



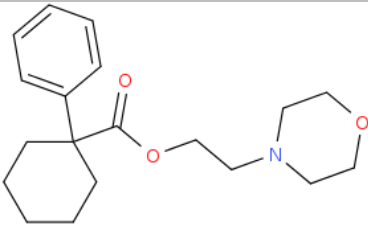
ANALYTICAL REPORT¹

PRE-084 (
C19H27NO3)

2-(morpholin-4-yl)ethyl 1-phenylcyclohexane-1-carboxylate

Remark – other NPS detected: **none**

Sample ID:	1544-16
Sample description:	powder - white
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	3/8/2016
Date of entry (M/D/Y) into NFL database:	4/18/2016
Report updates (if any) will be published here:	http://www.policija.si/apps/nfl_response_web/seznam.php

Substance identified - structure ² (base form)	
Systematic name	2-(morpholin-4-yl)ethyl 1-phenylcyclohexane-1-carboxylate
Other names	Pre-084; ChEMBL305881; 2-morpholin-4-ylethyl 1-phenylcyclohexane-1-carboxylate; Pre 084; Tocris-0589; Lopac-P-2607
Formula (per base form)	C19H27NO3
M _w (g/mol)	317,43
Salt form/anions detected	hydrochloride
StdInChIKey	RQHKZUBCUZVZEF-UHFFFAOYSA-N
Compound Class	Others
Other NPS detected	none
Add.info (purity..)	pure by GC, HPLC-TOF, minor impurities by 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 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 (RT=9.53 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 mm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 °C for 1 min, followed by heating up to 293 °C at a rate of 18 °C/min, hold for 6.1 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) 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 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

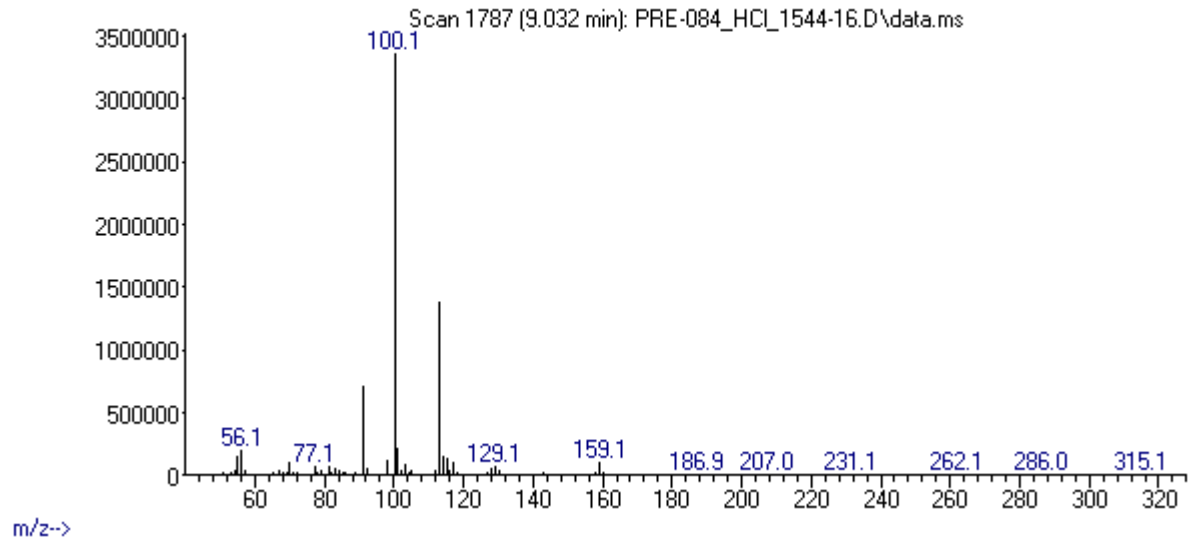
Solubility in	result/remark
CH ₂ Cl ₂	soluble
MeOH	soluble
H ₂ O	soluble

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 9,03 BP(1): 100; BP(2): 113,BP(3) :91,
HPLC-TOF	+	Exact mass (theoretical): 317,1991; measured value Δppm:-0,77; formula: C ₁₉ H ₂₇ NO ₃
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	after chromatographic separation
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

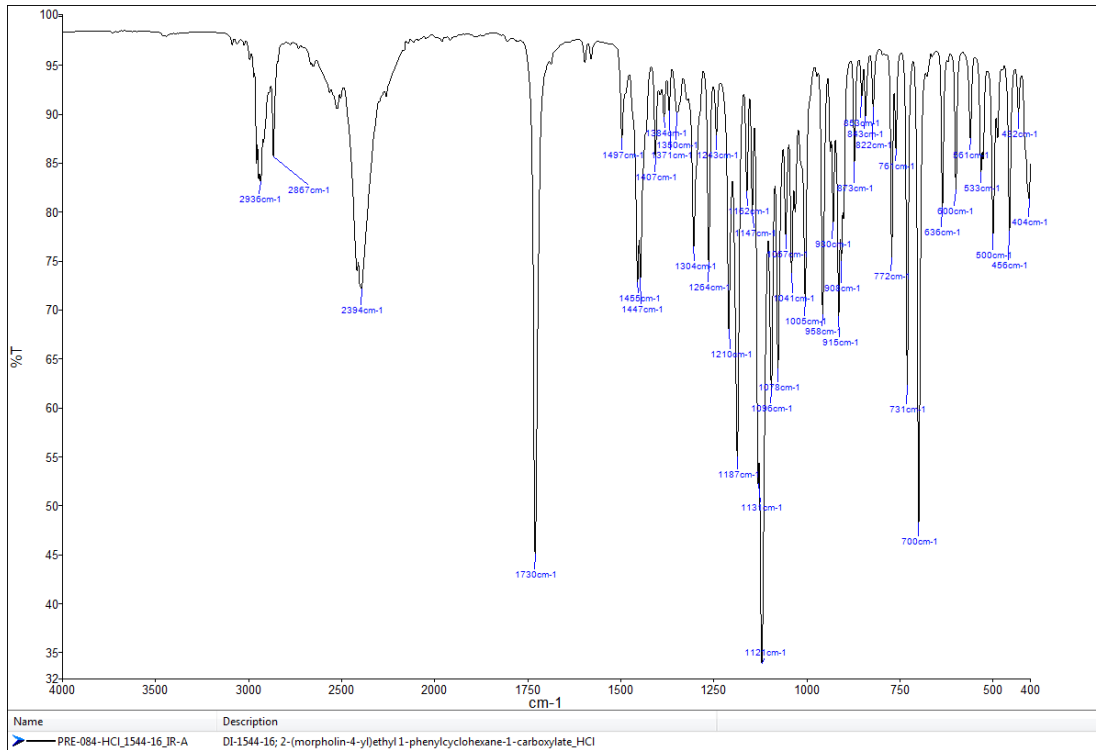
ANALYTICAL RESULTS

MS (EI)

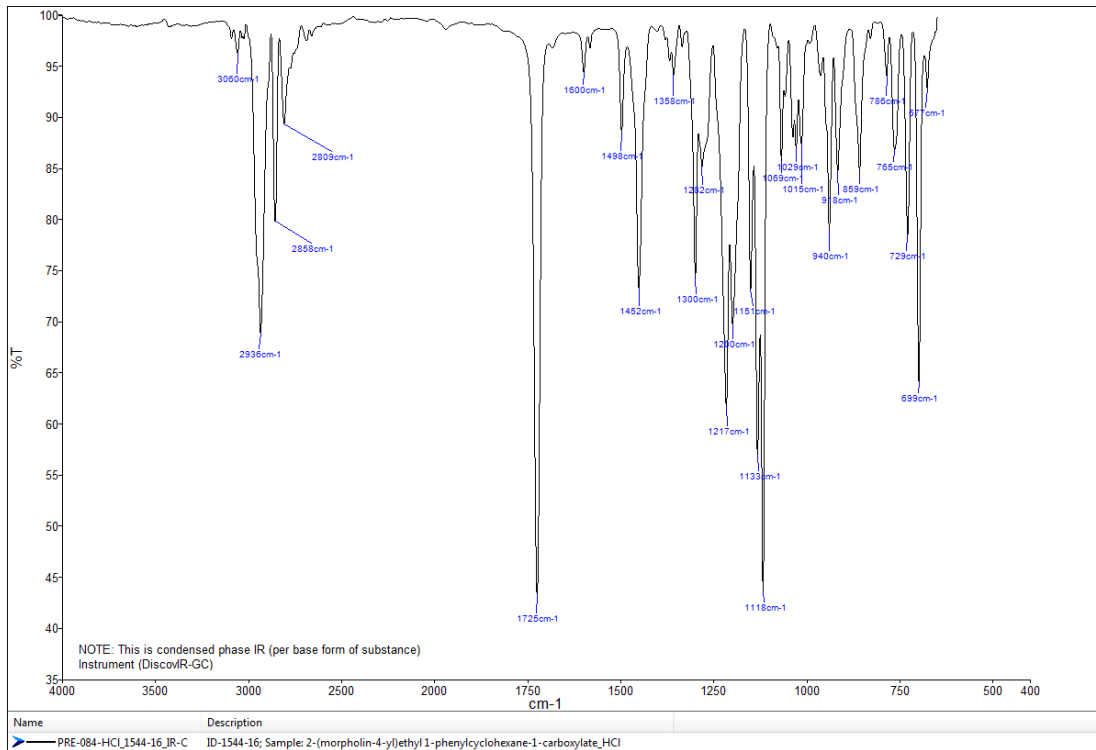
Abundance



FTIR-ATR - direct measurement (sample as received)



IR (condensed phase – after chromatographic separation)



TOF REPORT

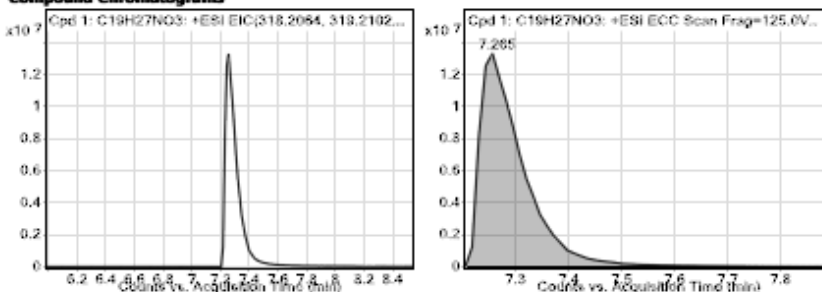
Data File	PRE-084_1544-16_TOF.d	Sample Name	ID_1544-16
Sample Type	Sample	Position	P1-E4
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-1512015-XDB-C18-ESI-poz-pod.m	Acquired Time	3/13/2016 10:52:27 AM
IRM Calibration Status	Success	DA Method	Drugs_NFL.m
Comment	extract in MeOH		

Compound Table

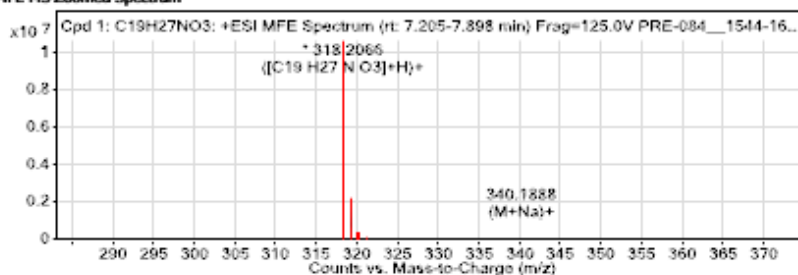
Label	Compound Name	Obs. RT	Obs. Mass
Cpd 1: C19H27NO3	C19H27NO3	7.265	317.1993

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
C19H27NO3	318.2066	7.265	317.1993	7.27	C19 H27 N O3	317.1991	-0.77

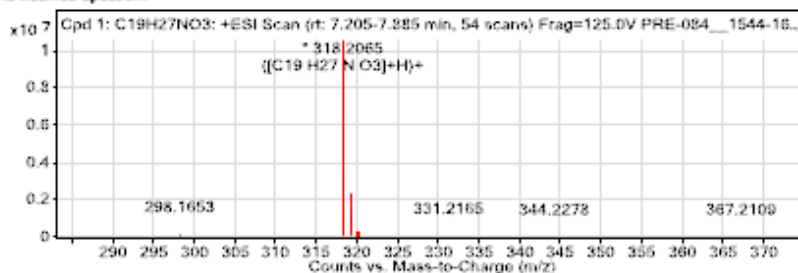
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

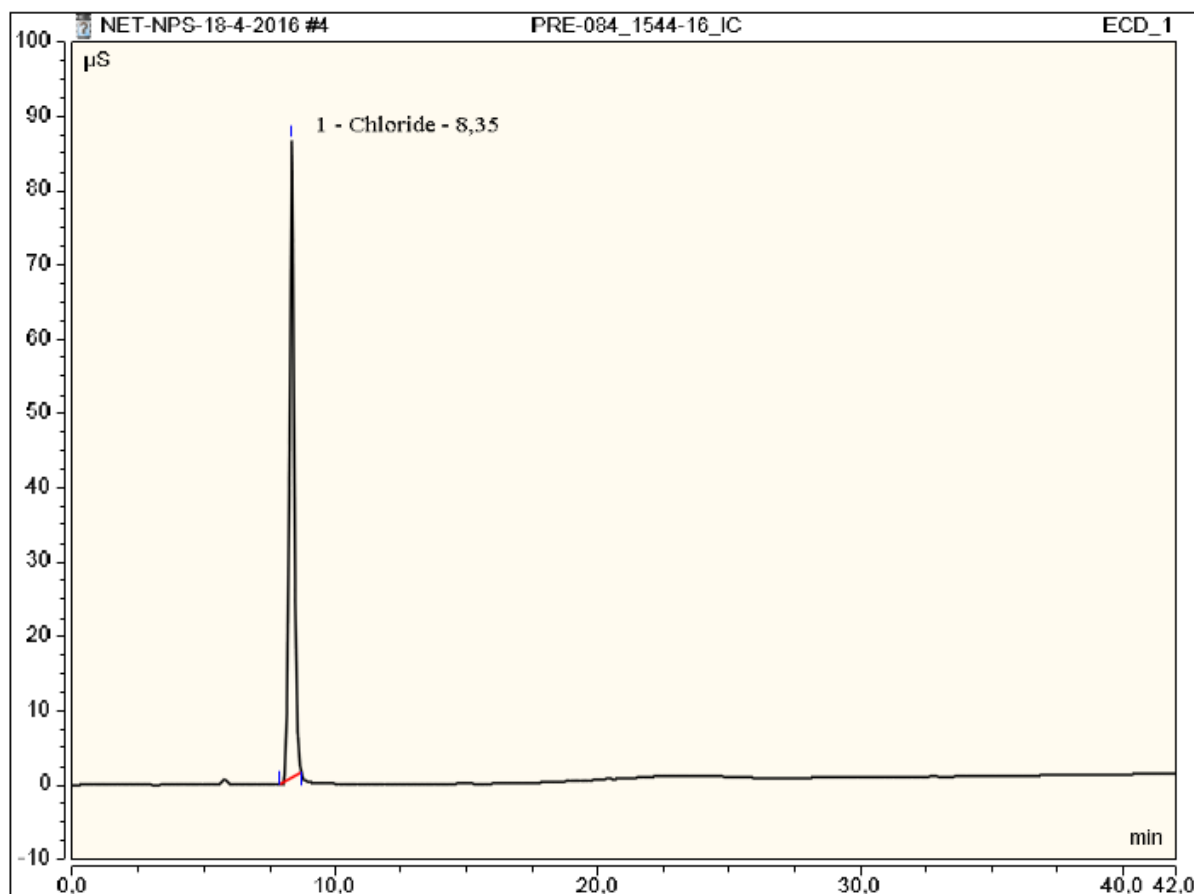
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
318.2066	1	10578969	C19 H27 N O3	(M+H)+
319.21	1	2220091.94	C19 H27 N O3	(M+H)+
320.2131	1	266340.17	C19 H27 N O3	(M+H)+
321.2153	1	25868.35	C19 H27 N O3	(M+H)+
322.2175	1	820.34	C19 H27 N O3	(M+H)+
340.1888	1	2508.58		(M+Na)+

--- End Of Report ---

Peak Integration Report

Sample Name:	PRE-084_1544-16_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	18-apr-2016 / 15:50	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	8,35	Chloride	BMB	18,66	85,75	n.a.
		TOTAL:		18,66	85,75	0,00





REPORT

Sample ID:	1544-16
Our notebook code:	P-1544-16
NMR sample preparation:	15 mg dissolved in 0.7 mL DMSO- d_6
NMR experiments:	^1H , ^{13}C , ^1H - ^1H <i>gs</i> -COSY, ^1H - ^{13}C <i>gs</i> -HSQC, ^1H - ^{13}C <i>gs</i> -HMBC, ^1H - ^{15}N <i>gs</i> -HMBC.
Proposed structure:	
Chemical name:	4-(2-((1-phenylcyclohexane-1-carbonyl)oxy)ethyl)morpholin-4-ium cation
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Sample contains some minor impurities as evident in ^1H NMR.
Supporting information:	Copies of ^1H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	April 11, 2016

P-1544-16



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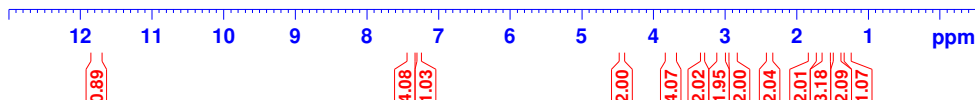
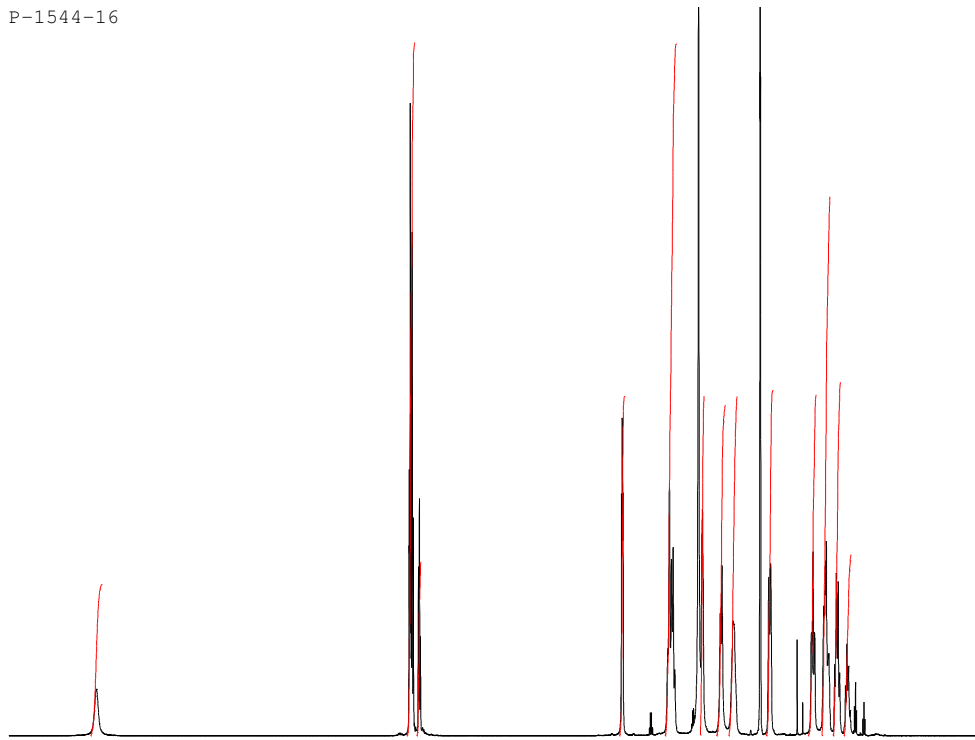
Current Data Parameters
NAME          P-1544-16
EXPNO         1
PROCNO        1

F2 - Acquisition Parameters
Date_         20160327
Time          12.01
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SWH           10000.000 Hz
FIDRES        0.152588 Hz
AQ            3.2768500 sec
RG            64
DW            50.000 usec
DE            6.50 usec
TE            300.0 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
SFO1          500.1330885 MHz
NUC1           1H
P1             8.90 usec
PLW1          26.00000000 W

F2 - Processing parameters
SI            65536
SF            500.1300000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00

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P-1544-16



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Current Data Parameters
NAME          P-1544-16
EXPNO         3
PROCNO        1

F2 - Acquisition Parameters
Date_         20160327
Time          14.36
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
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            1.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
SFO1          125.7703637 MHz
NUC1           13C
P1             9.00 usec
PLW1          122.00000000 W

===== CHANNEL f2 =====
SFO2          500.1320005 MHz
NUC2           1H
CPDPRG[2]     waltz16
PCPD2         80.00 usec
PLW2          26.00000000 W
PLW12         0.32179001 W
PLW13         0.16186000 W

F2 - Processing parameters
SI            32768
SF            125.7577885 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40

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