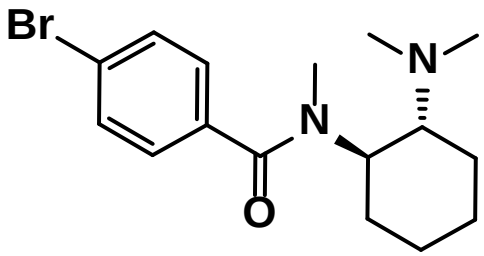
U08

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



1. GENERAL INFORMATION

IUPAC Name: N-((*1R,2R*)-2-(dimethylamino)cyclohexyl)-4-bromo-N-methylbenzamide

CAS#: 67579-31-1; 75570-38-6 (base)

Synonyms: U08

Source: **Synthesized Material** Lot# JLK008-137-U08

Appearance: White Crystals

UV_{max} (nm): Not Determined

2. CHEMICAL AND PHYSICAL DATA

2.1 CHEMICAL DATA

Form	Chemical Formula	Molecular Weight	Melting Point (^{o}C)
Base	$C_{16}H_{23}BrN_2O$	339.11	69.9 $\pm$ 0.56

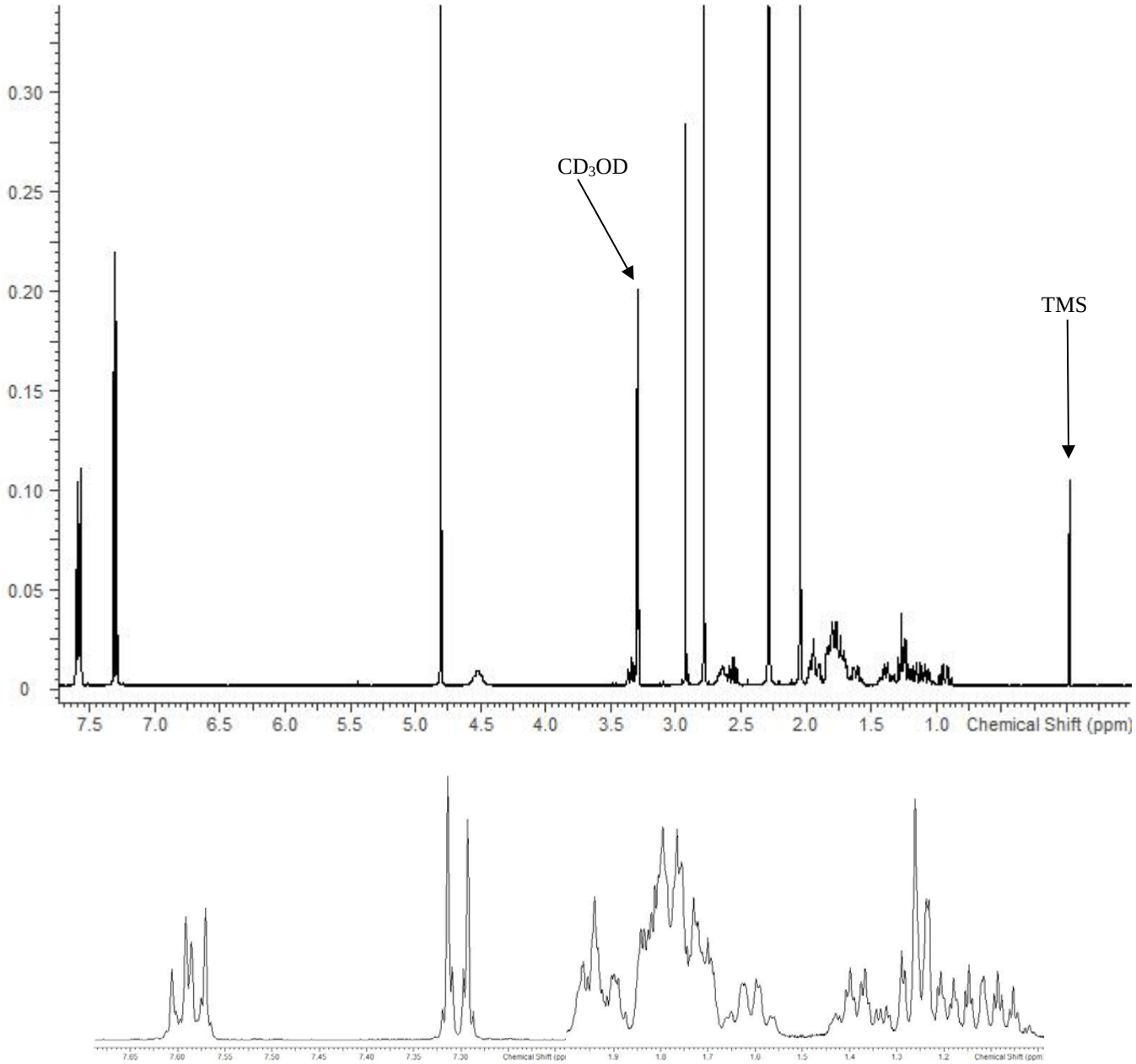
3. QUALITATIVE DATA

3.1 NUCLEAR MAGNETIC RESONANCE

Sample Preparation: Dilute analyte to ~5 mg/mL in deuterated chloroform:methanol ($CDCl_3$:CD_3OD, 1:5) + TMS.

Instrument: 400 MHz NMR spectrometer
Parameters: Spectral width: 6410.3 Hz containing -3 ppm through 13 ppm
Pulse angle: 90°
Delay between pulses: 30 seconds

^{1}H NMR: U08; Lot JLK008-137-U08; $CDCl_3$:CD_3OD (1:5) + TMS; 400 MHz

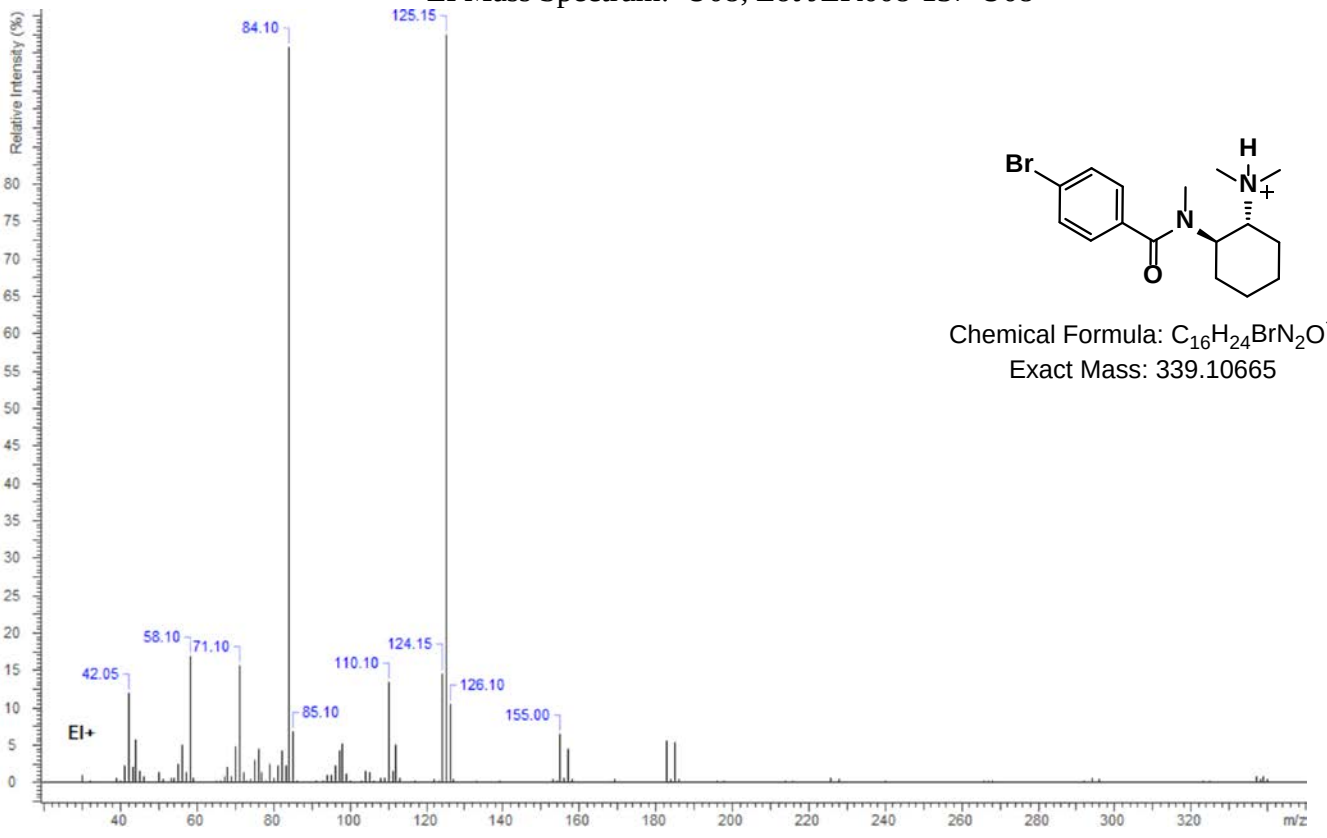


3.2 GAS CHROMATOGRAPHY/MASS SPECTROMETRY

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

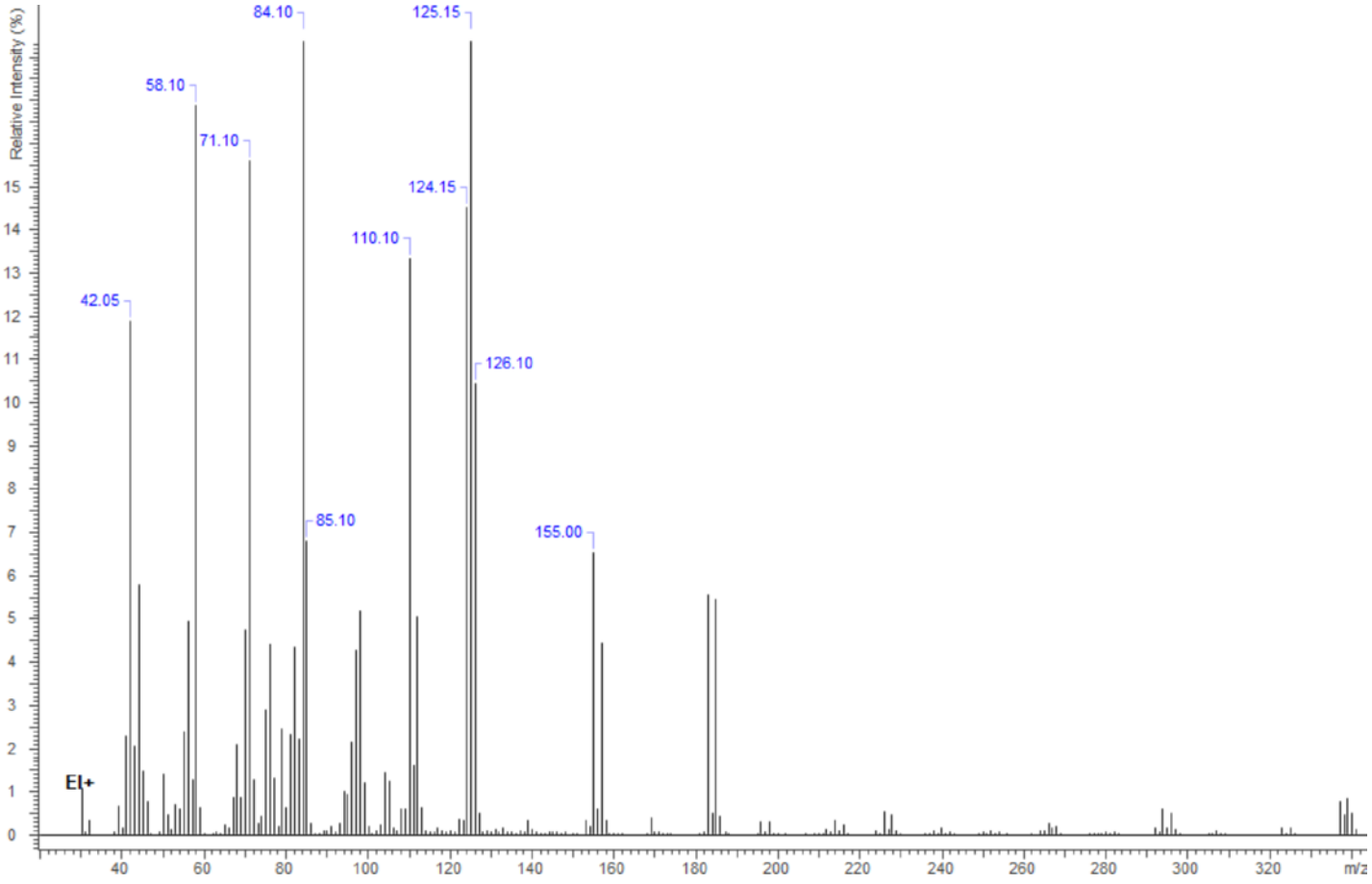
Instrument:	Shimadzu gas chromatograph operated in split mode with MS detector
Column:	Rtx5MS (a DB-5 equivalent); 30m x 0.25 mm x 0.25 µm
Carrier Gas:	Helium at 1 mL/min
Temperatures:	Injector: 280°C MSD transfer line: 280°C MS Source: 200°C Oven program: 1) 90°C initial temperature for 2.0 min 2) Ramp to 300°C at 14°C/min 3) Hold final temperature for 10.0 min
Injection Parameters:	Split Ratio = 1:15, 1 µL injected
MS Parameters:	Mass scan range: 34-550 amu Threshold: 100 Tune file: 050218_Tune.qgt Acquisition mode: scan
Retention Time:	15.65 min

EI Mass Spectrum: U08; Lot JLK008-137-U08



Chemical Formula: $C_{16}H_{24}BrN_2O^+$
Exact Mass: 339.10665

Zoomed view (84.10 and 125.15 are truncated in this view)

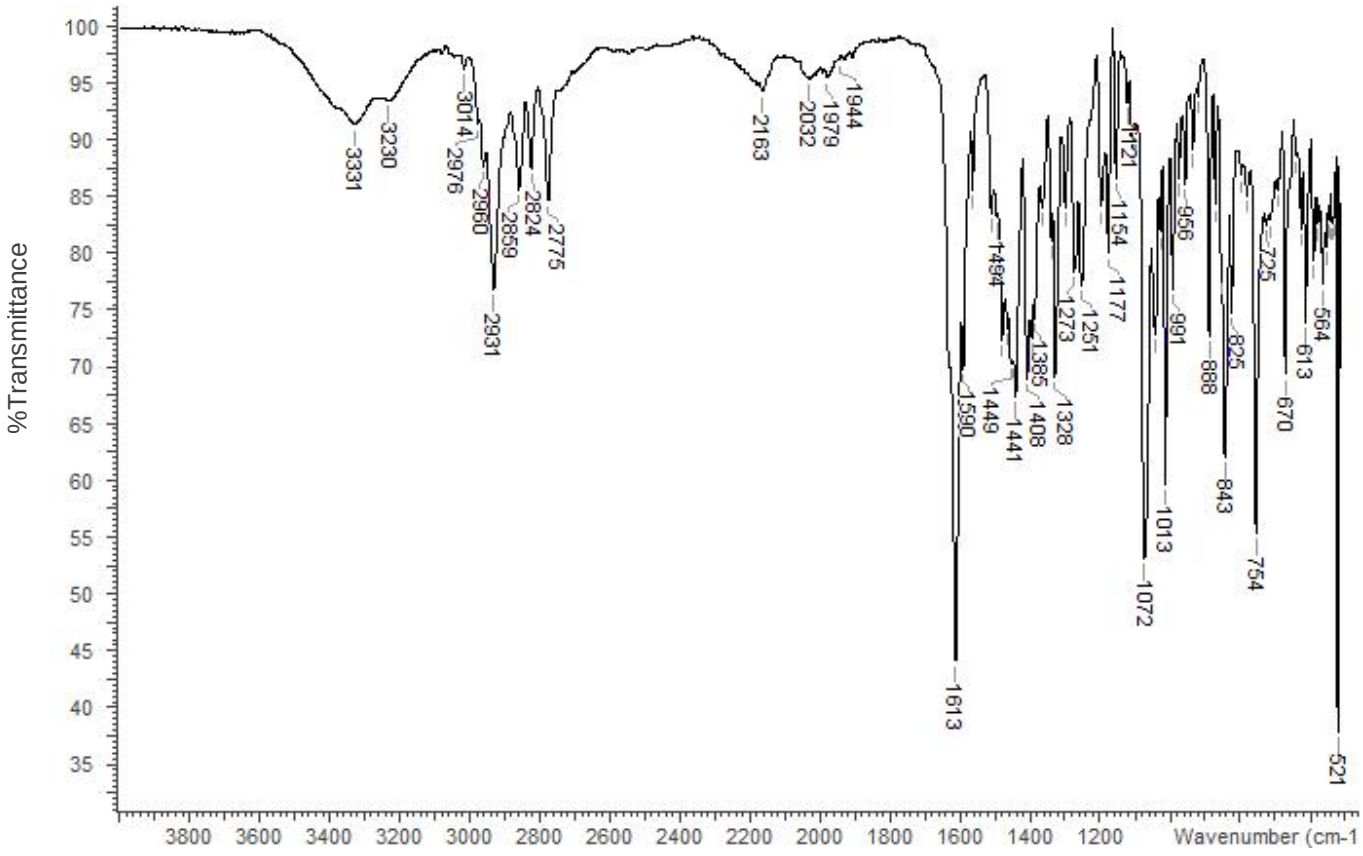


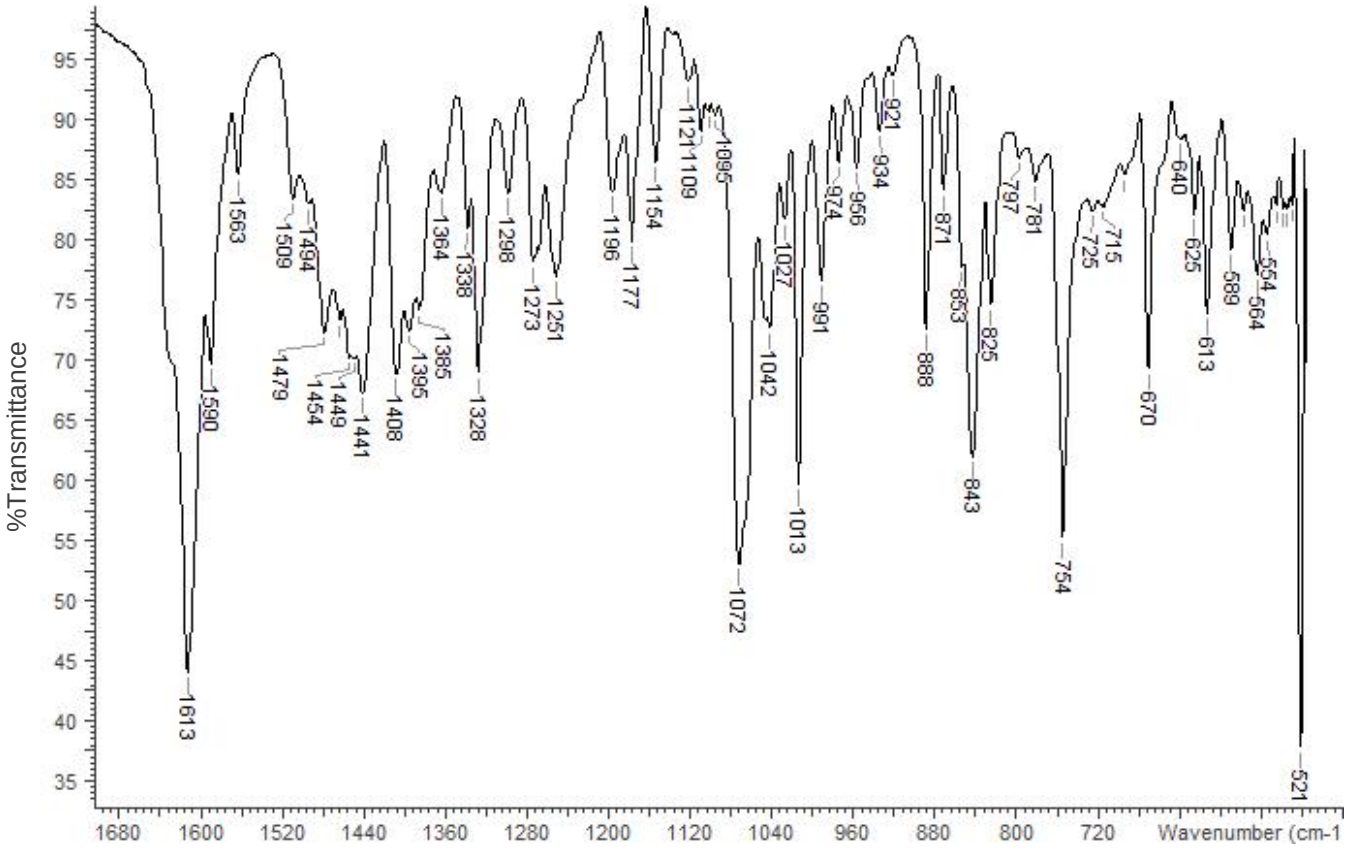
3.3 INFRARED SPECTROSCOPY (FTIR)

Instrument: FTIR with ZnSe ATR attachment (1 bounce)

Scan Parameters: Number of scans: 4
Number of background scans: 4
Resolution: 4 cm^{-1}
Sample gain: 8
Aperture: 150

FTIR ATR (ZnSe, 1 Bounce): U08; Lot JLK008-137-U08



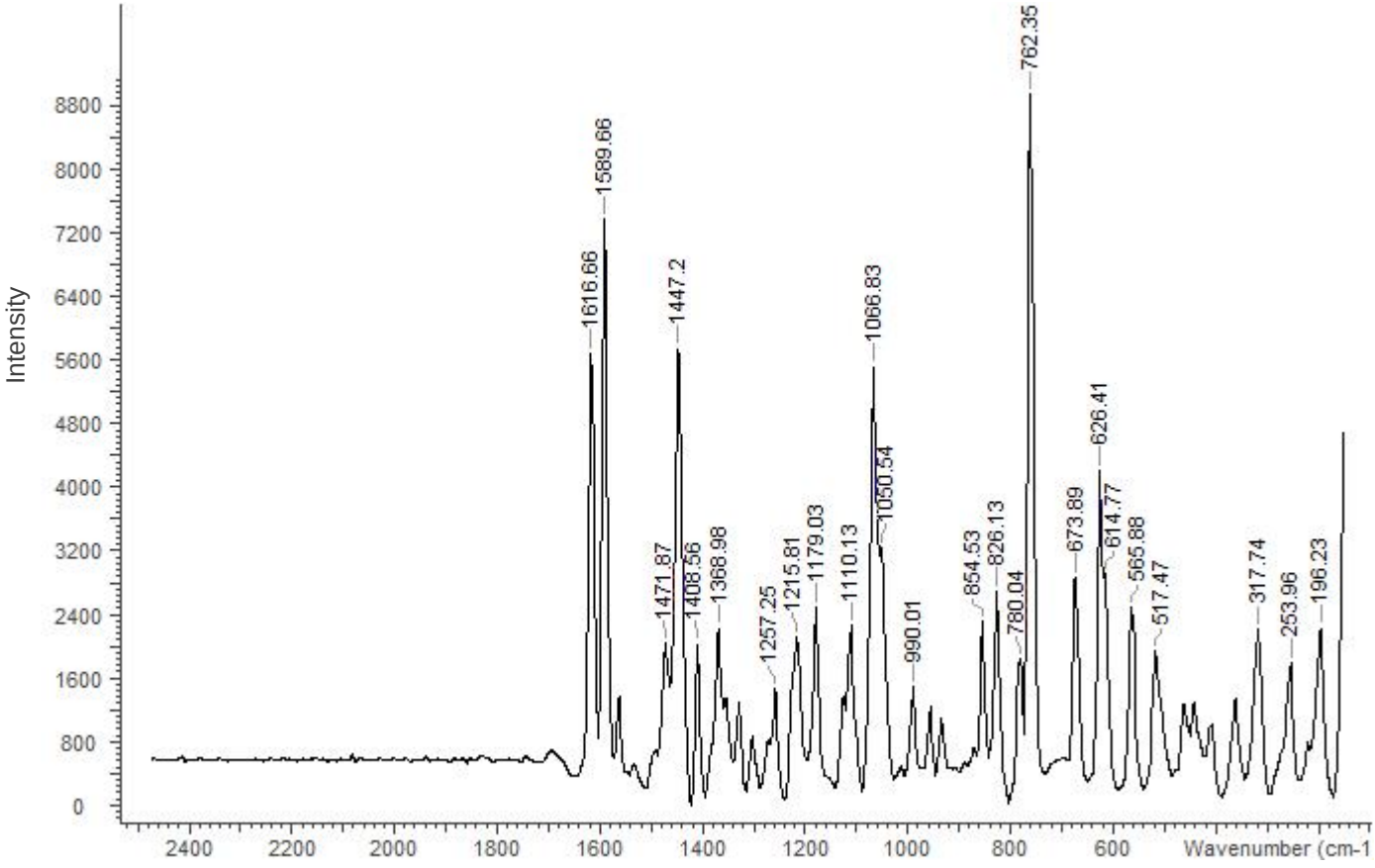

%Transmittance
Wavenumber (cm-1
1613
1590
1563
1509
1494
1479
1454
1449
1441
1408
1395
1385
1364
1338
1328
1298
1273
1251
1196
1177
1154
1121
1109
1095
1072
1042
1027
1013
991
974
956
934
921
888
871
853
843
825
797
781
754
725
715
670
640
625
613
589
564
554
521

3.4 RAMAN SPECTROSCOPY

Instrument: Rigaku Progeny 1064

Scan Parameters: Power (mW): 350
Exposure (ms): 1000
Averages: 30
Threshold: 0.80

Raman (1064 nm): U08; Lot JLK008-137-U08



4. ADDITIONAL RESOURCES

ANALGESIC N-(2-AMINOCYCLOALIPHATIC)BENZAMIDES
Szmuszkovicz
US Patent 4,098, 904 Jul. 4, 1978 Example 18

Benzeneacetamide amines: structurally novel non-mμ 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 μ-opiate receptor 1 (OPRM1) expressing cells. Pharmacol. Research & Perspectives 7: e00511 (2019) doi: 10.1002/prp2.511

5. ACKNOWLEDGEMENT

These data are from a project supported by Award No. 2016-R2-CX-0059, awarded by the National Institute of Justice, Office of Justice Programs, U.S. Department of Justice. The opinions, findings, and conclusions or recommendations expressed in this publication are those of the authors and do not necessarily reflect those of the Department of Justice. We also thank Rigaku Corporation for the loan of the Progeny 1064 Raman instrument.