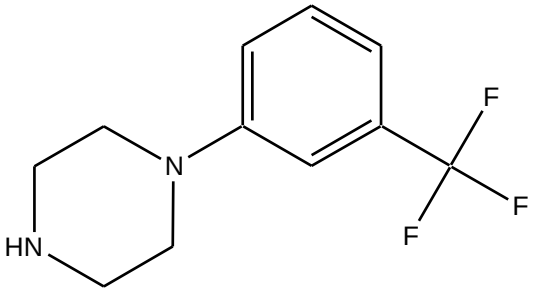
1-(3-TRIFLUOROMETHYLPHENYL)PIPERAZINE

Latest revision: June 27, 2005



1. SYNONYMS

CFR: N/A

CAS #: Hydrochloride: 16015-69-3

Other Names: TFMPP

2. CHEMICAL AND PHYSICAL DATA

2.1. CHEMICAL DATA

Form	Chemical Formula	Molecular Weight	Melting Point (°C)
Hydrochloride	$C_{11}H_{14}Cl\ F_3N_2$	266.70	237

2.2. SOLUBILITY

Form	A	C	E	H	M	W
Hydrochloride	SS	S	VSS	I	FS	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	No Reaction
Marquis	No reaction

3.2. CRYSTAL TESTS

REAGENT	COLOR PRODUCED
Platinic Bromide	Oils, then clusters of rods from center core (bundles of rods), (overgrown bow ties)

3.3. THIN-LAYER CHROMATOGRAPHY

Visualization

Acidified iodoplatinate solution

COMPOUND	RELATIVE R_1 System TLC5
BZP	0.6
TFMPP	**1.0**
2-MeOPP	0.7
3-MeOPP	0.8
4-MeOPP	0.7

3.4. GAS CHROMATOGRAPHY

Method PIPERAZINE-GCS1

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	0.267	3,4-methylenedioxymethamphetamine	1.004
methamphetamine	0.592	1-(2-methoxyphenyl)piperazine	1.112
dimethylphthalate	0.911	1-(4-methoxylphenyl)piperazine	1.239
benzylpiperazine	0.962	1-(3-methoxyphenyl)piperazine	1.254
1-(3-trifluoromethylphenyl)piperazine	**1.0(4.377 min)**	caffeine	1.310

4. SEPARATION TECHNIQUES

The solubility properties provided in the table 2.2 can be utilized to extract diluents and adulterants. For example, acetone may be used to separate TFMPP from 2-MeOPP or 3-MeOPP. 1-(3-Trifluoromethylphenyl)piperazine is slightly soluble in acetone whereas 1-(2-Methoxyphenyl)piperazine and 1-(3-Methoxyphenyl)piperazine are insoluble in acetone. Methanol can be used to separate TFMPP from 4-MeOPP since TFMPP is fairly soluble in methanol and 4-MeOPP is very slightly soluble.

5. QUANTITATIVE PROCEDURES

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-trifluoromethylphenyl)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 0.32 mm 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-Trifluoromethylphenyl)piperazine: 2.40 min Dimethylphthalate: 2.054 min
Linear Range:	0.1344 - 2.0064 mg/mL
Repeatability:	RSD less than 0.5%
Correlation Coefficient:	0.9999
Accuracy:	Error less than 5%

COMPOUND	RRT	COMPOUND	RRT
methamphetamine	0.439	1-(2-methoxyphenyl)piperazine	1.191
dimethylphthalate	0.854	1-(4-methoxylphenyl)piperazine	1.402
benzylpiperazine	0.931	1-(3-methoxyphenyl)piperazine	1.441
1-(3-trifluoromethylphenyl)piperazine	**1.0(2.398min)**	caffeine	1.545

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 s

Run Time: 6 min

Rinse Time: 1 min

Typical Migration Time: 1-(3-Trifluoromethylphenyl)piperazine: 4.994 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	0.630	1-(2-methoxyphenyl)piperazine	0.944
benzylpiperazine	0.706	1-(3-methoxyphenyl)piperazine	0.952
methamphetamine	0.889	**1-(3-trifluoromethylphenyl)piperazine**	**1**
1-(4-methoxylphenyl)-piperazine	0.915		

6. QUALITATIVE DATA

6.1. ULTRAVIOLET SPECTROPHOTOMETRY

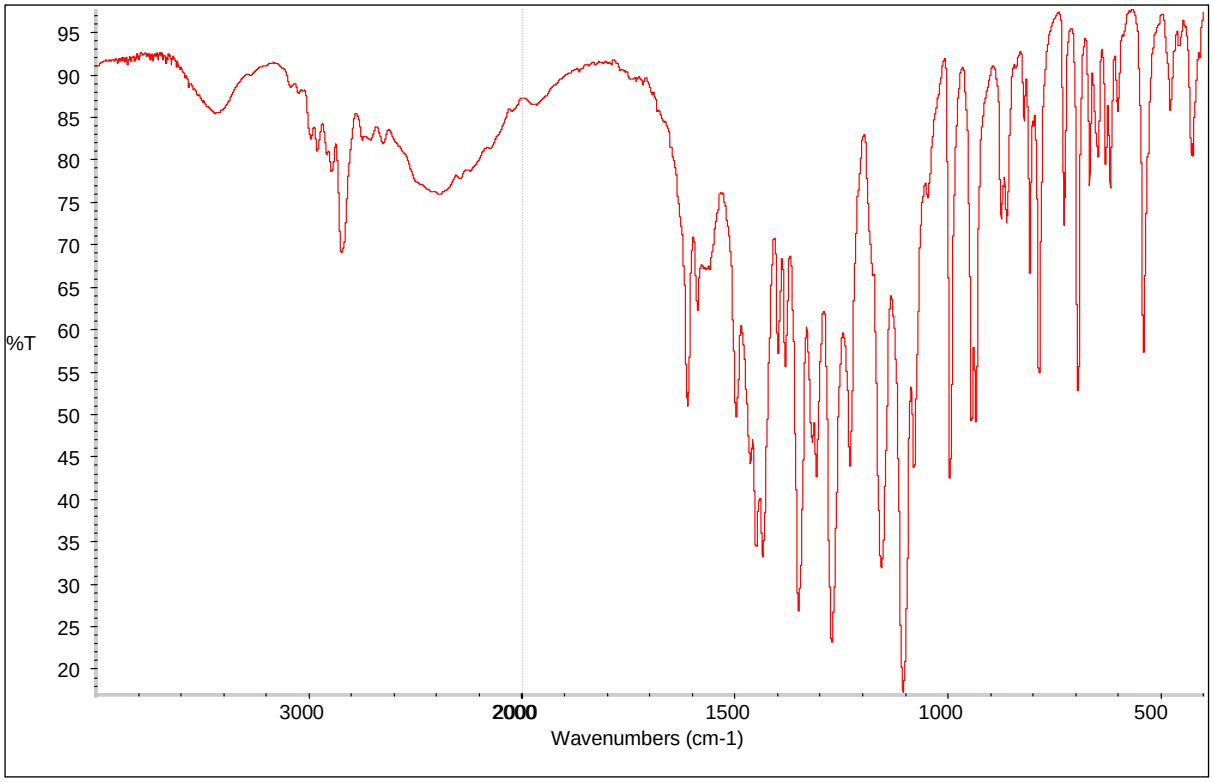
SOLVENT	MAXIMUM ABSORBANCE (NM)
Aqueous acid	202

7. ADDITIONAL RESOURCES

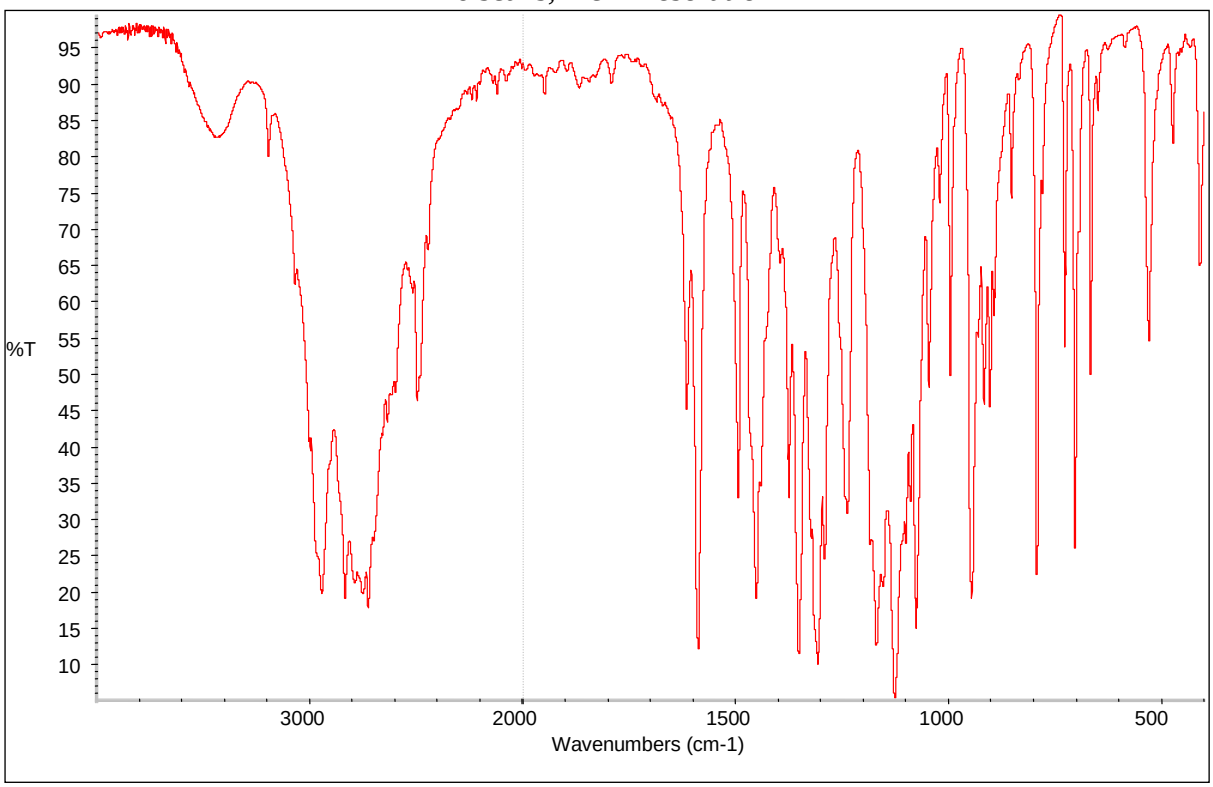
Forendex

Wikipedia

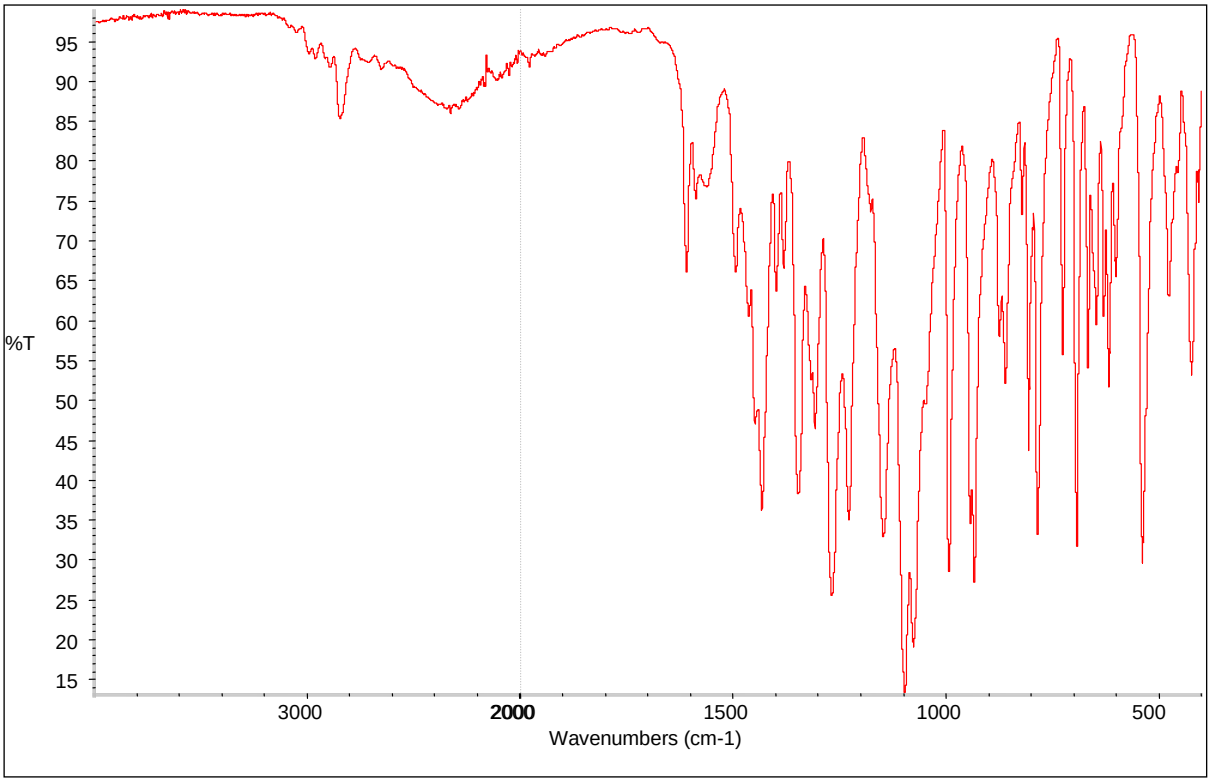
FTIR: 1-(3-Trifluoromethylphenyl)piperazine base on KBr
16 scans; 4 cm^{-1} resolution



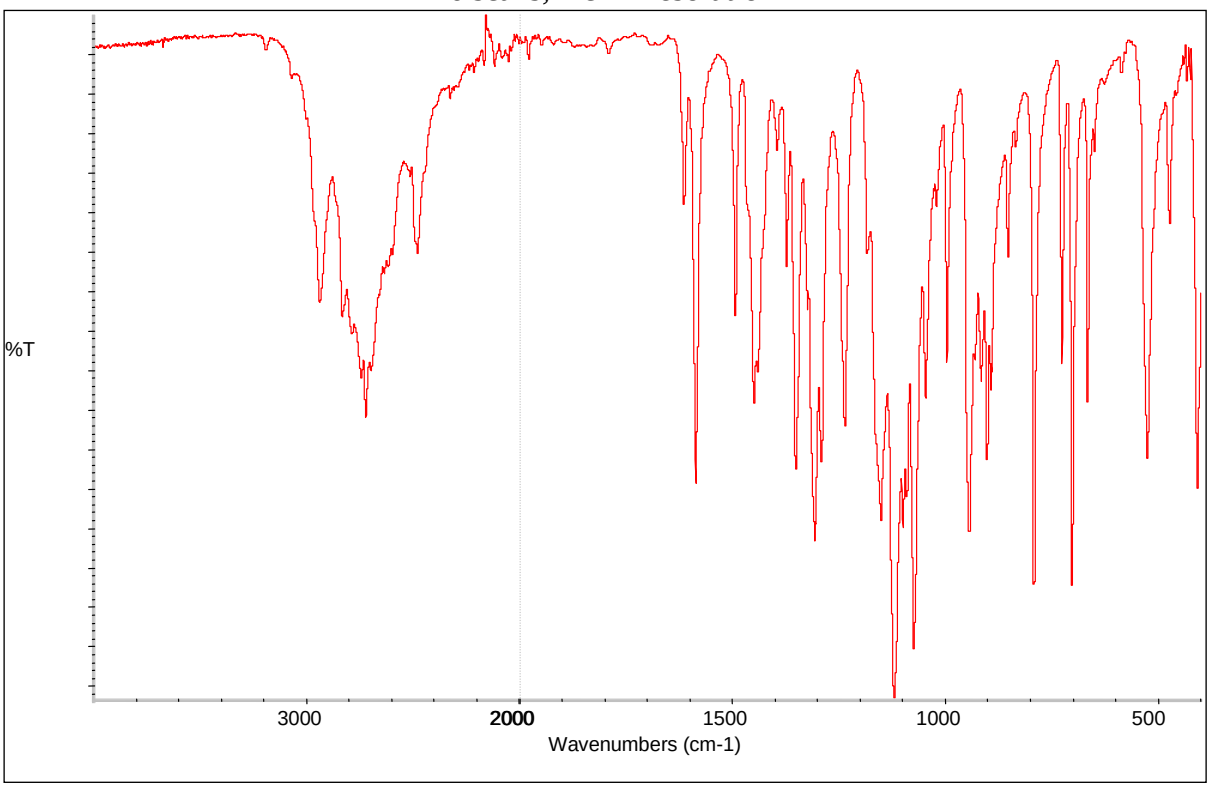
FTIR: 1-(3-Trifluoromethylphenyl)piperazine HCl in KBr
16 scans; 4 cm^{-1} resolution



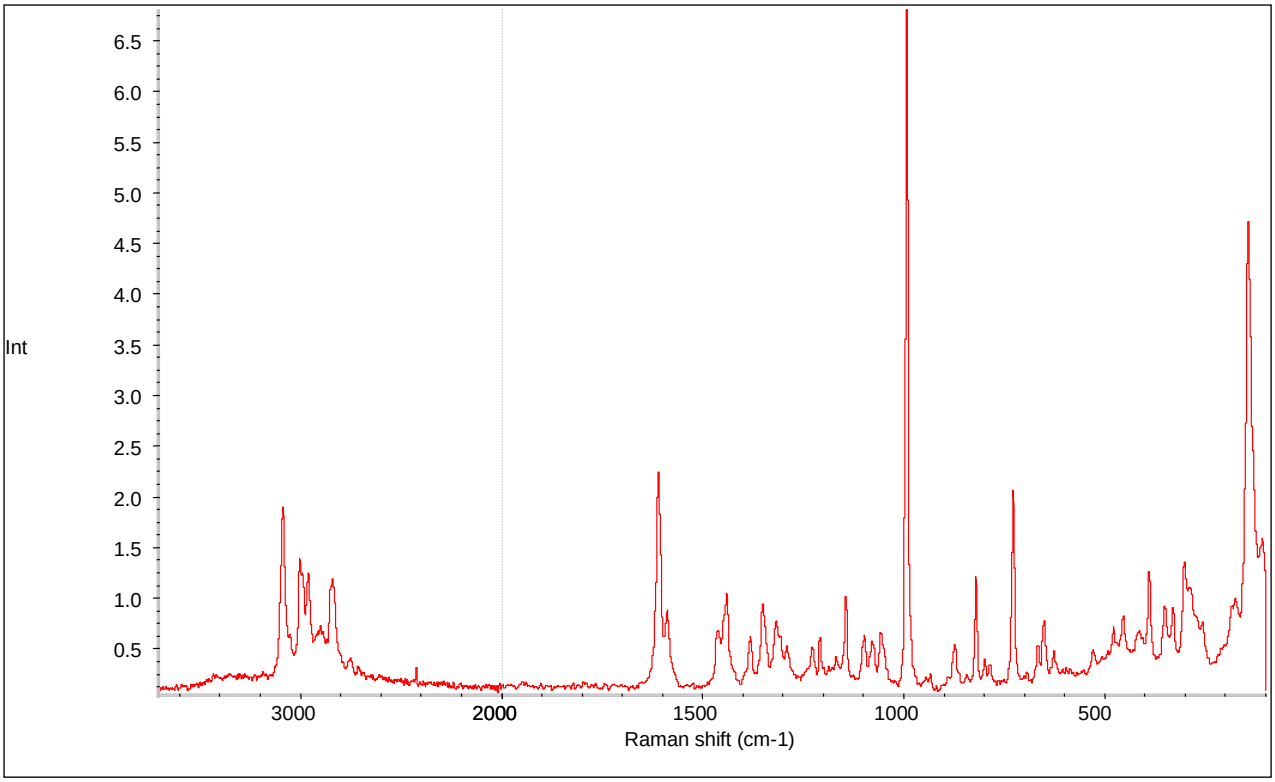
FTIR (ATR): 1-(3-Trifluoromethylphenyl)piperazine base
16 scans; 4 cm^{-1} resolution



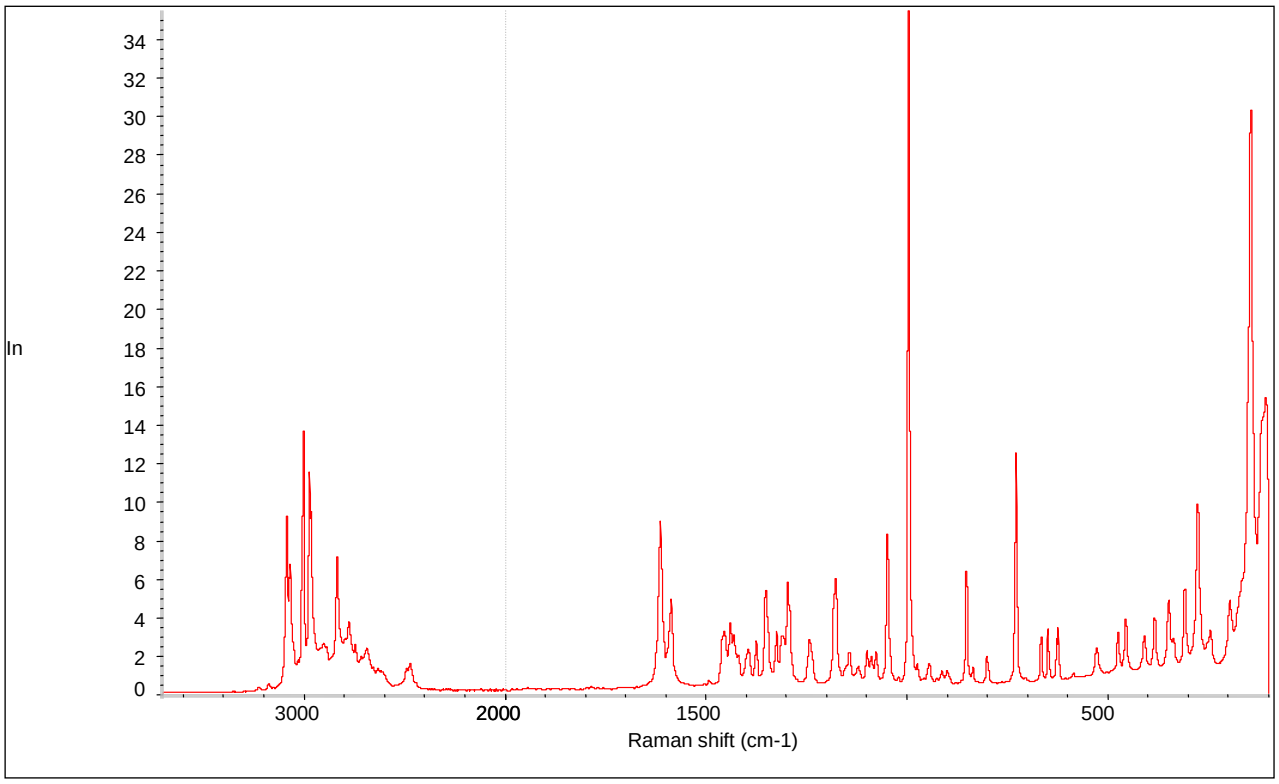
FTIR (ATR): 1-(3-Trifluoromethylphenyl)piperazine HCl
16 scans; 4 cm^{-1} resolution



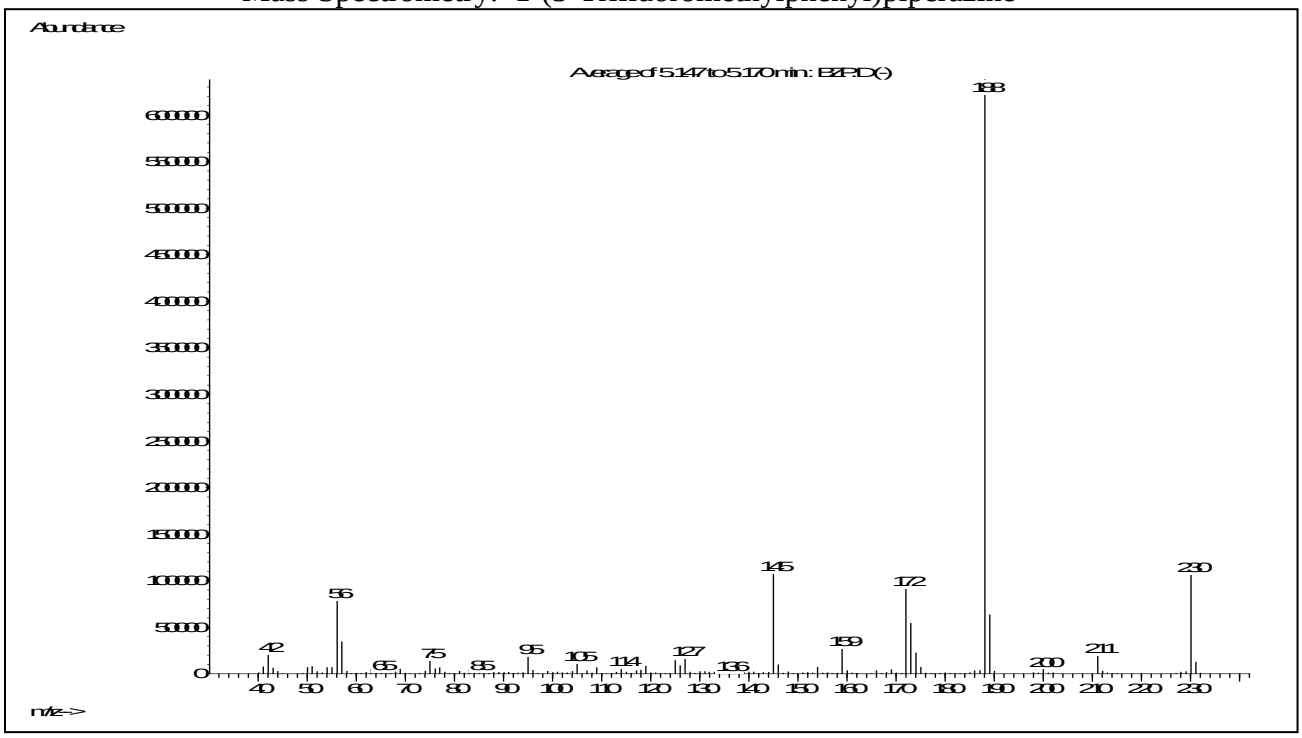
FT RAMAN: 1-(3-Trifluoromethylphenyl)piperazine base
256 scans; 4.0 nm resolution



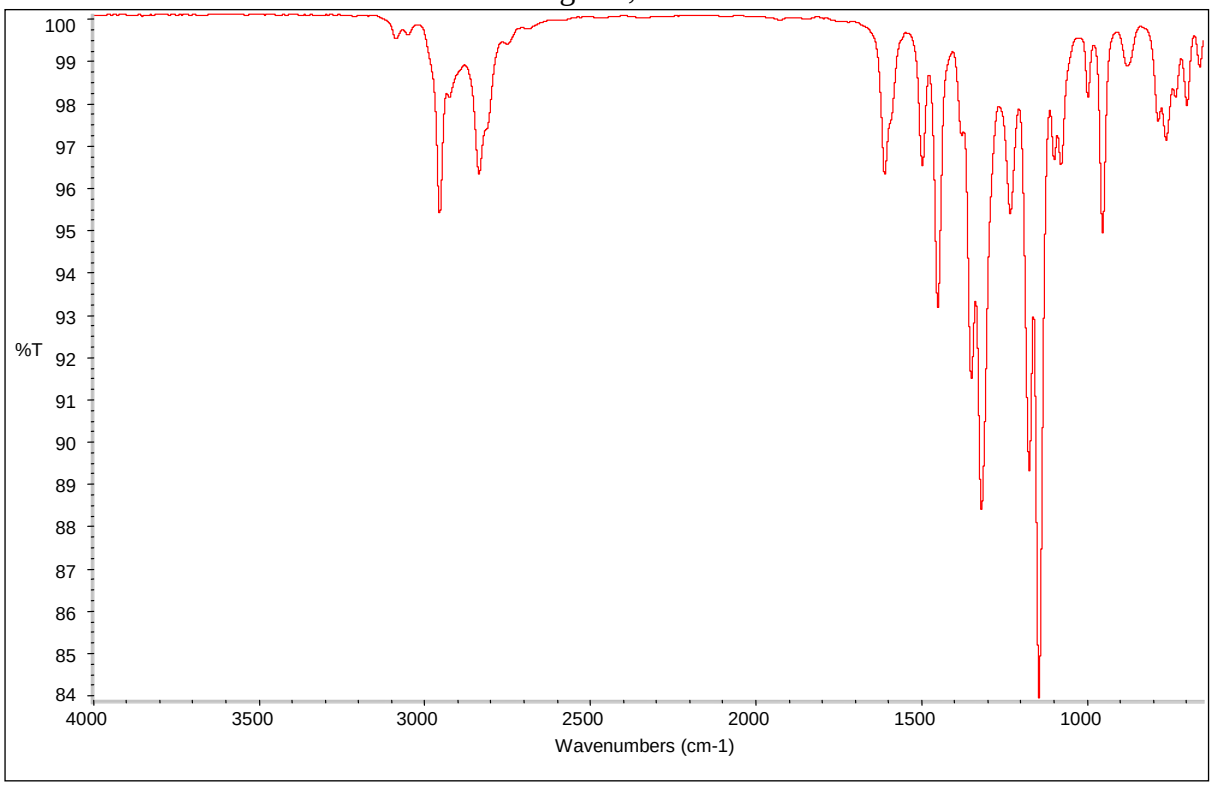
FT RAMAN: 1-(3-Trifluoromethylphenyl)piperazine HCl
256 scans; 4.0 nm resolution



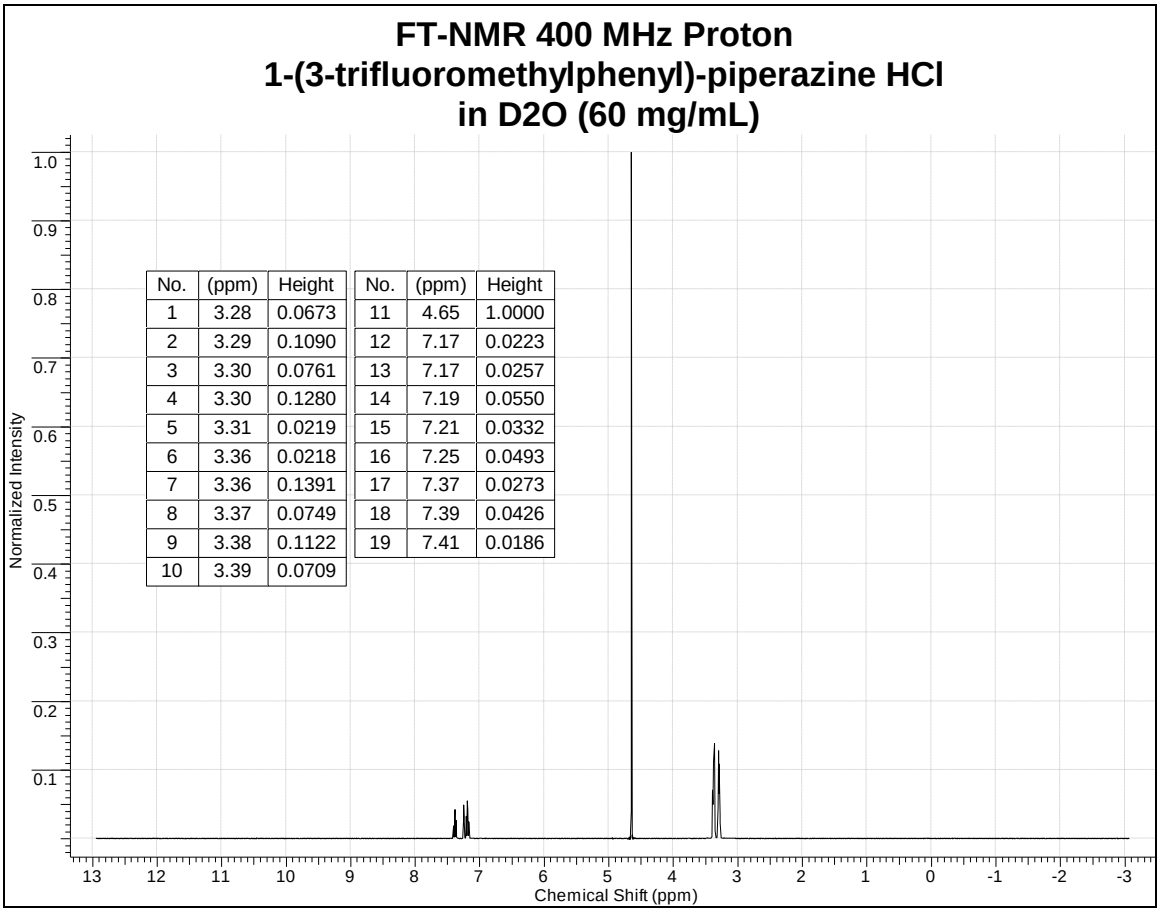
Mass Spectrometry: 1-(3-Trifluoromethylphenyl)piperazine



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

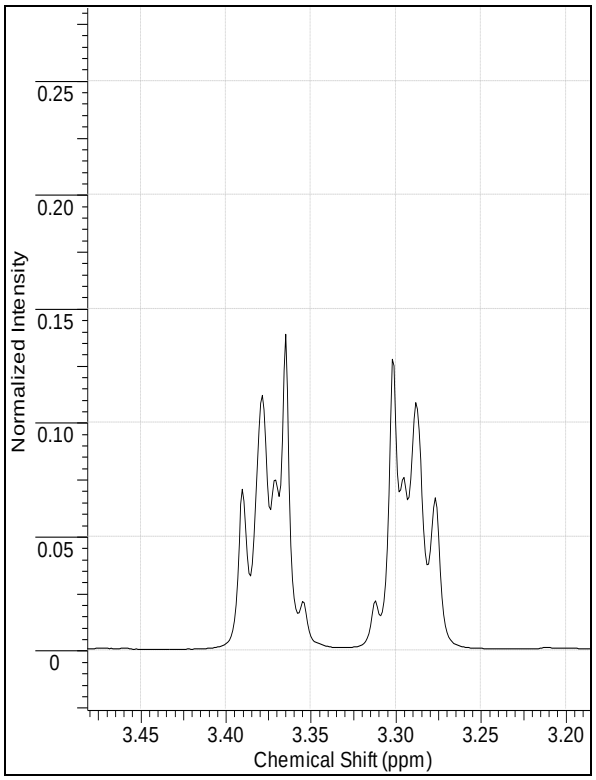


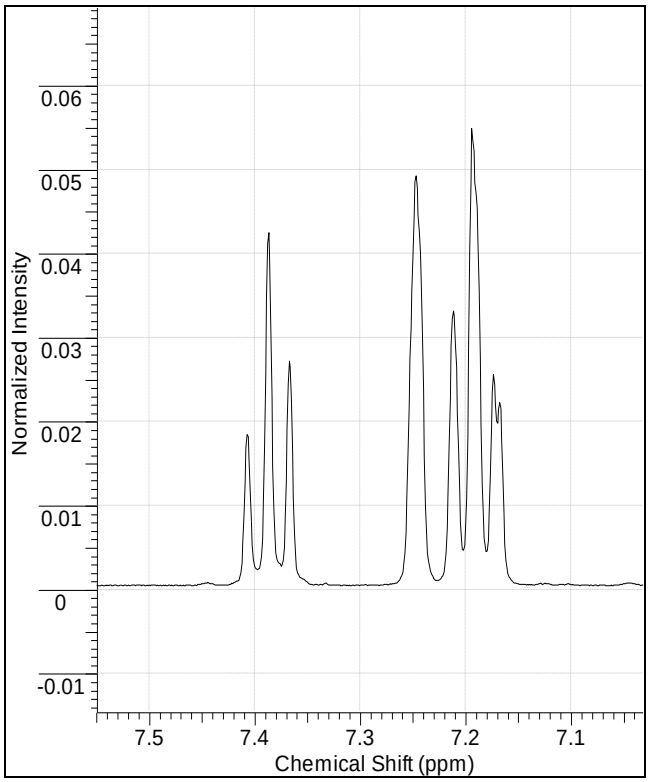
FT-NMR 400 MHz Proton
1-(3-trifluoromethylphenyl)-piperazine HCl
in D2O (60 mg/mL)



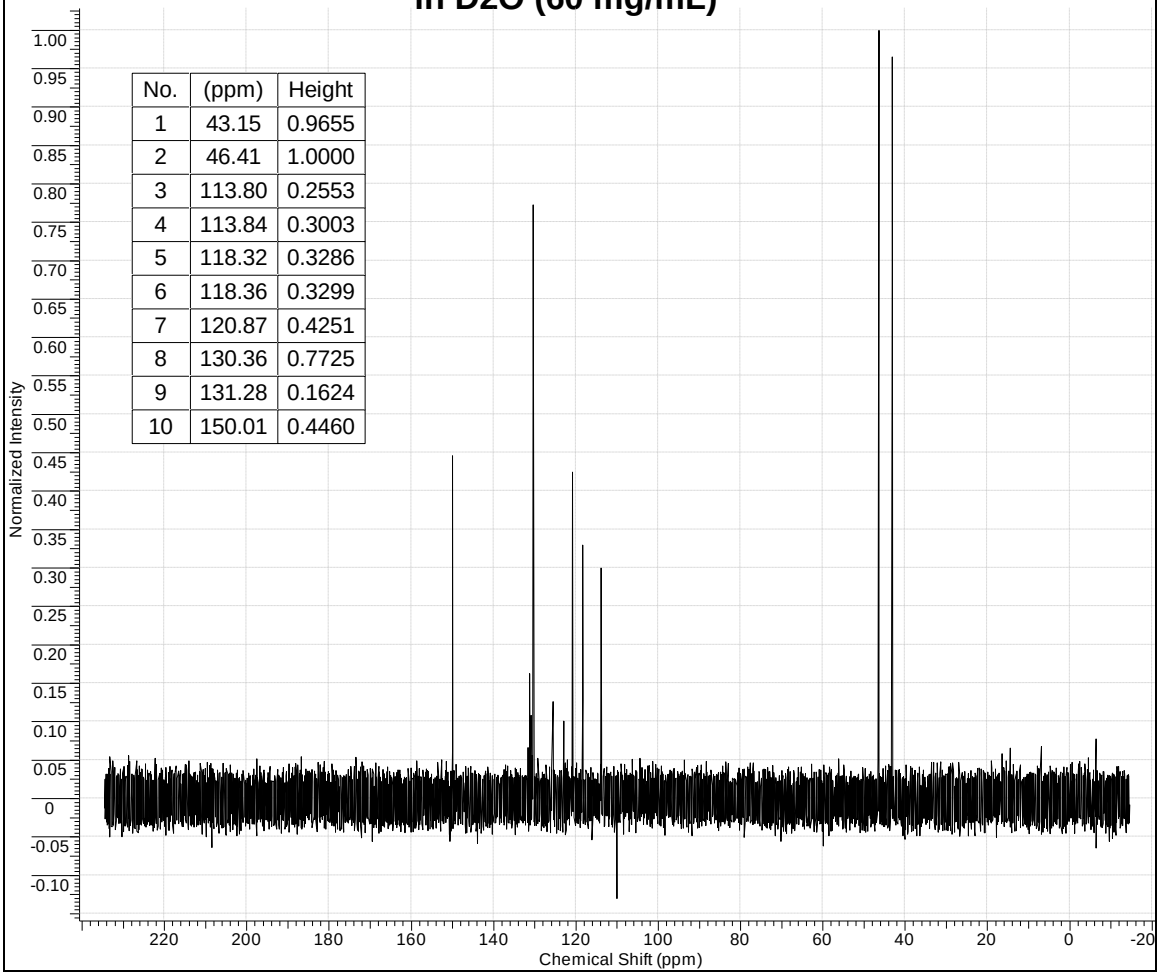
No.	(ppm)	Height
1	3.28	0.0673
2	3.29	0.1090
3	3.30	0.0761
4	3.30	0.1280
5	3.31	0.0219
6	3.36	0.0218
7	3.36	0.1391
8	3.37	0.0749
9	3.38	0.1122
10	3.39	0.0709

No.	(ppm)	Height
11	4.65	1.0000
12	7.17	0.0223
13	7.17	0.0257
14	7.19	0.0550
15	7.21	0.0332
16	7.25	0.0493
17	7.37	0.0273
18	7.39	0.0426
19	7.41	0.0186





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



No.	(ppm)	Height
1	43.15	0.9655
2	46.41	1.0000
3	113.80	0.2553
4	113.84	0.3003
5	118.32	0.3286
6	118.36	0.3299
7	120.87	0.4251
8	130.36	0.7725
9	131.28	0.1624
10	150.01	0.4460