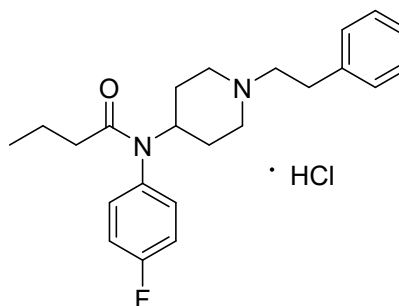


CERTIFICATE OF ANALYSIS

para-Fluorobutyryl fentanyl (hydrochloride)
N-(4-fluorophenyl)-N-[1-(2-phenylethyl)-4-piperidinyl]-butanamide,
monohydrochloride

Item #: 17049
Batch #: 0541382
CAS Registry Number: N/A

Expiry Date: 22OCT2024 (valid from date of certification)
Description: neat solid
Storage and Handling: Store at -20°C. Warm to room temperature prior to opening.
Safety: Poison



ISO/IEC 17025
#AT-1773
ISO Guide 34
#AR-1774



Compound Information

Chemical Formula	C ₂₃ H ₂₉ FN ₂ O • HCl
Formula Weight	405.00 amu
UV λ _{max}	202 nm

Quality Information

Qualifier	Method	Limit	Result	Meets Specification
Appearance	Visual inspection	White/off-white solid	White solid	Y
Chromatographic Purity, HPLC	Cayman Method TST SD139	≥98.00%	>99.90% ± 0.18%	Y
Identity, LCMS	Cayman Method TST SD13, +ESI	369.2 ± 0.5 amu	369.2 amu	Y
Identity, GCMS	Cayman Method TST SD12	Conforms	Conforms	Y
FTIR	USP<851> (diamond ATR)	Conforms	Conforms	Y
% LOD	Cayman Method TST SD24	≤5.00%	<0.10% ± 0.48%	Y
% ROI	Cayman Method TST SD06	≤3.00%	<0.10% ± 0.28%	Y
*Identity, NMR	¹ H NMR	Conforms	Conforms	Y

*NMR is provided as supplemental info but is not within scope of ISO accreditation

Property values are traceable to SI units through an unbroken chain of measurements.

Measurement Uncertainty

All measurement uncertainties are expressed as expanded uncertainties in accordance with ISO 17025 and Guide 34 at the approximate 95% confidence interval using the appropriate coverage factor.

Cayman Chemical certifies that this standard meets the specifications stated in this certificate and warrants this product to meet the stated acceptance criteria through the expiration date when stored unopened as recommended.

Approval:  Title: Cayman Chemical ISO Quality Manager

Certification Date: 22OCT2018

Cayman Chemical Co.

1180 E. Ellsworth Rd
Ann Arbor, MI 48108
www.caymanchem.com

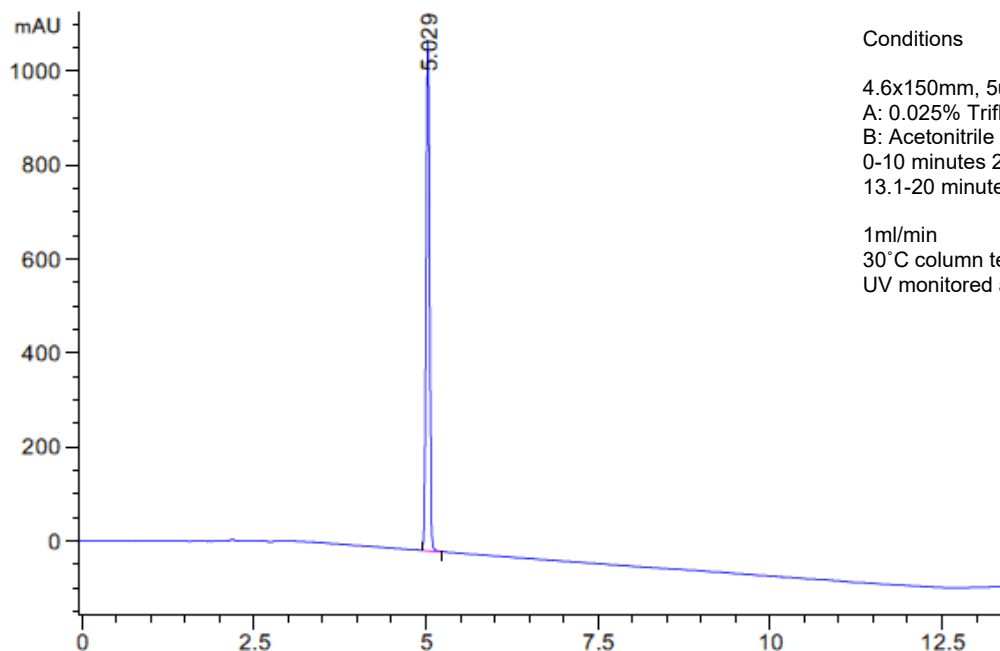
Contact Us:

1-800-364-9897 or (734)971-3335
Fax: (734)971-3640
Email: crmquality@caymanchem.com

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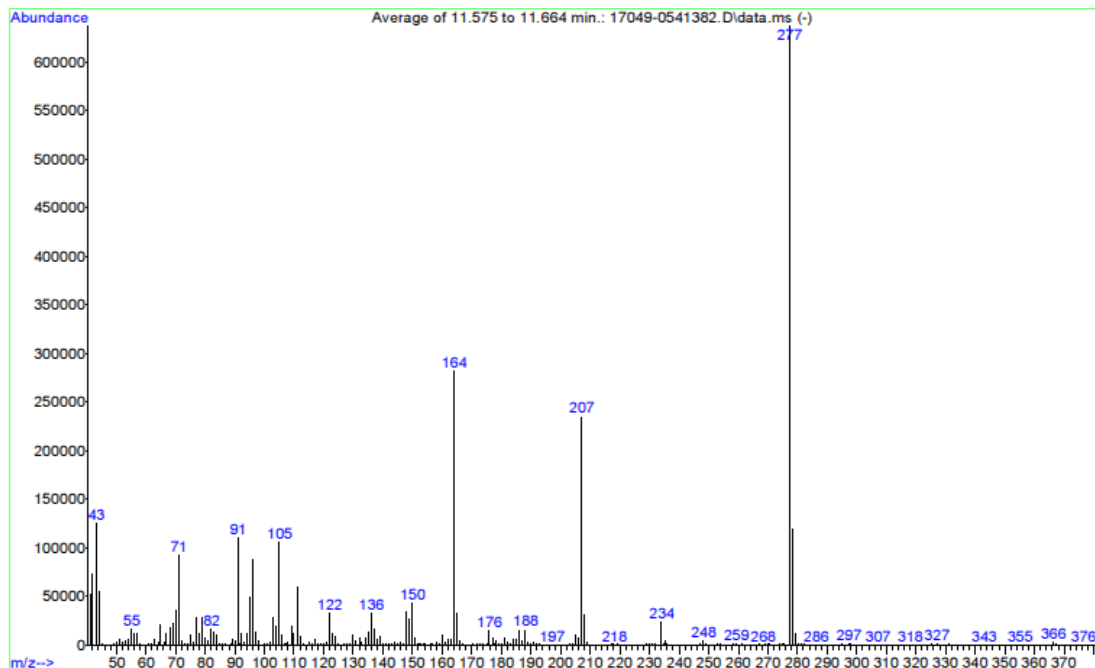
Supplemental Data**HPLC-UV**

DAD1 A, Sig=210,8 Ref=450,16 (C:\CAYMAN\... 17049 2018-10-01 09-23-16\SIG100002

**Conditions**

4.6x150mm, 5um Gemini C18 column
A: 0.025% Trifluoroacetic in H₂O
B: Acetonitrile
0-10 minutes 20-95% B, 10-13 minutes 95% B,
13.1-20 minutes 20% B

1ml/min
30°C column temp
UV monitored at 210nm

GC-MS**Conditions**

Agilent 6890 GC/
5973 MSD
30m x 0.32mm, 0.5um
Rtx-5MS column
He carrier gas;
Flow: 2mL/min
Inlet temp: 300°C
15:1 split
Oven Program:
50°C for 1minute,
ramp to 300°C at
30°C / minute, hold at
300°C to 15 minutes
Transfer line temp:
300°C
70ev EI MS
40-600 m/z
Apex spectrum -
background
(1 minute window in
front of peak)
Tune File: stune

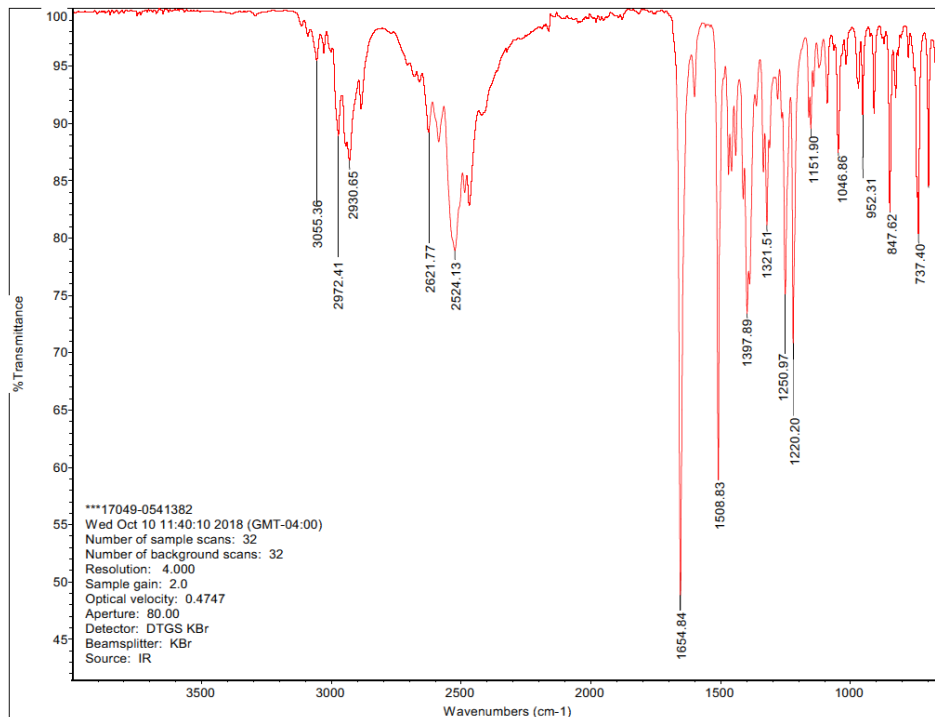
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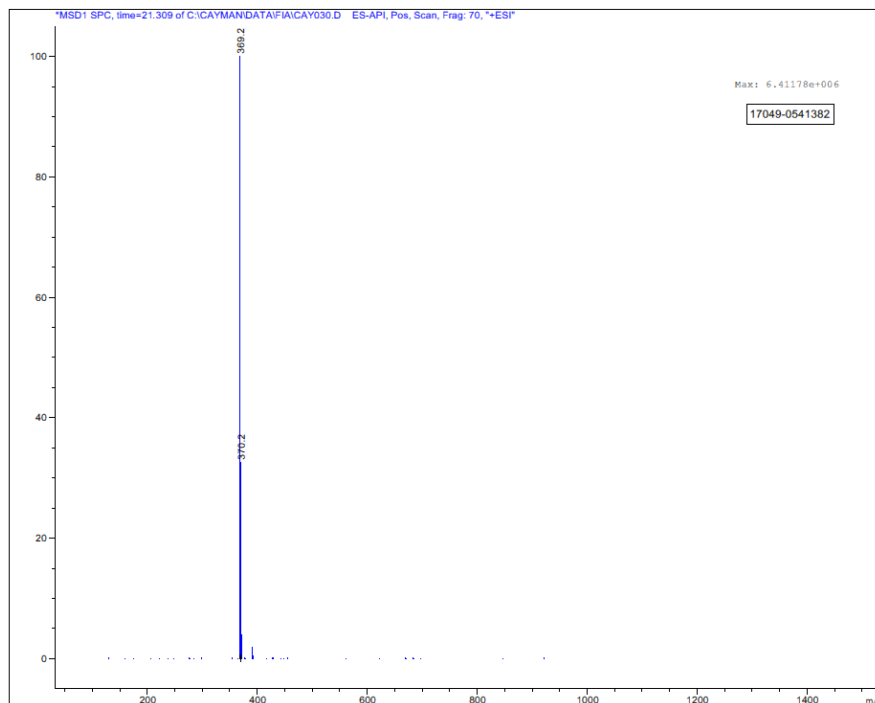
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FTIR**Conditions**

Thermo Nicolet iS10 FTIR /
Diamond SmartATR (single bounce)
32 scans / 32 background scans
Range: 650-4000cm⁻¹
Resolution: 4.000
ATR and background corrected

ESI-MS**Conditions**

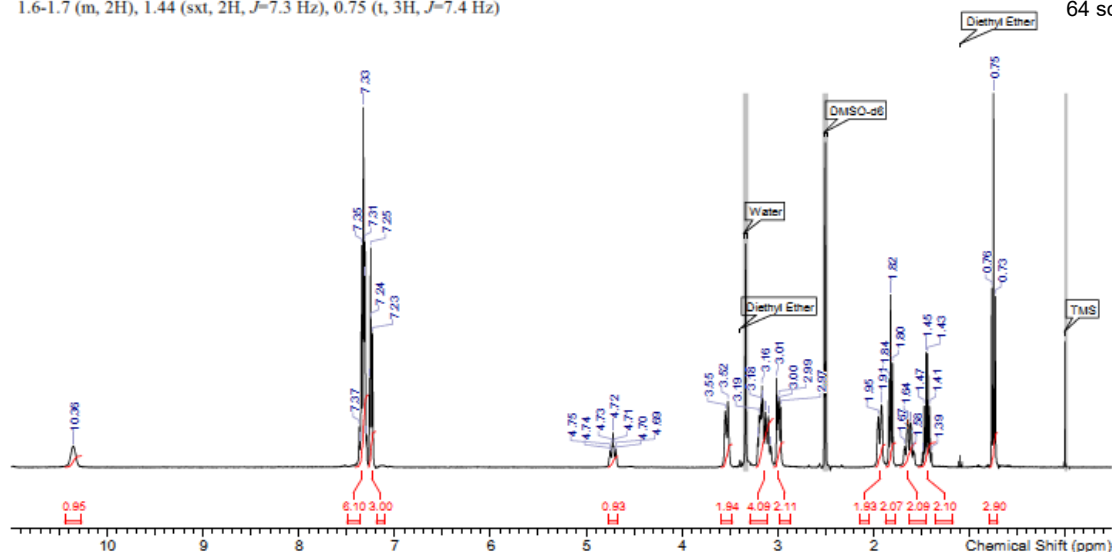
Agilent 1260 HPLC /
G6125B MSD
FIA-MS (no column)
Mobile phase: 50:50:0.1
MeOH/H₂O/acetic acid
Flow: 0.5mL/min
+ESI 100-1000m/z range
Nebulizer: 60psi
Desolvation Gas: 13L/min
Desolvation Temp: 350°C
Electrospray Voltage: 4kV
MS collected across peak width
at half height

NMR *not within scope of ISO 17025/Guide 34 accreditation

Date	Sep 26 2018
File Name	IsosulfuricAcid.mdata_2018\NKL-1820-007_20180926_NKL-1820-007_20180926_01\NKL-1820-007_20180926_001.fid.tif
Frequency (MHz)	399.9680
Nucleus	¹ H
Solvent	DMSO-d ₆
Temperature (degree C)	25.000
Number of Transients	32

¹H NMR (DMSO-d₆, 400 MHz) δ 10.36 (br s, 1H), 7.3-7.4 (m, 6H), 7.2-7.3 (m, 3H), 4.7-4.8 (m, 1H), 3.53 (br d, 2H, J=11.3 Hz), 3.0-3.2 (m, 4H), 2.9-3.0 (m, 2H), 1.93 (br d, 2H, J=13.3 Hz), 1.82 (t, 2H, J=7.4 Hz), 1.6-1.7 (m, 2H), 1.44 (sxt, 2H, J=7.3 Hz), 0.75 (t, 3H, J=7.4 Hz)

Conditions

Varian Inova 400MHz NMR
64 scans**Homogeneity**

A minimum sample size of 1.5 µg was used to determine homogeneity of the bulk solid. The recommended minimum quantity for use is 1.5 µg. Quantities below this have not been evaluated.

Short Term Stability Summary

No decrease in purity was observed at ambient conditions or 60°C after two weeks. This data supports shipping of this product at ambient temperature.

Long Term Stability

Long term stability data predicts 6 years stability at the -20°C storage temperature.

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Revision History

Revision #	Date	Reason for Revision
01	22OCT2018	Initial version

Disclaimers**Material Safety Data**

This material should be considered hazardous until information to the contrary becomes available. Do not ingest, swallow, or inhale. Do not get in eyes, on skin, or on clothing. Wash thoroughly after handling. This information contains some but not all of the information required for the safe and proper use of this material. Before use, review the complete Material Safety Data Sheet, which has been sent via email to your institution.

Warranty and Limitation of Remedy

Cayman Chemical Company makes no warranty or guarantee of any kind, whether written or oral, expressed or implied, including without limitation, any warranty of fitness for a particular purpose, suitability and merchantability, which extends beyond the description of the chemicals hereof. Cayman warrants only to the original customer that the material will meet our specifications at the time of delivery.

Cayman will carry out its delivery obligations with due care and skill. Thus, in no event will Cayman have any obligation or liability, whether in tort (including negligence) or in contract, for any direct, indirect, incidental or consequential damages, even if Cayman is informed about their possible existence.

This limitation of liability does not apply in the case of intentional acts or negligence of Cayman, its directors or its employees.

Buyer's exclusive remedy and Cayman's sole liability hereunder shall be limited to a refund of the purchase price, or at Cayman's option, the replacement, at no cost to Buyer, of all material that does not meet our specification.

Said refund or replacement is conditioned on Buyer giving written notice to Cayman within thirty (30) days after arrival of the material at its destination. Failure of Buyer to give said notice within thirty (30) days shall constitute a waiver of Buyer of all claims hereunder with respect to said material.

For further details, please refer to our Warranty and Limitations of Remedy located on our website and in our catalog.

This Certificate shall not be reproduced except in full, without written approval from the Cayman Chemical ISO Quality Manager

ISO CRT SD01 v 1.1

