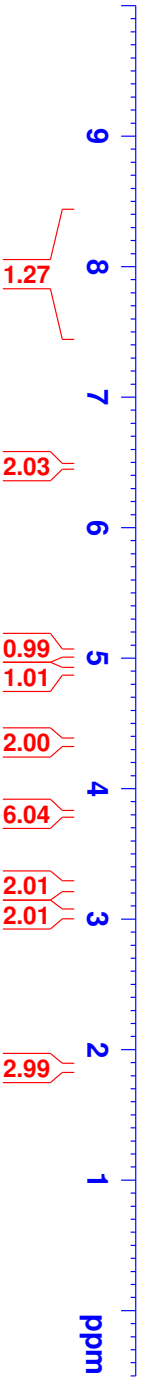
Sample ID:	1441-16
Our notebook code:	P-1441-16
NMR sample preparation:	15 mg dissolved in 0.7 mL CDCl ₃
NMR experiments:	¹ H, ¹³ C, ¹ H- ¹ H <i>gs</i> -COSY, ¹ H- ¹³ C <i>gs</i> -HSQC, ¹ H- ¹³ C <i>gs</i> -HMBC, ¹ H- ¹⁵ N <i>gs</i> -HMBC.
Proposed structure:	
Chemical name:	2-(3,5-dimethoxy-4-((2-methylallyl)oxy)phenyl)ethan-1-amine
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Sample is pure according to the NMR.
Supporting information:	Copies of ¹ H and ¹³ C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	January 23, 2017



Current Data Parameters
 NAME p-1441-16
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160807
 Time 11.12
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10000.000 Hz
 FIDRES 0.152588 Hz
 AQ 3.2768500 sec
 RG 80.6
 DW 50.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 500.1330885 MHz
 NUQ1 1H
 P1 8.90 usec
 PLW1 26.00000000 W
 F2 - Processing parameters
 SI 65536
 SF 500.1300076 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





Current Data Parameters
 NAME P-1441-16
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160807
 Time 14.55
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 4096
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010048 sec
 RG 2050
 DW 16.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 ID0 1

==== CHANNEL f1 =====
 SF01 125.7703637 MHz
 NUC1 13C
 P1 9.00 usec
 P1M1 122.00000000 W
 ===== CHANNEL f2 =====
 SF02 500.1320005 MHz
 NUC2 1H
 CPDPRG12 waltz16
 PCPD2 80.00 usec
 P1M2 26.00000000 W
 P1M12 0.32179001 W
 P1M13 0.16186000 W
 F2 - Processing parameters
 SI 32768
 SF 125.7577878 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

