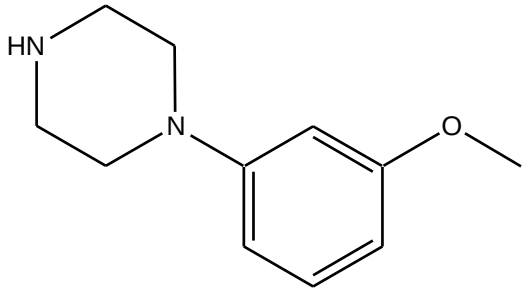
1-(3-METHOXYPHENYL)PIPERAZINE

Latest revision: June 27, 2005



1. SYNONYMS

CFR: N/A

CAS #: Dihydrochloride: 6968-76-9

Other Names: N/A

2. CHEMICAL AND PHYSICAL DATA

2.1. CHEMICAL DATA

Form	Chemical Formula	Molecular Weight	Melting Point (°C)
Dihydrochloride	$C_{11}H_{18}Cl_2N_2O$	265.18	214

2.2. SOLUBILITY

Form	A	C	E	H	M	W
Dihydrochloride	I	VSS	VSS	I	S	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

3. SCREENING TECHNIQUES

3.1. COLOR TESTS

REAGENT	COLOR PRODUCED
Cobalt thiocyanate	No reaction

Nitroprusside	Blue
Marquis	No reaction

3.2. CRYSTAL TESTS

REAGENT	COLOR PRODUCED
Platinic Bromide	Crosses with comb edges

3.3. THIN-LAYER CHROMATOGRAPHY

Visualization

Acidified iodoplatinate solution

COMPOUND	RELATIVE R_1 System TLC5
BZP	0.8
TFMPP	1.1
2-MeOPP	0.8
3-MeOPP	**1.0**
4-MeOPP	0.9

3.4. GAS CHROMATOGRAPH

Method PIPERAZINE-GCS

Instrument:	Gas chromatograph operated in split mode with FID
Column:	5% phenyl/95% methyl silicone 10 m x 0.32 mm x 0.52 µm
Carrier gas:	Hydrogen at 1.8 mL/min
Temperatures:	Injector: 280°C Detector: 280°C Oven program: 1) 100°C initial temperature for 1.0 min 2) Ramp to 280°C at 25°C/min 3) Hold final temperature for 3.0 min
Injection Parameters:	Split Ratio = 50:1, 1 µL injected

Samples are to be dissolved in methanol.

COMPOUND	**RRT**	**COMPOUND**	**RRT**
dimethyl sulfone	0.2128	3,4-methylenedioxymethamphetamine	0.800
methamphetamine	0.4724	1-(2-methoxyphenyl)piperazine	0.8865
dimethylphthalate	0.7298	1-(4-methoxylphenyl)piperazine	0.9879
benzylpiperazine	0.7670	**1-(3-methoxyphenyl)piperazine**	**1.0 (5.491 min)**
1-(3-trifluoromethylphenyl)piperazine	0.7971	Caffeine	1.044

4. SEPARATION TECHNIQUES

The solubility properties provided in the table 2.2 can be utilized to extract diluents and adulterants including other isomeric forms of 3-MeOPP. For example, chloroform may be used to separate 3-MeOPP from 2-MeOPP. 1-(3-methoxyphenyl)piperazine is very slightly soluble in $CHCl_3$ whereas 1-(2-methoxyphenyl)piperazine is fairly soluble in $CHCl_3$.

5. QUANTITATIVE PROCEDURE

5.1. GAS CHROMATOGRAPHY

Method PIPERAZINE1-GCQ1

Internal Standard Stock Solution:
0.25 mg/mL dimethylphthalate in methanol.

Standard Solution Preparation:
Accurately weigh and prepare a standard solution of 1-(3-methoxyphenyl)piperazine at approximately 1.0 mg/mL using above internal standard stock solution.

Sample Preparation:
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.

Instrument:	Gas chromatograph operated in split mode with FID
Column:	5% phenyl/95% methyl silicone 10 m x 0.3 2mm x 0.52 µm film thickness
Carrier gas:	Hydrogen at 1.0 mL/min
Temperatures:	Injector: 280°C Detector: 280°C Oven program: 1) 130°C initial temperature for 1.0 min 2) Ramp to 200°C at 25°C/min 3) Hold final temperature for 1.0 min
Injection Parameters:	Split Ratio = 50:1, 1 µL injected
Typical Retention Time:	1-(3-Methoxyphenyl)piperazine: 3.460 min Dimethylphthalate: 2.055 min
Linear Range:	0.0752 - 2.0824 mg/mL
Repeatability:	RSD less than 0.5%
Correlation Coefficient:	0.999
Accuracy:	Error less than 5%

COMPOUND	RRT	COMPOUND	RRT
methamphetamine	0.304	1-(2-methoxyphenyl)piperazine	0.826
dimethylphthalate	0.592	1-(4-methoxylphenyl)piperazine	0.973
benzylpiperazine	0.646	**1-(3-methoxyphenyl)piperazine**	**1.0(3.456min)**
1-(3-trifluoromethylphenyl)-piperazine	0.693	caffeine	1.072

5.2. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Method 3MEOPP-LCQ1

Sample Preparation:
Accurately weigh an amount of sample into a volumetric flask and dilute with 0.01N HCl. If necessary, dilute the sample so the final concentration approximates the standard concentration.

Instrument:	High performance liquid chromatograph equipped with diode array
Column:	4.6 mm x 250 mm, 10 µm C18(2)
Detector:	UV, 210 nm
Flow:	1.00 mL/min
Injection Volume:	3.0 µL
Buffer:	4000 mL distilled water, 10 g sodium hydroxide, 30.0 mL phosphoric acid and 8.0 mL hexylamine (NaHAP buffer)
Mobile Phase:	86% NaHAP Buffer: 14% Acetonitrile
Linear Range:	0.051-0.508 mg/mL
Repeatability:	Less than 3% RSD
Correlation Coefficient:	0.9999
Accuracy:	Error less than 5%

COMPOUND	RRT	COMPOUND	RRT
BZP	0.45	3-MeOPP	1.10
TFMPP	5.11	4-MeOPP	0.87
2-MeOPP	1.00 (5.135 min)		

5.3. CAPILLARY ELECTROPHORESIS

Method PIP-CEQ1

Internal Standard Stock Solution:
Thiamine hydrochloride internal standard at a concentration of 0.2 mg/mL.

Standard Solution Preparation:
Accurately weigh and prepare a standard solution at approximately 0.4mg/mL using the internal standard stock solution.

Sample Preparation:
Accurately weigh an amount of sample and dilute with internal standard stock solution. The sample concentration should approximate the standard.

Mode: Free zone

Column: 34 cm x 50 µm fused silica capillary

Run Buffer: 100 mM lithium phosphate buffer at pH 2.3

Detector: UV, 210 nm

Voltage: 20 kV

Temperature: 20°C air cooled

Injection: Hydrodynamic, 50 mbar for 2.5 seconds

Run Time: 6 min

Rinse Time: 1 min

Typical Migration Time: **1-(3-Methoxyphenyl)piperazine:** 4.754 min.
Thiamine: 3.144

Linear Range: 0.05 – 1.2 mg/mL

Repeatability: RSD less than 3%

Correlation Coefficient: 0.999

Accuracy: Error less than 5%

COMPOUND	RMT	COMPOUND	RMT
thiamine	0.661	1-(2-methoxyphenyl)piperazine	0.991
benzylpiperazine	0.741	**1-(3-methoxyphenyl)piperazine**	**1**
methamphetamine	0.934	1-(3-trifluoromethylphenyl)-piperazine	1.050
1-(4-methoxylphenyl)piperazine	0.961		

6. QUALITATIVE DATA

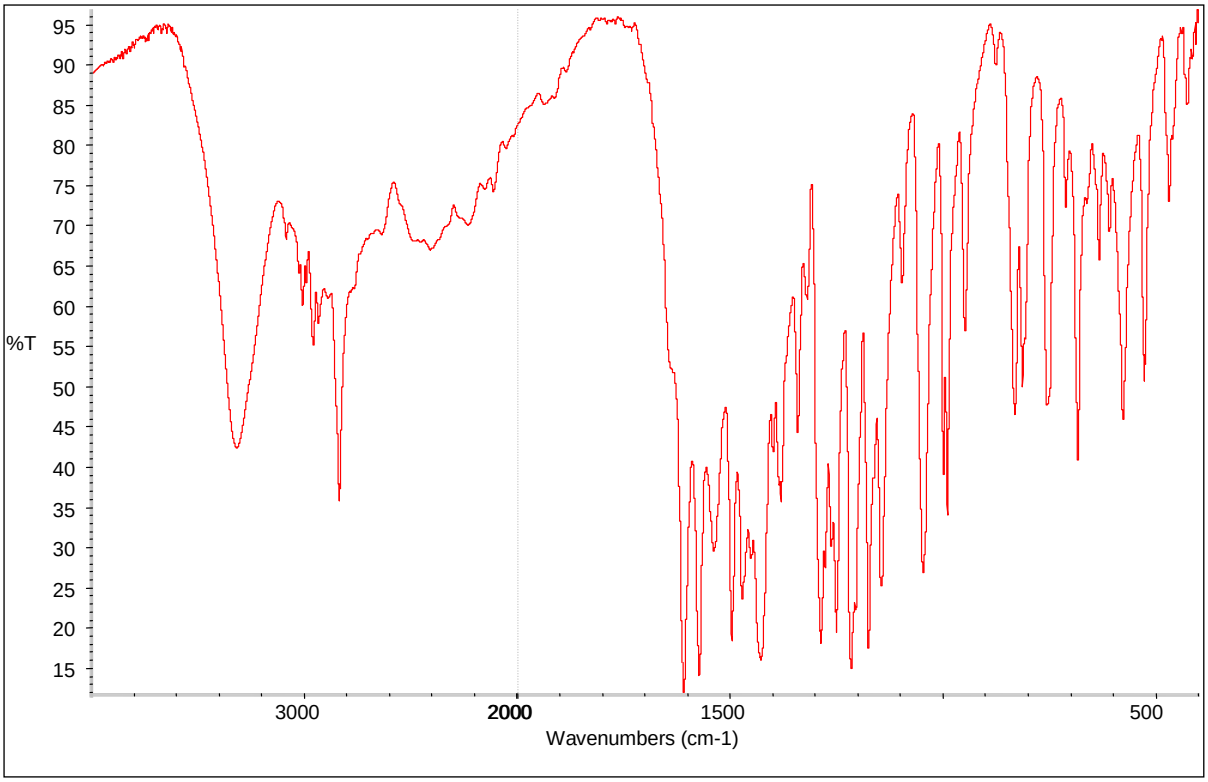
6.1. ULTRAVIOLET SPECTROPHOTOMETRY

SOLVENT	MAXIMUM ABSORBANCE (NM)
Aqueous acid	210

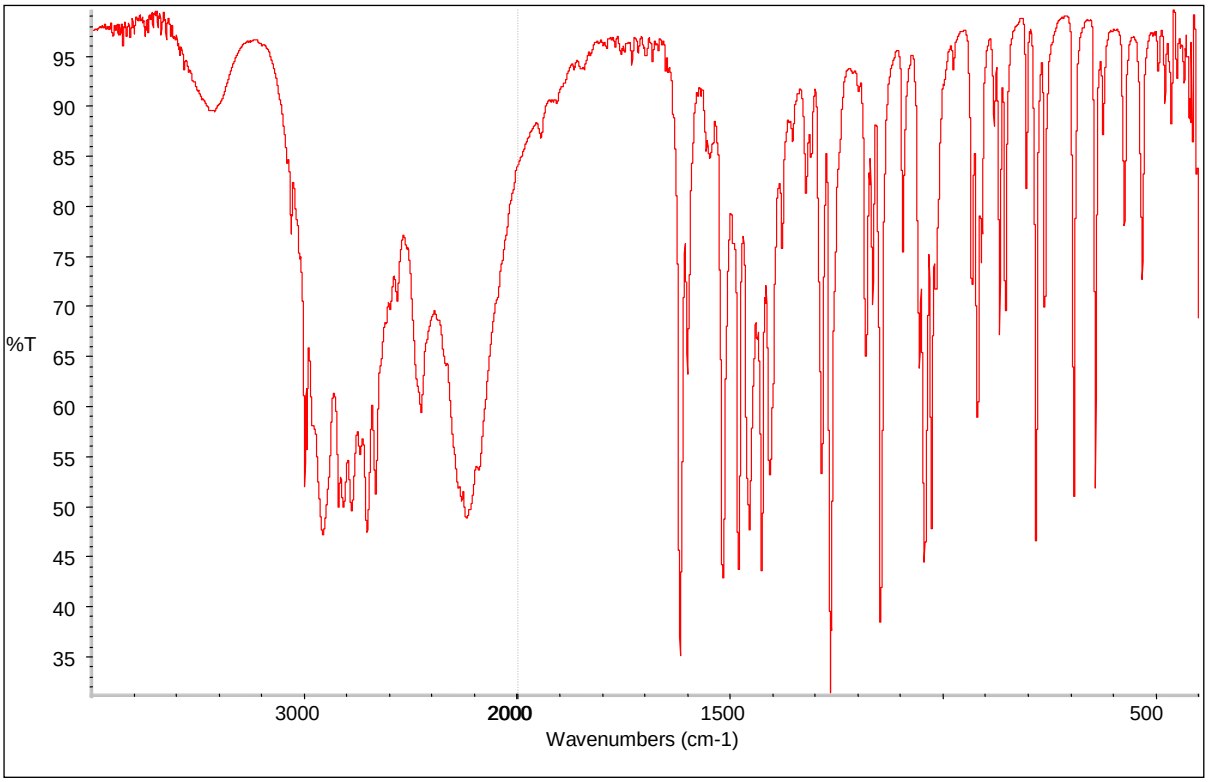
7. REFERENCES

https://fscimage.fishersci.com/msds/21631.htm

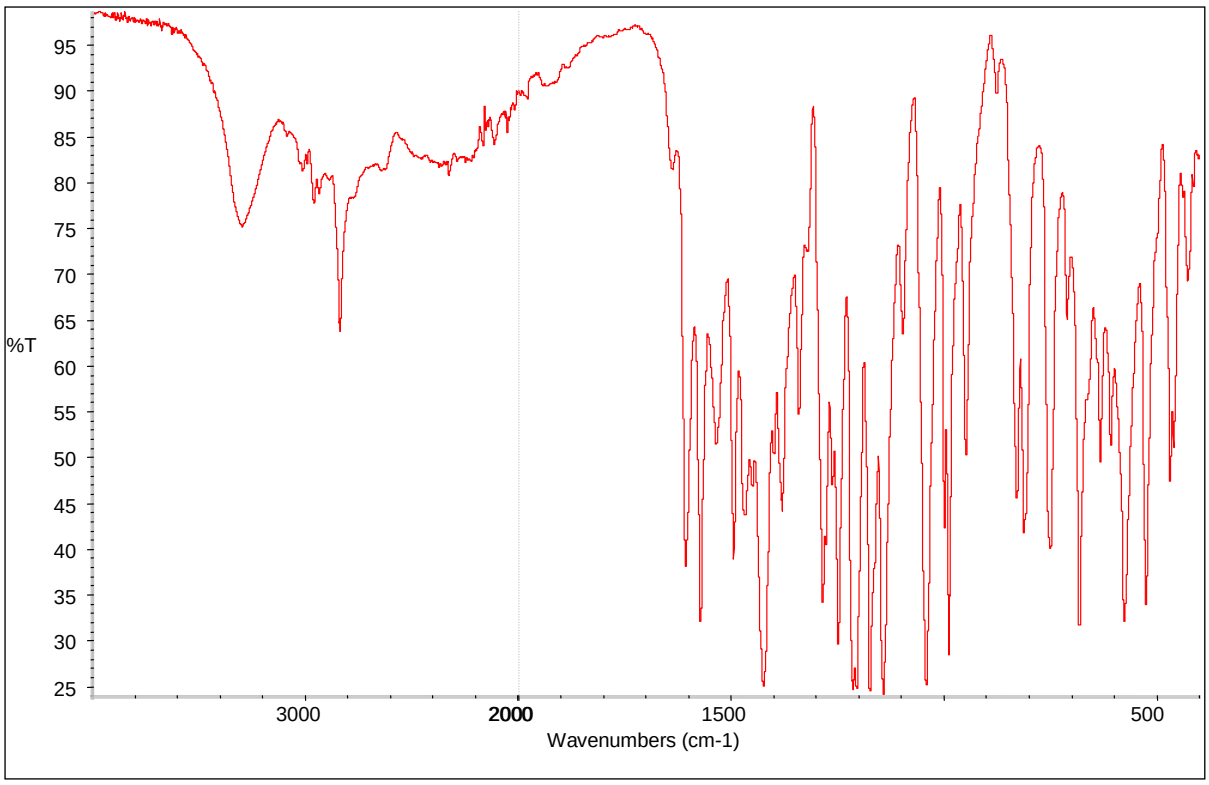
FTIR: 1-(3-Methoxyphenyl)piperazine base on KBr
16 scans; 4 cm^{-1} resolution



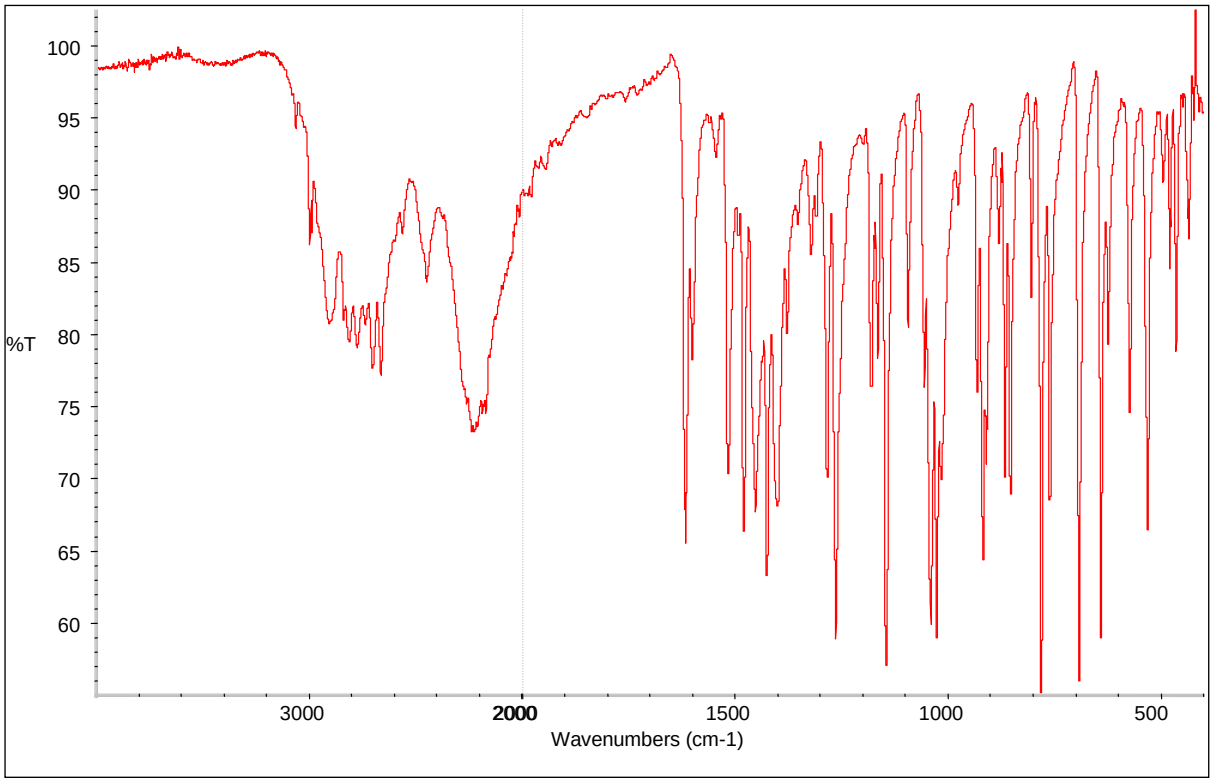
FTIR: 1-(3-Methoxyphenyl)piperazine diHCl in KBr
16 scans; 4 cm^{-1} resolution



FTIR (ATR): 1-(3-Methoxyphenyl)piperazine base
16 scans; 4 cm^{-1} resolution

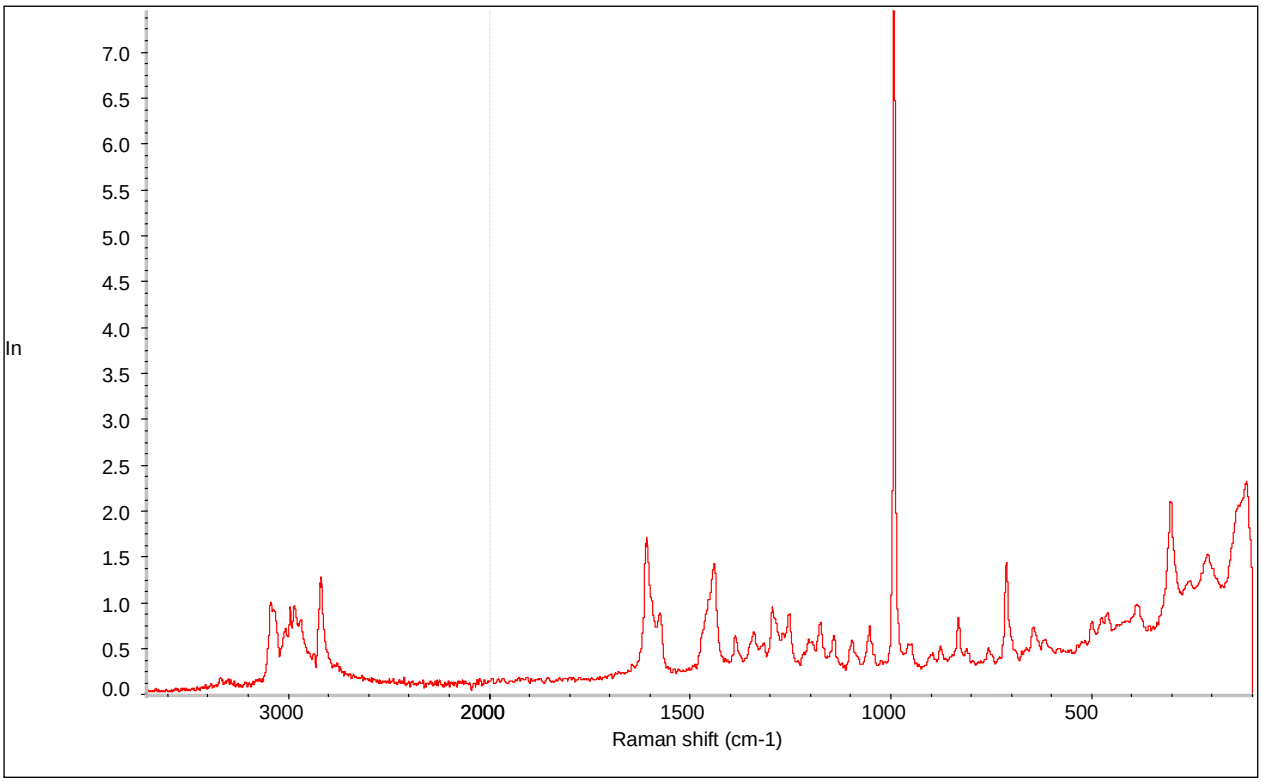


FTIR (ATR): 1-(3-Methoxyphenyl)piperazine diHCl
16 scans; 4 cm^{-1} resolution

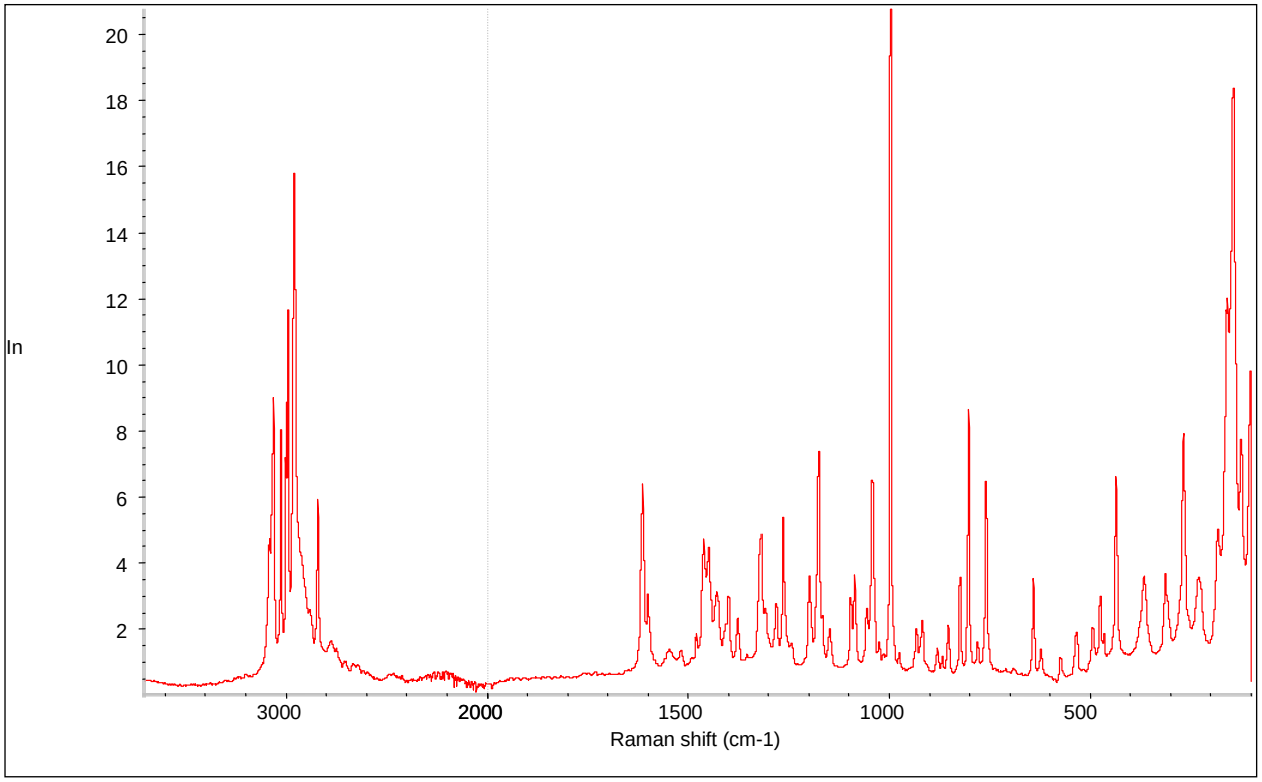


FT RAMAN: 1-(3-Methoxyphenyl)piperazine base

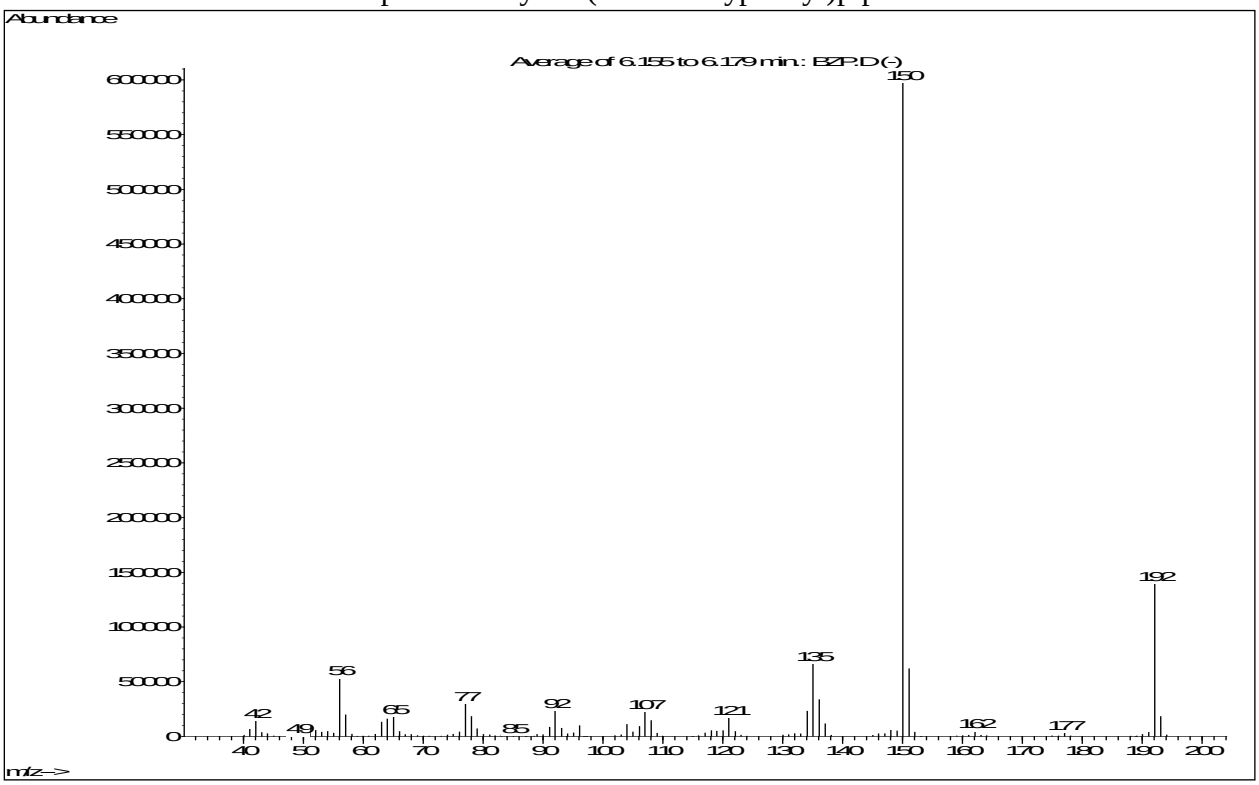
256 scans; 4.0 nm resolution



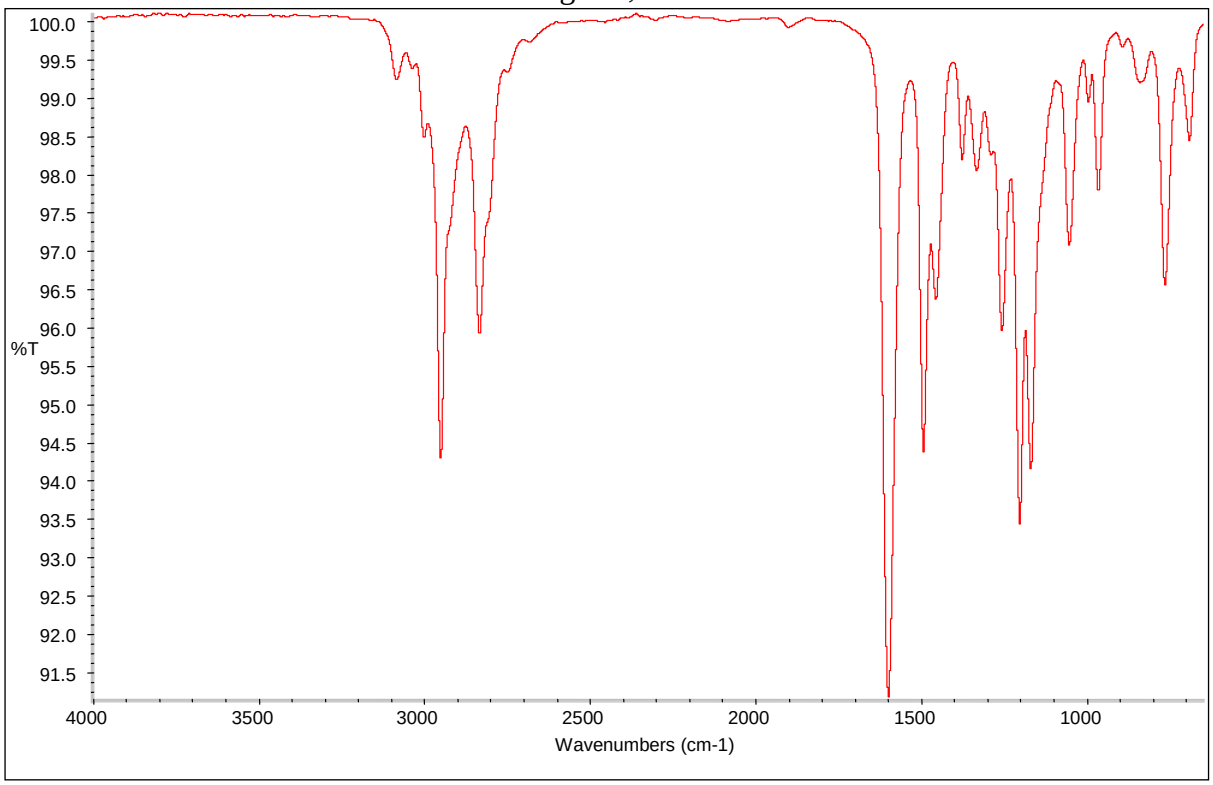
FT RAMAN: 1-(3-Methoxyphenyl)piperazine diHCl
256 scans; 4.0 nm resolution

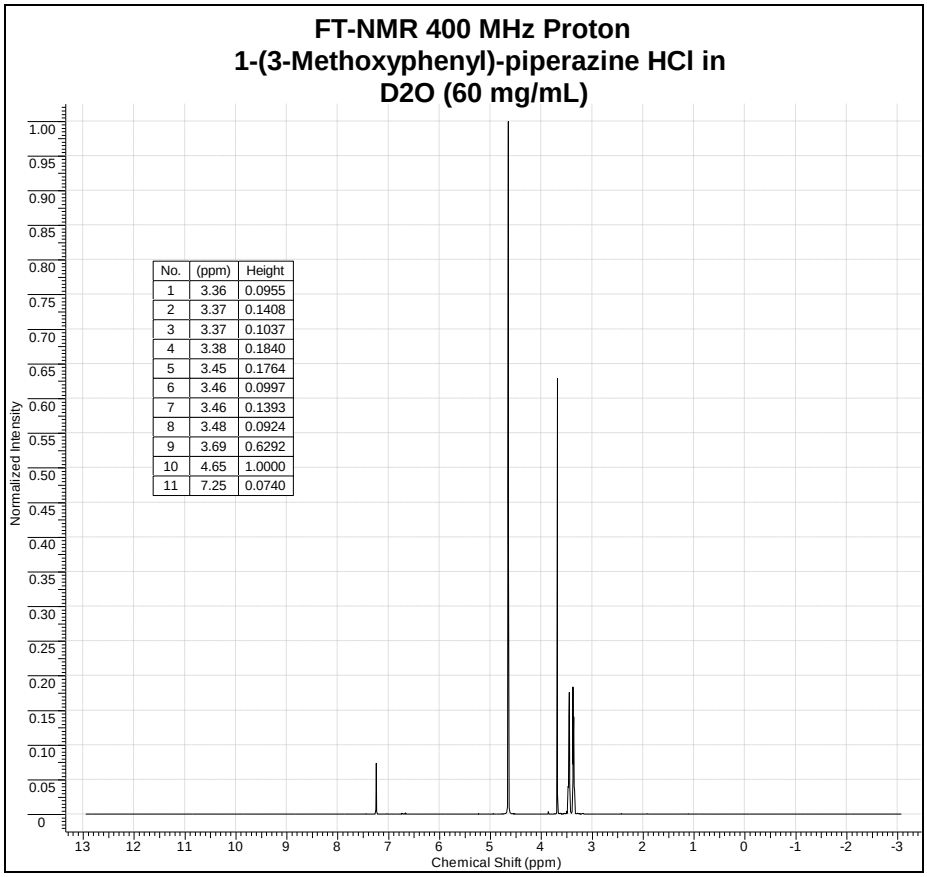


Mass Spectrometry: 1-(3-Methoxyphenyl)piperazine

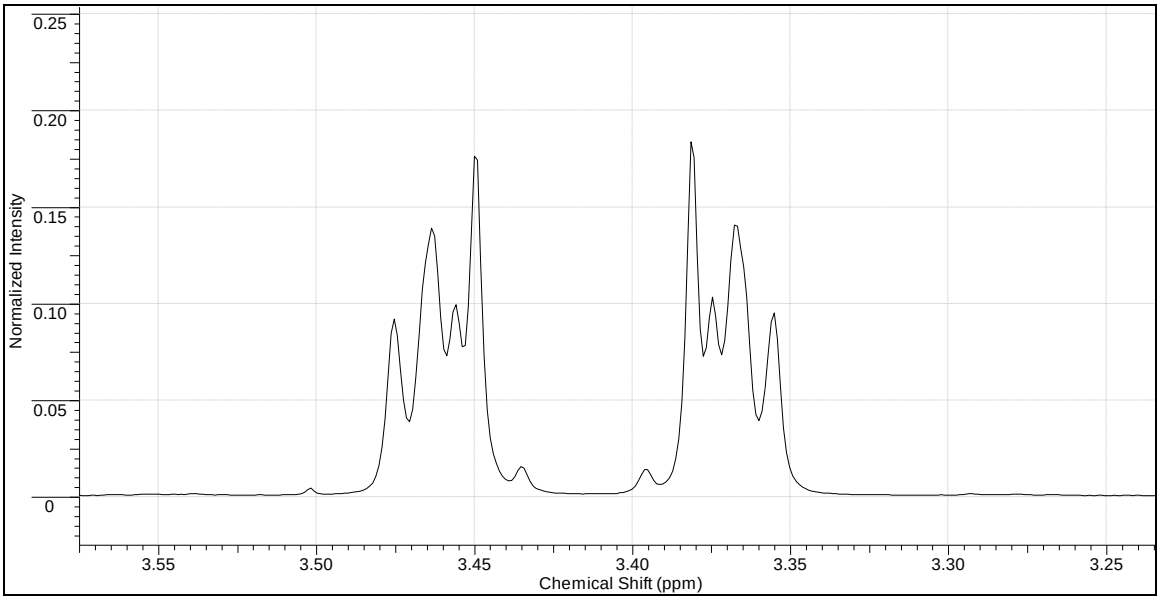


Vapor Phase IR: 1-(3-Methoxyphenyl)piperazine
0.5mg/mL, 8cm^{-1}

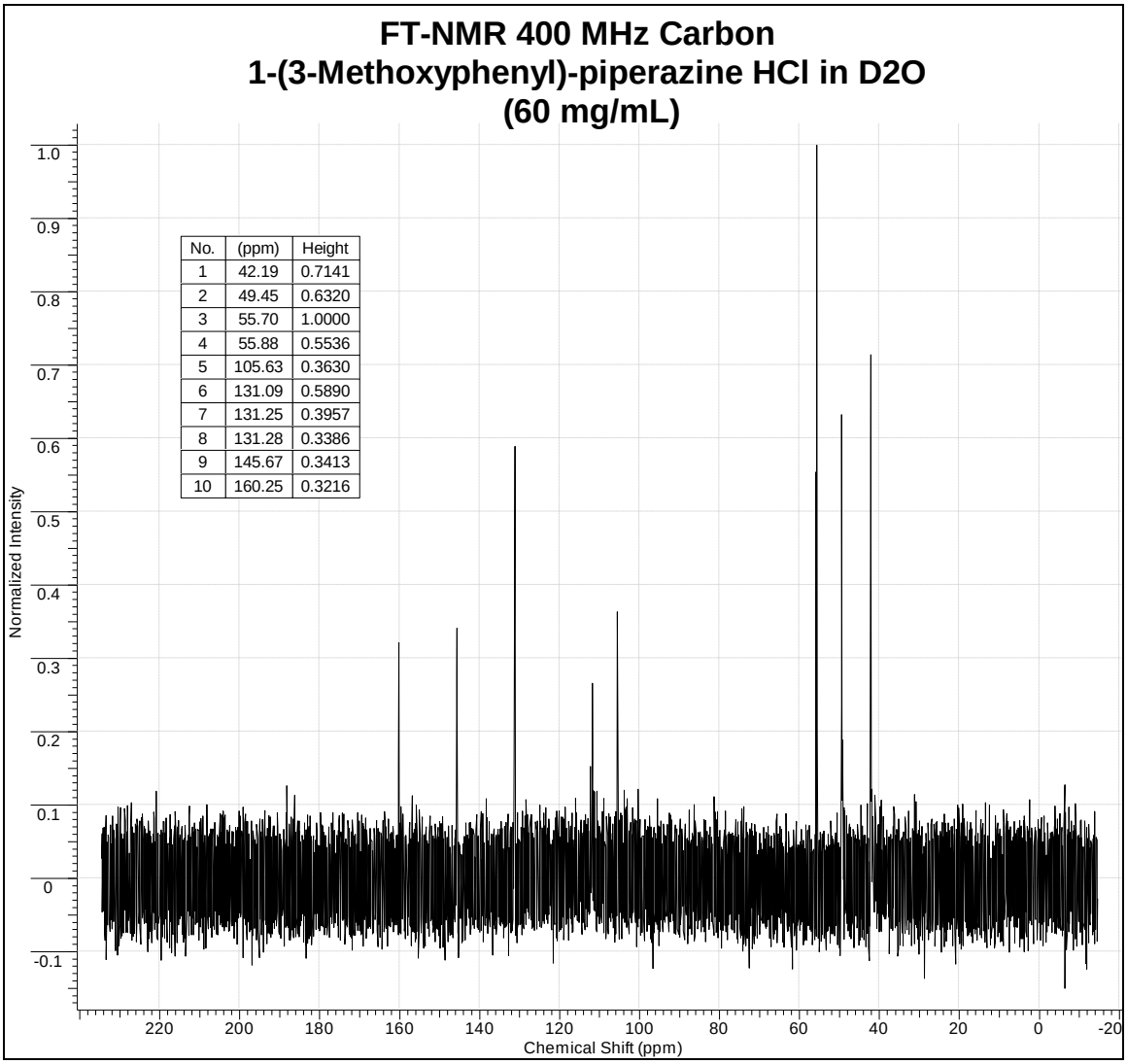




No.	(ppm)	Height
1	3.36	0.0955
2	3.37	0.1408
3	3.37	0.1037
4	3.38	0.1840
5	3.45	0.1764
6	3.46	0.0997
7	3.46	0.1393
8	3.48	0.0924
9	3.69	0.6292
10	4.65	1.0000
11	7.25	0.0740



FT-NMR 400 MHz Carbon
1-(3-Methoxyphenyl)-piperazine HCl in D2O
(60 mg/mL)



No.	(ppm)	Height
1	42.19	0.7141
2	49.45	0.6320
3	55.70	1.0000
4	55.88	0.5536
5	105.63	0.3630
6	131.09	0.5890
7	131.25	0.3957
8	131.28	0.3386
9	145.67	0.3413
10	160.25	0.3216