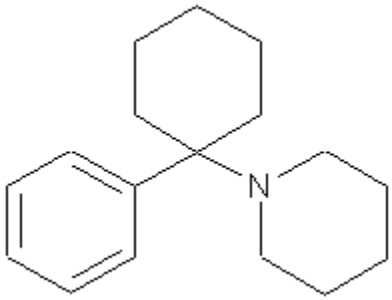
PHENCYCLIDINE

Latest Revision: May 16, 2005



1. SYNONYMS

CFR: Phencyclidine

CAS #: Base: 77-10-1
Hydrochloride: 956-90-1

Other Names: 1-(1-Phenylcyclohexyl) piperidine
PCP
Angel dust
CI-395
Sernylan
Sernyl

2. CHEMICAL AND PHYSICAL DATA

2.1. CHEMICAL DATA

Form	Chemical Formula	Molecular Weight	Melting Point (°C)
Base	$C_{17}H_{25}N$	243.4	46-46.5
Hydrochloride	$C_{17}H_{26}NCl$	279.9	233-235

2.2. SOLUBILITY

Form	A	C	E	H	M	W
Base	FS	FS	FS	FS	S	VSS
Hydrochloride	SS	FS	I	I	FS	FS

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

3. SCREENING TECHNIQUES

3.1. COLOR TESTS

REAGENT	COLOR PRODUCED
p-Dimethylaminobenzaldehyde	Red

3.2. CRYSTAL TESTS

REAGENT	CRYSTALS FORMED
Potassium permanganate	Bow-tie shaped

3.3. THIN-LAYER CHROMATOGRAPHY

Visualization

Acidified iodoplatinate spray Dragendorff spray

COMPOUND	RELATIVE R_1		
	System TLC17	System TLC11	System TLC16
piperidine	0.4	0.2	0.1
PCP	1.0	1.0	1.0
piperidinocyclohexylcarbonitrile (PCC)	4.5	1.7	1.7

Both iodoplatinate and Dragendorff sprays will detect the three components. Iodine vapor produces a white spot outlined in brown for PCC, where as PCP and piperidine both give brown spots.

3.4. GAS CHROMATOGRAPHY

Method PCP-GCS1

Instrument: Gas chromatograph operated in split mode with FID

Column: 5% phenyl/95% methyl silicone 12 m x 0.2 mm x 0.33 µm film thickness

Carrier gas: Helium at 1.0 mL/min

Temperatures: Injector: 270°C
Detector: 280°C
Oven program:
1) 175°C initial temperature for 1.0 min
2) Ramp to 275°C at 15°C/min
3) Hold final temperature for 3.0 min

Injection Parameters: Split Ratio = 60:1, 1 µL injected

Samples are to be dissolved or diluted in chloroform and filtered.

COMPOUND	RRT	COMPOUND	RRT
nicotinamide	0.31	theophylline	1.15
benzocaine	0.49	chlorpheniramine	1.19
PCC	0.50	procaine	1.21
ibuprofen	0.53	methaqualone	1.44
acetaminophen	0.63	cocaine	1.52
phenacetin	0.65	tetracaine	1.55
pentobarbital	0.72	tetracosane	1.73
secobarbital	0.80	codeine	1.81
caffeine	0.88	morphine	1.89
diphenhydramine	0.95	acetylcodeine	2.00
antipyrine	0.97	O^6-monoacetylmorphine	2.02
lidocaine	0.97	heroin	2.19
phencyclidine	**1.00 (3.50 min)**	quinidine	2.45
aminopyrine	1.06	quinine	2.46
phenobarbital	1.11		

3.5. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Method PCP-LCS1

Instrument: High performance liquid chromatograph equipped with diode array

Column: 5 µm ODS, 150 mm x 4.6 mm

Detector: UV, 210 nm

Flow: 1.0 mL/min

Injection Volume: 5.0 µL

Buffer: 4000 mL distilled water, 10 g sodium hydroxide, 30.0 mL phosphoric acid and 8.0 mL hexylamine

Mobile Phase:
1) Initially, buffer: acetonitrile 98:2 for 2 min
2) Gradient to buffer: acetonitrile 80:20 over 12 min
3) Gradient to buffer: acetonitrile 60:40 over 13 min
4) Hold buffer: acetonitrile 60:40 for 5 min

Samples are to be dissolved in buffer: acetonitrile 90:10, sonicated, then filtered with a 0.45-micron filter.

COMPOUND	RRT	COMPOUND	RRT
isonicotinamide	0.11	tropacocaine	0.71
nicotinamide	0.11	benzoylecgonine	0.72
morphine	0.19	antipyrine	0.76
phenylpropanolamine	0.19	cocaine	0.77
ephedrine	0.23	acetylcodeine	0.79
aminopyrine	0.25	heroin	0.83
procaine	0.27	**phencyclidine**	**1.00**
amphetamine	0.29	aspirin	1.08
methamphetamine	0.33	diazepam	1.13
codeine	0.35	*t*-cinnamoylcocaine	1.14
methylenedioxy-amphetamine	0.38	phenobarbital	1.19
methylenedioxy-methamphetamine	0.41	tetracaine	1.19

lidocaine	0.42	phenacetin	1.21
quinine	0.44	diphenhydramine	1.22
O^6-monoacetylmorphine	0.49	phenyl-2-propanone	1.23
acetaminophen	0.51	benzocaine	1.29
strychnine	0.62	amobarbital	1.52
caffeine	0.65	methaqualone	1.56
barbital	0.67	secobarbital	1.65

4. SEPARATION TECHNIQUES

In general, phencyclidine can be extracted by dissolving the sample in dilute acid, making the solution basic, extracting with petroleum ether and recrystallizing as the hydrochloride salt form.

Plant material that has been impregnated with phencyclidine in solution can be extracted using column chromatography. The plant material is incorporated directly into a 1 N HCl celite column. Elute phencyclidine from the column with water-washed chloroform. Evaporate the eluent to dryness. The resulting phencyclidine hydrochloride residue is then cleaned by washing with acetone, which will remove plant material related impurities.

Contaminants from synthesis mixtures of phencyclidine in solution can be removed by an acid-base extraction procedure in which phencyclidine hydrochloride ion-pairs in chloroform. To a 125 mL separatory funnel add 2 mL of PCP sample, 50 mL of water, 50 mL of diethyl ether, and 5-7 drops of concentrated sodium hydroxide. Check pH to ensure aqueous phase is basic. Shake the funnel well, allow the layers to separate, and discard the aqueous layer leaving the ether (top layer) in the funnel. Wash the ether layer with 50 mL of water, shake, separate the layers and again discard the water, leaving the ether in the funnel. Add another 50 mL portion of water to the separatory funnel, and slowly add 5-7 drops of sulfuric acid to acidify the aqueous phase. Check the pH and add more acid if necessary to ensure that the aqueous phase is acidic. Shake the funnel, allow layers to separate and discard the ether portion. Return the aqueous layer to the separatory funnel and wash with a second portion of ether. Place the aqueous phase back into the separatory funnel and add one gram of sodium chloride to the acidic aqueous phase. Mix the solution until all the sodium chloride is dissolved. Add 50 mL of chloroform and extract. Save the chloroform (bottom layer) in a beaker. Perform a second chloroform extraction and combine with the first chloroform fraction. Evaporate the chloroform to dryness to recover the phencyclidine hydrochloride.

5. QUANTITATIVE PROCEDURES

5.1. GAS CHROMATOGRAPHY

Method PCP-GCQ1

Internal Standard Stock Solution:
0.4 mg/mL docosane in chloroform.

Standard Solution Preparation:
Accurately weigh and prepare a standard solution of phencyclidine (hydrochloride or base) at approximately 0.4 mg/mL using above internal standard stock solution.

Sample Preparation:
Powder: Accurately weigh an amount of sample into a volumetric flask and dilute with internal standard stock solution. If necessary, dilute the sample so the final concentration approximates the standard concentration.

Liquid: Accurately pipette an aliquot of the sample into a volumetric flask and dilute to volume with internal standard stock solution. Dilute the sample so the final concentration approximates the standard concentration.

Plant material: Accurately weigh an amount of sample into a container and add an accurate volume of internal standard stock solution. Allow the phencyclidine to extract from the plant material for at least two hours. If necessary, dilute the sample so the final concentration approximates the standard concentration. Filter sample prior to injection.

Instrument:	Gas chromatograph operated in split mode with FID
Column:	5% phenyl/95% methyl silicone 12 m x 0.20 mm x 0.33µm film thickness
Carrier gas:	Helium 1.0 mL/min
Temperatures:	Injector: 230°C Detector: 280°C Oven program: 1) 200°C initial temperature for 1.2 min 2) Ramp to 270°C at 30°C/min 3) Hold final temperature for 2.0 min
Injection Parameters:	Split Ratio = 50:1, 1 µL injected
Typical Retention Time:	Phencyclidine: 2.15 min Docosane: 2.95 min
Linear Range:	Base: 0.1 - 3.0 mg/mL Hydrochloride: 0.125 - 2.0 mg/mL
Repeatability:	Base: RSD less than 0.3% Hydrochloride: RSD less than 0.6%
Correlation Coefficient:	Base: 0.999 Hydrochloride: 0.999
Accuracy:	Base: Error less than 5% Hydrochloride: Error less than 5%

The following compounds typically found in liquid phencyclidine samples were found to separate with a resolution greater than 1.5:

piperidine	1-piperidinocyclohexene
cyclohexanone	biphenyl
bromobenzene	1-phenylcyclohexanol
phenol	1-pPhenylcyclohexene
1-phenethanol	1-piperidinocyclohexylcarbonitrile
3,5-dimethylpiperidine	1-cyclohexylpiperidine

Method PCP-GCQ2

Internal Standard Stock Solution:
0.4 mg/mL docosane in chloroform.

Standard Solution Preparation:
Accurately weigh and prepare a standard solution of phencyclidine (hydrochloride or base) at approximately 0.4 mg/mL using above internal standard stock solution.

Sample Preparation:
Powder: Accurately weigh an amount of sample into a volumetric flask and dilute with internal standard stock solution. If necessary, dilute the sample so the final concentration approximates the standard concentration.

Liquid: Accurately pipette an aliquot of the sample into a volumetric flask and dilute to volume with internal standard stock solution. Dilute the sample so the final concentration approximates the standard concentration.
Plant material: Accurately weigh an amount of sample into a container and add an accurate volume of internal standard stock solution. Allow the phencyclidine to extract from the plant material for at least two hours. If necessary, dilute the sample so the final concentration approximates the standard concentration. Filter sample prior to injection.

Instrument:	Gas chromatograph operated in split mode with FID
Column:	5% phenyl/95% methyl silicone 30 m x 0.32 mm x 0.25 µm film thickness
Carrier gas:	Helium 2.0 mL/min
Temperatures:	Injector: 280°C Detector: 280°C Oven program: 250°C isothermal
Injection Parameters:	Split Ratio = 60:1, 1 µL injected
Typical Retention Time:	Phencyclidine: 1.86 min Docosane: 2.39 min

Linear Range: Base: 0.1 - 2.5 mg/mL
Hydrochloride: 0.125 - 2.0 mg/mL

Repeatability: Base: RSD less than 0.3%
Hydrochloride: RSD less than 1.2%

Correlation Coefficient: Base: 0.999
Hydrochloride: 0.999

Accuracy: Base: Error less than 5%
Hydrochloride: Error less than 5%

The following compounds typically found in liquid phencyclidine samples were found to separate with a resolution greater than 1.5:

piperidine	1-piperidinocyclohexene
cyclohexanone	biphenyl
bromobenzene	1-phenylcyclohexanol
phenol	1-phenylcyclohexene
1-phenethanol	1-piperidinocyclohexylcarbonitrile
3,5-dimethylpiperidine	1-cyclohexylpiperidine

Method PCP-GCQ3

Internal Standard Stock Solution:
0.4 mg/mL docosane in chloroform.

Standard Solution Preparation:
Accurately weigh and prepare a standard solution of phencyclidine (hydrochloride or base) at approximately 0.4 mg/mL using above internal standard stock solution.

Sample Preparation:
Powder: Accurately weigh an amount of sample into a volumetric flask and dilute with internal standard stock solution. If necessary, dilute the sample so the final concentration approximates the standard concentration.

Liquid: Accurately pipette an aliquot of the sample into a volumetric flask and dilute to volume with internal standard stock solution. Dilute the sample so the final concentration approximates the standard concentration.

Plant material: Accurately weigh an amount of sample into a container and add an accurate volume of internal standard stock solution. Allow the phencyclidine to extract from the plant material for at least two hours. If necessary, dilute the sample so the final concentration approximates the standard concentration. Filter sample prior to injection.

Instrument: Gas chromatograph operated in split mode with FID

Column: 100% methyl siloxane 12 m x 0.20 mm x 0.33 µm film thickness

Carrier gas: Helium 1.0 mL/min

Temperatures: Injector: 270°C
Detector: 280°C
Oven program: 250°C isothermal

Injection Parameters: Split Ratio = 40:1, 1 µL injected

Typical Retention Time: Phencyclidine: 0.89 min
Docosane: 1.29 min

Linear Range: Base: 0.1 - 3.0 mg/mL
Hydrochloride: 0.125 - 4.0 mg/mL

Repeatability: Base: RSD less than 0.3%
Hydrochloride: RSD less than 1.0%

Correlation Coefficient: Base: 0.999
Hydrochloride: 0.999

Accuracy: Base: Error less than 5%
Hydrochloride: error less than 5%

The following compounds typically found in liquid phencyclidine samples were found to separate with a resolution greater than 1.5:

piperidine	1-piperidinocyclohexene
cyclohexanone	biphenyl
bromobenzene	1-phenylcyclohexanol
phenol	1-phenylcyclohexene
1-phenethanol	1-piperidinocyclohexylcarbonitrile
3,5-dimethylpiperidine	1-cyclohexylpiperidine

5.2. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Method PCP-LCQ1

Internal Standard Stock Solution:
0.1 mg/mL strychnine in mobile phase.

Standard Solution Preparation:
Accurately weigh and prepare a standard solution of phencyclidine (hydrochloride or base) at approximately 0.2 mg/mL using internal standard stock solution.

Sample Preparation:
Powder: Accurately weigh an amount of sample into a volumetric flask and dilute with internal standard stock solution. If necessary, dilute the sample so the final concentration approximates the standard concentration.

Filter sample with 0.45-micron filter prior to injection.

Instrument: High performance liquid chromatograph equipped with diode array

Column: 5µm ODS, 150 mm x 4.6 mm

Detector: UV, 210 nm

Flow: 1.5 mL/min

Injection Volume: 5.0 µL

Buffer: 4000 mL distilled water, 10 g sodium hydroxide, 30.0 mL phosphoric acid and 8.0 mL hexylamine

Mobile Phase: Buffer: acetonitrile 80:20

Typical Retention Time: Phencyclidine: 4.42 min
Strychnine: 1.70 min

Linear Range: 0.062 - 1.5 mg/mL

Repeatability: RSD less than 2%

Correlation Coefficient: 0.999

Accuracy: Error less than 5%

The following compounds typically found in liquid phencyclidine samples were found to separate with a resolution greater than 1.5:

piperidine	1-piperidinocyclohexene
cyclohexanone	biphenyl
bromobenzene	1-phenylcyclohexanol
phenol	1-phenylcyclohexene
1-phenethanol	1-piperidinocyclohexylcarbonitrile
3,5-dimethylpiperidine	1-cyclohexylpiperidine

6. QUALITATIVE DATA

6.1. ULTRAVIOLET SPECTROPHOTOMETRY

SOLVENT	MAXIMUM ABSORBANCE (NM)

Aqueous Acid	258

See spectra on the following pages for FT-IR, FT-Raman, Mass Spectrometry, Nuclear Magnetic Resonance, and Vapor Phase IR.

7. REFERENCES

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Kalir A., Edery H., Pelah Z., Balderman D. and Porath G., *Journal of Medicinal Chemistry*, Vol. 12, 1969, pp.473-477.

Maddox V., Godefroi E. and Parcell R., *Journal of Medicinal Chemistry*, Vol. 8, 1965, pp. 230-235.

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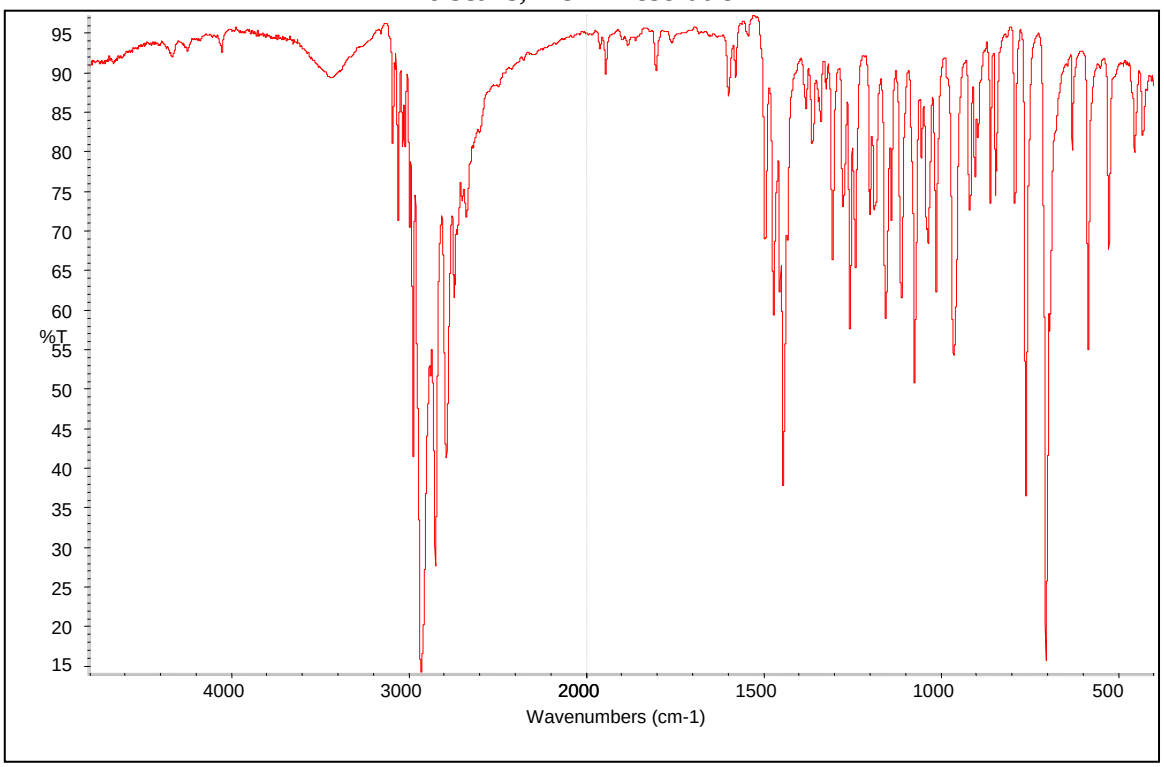
Raney J., Skowronski G. and Wagenhofer R., *Microgram*, Vol. XIV, 1981, pp.78-86.

8. ADDITIONAL RESOURCES

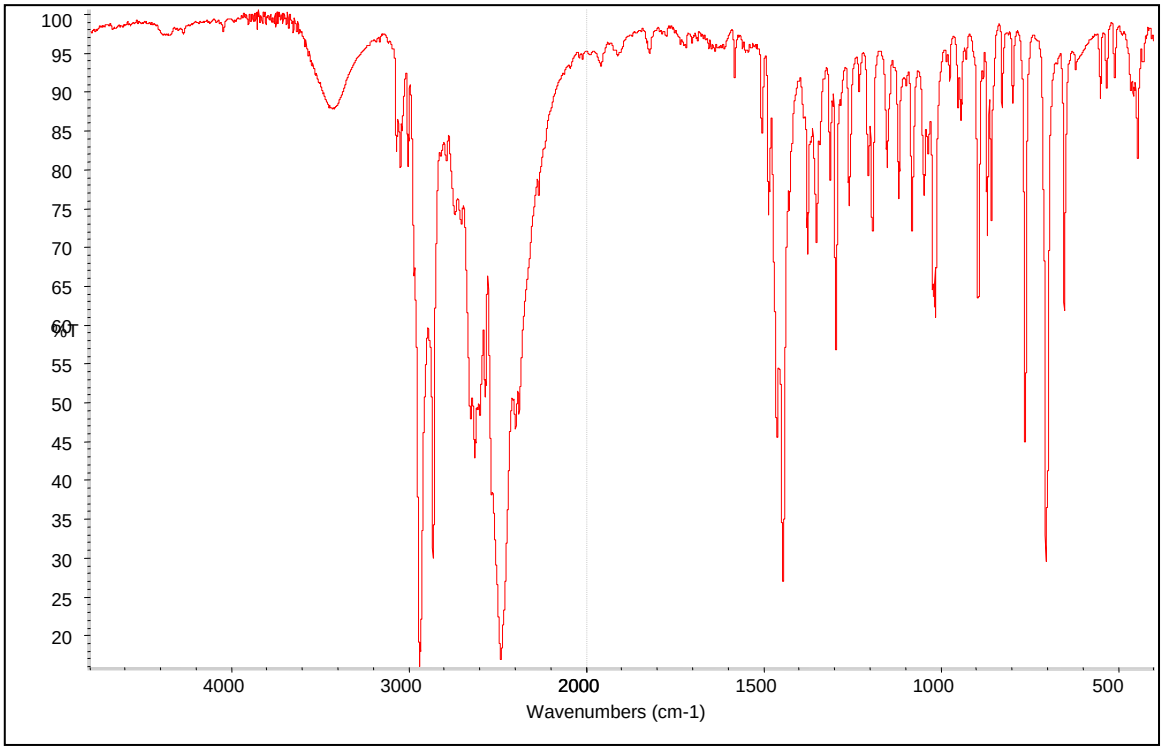
Forendex

Wikipedia

FTIR: Phencyclidine Base in KBr
16 scans, 4 cm^{-1} resolution

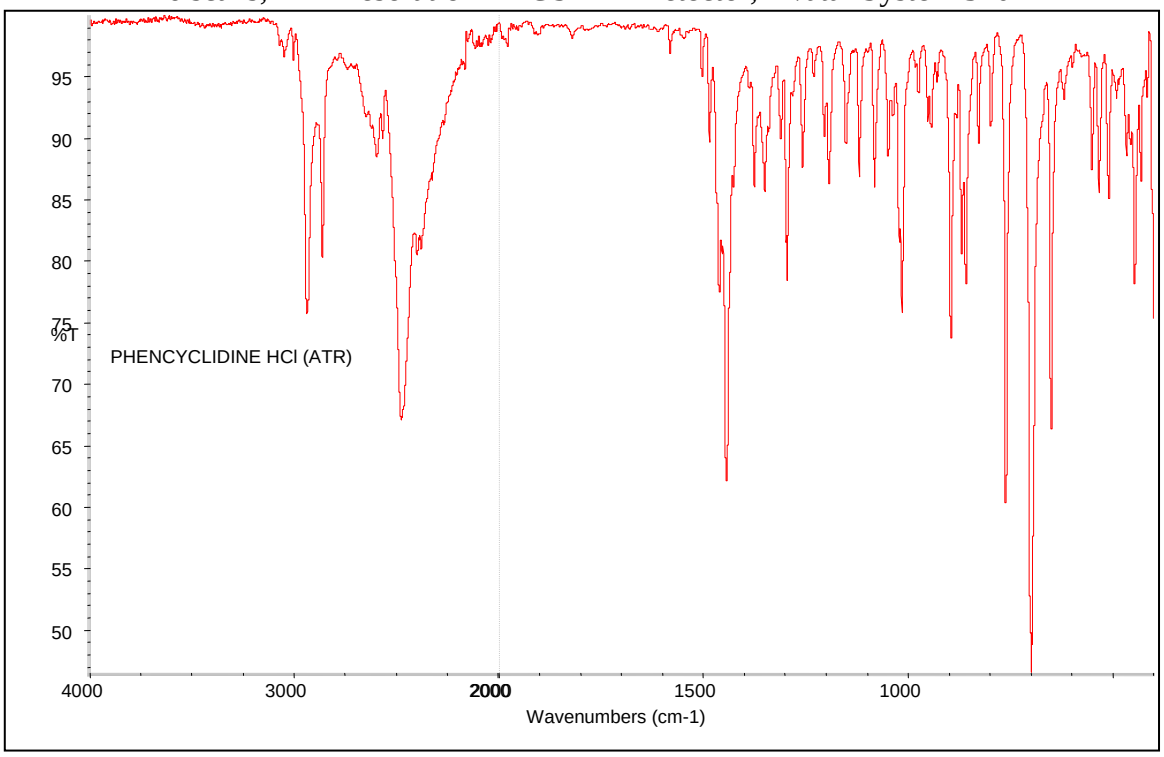


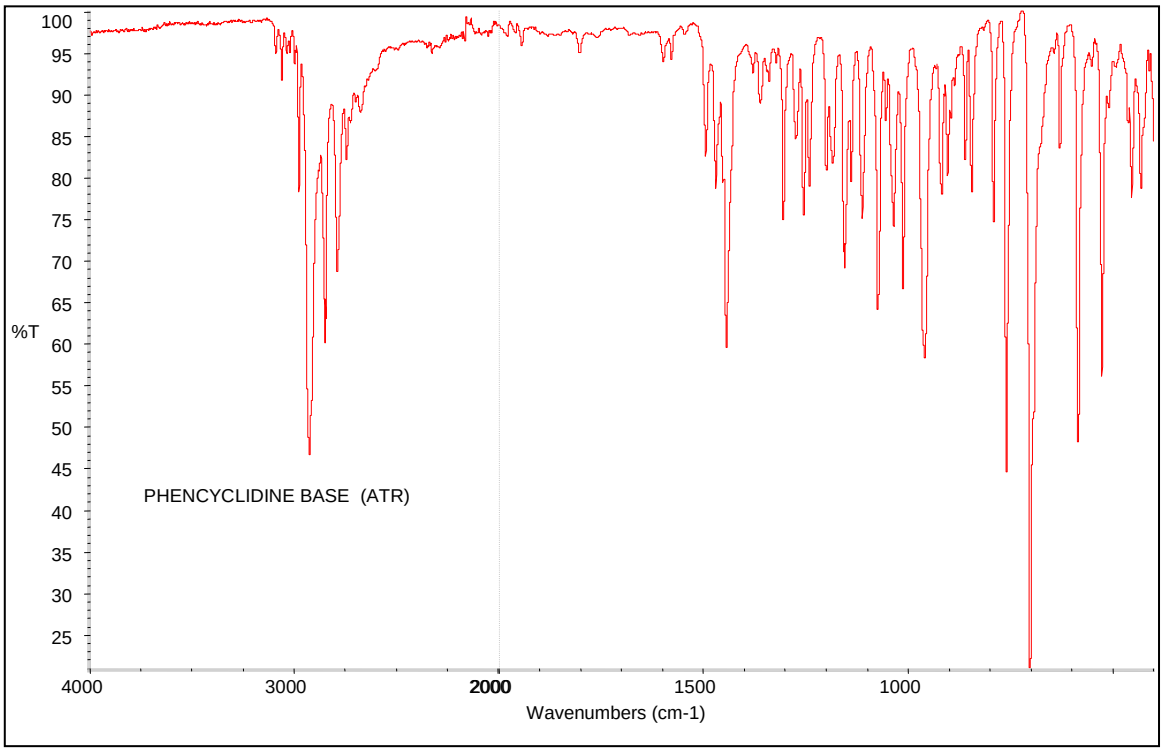
FTIR: Phencyclidine hydrochloride in KBr
16 Scans, 4 cm^{-1} resolution



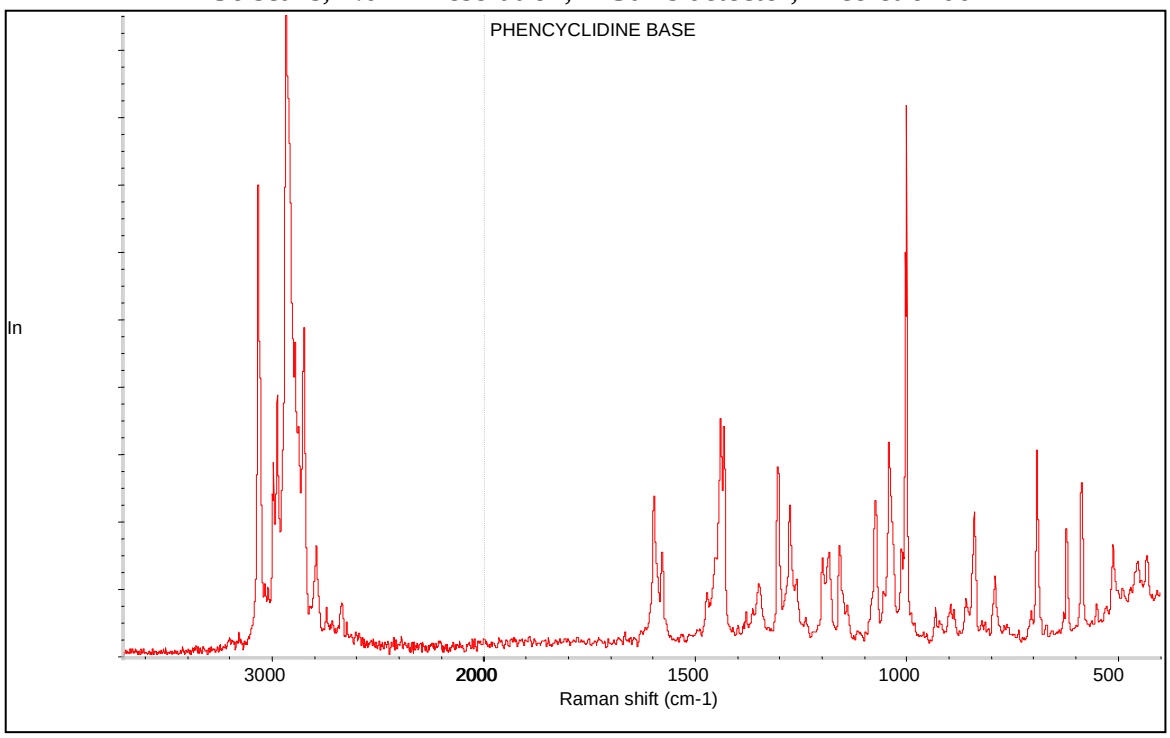
FTIR (ATR): Phencyclidine

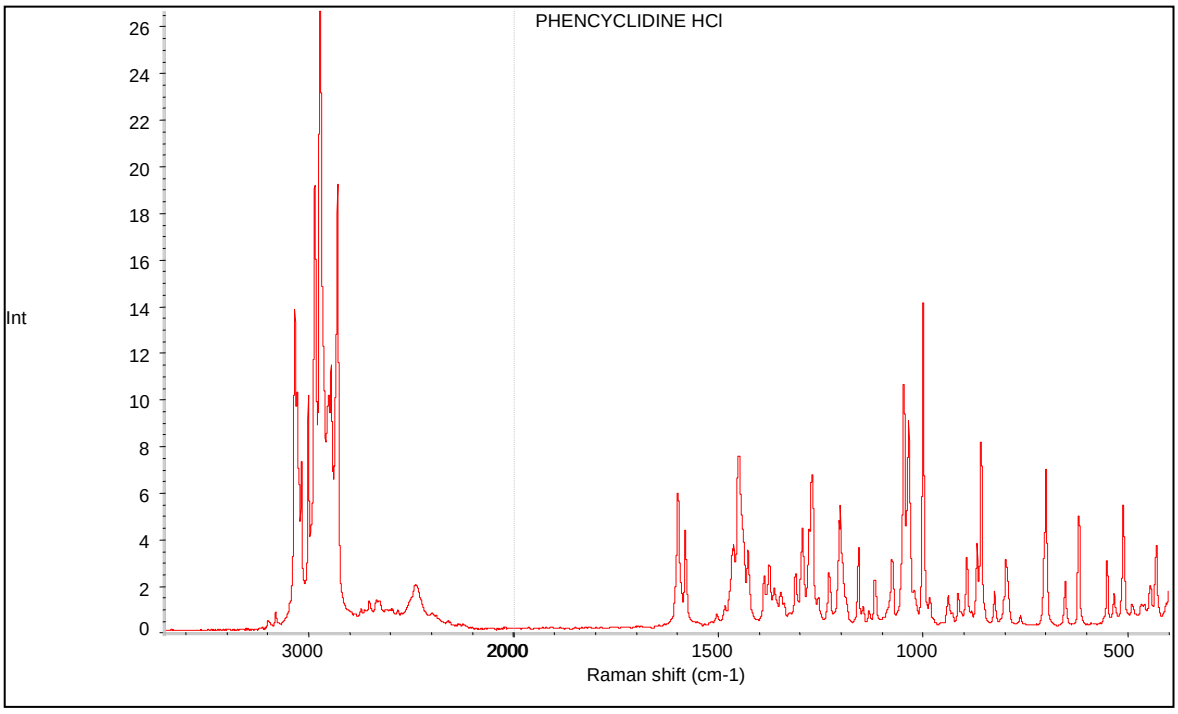
16 scans; 4 nm resolution DTGS KBr Detector, Avatar System 370



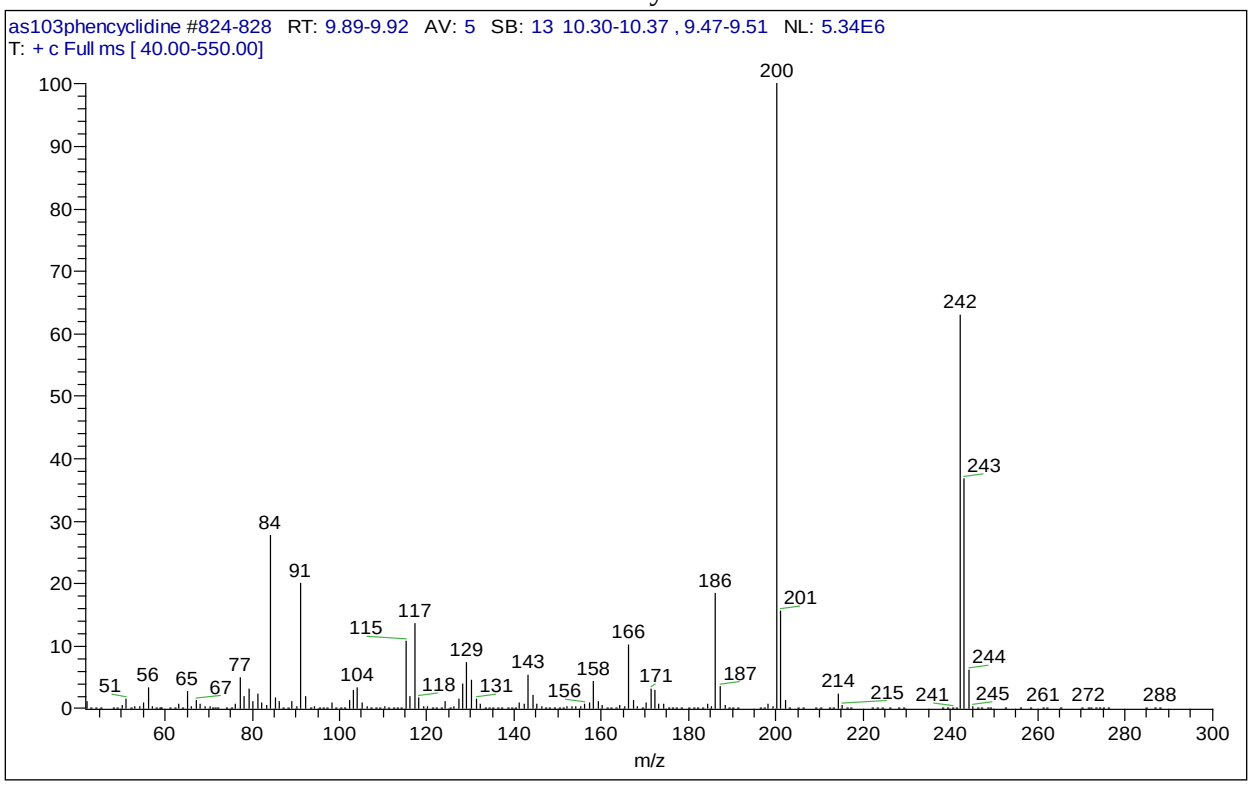


RAMAN: Phencyclidine
256 scans; 4.0 nm resolution, InGaAs detector, Nicolet 6700

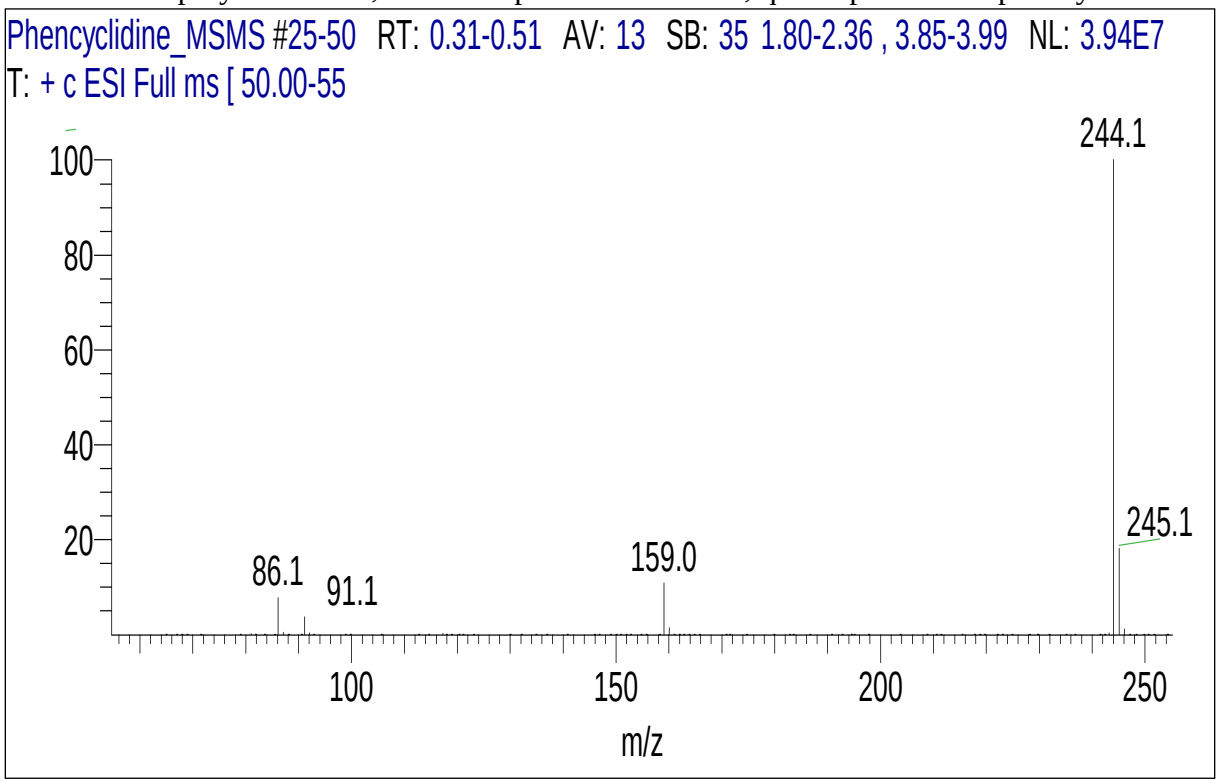




MS: Phencyclidine

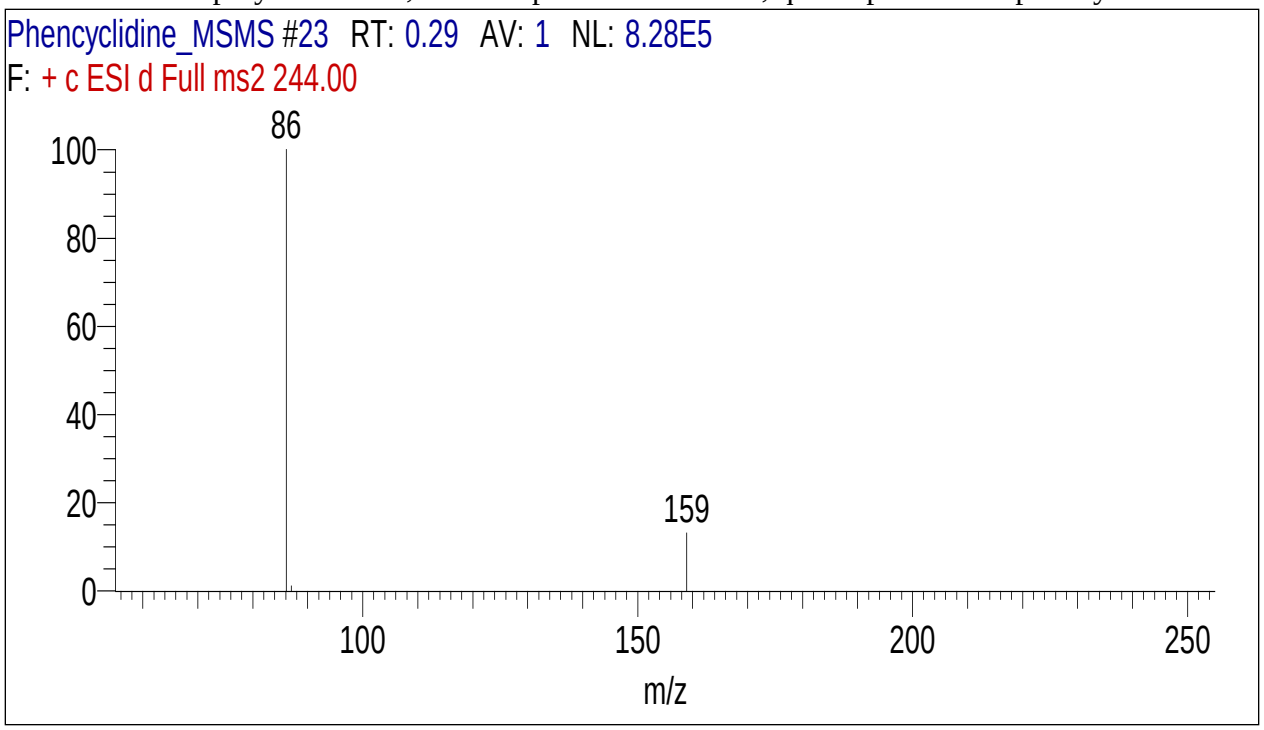


MS (ESI): Phencyclidine in MeOH;
Electrospray ionization; Full-scan positive ion mode; quadrupole ion-trap analyzer.

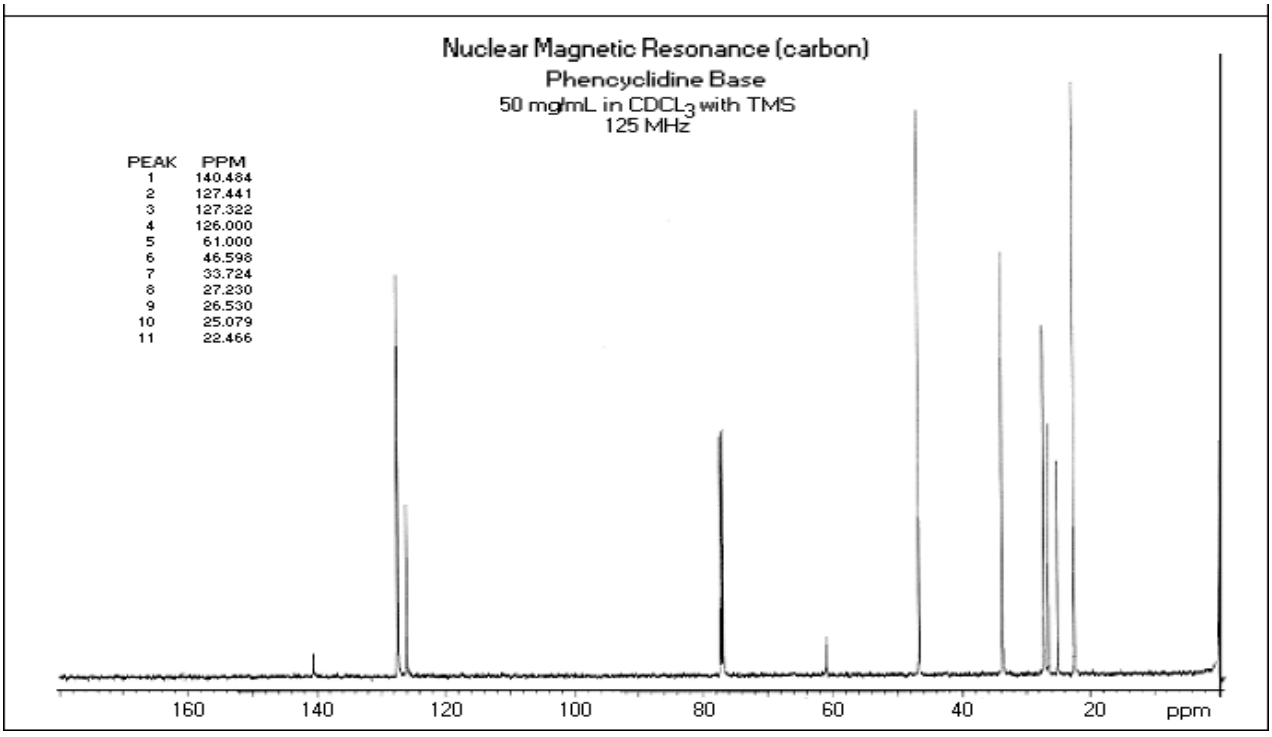


Full scan: molecular weight information (M+H+)+

MS/MS: Phencyclidine in MeOH;
Electrospray ionization; MS/MS positive ion mode; quadrupole ion-trap analyzer.



Tandem MS: MS/MS using standard collision energy of 35 eV.

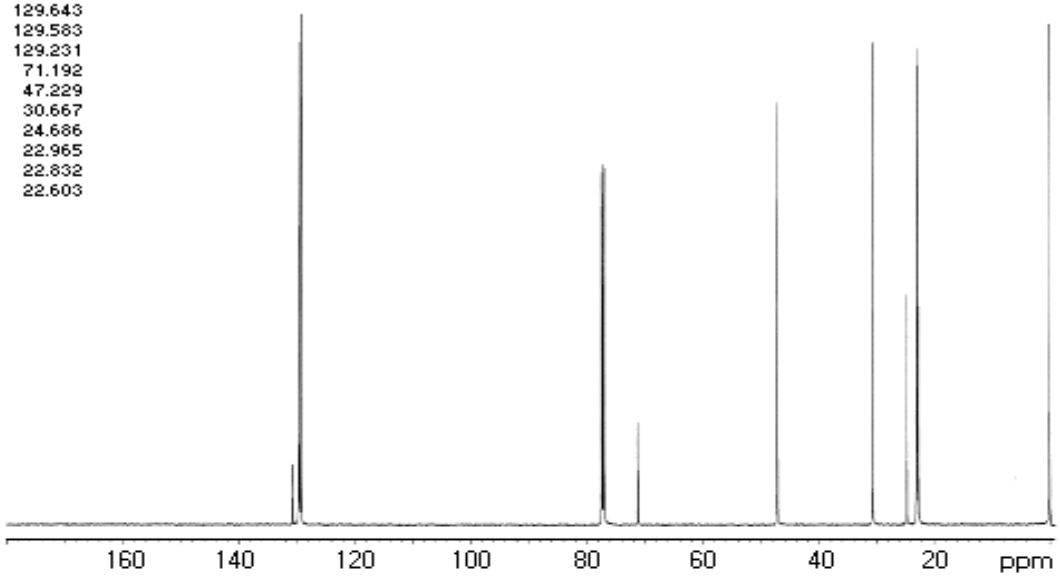


Nuclear Magnetic Resonance (carbon)

Phencyclidine Hydrochloride

50 mg/mL in $CDCL_3$ with TMS
125 MHz

PEAK	PPM
1	130.682
2	129.643
3	129.583
4	129.231
5	71.192
6	47.229
7	30.667
8	24.686
9	22.965
10	22.832
11	22.603

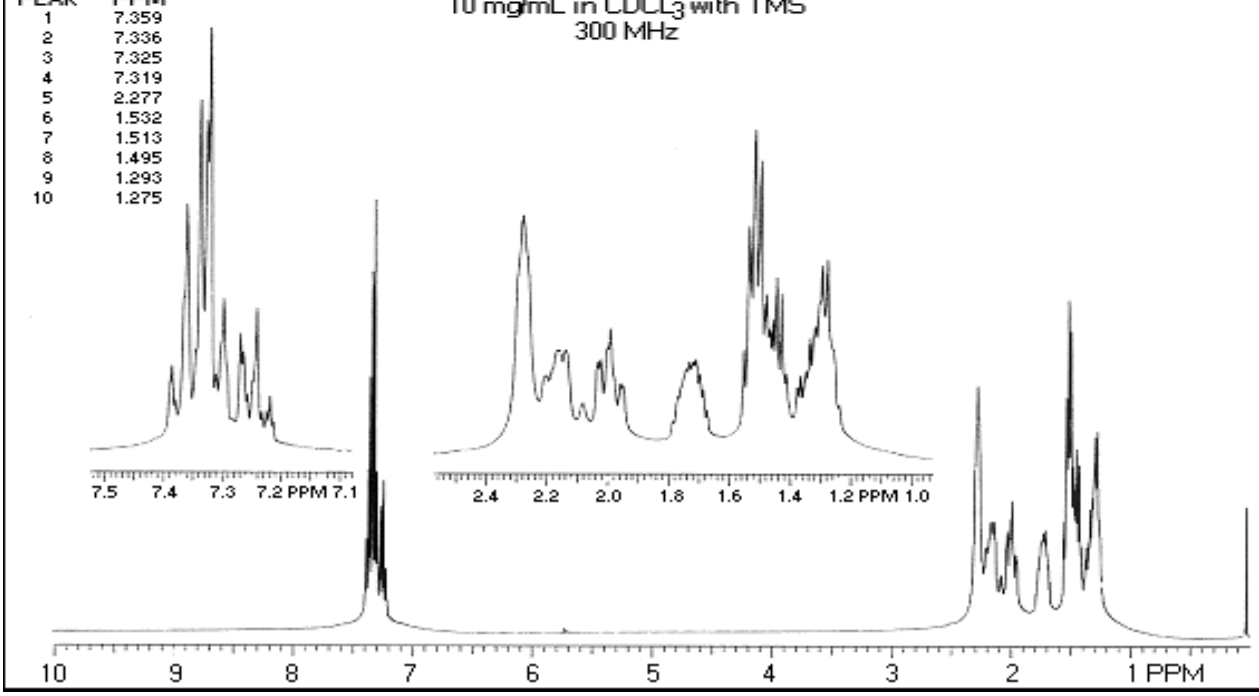


Nuclear Magnetic Resonance (proton)

Phencyclidine Base

10 mg/mL in $CDCL_3$ with TMS
300 MHz

PEAK	PPM
1	7.359
2	7.336
3	7.325
4	7.319
5	2.277
6	1.532
7	1.513
8	1.495
9	1.293
10	1.275



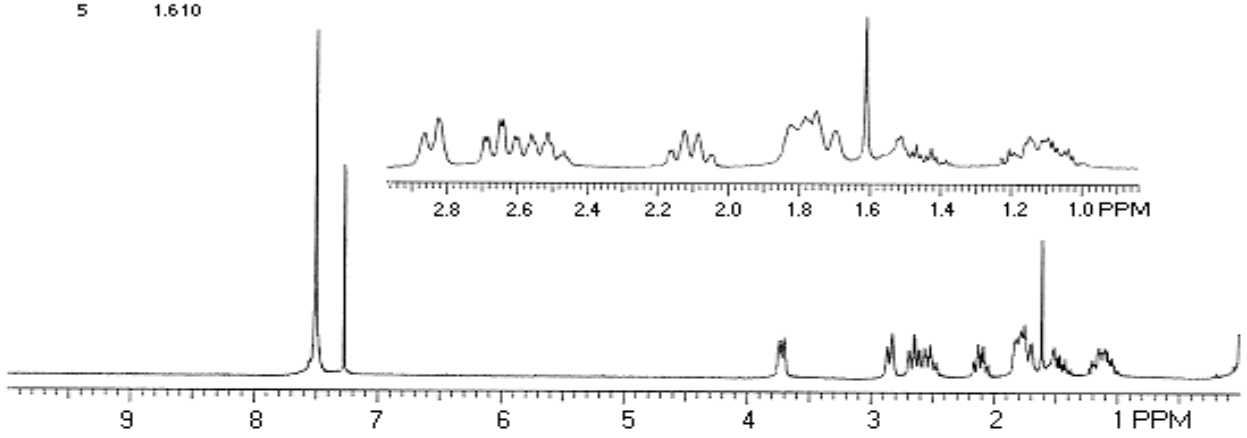
Nuclear Magnetic Resonance (proton)

Phencyclidine Hydrochloride

10 mg/mL in $CDCL_3$ with TMS

300 MHz

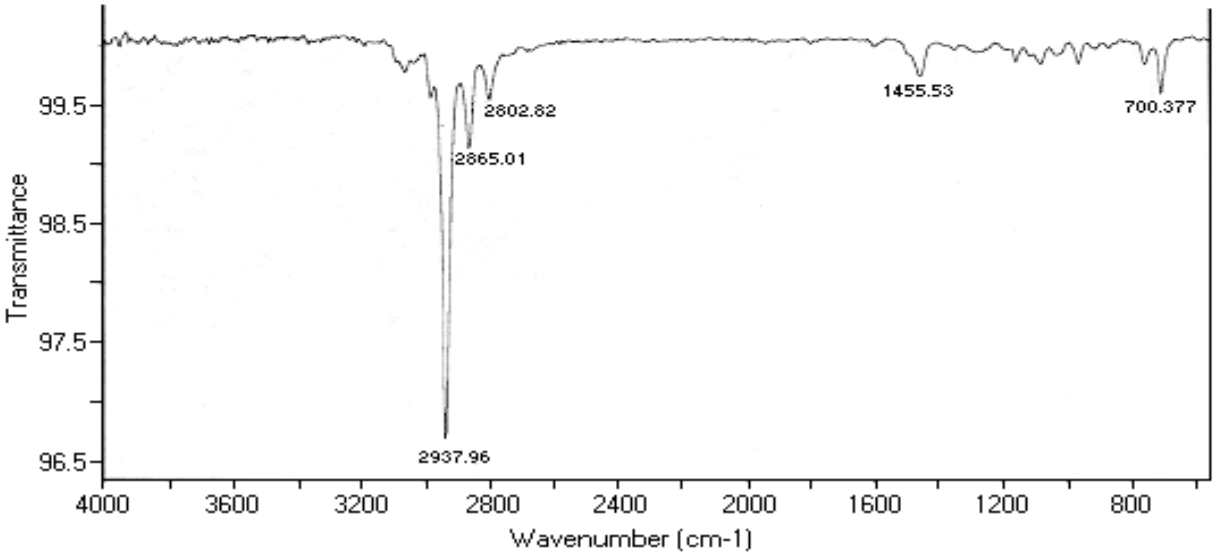
PEAK	PPM
1	7.511
2	7.499
3	1.781
4	1.751
5	1.610



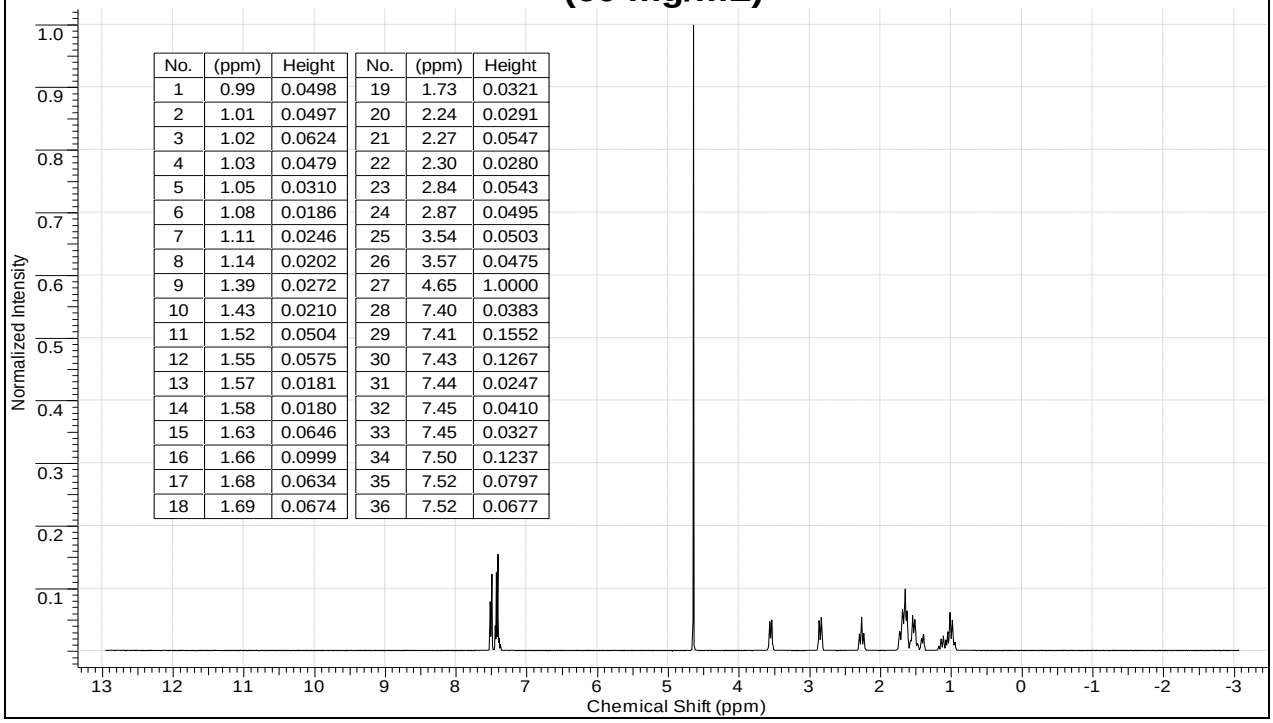
Vapor Phase IR

Phencyclidine

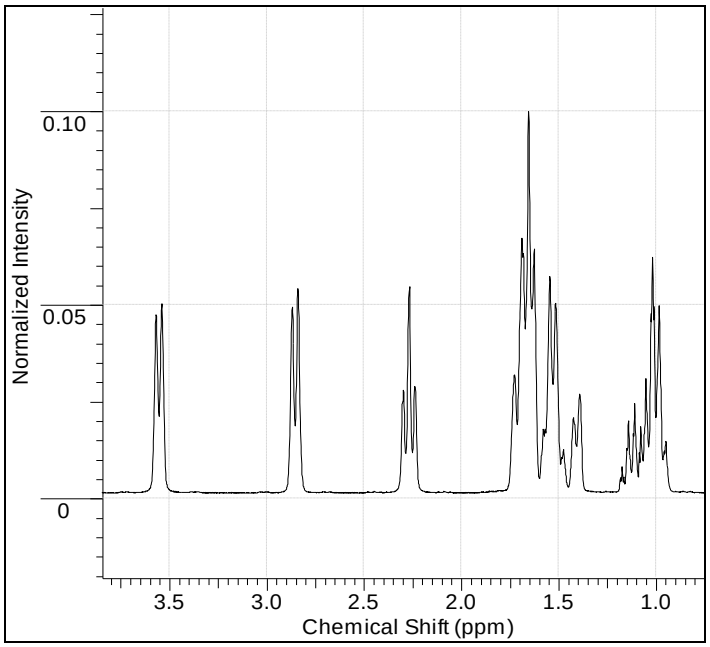
1.8 mg/mL in CH_3OH

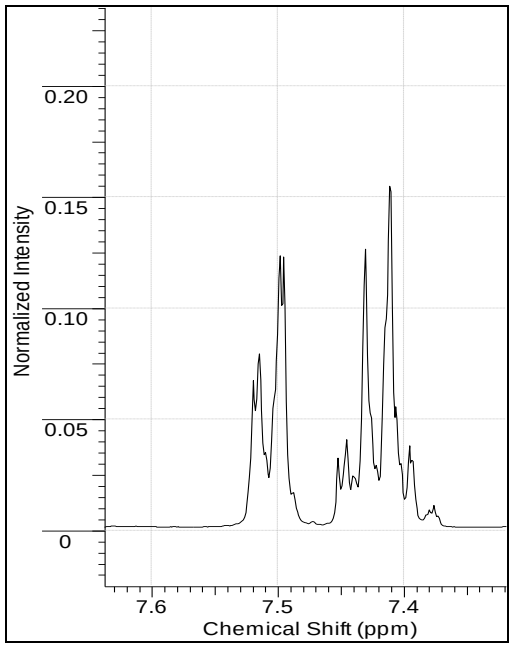


FT-NMR 400 MHz Proton
Phencyclidine HCl in D2O
(60 mg/mL)

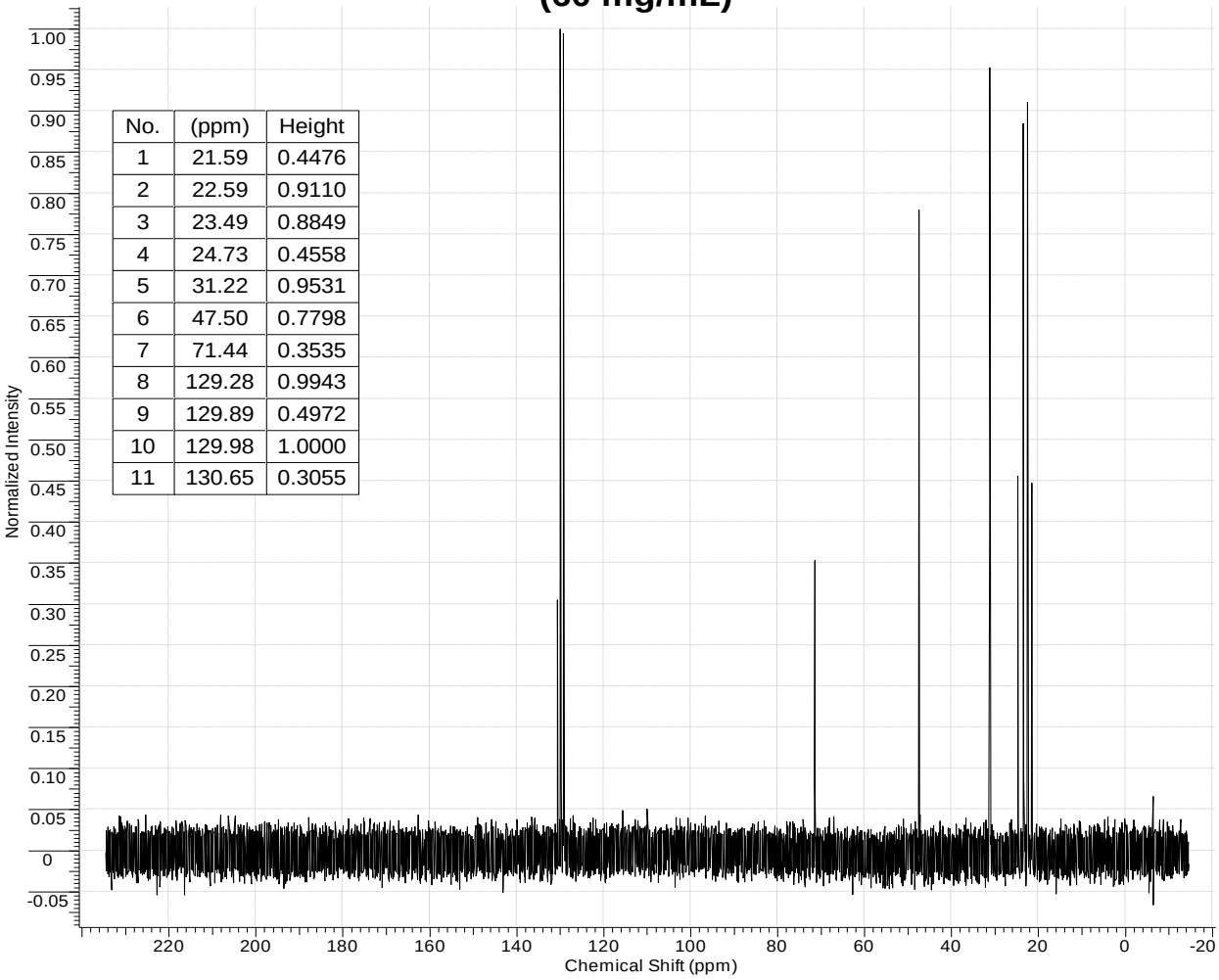


No.	(ppm)	Height	No.	(ppm)	Height
1	0.99	0.0498	19	1.73	0.0321
2	1.01	0.0497	20	2.24	0.0291
3	1.02	0.0624	21	2.27	0.0547
4	1.03	0.0479	22	2.30	0.0280
5	1.05	0.0310	23	2.84	0.0543
6	1.08	0.0186	24	2.87	0.0495
7	1.11	0.0246	25	3.54	0.0503
8	1.14	0.0202	26	3.57	0.0475
9	1.39	0.0272	27	4.65	1.0000
10	1.43	0.0210	28	7.40	0.0383
11	1.52	0.0504	29	7.41	0.1552
12	1.55	0.0575	30	7.43	0.1267
13	1.57	0.0181	31	7.44	0.0247
14	1.58	0.0180	32	7.45	0.0410
15	1.63	0.0646	33	7.45	0.0327
16	1.66	0.0999	34	7.50	0.1237
17	1.68	0.0634	35	7.52	0.0797
18	1.69	0.0674	36	7.52	0.0677





FT-NMR 400 MHz Carbon
Phencyclidine HCl in D2O
(60 mg/mL)



No.	(ppm)	Height
1	21.59	0.4476
2	22.59	0.9110
3	23.49	0.8849
4	24.73	0.4558
5	31.22	0.9531
6	47.50	0.7798
7	71.44	0.3535
8	129.28	0.9943
9	129.89	0.4972
10	129.98	1.0000
11	130.65	0.3055