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RESPONSE 2 to challenges in forensic drugs analyses

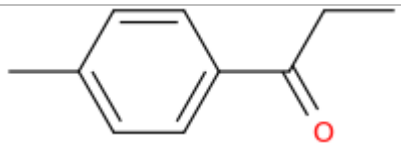
ANALYTICAL REPORT

4'-Methylpropiophenone (C10H12O)

1-(4-methylphenyl)propan-1-one

Remark – other NPS detected: none

Sample ID:	2063-19
Sample description:	liquid
Sample type:	seized /KR
Date of entry (DD/MM/YYYY) into NFL database:	13/08/2019
Report updates (if any) will be published here:	http://www.policija.si/apps/nfl_response_web/seznam.php

Substance identified - structure[1] (base form)	
Systematic name	1-(4-methylphenyl)propan-1-one
Other names	4'-Methylpropiophenone; 4-Methylpropiophenone; 1-(p-tolyl)propan-1-one; p-Methylpropiophenone; p-Methylpropiophenone; 1-(4-methylphenyl)propan-1-one; 1-Propanone, 1-(4-methylphenyl)-; p-Tolyl ethyl ketone
Formula (per base form)	C10H12O
M_w (g/mol)	148,2
Salt form/anions detected	base
StdInChIKey (per base form)	PATYHUUYADUHQS-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	>98% purity of a sample, based on 1H NMR spectra

[1] 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 0C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickens 0.25 µm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 ^{0}C for 1 min, followed by heating up to 190 ^{0}C at rate 8 ^{0}C/min, then heating up to 293 0C at a rate of 18 ^{0}C/min, hold for 7.1 min, then heating at 50 ^{0}C/min up to 325 ^{0}C and finally 6.1 min isothermal. MSD source EI = 70 eV. GC-MS transfer line T= 235^{0}C, source and quadropole temperatures 280^{0}C and 180^{0}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 ^{0}C. Chromatographic separation as above **(1).** Split MS : IR = 1: 9.
MSD source EI = 70 eV. GC-MS transfer line T= 235^{0}C, source and quadropole temperatures 280^{0}C and 180^{0}C, respectively. Scan range m/z scan range: from 50 (30 until 6 min.) to 550 (300) amu.
IR (condesed (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 KOH 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

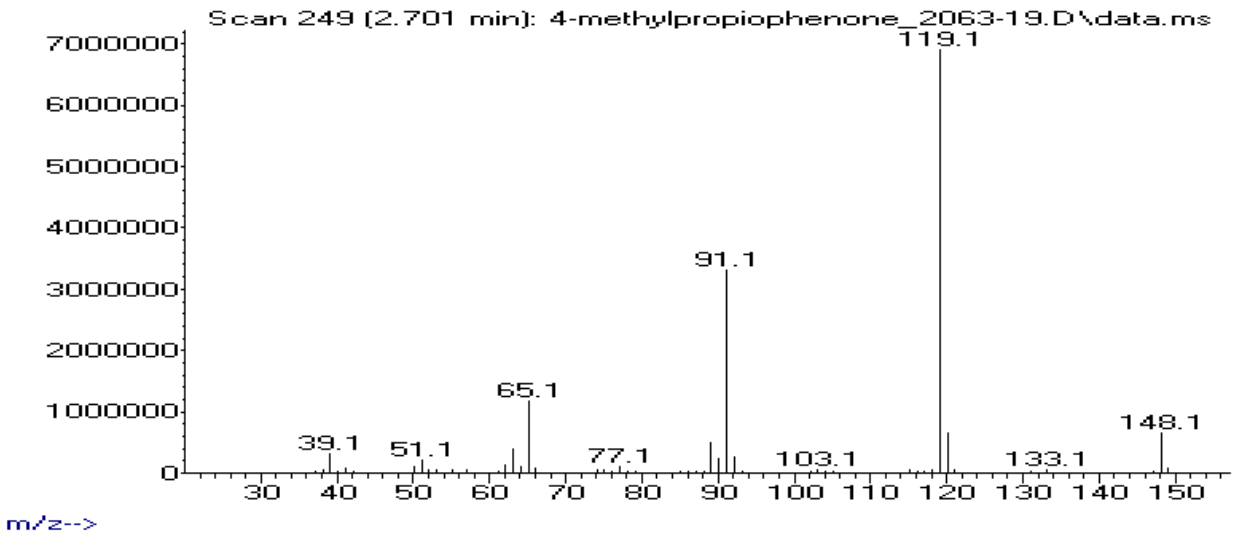
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
H_2O	not tested

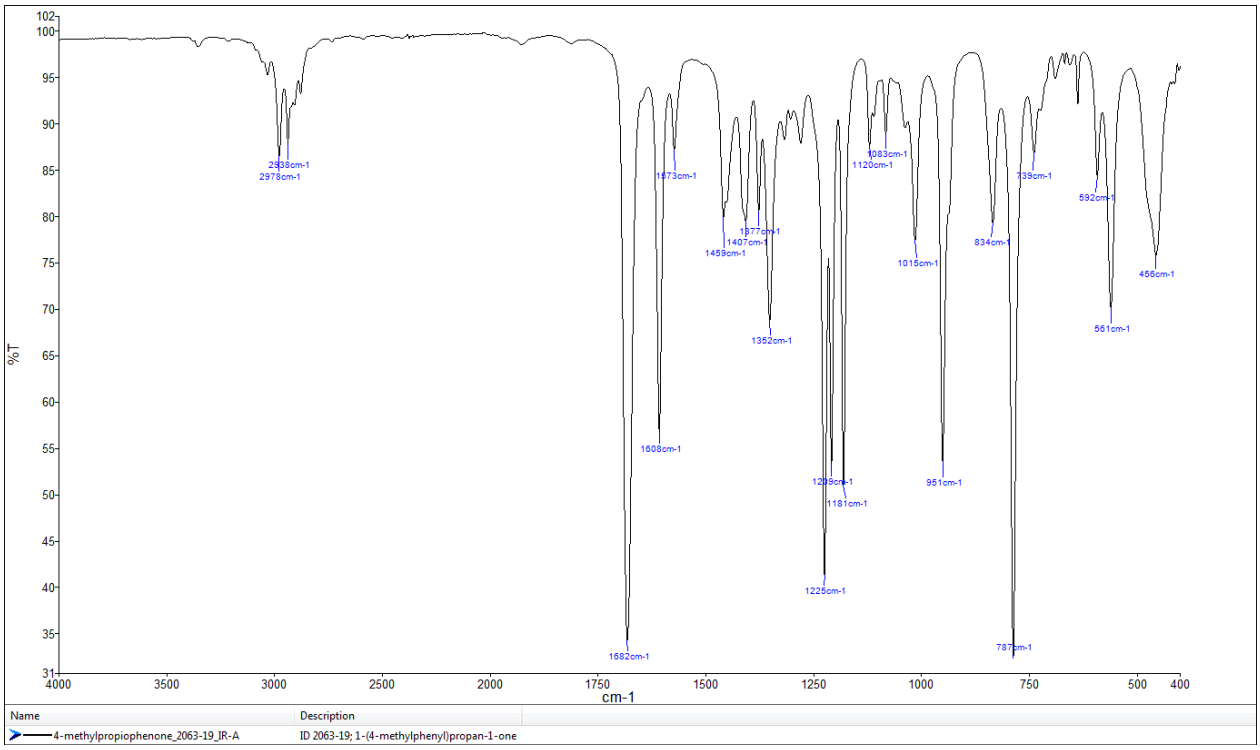
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 2,7 BP(1): 119; BP(2): 91,BP(3) :65,
HPLC-TOF	+	Exact mass (theoretical): 148,0888; measured value Δppm:-3,2; formula:C10H12O
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	-	
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

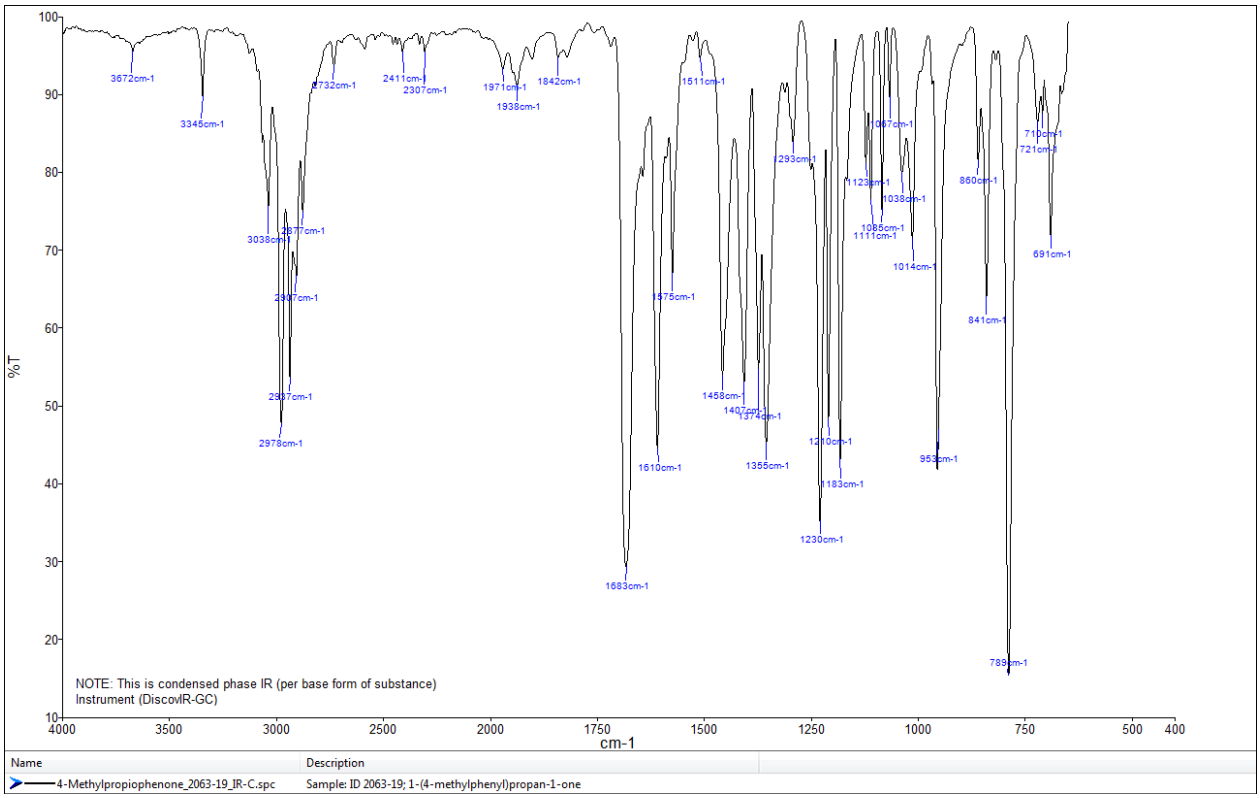
MS (EI)



FTIR-ATR - direct measurement (sample as received)



IR (solid phase – after chromatographic separation)



TOF REPORT

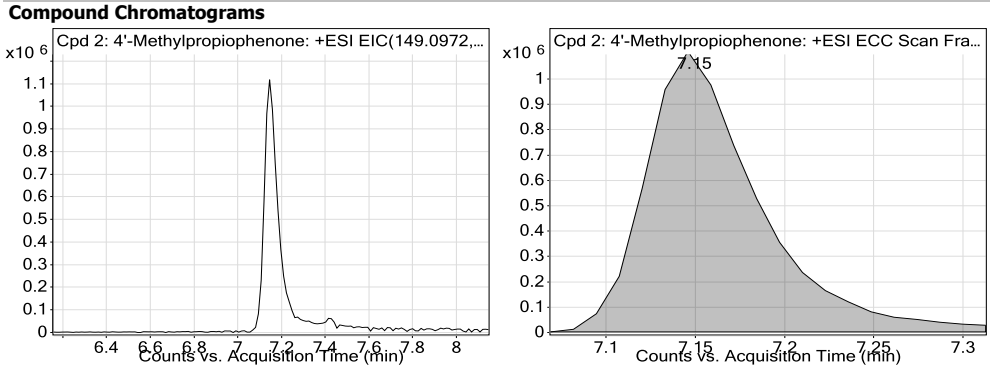
Data File	233-1094-19_1.d	**Sample Name**	vzorec 1
Sample Type	Sample	**Position**	P1-A2
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-8_1_2019-XDB-C18-ESI+.m	**Acquired Time**	3/20/2019 10:42:43 AM
IRM Calibration Status	Success	**DA Method**	a-Drugs_NFL.m
Comment	extract in MeOH		

Compound Table

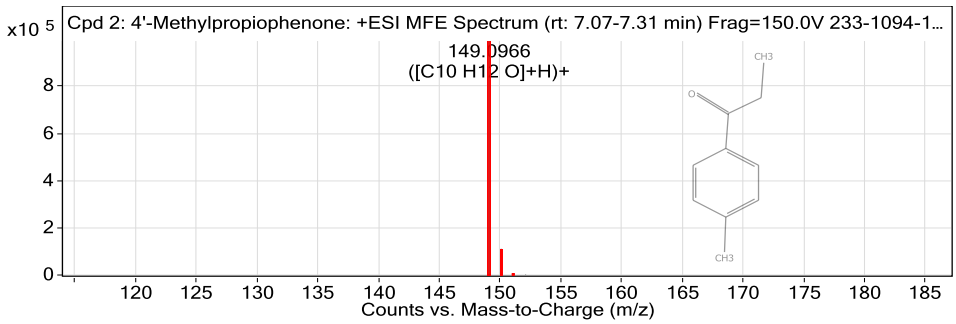
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 2: 4'-Methylpropiophenone	4'-Methylpropiophenone	C10 H12 O	7.15	148.0893

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
4'-Methylpropiophenone	149.0966	7.15	148.0893	7.15	C10 H12 O	148.0888	-3.2

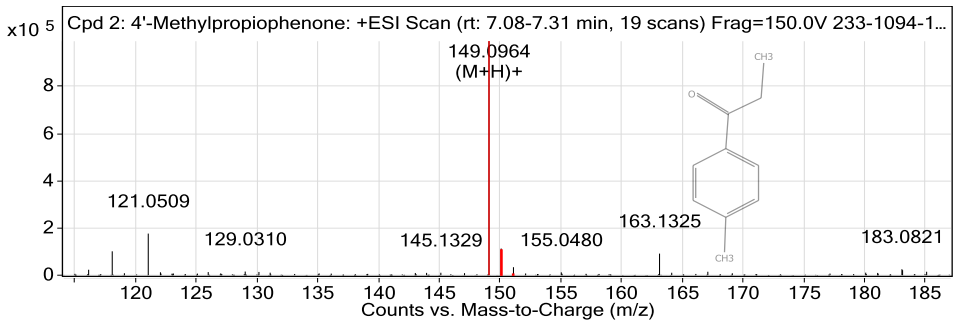
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
149.0966	1	991061.31	C10 H12 O	(M+H)+
150.0996	1	110851.85	C10 H12 O	(M+H)+
151.1079	1	8362.38	C10 H12 O	(M+H)+
152.1101	1	1113.03	C10 H12 O	(M+H)+

--- End Of Report ---

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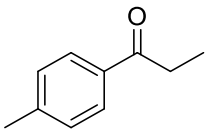
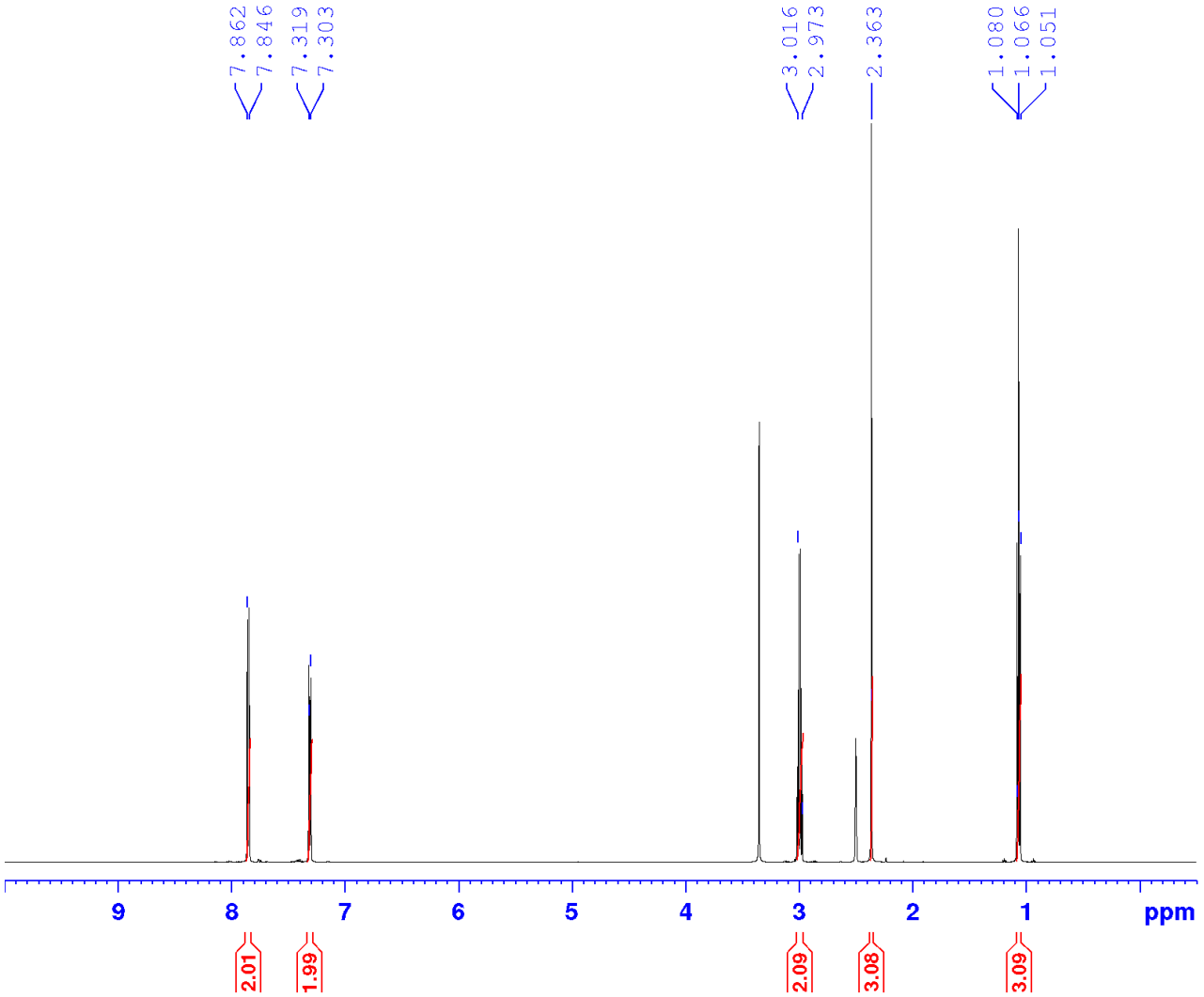
University
of Ljubljana

Faculty *of Chemistry*
and Chemical Technology

R E P O R T

Contract No.	C1714-17-460078 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	**233-1094-19_1**
Received date:	April 1, 2019
Our notebook code:	NFL-233-1094-19_1
NMR sample preparation:	21.0 mg dissolved in 0.7 mL DMSO-d_6
NMR experiments:	^{1}H, ^{13}C, ^{1}H–^{1}H *gs*-COSY, ^{1}H–^{13}C *gs*-HSQC, ^{1}H–^{13}C *gs*-HMBC
Proposed structure with formula, exact mass, molecular weight:	 Chemical Formula: $C_{10}H_{12}O$ Exact Mass: 148,0888 Molecular Weight: 148,2050
Chemical name:	1-(*p*-tolyl)propan-1-one
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. - >98% purity of a sample, based on ^{1}H NMR spectrum.
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra, ^{1}H and ^{13}C FIDs.
Principal investigator:	Prof. Dr. Janez Košmrlj
Date of report:	April 5, 2019

NFL-233-1094-19_1
1H

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Current Data Parameters
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EXPNO                    11
PROCNO                    1

F2 - Acquisition Parameters
Date_            20190401
Time                18.12
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              3.2767999 sec
RG                     57
DW                 50.000 usec
DE                  6.50 usec
TE                 296.0 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          500.1330885 MHz
NUC1                   1H
P1                  8.70 usec
PLW1          26.00000000 W

F2 - Processing parameters
SI                  65536
SF            500.1300045 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
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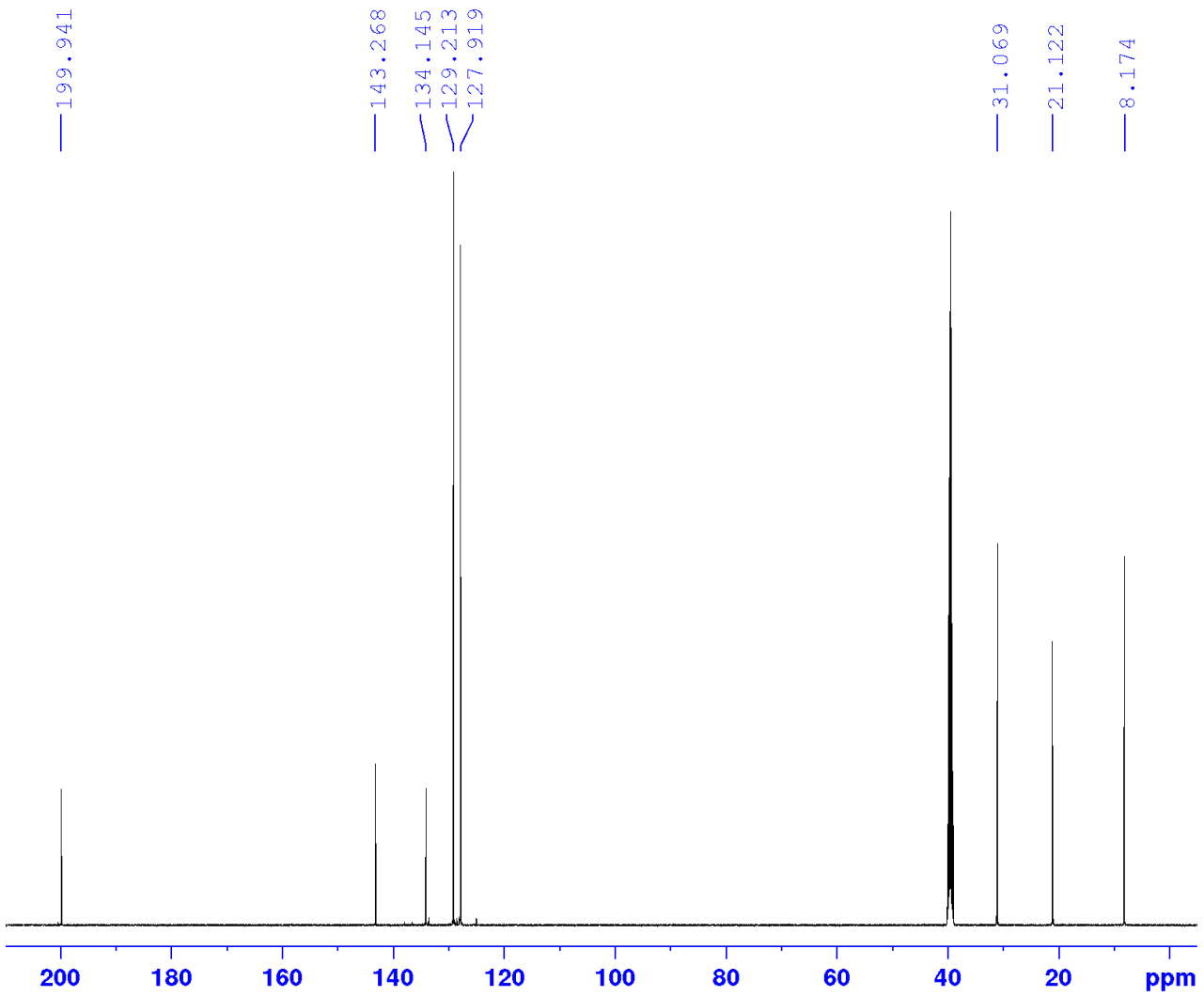
NFL-233-1094-19_1
13C

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EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
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Time                16.38
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT             DMSO
NS                  3072
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               296.0 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                13C
P1                 8.70 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         0.30046001 W
PLW13         0.15113001 W

F2 - Processing parameters
SI                 32768
SF           125.7578458 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
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