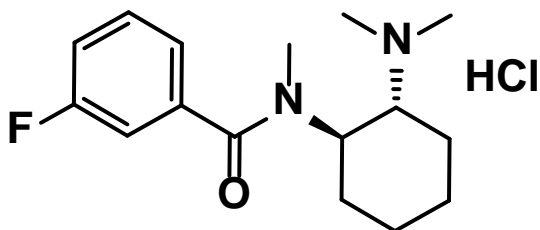


## U16 hydrochloride

The Krstenansky lab at the KGI School of Pharmacy and Health Sciences generated this monograph using synthesized material



### 1. GENERAL INFORMATION

<b>IUPAC Name:</b>	N-((1 <i>R</i> ,2 <i>R</i> )-2-(dimethylamino)cyclohexyl)-3-fluoro-N-methylbenzamide; hydrochloride
<b>CAS#:</b>	N/A
<b>Synonyms:</b>	U16
<b>Source:</b>	Synthesized Material Lot# JLK010-048-U16
<b>Appearance:</b>	White Crystals (HCl)
<b>UV<sub>max</sub> (nm):</b>	Not Determined

### 2. CHEMICAL AND PHYSICAL DATA

#### 2.1 CHEMICAL DATA

Form	Chemical Formula	Molecular Weight	Melting Point (°C)
HCl	C <sub>16</sub> H <sub>23</sub> FN <sub>2</sub> O·HCl	314.83	108.8 ± 1.04
Base	C <sub>16</sub> H <sub>23</sub> FN <sub>2</sub> O	278.36	Not determined

### 3. QUALITATIVE DATA

#### 3.1 NUCLEAR MAGNETIC RESONANCE

*Sample Preparation:* Dilute analyte to ~5 mg/mL in deuterated chloroform:methanol ( $\text{CDCl}_3:\text{CD}_3\text{OD}$ , 1:5) + TMS.

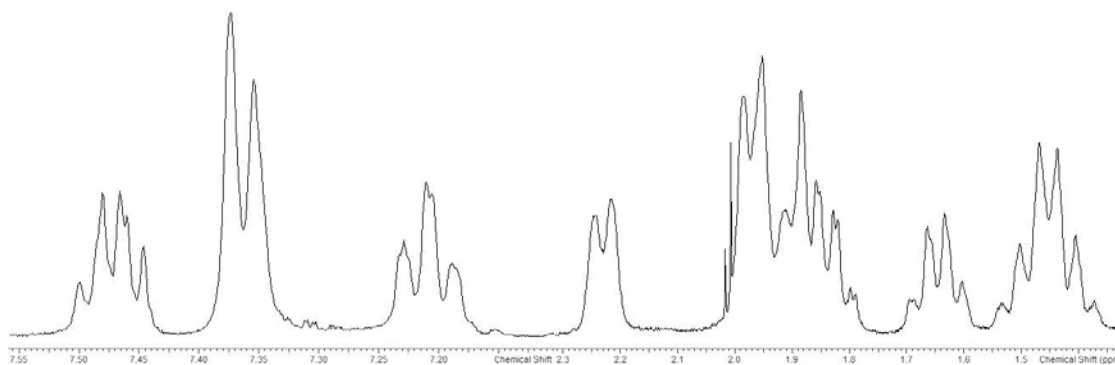
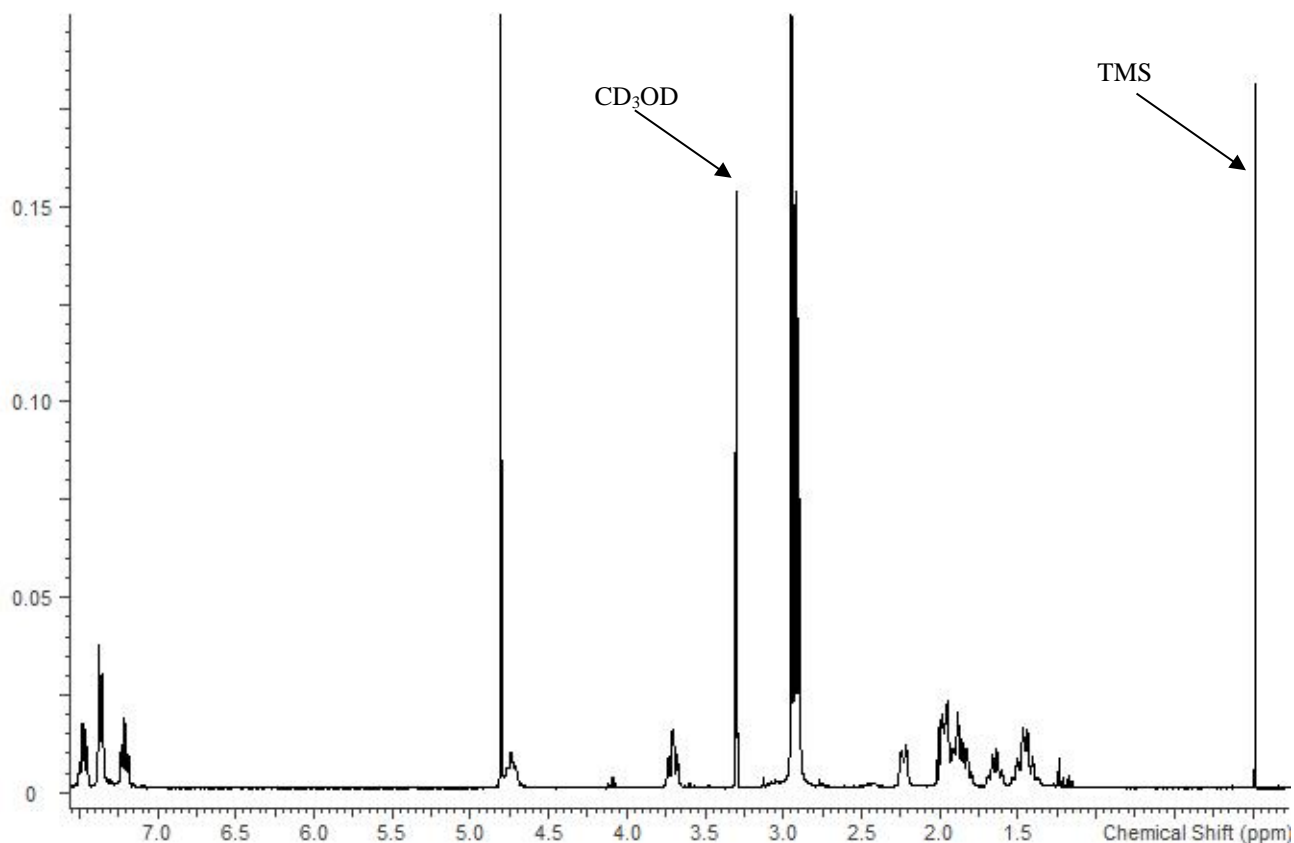
*Instrument:* 400 MHz NMR spectrometer

*Parameters:* Spectral width: 6410.3 Hz containing -3 ppm through 13 ppm

Pulse angle:  $90^\circ$

Delay between pulses: 30 seconds

$^1\text{H}$  NMR: U16 HCl; Lot JLK010-048-U16;  $\text{CDCl}_3:\text{CD}_3\text{OD}$  (1:5) + TMS; 400 MHz



## U16 hydrochloride

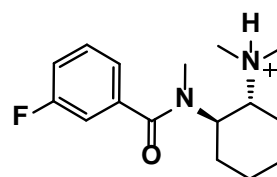
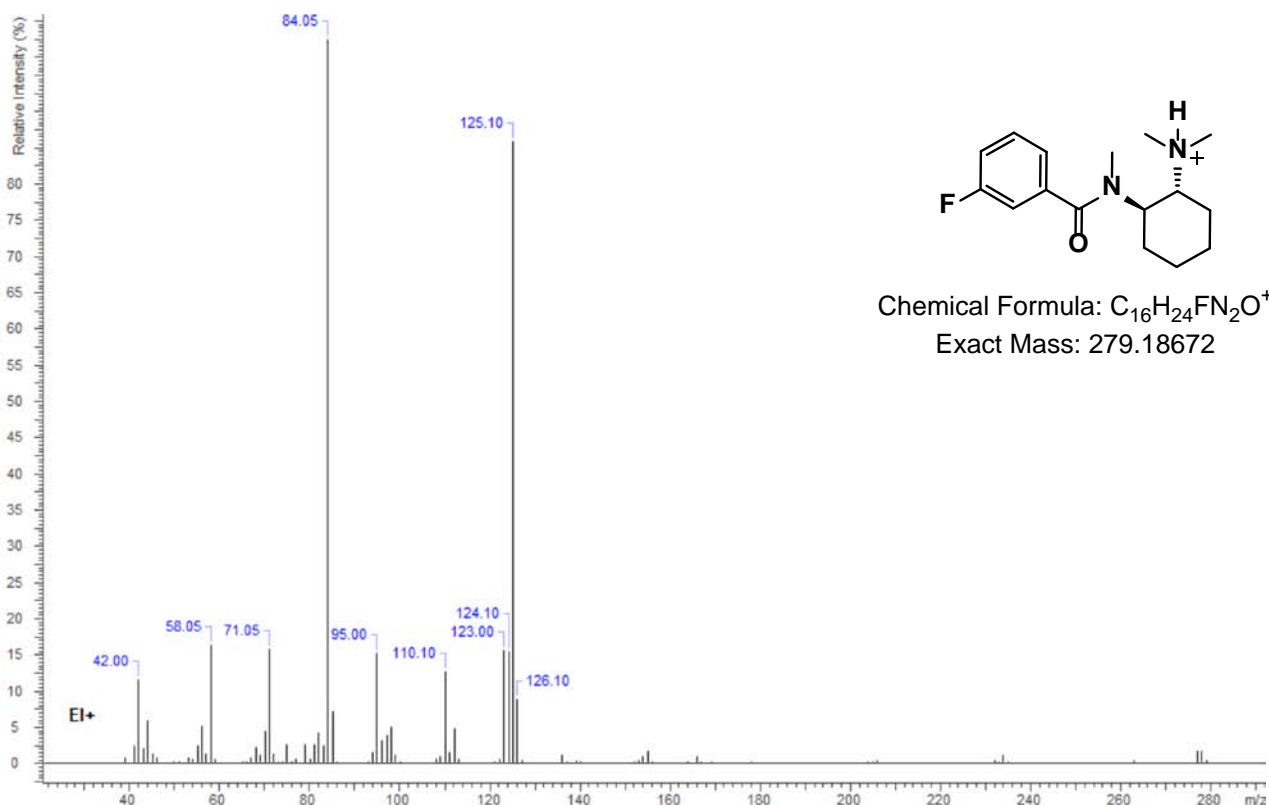
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### 3.2 GAS CHROMATOGRAPHY/MASS SPECTROMETRY

**Sample Preparation:** Dilute analyte ~ 1 mg/mL in methanol

<b>Instrument:</b>	Shimadzu gas chromatograph operated in split mode with MS detector
<b>Column:</b>	Rtx5MS (a DB-5 equivalent); 30m x 0.25 mm x 0.25 μm
<b>Carrier Gas:</b>	Helium at 1 mL/min
<b>Temperatures:</b>	Injector: 280°C MSD transfer line: 280°C MS Source: 200°C Oven program: 1) 90°C initial temperature for 2.0 min 2) Ramp to 300°C at 14°C/min 3) Hold final temperature for 10.0 min
<b>Injection Parameters:</b>	Split Ratio = 1:15, 1 μL injected
<b>MS Parameters:</b>	Mass scan range: 34-550 amu Threshold: 100 Acquisition mode: scan
<b>Retention Time:</b>	13.50 min

EI Mass Spectrum: U16 HCl; Lot JLK010-048-U16

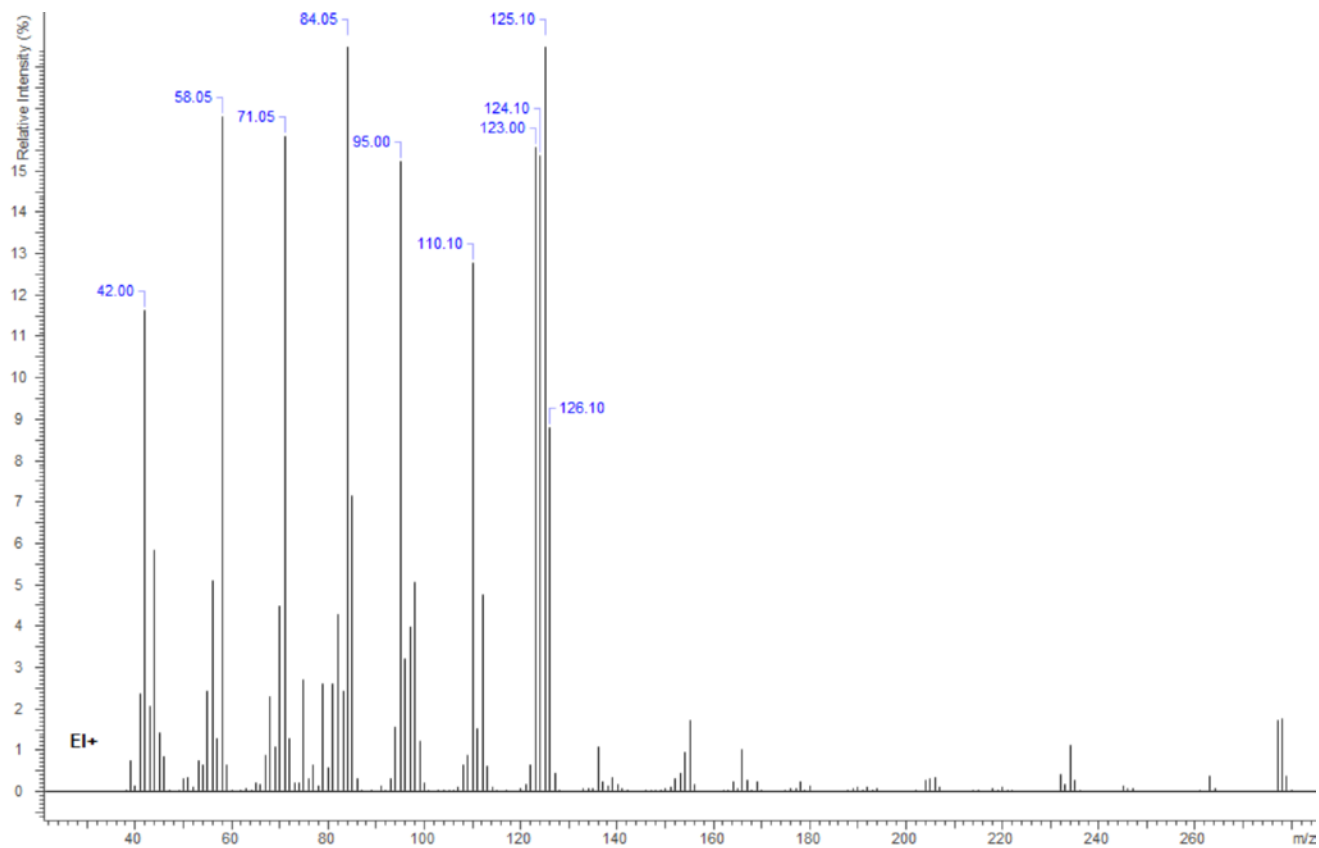


Chemical Formula: C<sub>16</sub>H<sub>24</sub>FN<sub>2</sub>O<sup>+</sup>  
Exact Mass: 279.18672

# U16 hydrochloride

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Zoomed view (84.05 and 125.10 are truncated in this view)



## U16 hydrochloride

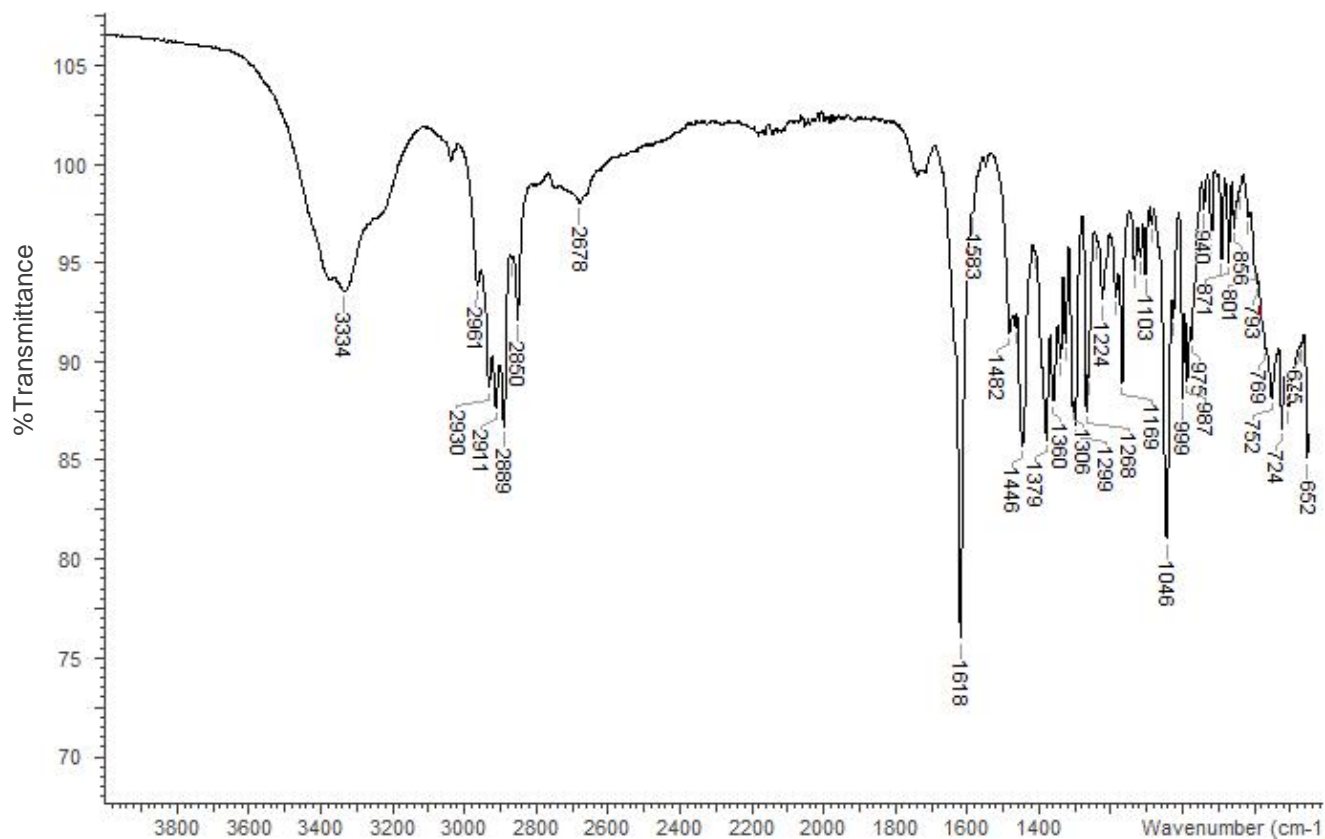
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### 3.3 INFRARED SPECTROSCOPY (FTIR)

**Instrument:** FTIR with ZnSe ATR attachment (1 bounce)

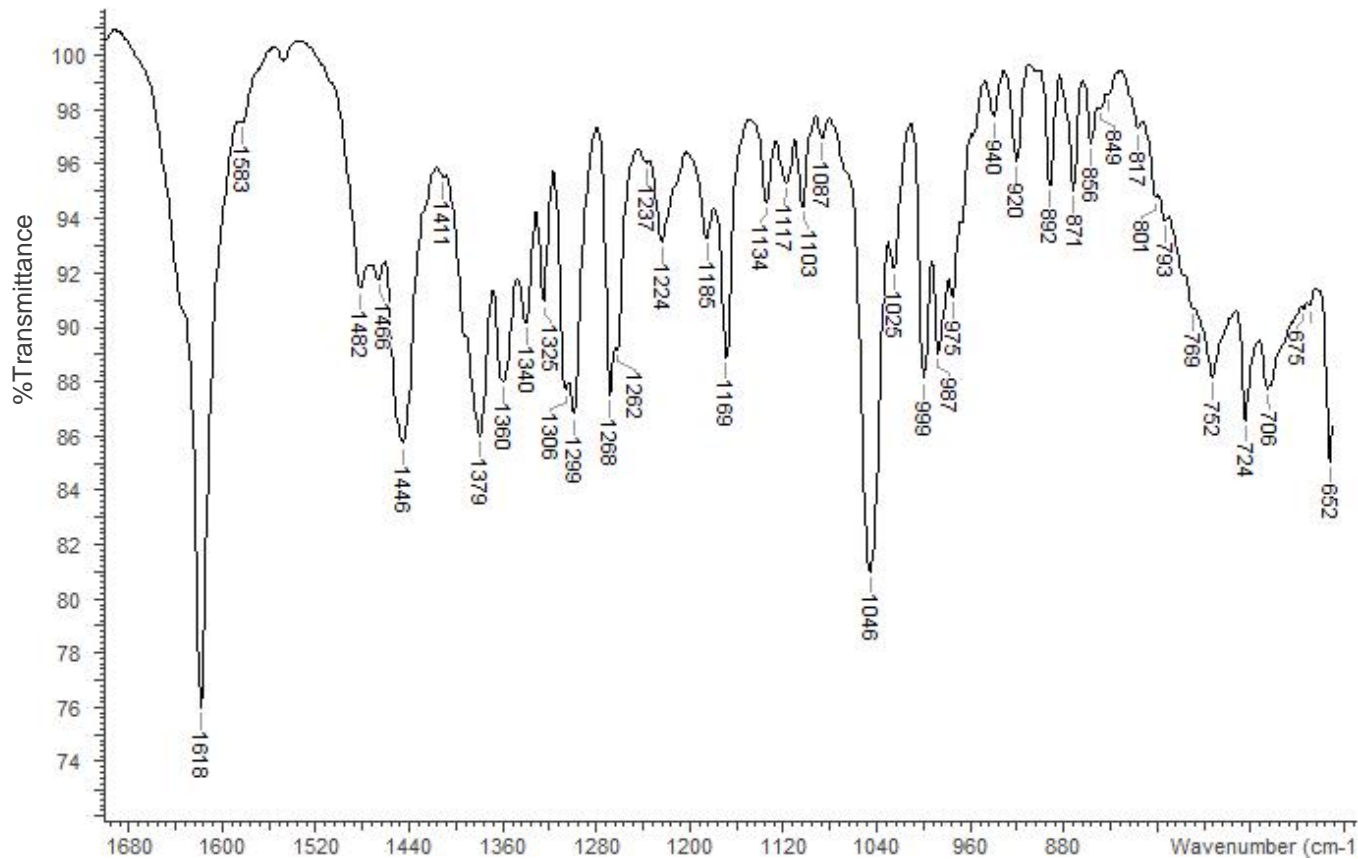
**Scan Parameters:**  
 Number of scans: 4  
 Number of background scans: 4  
 Resolution: 4 cm<sup>-1</sup>  
 Sample gain: 8  
 Aperture: 150

FTIR ATR (ZnSe, 1 Bounce): U16 HCl; Lot JLK010-048-U16



## U16 hydrochloride

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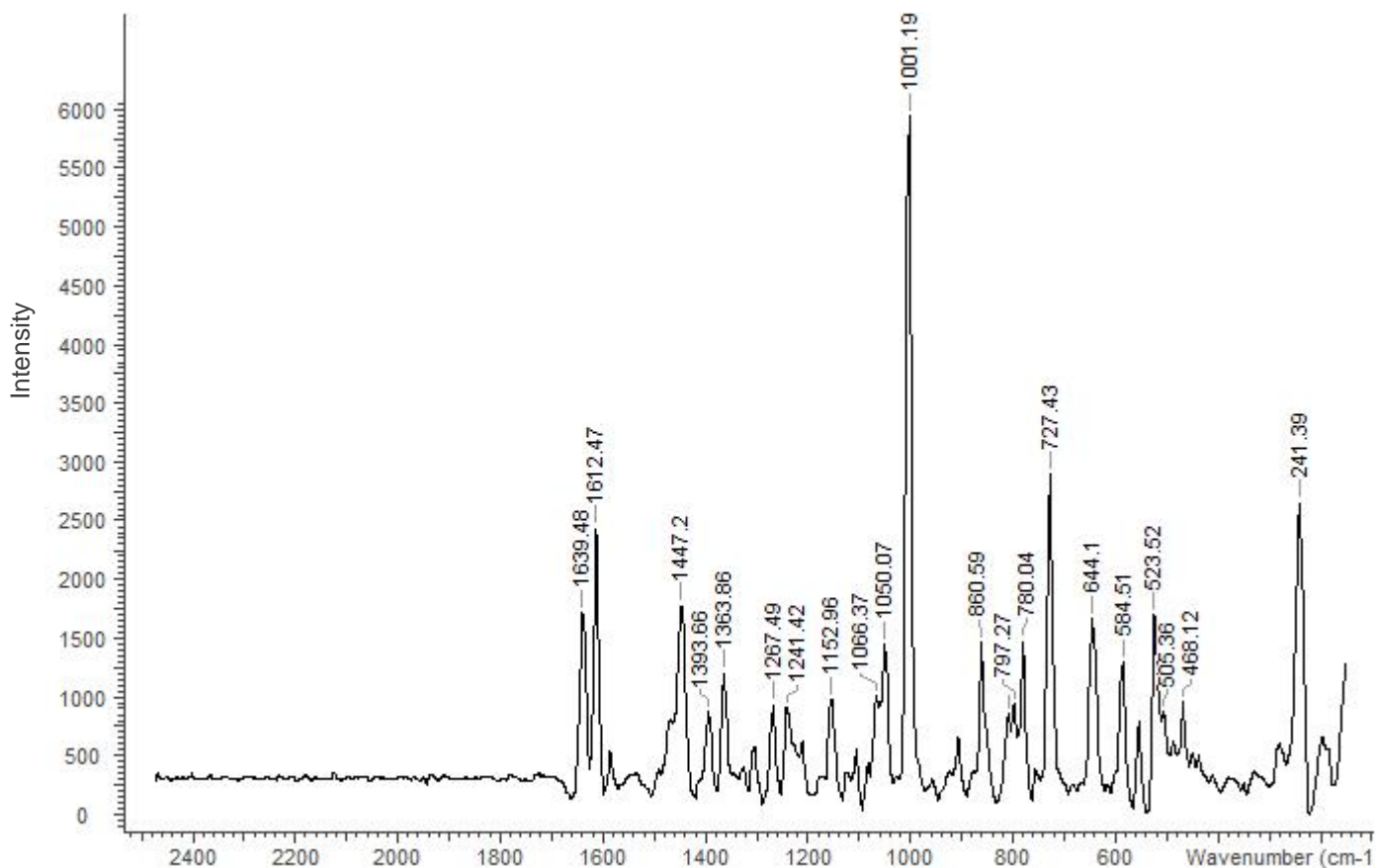
# U16 hydrochloride

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## 3.4 RAMAN SPECTROSCOPY

**Instrument:** Rigaku Progeny 1064  
**Scan Parameters:** Power (mW): 350  
Exposure (ms): 1000  
Averages: 30  
Threshold: 0.80

Raman (1064 nm): U16 HCl; Lot JLK010-048-U16



#### 4. ADDITIONAL RESOURCES

##### ANALGESIC N-(2-AMINOCYCLOALIPHATIC)BENZAMIDES

Szmuszkovicz

US Patent 4,098, 904 Jul. 4, 1978

Benzeneacetamide amines: structurally novel non- $\mu$  opioids

J. Szmuszkovicz, and P.F. Von Voigtlander

Journal of Medicinal Chemistry 1982, 25 (10), 1125–1126

DOI: 10.1021/jm00352a005

Factors affecting binding of trans-N-[2-(methylamino)cyclohexyl]benzamides at the primary morphine receptor

B.V. Cheney, J. Szmuszkovicz, R.A. Lahti and D.A. Zichi

Journal of Medicinal Chemistry 1985, 28 (12), 1853–1864

DOI: 10.1021/jm00150a017

Single stereoisomer analogs in the U-47700 series:

Tom Hsu, Jayapal Reddy Mallareddy, Kayla Yoshida, Vincent Bustamante, Tim Lee, John L. Krstenansky, Alexander C. Zambon, Synthesis and pharmacological characterization of ethylenediamine synthetic opioids in human  $\mu$ -opiate receptor 1 (OPRM1) expressing cells. *Pharmacol. Research & Perspectives* 7: e00511 (2019) doi: 10.1002/prp2.511

#### 5. ACKNOWLEDGEMENT

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