NACIONALNI FORENZIČNI LABORATORIJ
NATIONAL FORENSIC LABORATORY
Vodovodna 95, 1000 LJUBLJANA, SLOVENIA
E: nfl@policija.si

RESPONSE 2
to challenges in forensic drugs analyses

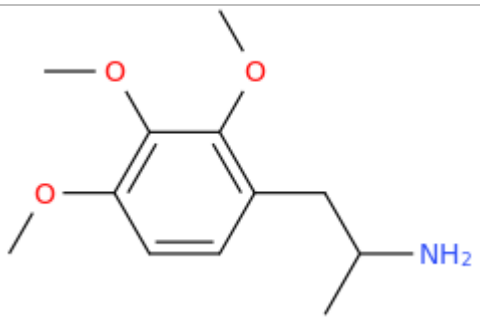
ANALYTICAL REPORT

2,3,4-TMA (C12H19NO3)

1-(2,3,4-trimethoxyphenyl)propan-2-amine

Remark – other active cpd. detected none

Sample ID:	2069-19
Sample description:	powder - white
Sample type:	RM-reference material
Comments:	CAY Lot#0550773-3,
Date of entry (DD/MM/YYYY):	06/06/2019

Substance identified-structure[1] (base form)	
Systematic name:	1-(2,3,4-trimethoxyphenyl)propan-2-amine
Other names:	TMA-3; 2,3,4-Trimethoxyamphetamine; 2,3,4-trimethoxy-α-methyl-benzeneethanamine
Formula (per base form)	C12H19NO3
M_w (g/mol)	225,29
Salt form:	HCl
StdInChIKey (per base form)	LWDQPPLPHGXYLG-UHFFFAOYSA-N
Other active cpd. detected	none
Add.info (purity..)	≥98%

[1] Created by OPSIN free tool: http://opsin.ch.cam.ac.uk/ **DOI:** 10.1021/ci100384d

Report updates

date	comments (explanation)

Supporting information

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 4,61 BP(1): 44; BP(2): 182,BP(3) :167,
FTIR-ATR	+	direct measurement
GC-IR (condensed phase)	+	always as base form
HPLC-TOF	+	exact mass theoretical: 225,1365 / measured Δppm: 3,99

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.

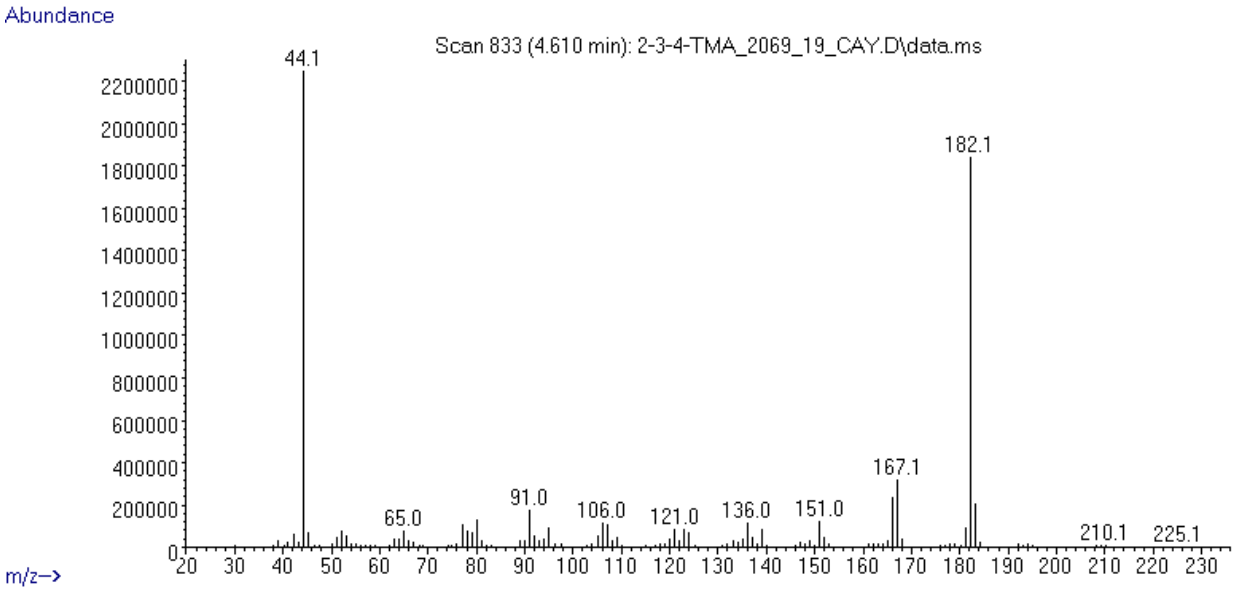
2. FTIR-ATR (Perkin Elmer): scan range 4000-400 cm^{-1}; resolution 4cm^{-1}

3. 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}.

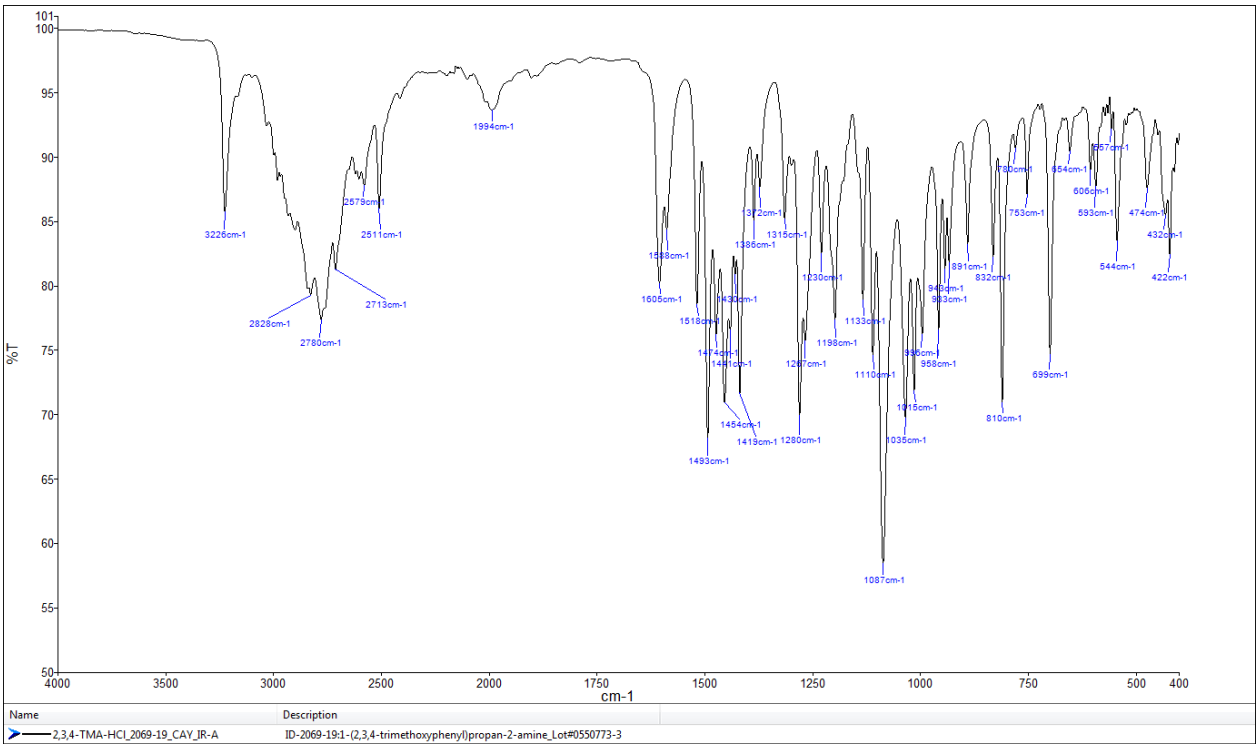
4. **HPLC-TOF** (Agilent): 6230B TOF with Agilent 1260 Infinity HPLC with binary pump, column: Zorbax Eclipse XDB-C18, 50 x 4.6 mm, 1.8 micron. Mobile phases (A) 0.1% formic acid and 1mM ammonium formate in water; (B) 0.1% formic acid in methanol (B). Gradient: starting at 5% B, changing to 40% B over 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.

ANALYTICAL RESULTS

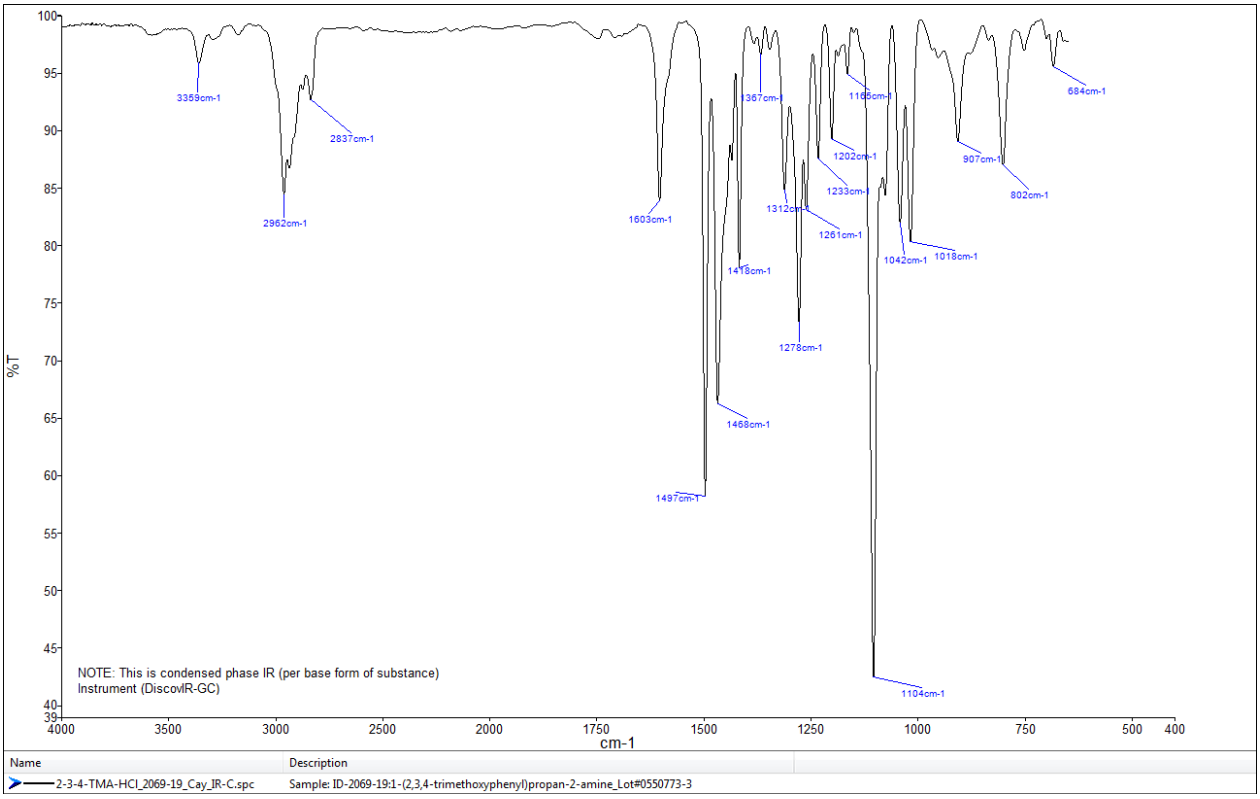
MS (EI)



FTIR-ATR – direct measurement



IR- (condensed (solid) phase – after chromatographic separation) - spectrum per base form



TOF REPORT

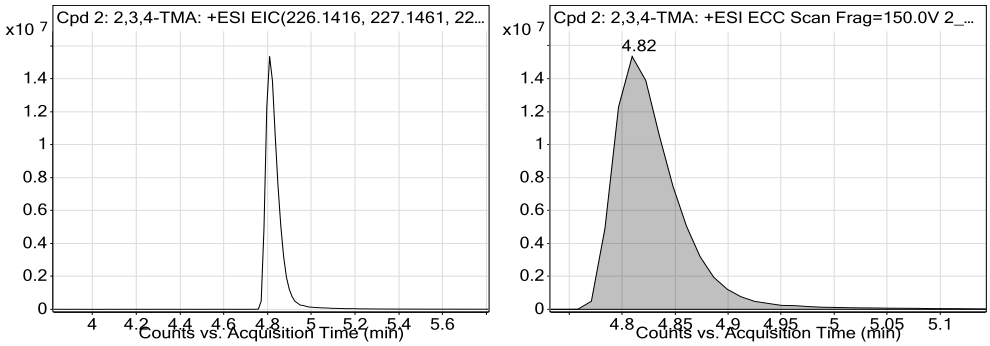
Data File	2_3_4-TMA_2069_19.d	**Sample Name**	ID-2069-19
Sample Type	Sample	**Position**	P1-B5
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-8_1_2019-XDB-C18-ESI+.m	**Acquired Time**	5/14/2019 8:02:21 AM
IRM Calibration Status	Success	**DA Method**	a-Drugs_NFL.m
Comment	MeOH		

Compound Table

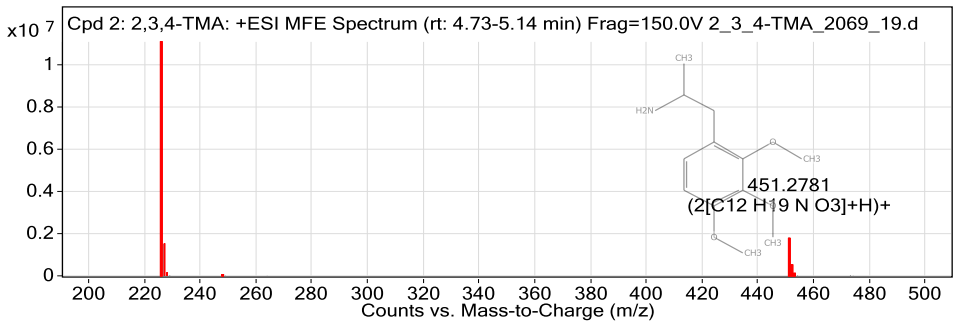
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 2: 2,3,4-TMA	2,3,4-TMA	C12 H19 N O3	4.82	225.1356

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
2,3,4-TMA	226.1431	4.82	225.1356	4.82	C12 H19 N O3	225.1365	3.99

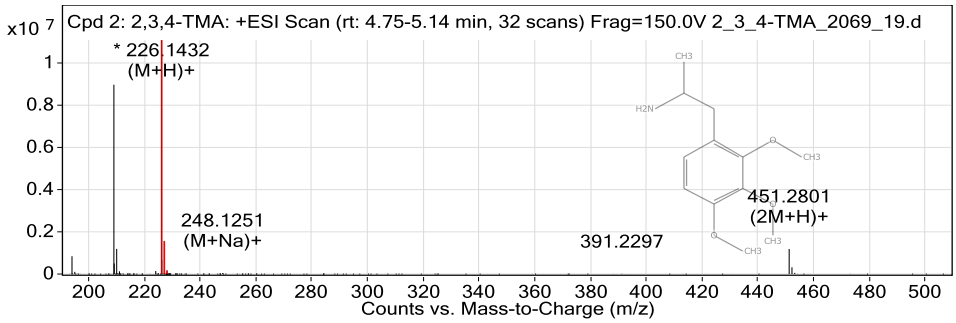
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
226.1431	1	11066632	C12 H19 N O3	(M+H)+
227.1447	1	1508017.89	C12 H19 N O3	(M+H)+
228.1469	1	161165.41	C12 H19 N O3	(M+H)+
229.15	1	14459.12	C12 H19 N O3	(M+H)+
248.123	1	50174.8	C12 H19 N O3	(M+Na)+
451.2781	1	1805327.5	C12 H19 N O3	(2M+H)+
452.2814	1	484655.43	C12 H19 N O3	(2M+H)+
453.2837	1	84737.05	C12 H19 N O3	(2M+H)+
454.2857	1	11782.06	C12 H19 N O3	(2M+H)+
473.2603	1	15234.68	C12 H19 N O3	(2M+Na)+

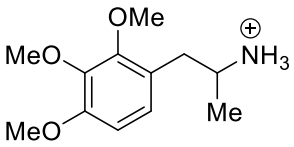
--- End Of Report ---

Večna pot 113
P. O. Box 537
SI-1001 Ljubljana
Slovenia
Phone: +386 1 479 8558
janez.kosmrlj@fkkt.uni-lj.si

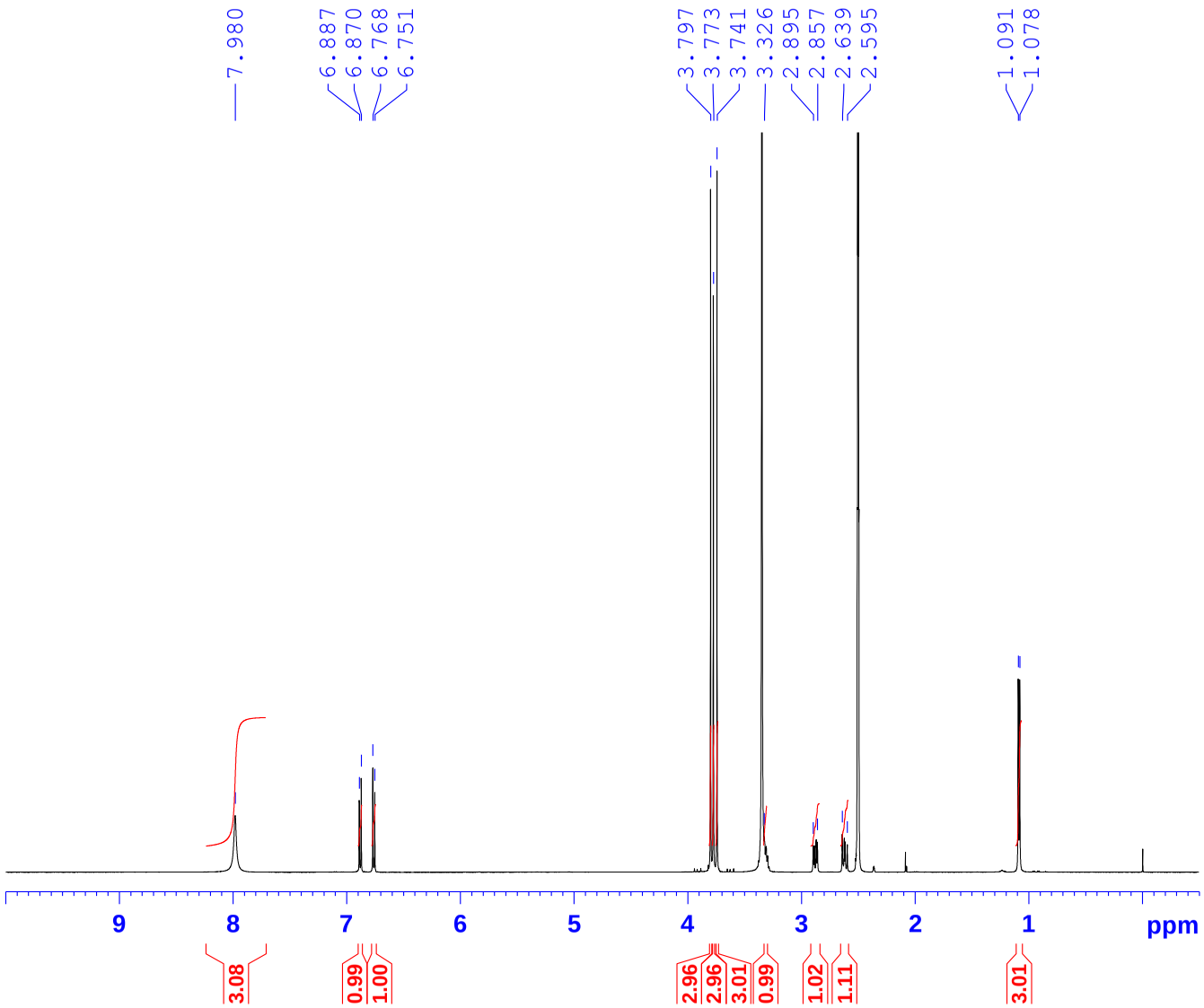
University
of Ljubljana

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:	**2069-19**
Received date:	May 20, 2019
Our notebook code:	NFL-2069-19
NMR sample preparation:	2.9 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, ^{1}H–^{15}N *gs*-HMBC
Proposed structure with formula, exact mass, molecular weight:	 Chemical Formula: $C_{12}H_{20}NO_3^+$ Exact Mass: 226,1438 Molecular Weight: 226,2955
Chemical name:	*N*-protonated 1-(2,3,4-trimethoxyphenyl)propan-2-amine
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. - >96% 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:	May 28, 2019

NFL-2069-19
1H



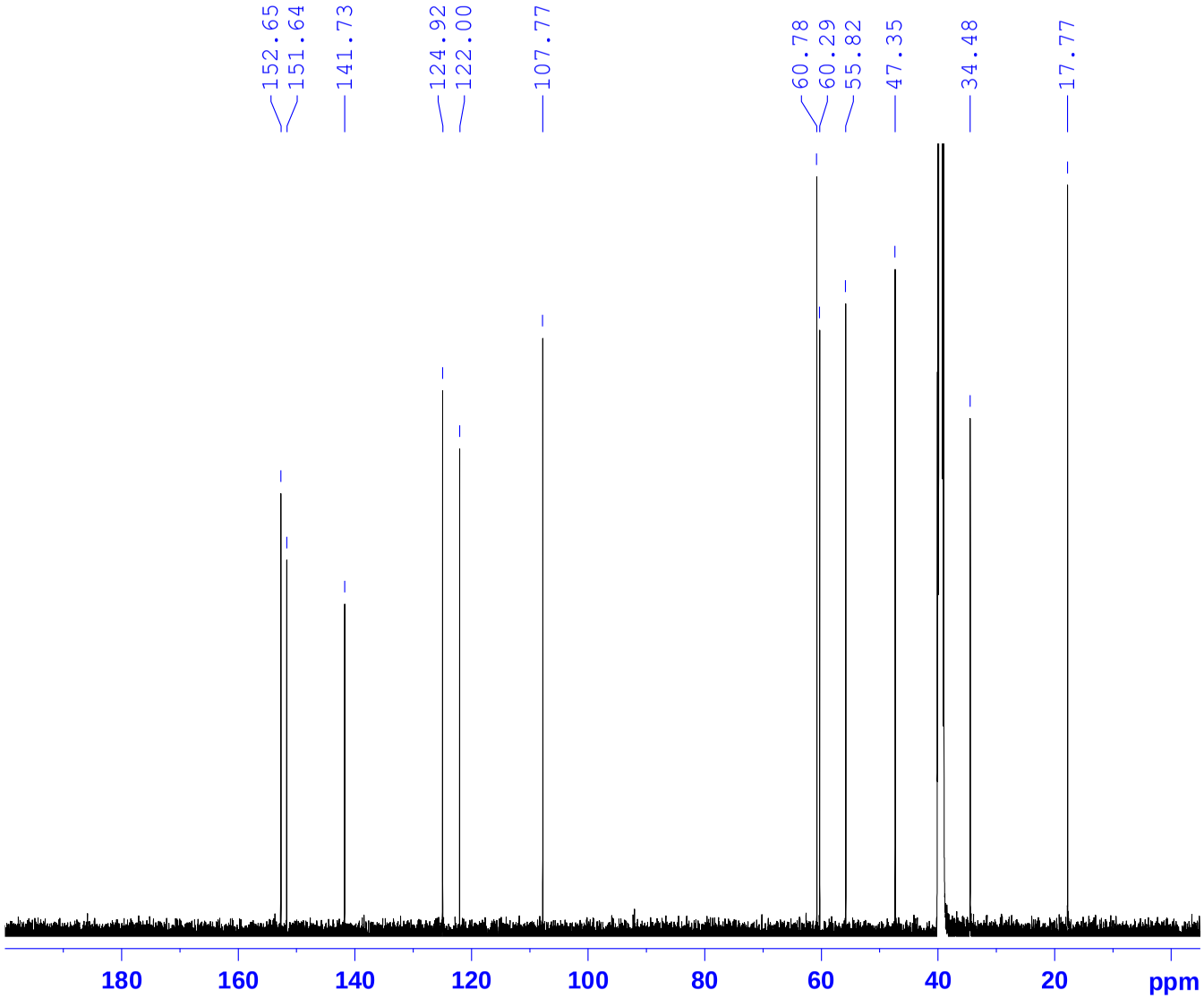

```
Current Data Parameters
NAME         NFL-2069-19
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190520
Time               15.31
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    64
DS                     2
SWH            10000.000 Hz
FIDRES             0.152588 Hz
AQ             3.2767999 sec
RG                   128
DW                50.000 usec
DE                 6.50 usec
TE                296.0 K
D1             2.00000000 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            500.1300047 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

NFL-2069-19
13C




```
Current Data Parameters
NAME         NFL-2069-19
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190520
Time              18.33
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT             DMSO
NS                  8192
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           125.7578452 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```