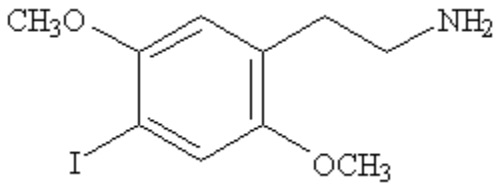
2,5-DIMETHOXY-4-IODOPHENETHYLAMINE

Latest Revision: August 22, 2005



1. SYNONYMS

CFR: Not Available

CAS #: Base: Not Available
Hydrochloride: 69587-11-7

Other Names: 4-Iodo-2,5-dimethoxyphenethylamine
4-Iodo-2,5-dimethoxybenzeneethanamine
2,5-Dimethoxy-4-iodo-beta-phenethylamine
2C-I

2. CHEMICAL AND PHYSICAL DATA

2.1. CHEMICAL DATA

Form	Chemical Formula	Molecular Weight	Melting Point (°C)
Base	$C_{10}H_{14}INO_2$	307.13	Not Available
Hydrochloride	$C_{10}H_{14}INO_2 \cdot HCl$	343.59	248-250

2.2. SOLUBILITY

Form	A	C	E	H	M	W
Base	NA	NA	NA	NA	NA	NA
Hydrochloride	PS	S	S	NA	VS	VS

A = acetone, C = chloroform, E = ether, H = hexane, M = methanol and W = water, VS = very soluble, FS = freely soluble, S = soluble, PS = sparingly soluble, SS = slightly soluble, VSS = very slightly soluble and I = insoluble, NA = Not Available

3. SCREENING TECHNIQUES

3.1. COLOR TESTS

REAGENT	COLOR PRODUCED
Marquis	Blue
Mecke	Brown-Black

3.2. GAS CHROMATOGRAPHY

Method SFL4 Screen

Instrument:	Gas chromatograph operated in split mode with FID
Column:	100% dimethylpolysiloxane gum 30 m x 0.25 mm i.d. x 0.25 µm film thickness
Carrier gas:	FID: Hydrogen at 1.3 mL/min
Makeup gas:	FID: Nitrogen at 40.0 mL/min
Temperatures:	Injector: 250°C Detector: 300°C Oven program: 1) 100°C initial temperature 2) Ramp to 295°C at 35°C/min 3) Hold final temperature for 6.43 min
Injection Parameters:	Split Ratio: 100:1 1 µL injection

Sample dissolved in water and base extracted with 1-5 N sodium hydroxide into an organic solvent.

COMPOUND	RRT	COMPOUND	RRT
amphetamine	0.474	4-MeOPP	0.904
methamphetamine	0.514	2C-B	0.935
nicotinamide	0.633	caffeine	0.945
3,4-MDA	0.716	**2C-I**	**1.000 (4.524 min)**
TFMPP	0.744	2C-T-2	1.014
3,4-MDMA	0.761	2C-T-7	1.063

benzocaine	0.772	procaine	1.080
3,4-MDEA	0.797	tetracaine	1.201
acetaminophen	0.847	quinine	1.572

3.3. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Method Phen01

Instrument: High performance liquid chromatograph equipped with mass spectrometer

Column: 5 µm ODS, 150 mm x 4.6 mm

Detector: Mass Spectrometer

Flow: 400 µL/min

Injection Volume: 5.0 µL

Buffer: 10 mM ammonium acetate in water

Mobile Phase: 1) Initially, CH_3OH: buffer 5:95 held for 10 min
2) Gradient to CH_3OH: buffer 80:20 over 10 min
3) Gradient to CH_3OH: buffer 5:95 over 10 min

Samples are to be dissolved in buffer solution, sonicated, and then filtered with a 0.45 µm filter.

COMPOUND	RRT	COMPOUND	RRT
ephedrine/pseudoephedrine	0.762	**2C-I**	**1.000 (13.29 min)**
amphetamine	0.834	2C-T-2	1.005
methamphetamine	0.845	3,4-MDMA	1.027
3,4-MDEA	0.862	2C-T-7	1.071
2C-B	0.969		

4. SEPARATION TECHNIQUES

5. QUANTITATIVE PROCEDURES

5.1. GAS CHROMATOGRAPHY

Method SFL4 4dimeth1

Internal Standard Stock Solution:
1.00 mg/mL tetradecane (C_{14}) in methylene chloride.

Standard Solution Preparation:
Prepare a standard solution of 2C-I·HCl within the linearity range listed below.

Sample Preparation:
Accurately weigh an amount of sample into a volumetric flask so that the final 2C-I·HCl concentration is approximately equivalent to that of the standard solution. Dilute to volume with deionized water. A 2 mL aliquot of the sample is then extracted with 2 mL of 1 M-5 M sodium hydroxide into 2 mL of internal standard stock solution.

Instrument:	Gas chromatograph operated in split mode with FID
Column:	100% dimethylpolysiloxane gum, 30 m x 0.25 mm x 0.25 µm film thickness
Carrier gas:	Hydrogen at 1.2 mL/min
Make-Up gas:	Nitrogen at 30 mL/min
Temperatures:	Injector: 265°C Detector: 275°C Oven program: 220°C isothermal
Injection Parameters:	Split Ratio: 50:1 1 µL injection
Typical Retention Time:	2C-I HCl: 2.06 min C_{14}: 1.30 min
Linear Range:	0.581 – 4.648 mg/mL
Repeatability:	RSD less than 3%
Correlation Coefficient:	r^2 greater than 0.998
Accuracy:	Error less than 5%

COMPOUND	RRT	COMPOUND	RRT
amphetamine	0.586	2C-B	0.874
methamphetamine	0.594	caffeine	0.895
C_{14}	0.630	**2C-I**	**1.000 (2.06 min)**
3,4-MDA	0.667	2C-T-2	1.028
TFMPP	0.684	2C-T-7	1.160
3,4-MDMA	0.689	procaine	1.205
3,4-MDEA	0.711	tetracaine	1.783

5.2. NUCLEAR MAGNETIC RESONANCE

Method SFL1 NMR1-1

Reagents:
Deuterium oxide (D_2O) containing DSS or TSP for 0 ppm reference

Internal Standard Stock Solution (ISSS):
5 mg/mL maleic acid (accurately weighed) in deuterium oxide (D_2O) containing DSS or TSP for 0 ppm reference
Sample Preparation:
Accurately weigh an amount of sample, usually 10-30 mg, into a capped test tube and accurately add a volume, normally 1.0 mL, of the ISSS. Vortex the sample for several seconds. If insolubles are present, add 1.0 mL D_2O (not containing maleic acid or the reference compound), vortex and sonicate 15 min. Filter if necessary. Place in NMR sample tube.

Instrument: Varian Mercury 400 MHz NMR spectrometer with proton detection probe

Parameters:
Spectral width: at least containing -3 ppm through 13 ppm
Pulse width: lesser of 10 µs or 90°
Delay between pulses: 45 seconds
Number of scans (NT): multiple of 4
Number of steady state scans: 0
Oversampling: 4 or more
Shimming: automatic gradient shimming of Z1-4 shims
Phasing, Drift Correction: automatic or manual
l

Total Run Time per Sample:
6 min (NT = 4)
14 min (NT = 16)

Uniformity within spectral width: 0.3% RSD (-0.6 to 11.4 ppm)

Linear Range: 0.6 - 60 mg/mL

Repeatability: less than 4%

Correlation Coefficient: 1.000

Accuracy: less than 3%

Signals used for quantitation (position in ppm with number of protons): 7.4 s (1)
These can have interferences: 6.9s(1)
3.8s+s(6)
3.2t(2)
2.9t(2)

6. QUALITATIVE DATA

6.1. ULTRAVIOLET SPECTROPHOTOMETRY

SOLVENT	MAXIMUM ABSORBANCE (NM)
Water	232, 296 (0.06 mg/mL)

6.2. LIQUID CROMATOGRAPHY/MASS SPECTROMETRY

Method Phen01

Sample Preparation:
Dilute analyte in an appropriate volume of HPLC-grade water and pass through 0.45 µm polypropylene filter. Introduce solution via divert valve of the mass spectrometer with a flow rate of 400 µL/minute of HPLC-grade water.

Instrument: LCQ Advantage MAX in ESI Mode

Sheath Gas (arb): 10

Auxiliary/Sweep Gas (arb): 0

Spray Voltage (kV): 4.50

Spray Current (µA): 0.29

Capillary Temperature (°C): 250.0

Capillary Voltage (V):	13.00
Tube Lens Offset (V):	-25.00
Scan Mode:	MS or MS^3 (depending on experiment being performed)
Mass Range:	Normal; MS: 50-550 amu; MS^3: 60 – 550 amu
Scan Type:	Full
Scan Time (microscans):	1
Maximum Injection Time (ms):	1000.0
Source Fragmentation:	Off
For MS^3:	
Parent Masses (m/z):	MS^2: 308.0 MS^3: 291.0
Isolation Width (m/z):	1.0
Normalized Collision Energy (%):	MS^2: 25.0 MS^3: 35.0
Activation Q:	0.250
Activation Time (msec):	30.0

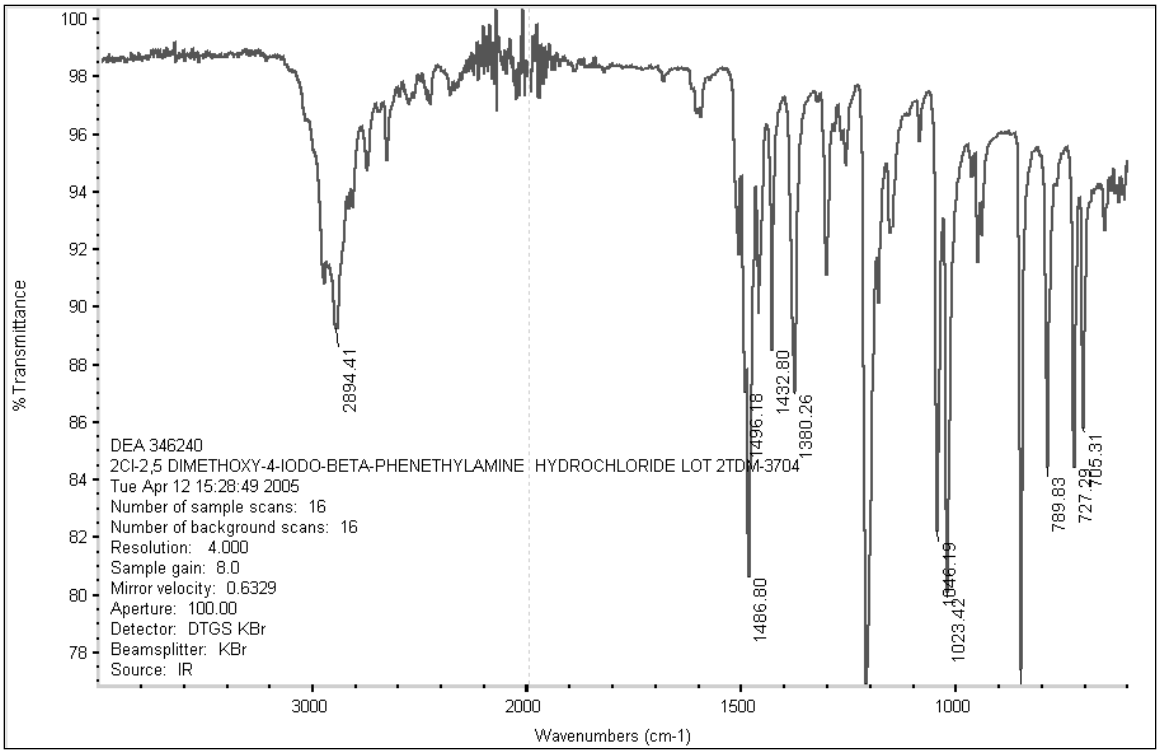
See spectra on the following pages for FTIR ATR, Vapor Phase IR, GC Mass Spectrometry, Mass Spectrometry (MS^1), Mass Spectrometry (MS^3), and Nuclear Magnetic Resonance.

7. ADDITIONAL RESOURCES

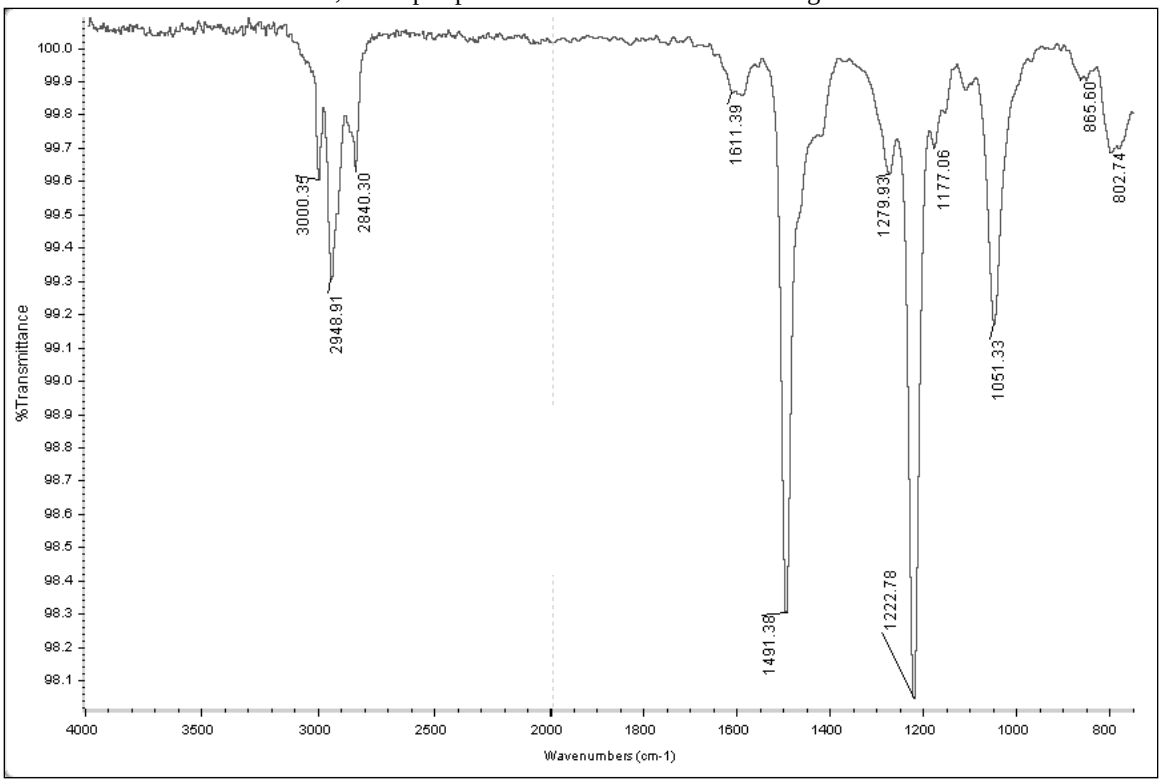
Forendex

Wikipedia

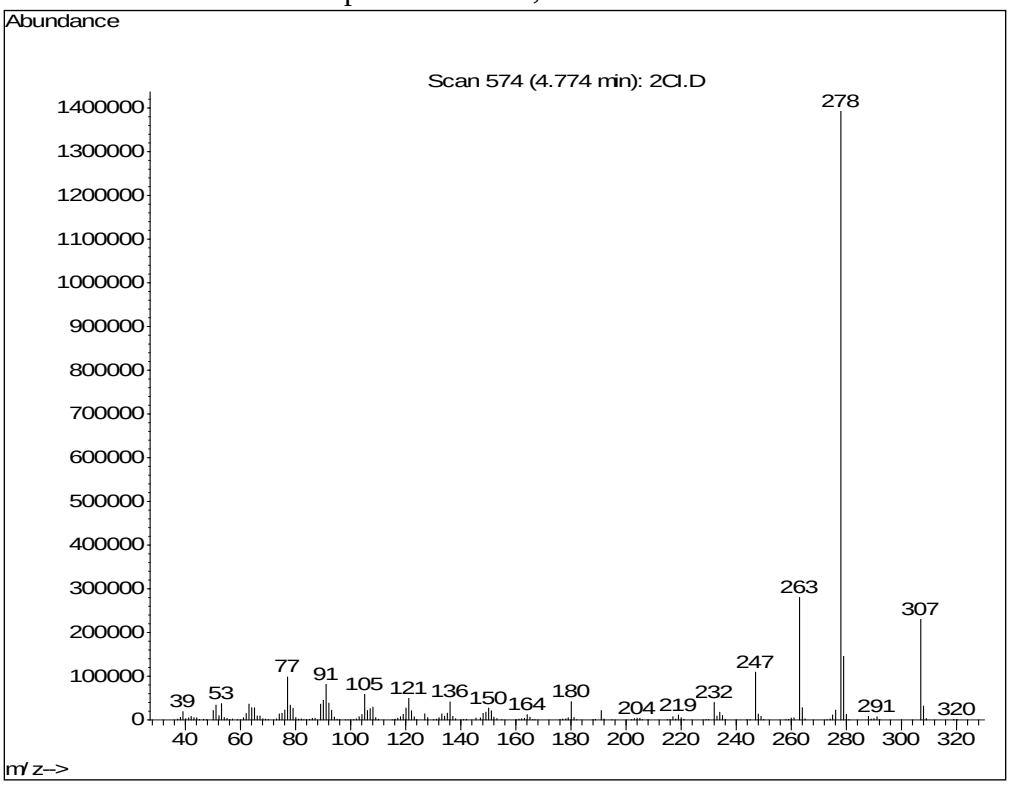
FTIR (Diamond ATR, 3 Bounce): 2C-I HCL Lot # 2TDM-37-04
32 scans, 4cm^{-1} resolution



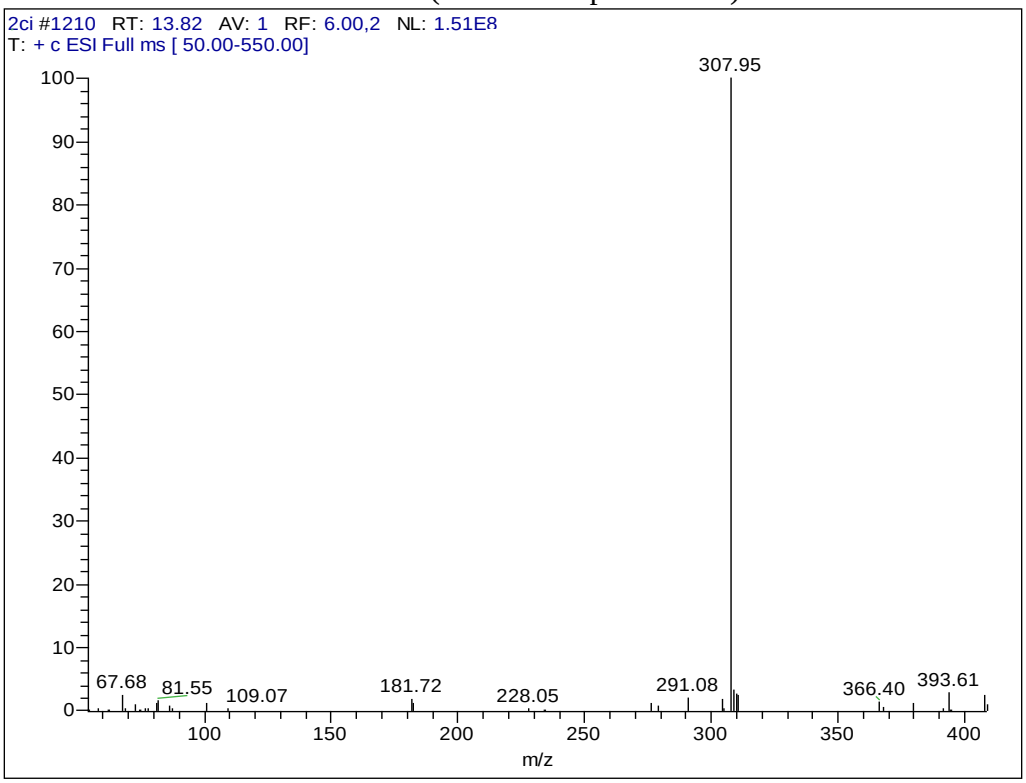
Vapor Phase IR: C-I Lot # 2TDM-37-04
280°C, 8 cm^{-1} resolution
*Under the above conditions, the vapor phase IR cannot be used to distinguish between 2C-I and 2C-B.



EI Mass Spectrum: 2C-I, Lot # 2TDM-37-04

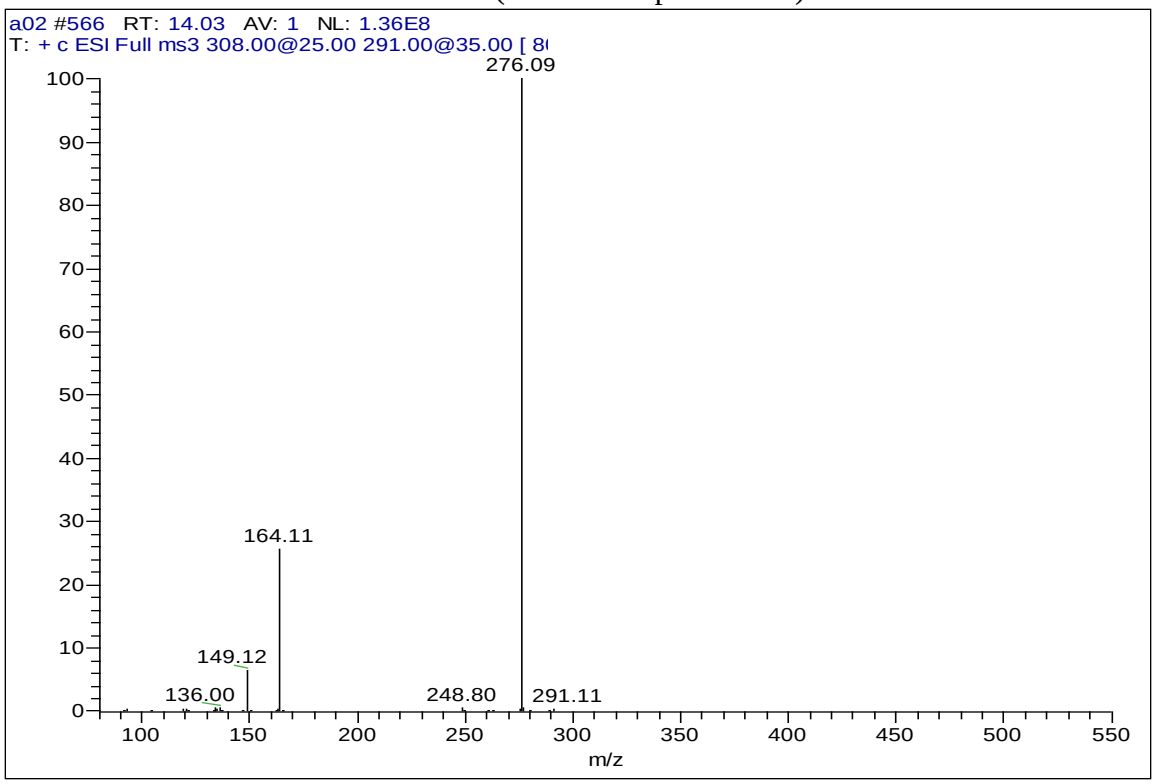


API-ESI Mass Spectrum: 2C-I, Lot # 2TDM-37-04
MS^1 mode (see text for parameters)



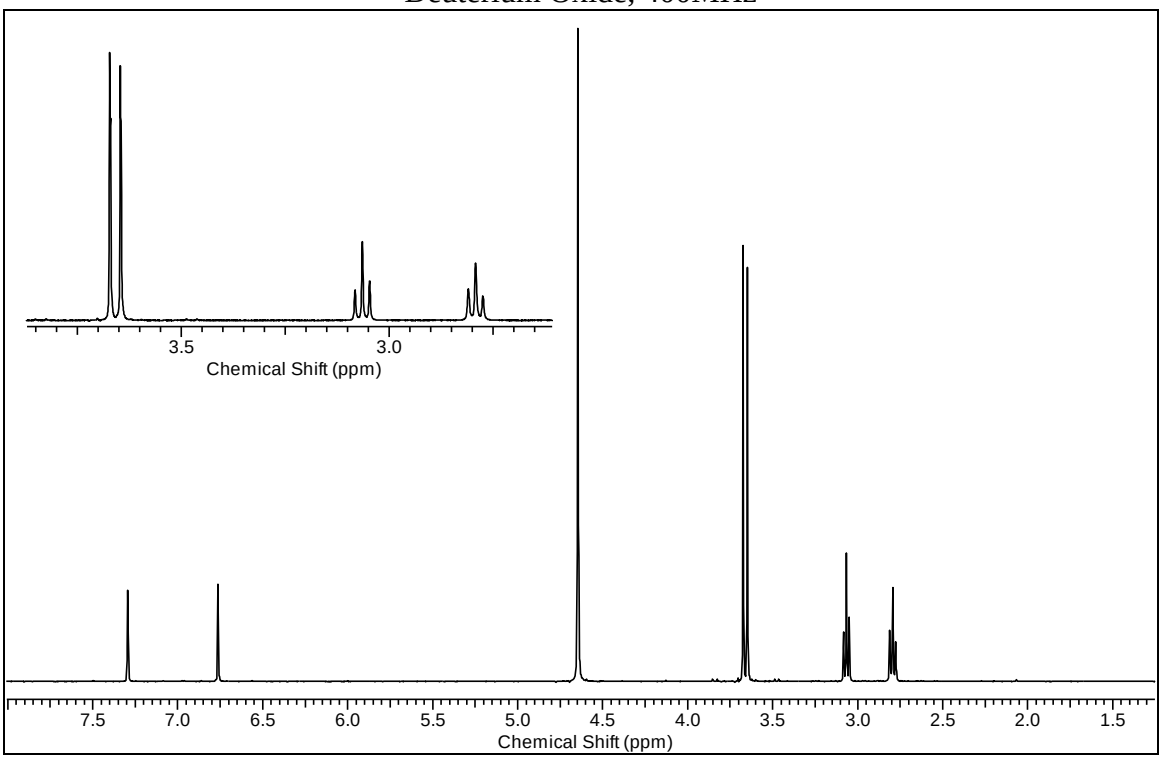
API - ESI Mass Spectrum: 2C-I, Lot # 2TDM-37-04

MS^3 mode (see text for parameters)

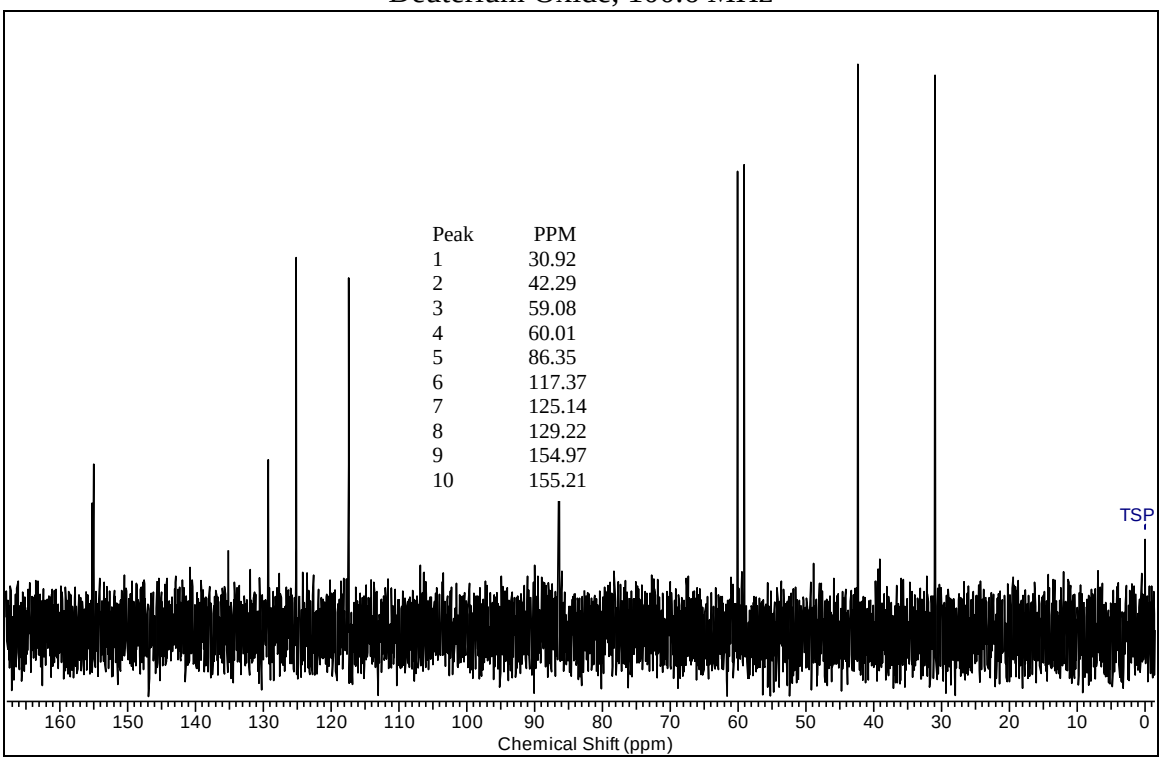


1H NMR: 2C-I Lot # 2TDM-37-0

Deuterium Oxide, 400MHz



^{13}C NMR: 2C-I HCl Lot # 2TDM-37-04
Deuterium Oxide, 100.6 MHz



Abbreviations used:
BZP = 1-benzylpiperazine
2C-B = 4-bromo-2,5-dimethoxyphenethylamine
2C-T-2 = 2,5-dimethoxy-4-ethylthiophenethylamine
2C-T-7 = 2,5-dimethoxy-(4-N-propylthio)-beta-phenethylamine
2C-I = 4-iodo-2,5-dimethoxy-beta-phenethylamine
4-MeOPP = 1-(4-methoxyphenyl)piperazine
TFMPP = trifluoromethylphenylpiperazine