

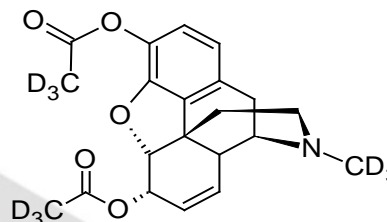
Certificate of Analysis

Heroin-D₉

7,8-Didehydro-4,5-epoxy-17-trideuteromethylmorphinan-3,6-diol di[trideuteroacetate]

Cerilliant Quality
ISO GUIDE 34
ISO/IEC 17025
ISO 13485
ISO 9001
GMP/GLP

Catalog Number: H-036
Solution Lot: FE093012-08
Expiration Date: October 2017
Solvent: Acetonitrile
Volume per Ampule: Not less than 1 mL
Storage: Store unopened in freezer.
Shipping: Ambient. See Stability Section.
Intended Use: For R&D/ analytical purposes only. Not suitable for human or animal consumption.
Regulatory: USDEA Exempt | Canadian TK# 61-274



Safety: Flammable, Poison

- Expiration Date has been established through real time stability studies.
- Ampules are overfilled to ensure a minimum 1 mL volume can be transferred when using a 1 mL Class A volumetric pipette. We advise laboratories to quantitatively transfer desired volumes of this standard using established good laboratory practices to dilute to the desired concentration.

Component	Solution Purity	Certified Concentration
Heroin-D ₉	99.7%	100.0 ± 0.6 µg/mL
<ul style="list-style-type: none"> Uncertainty of the concentration is expressed as an expanded uncertainty in accordance with ISO 17025 and Guide 34 at the approximate 95% confidence interval using a coverage factor of k = 2 and has been calculated by statistical analysis of our production system and incorporates uncertainty of the purity factor, material density, and balance and weighing technique. This standard is prepared gravimetrically and mass results are reported on the conventional basis for weighing in air. Concentration is calculated based on: the actual measured mass; Purity Factor of the analyte(s); measured mass of the solution; and the density of the pure diluent at 20°C. Concentration is corrected for chromatographic purity, residual water, residual solvents and residual inorganics. 		

Solution Standard Verification and Homogeneity

Standard Solution	Lot Number	Verified Concentration (µg/mL)		%RSD - Homogeneity	
		Actual Results	Acceptance Criteria	Actual Results	Acceptance Criteria
New Lot	FE093012-08	100.8	± 3%	0.7	≤ 3%
Previous Lot	FE012111-05	99.11	± 3%		
<ul style="list-style-type: none"> Concentration is verified through multiple analyses and is calculated as the average of multiple analyses compared to an independently prepared calibration solution. Homogeneity of the New Lot is ensured through rigorous production process controls statistically analyzed to evaluate risk and verified by analysis. The % RSD of samples pulled from across the lot demonstrate homogeneity of the New Lot. 					

Traceability

<ul style="list-style-type: none"> Gravimetrically prepared using qualified balances calibrated semi-annually by Mettler Toledo using NIST traceable weights. Calibration verification performed weekly and prior to each use utilizing NIST traceable weights. Each balance has been assigned a minimum weighing by Mettler Toledo taking into consideration the balance and installed environmental conditions to ensure weighing complies with USP tolerances of no more than 0.1% relative error. Concentration is verified against an independently prepared calibration solution gravimetrically prepared using balances calibrated to NIST. In addition, each neat material utilized has been identified and thoroughly characterized through the use of multiple analytical techniques. Spectral data is provided on subsequent pages of the COA.
--

Cerilliant certifies that this standard meets the specifications stated in this certificate and warrants this product to meet the stated acceptance criteria through the expiration/retest date when stored unopened as recommended. Product should be used shortly after opening to avoid concentration changes due to evaporation. Warranty does not apply to ampoules stored after opening.




Lara Sparks, Quality Assurance Director

October 29, 2012

Date

<i>Standard Solution Assay Parameters</i>		<i>Calibration Curve</i>	
Analysis Method:	UV/Vis	Calibration Curve:	Linear Regression
Wavelength:	205 nm	Number of Points:	4
Slit Width:	1.0 nm	Linearity (r):	1.000
Response:	0.5 s		

<i>Neat Material Data</i>			
Compound Name:	Heroin-D ₉	Chemical Formula:	C ₂₁ H ₁₄ D ₉ NO ₅
Compound Lot:	FC081010-01	CAS Number:	NA
		Molecular Weight:	378.35

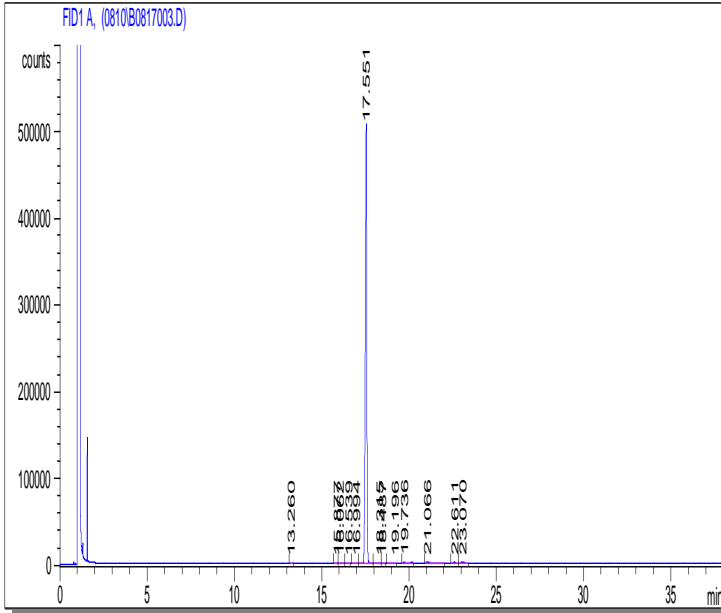
<i>Neat Material Characterization Summary</i>		
Analytical Test	Method	Results
Primary Chromatographic Purity by GC/FID Analysis	SP10-0101	99.6%
Secondary Chromatographic Purity by HPLC/PDA Analysis	SP10-0102	99.7%
Identity by GC/MS Analysis	SP10-0105	Consistent with Structure
Isotopic Purity by GC/MS SIM Analysis	SP10-0105	0.01% D ₀ vs D ₉
Identity by ¹ H-NMR Analysis	USP <761>, SP10-0116	Consistent with Structure
Residual Solvent Analysis by GC/FID Headspace	AM1087 ¹	0.80%
Residual Water Analysis by Karl Fischer Coulometry	USP <921>, SP10-0103	0.11%
Inorganic Content by Microash Analysis	SP10-0135	< 0.2%
Purity Factor		98.73%

- ♦ Primary purity is calculated as the average of two independently performed analyses utilizing two different methods. Acceptance criteria requires the purity values to be within 0.5% of each other.
- ♦ The primary chromatographic purity value is used to calculate the Purity Factor.
- ♦ A secondary chromatographic purity method is utilized as a control.
- ♦ Purity Factor = [(100 - wt% residual solvent - wt% residual water - wt% residual inorganics) x Chromatographic Purity/100].
- ♦ Purity factor does not include adjustment for chiral and/or isotopic purity.

¹ Validated analytical method.

Spectral and Physical Data

GC/FID

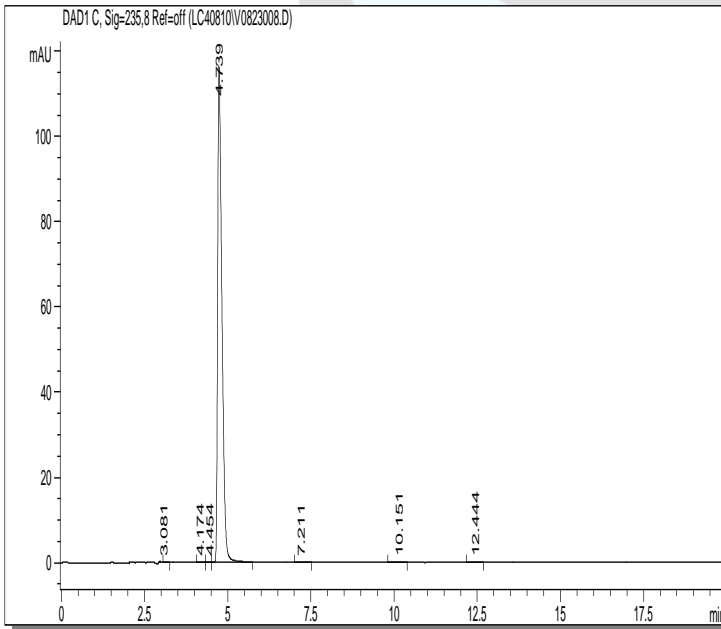


Column: DB-5ms, 30 m x 0.53 mm ID, 1.5 µm film thickness
Temp Program: 40°C to 200°C at 40°C/min
 200°C to 280°C at 5°C/min (hold 18 min)
Injector Temp: Cool-on-Column
Detector Temp: 325°C

Data File Name: S:\GC\GC2\2010\0810\B0817003.D
Operator: RPC
Instrument: GC#2
Sample Name: FC081010-01
Method File: AM1045.M
Acquired: August 17, 2010 10:51 AM

Peak #	Ret Time	Area	Height	Area %
1	13.26	115	22	0.00
2	15.88	301	37	0.01
3	16.06	1967	266	0.07
4	16.54	219	22	0.01
5	16.99	1495	150	0.05
6	17.55	2948750	505389	99.55
7	18.32	1476	134	0.05
8	18.49	1409	177	0.05
9	19.20	749	47	0.03
10	19.74	2378	217	0.08
11	21.07	1357	131	0.05
12	22.61	422	44	0.01
13	23.07	1374	143	0.05

HPLC/PDA



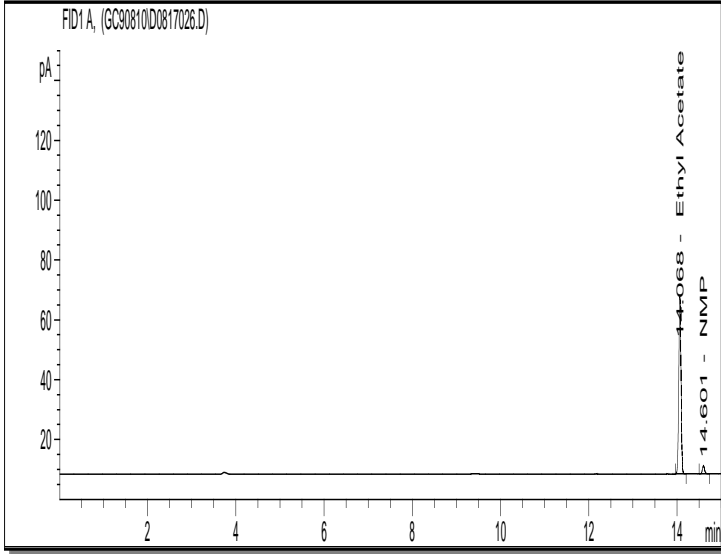
Column: Luna Phenyl Hexyl, 4.6 x 250 mm
Mobile Phase: Acetonitrile::10 mM Potassium phosphate buffer (35::65)
Flow Rate: 1.0 mL/min
Wavelength: 235 nm

Data File Name: S:\HPLC\HPLC4\2010\LC40810\0823008.D
Operator: TNT
Instrument: LC#4
Sample Name: FC081010-01
Method File: RMH003-2.M
Acquired: August 23, 2010 2:34 PM

Peak #	Ret Time	Area	Height	Area %
1	3.08	0.87	0.11	0.09
2	4.17	0.36	0.05	0.04
3	4.45	0.13	0.02	0.01
4	4.74	1023.01	116.42	99.72
5	7.21	0.36	0.04	0.04
6	10.15	0.67	0.04	0.07
7	12.44	0.52	0.03	0.05

Spectral and Physical Data (cont.)

Residual Solvent Analysis by GC/FID Headspace



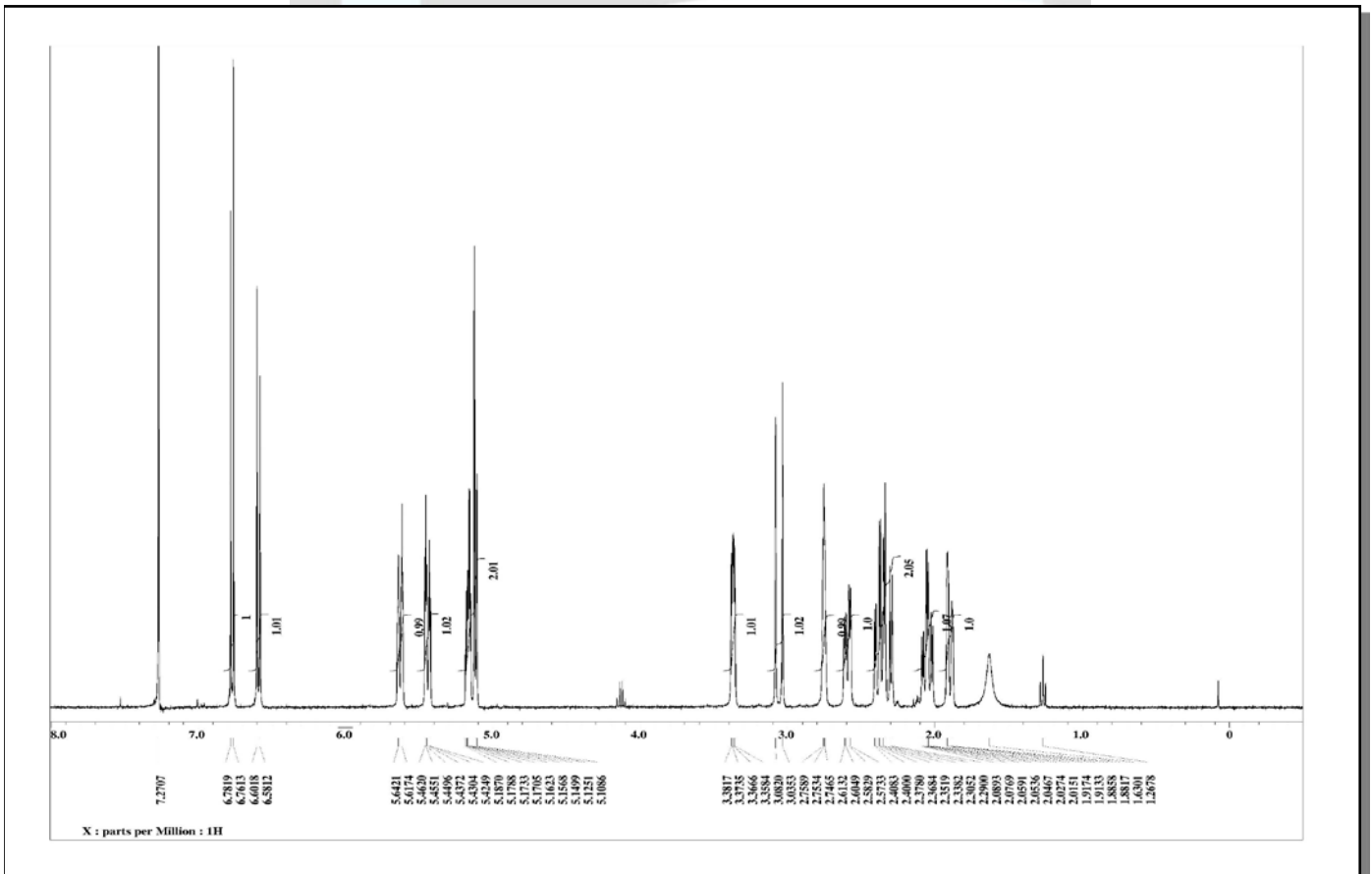
Column: DB-ALC1 30 m x 0.53 mm, 3 µm film thickness
Temp Program: 40°C (12 min) to 220°C at 40°C/min (5.5 min)
Carrier Gas: Helium
Flow Rate: 2.0 mL/min
Detector Heater Temp: 250°C
Injector: Headspace Sampler
HS Oven Temp: 60°C
Vial Equilibration: 10 minutes

Data File Name: C:\CHEM32\1\DATA\GC90810\D0817026.D
Operator: BD
Instrument: GC#9
Sample Name: FC081010-01
Acquired: August 18, 2010 3:31 AM

Peak	Compound	Area	Weight %
1	Ethyl acetate	211.05679	0.80
2	NMP	NA	NA
Total			0.80

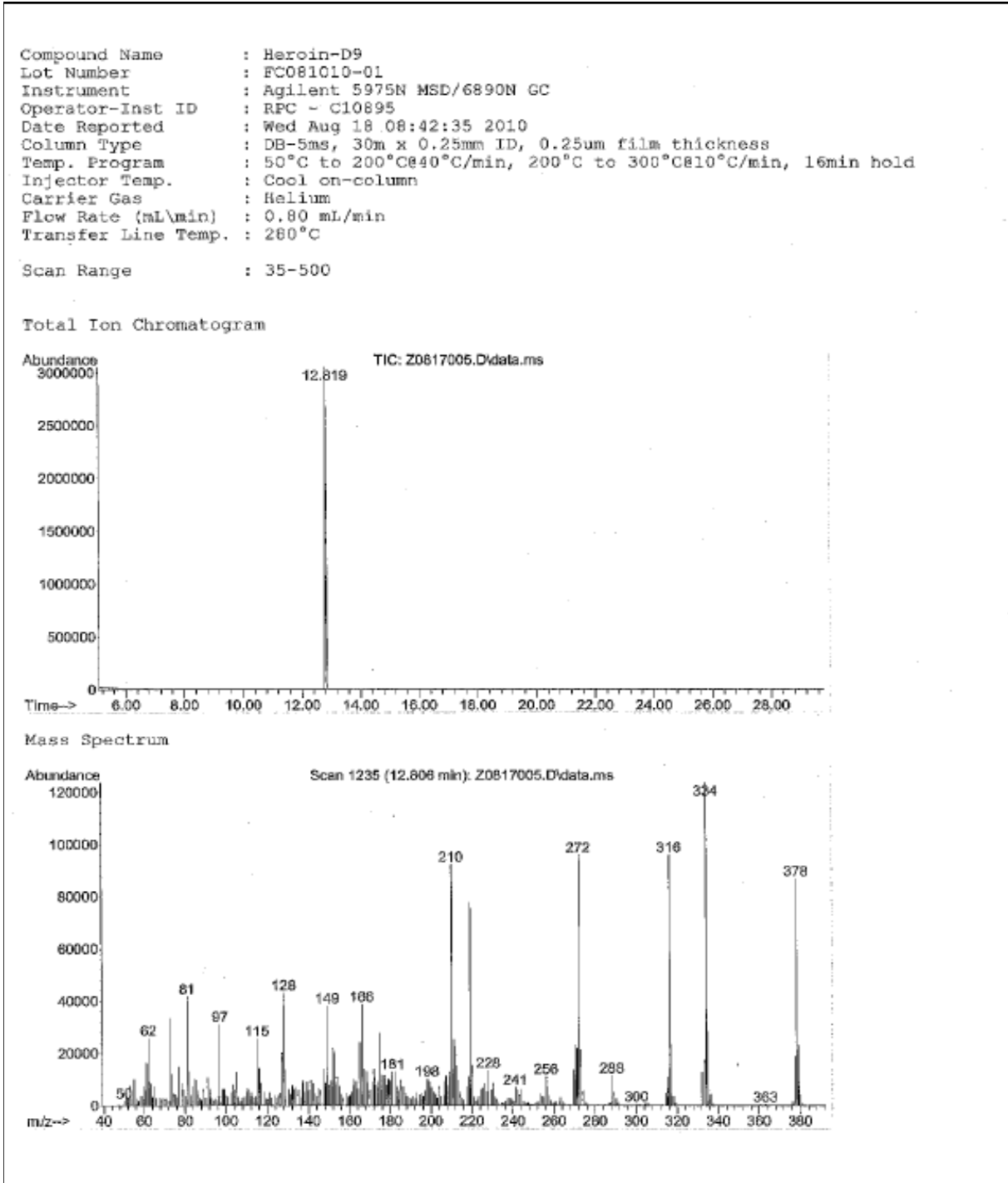
¹H NMR

Instrument: JEOL ECS 400
Solvent: Chloroform-D



Spectral and Physical Data (cont.)

GC/MS



Stability

Short Term Stability : A summary of accelerated stability findings for a related product (Heroin) is listed below.

Storage Condition	Mean Kinetic Temperature (MKT)	Time Period
Freezer	-15°C	No decrease in purity was noted after one week.
Refrigerator	4°C	
Room Temperature	21°C	
40°C	40°C	

Transport/Shipping : Stability data supports transport of this product at ambient conditions.

Short Term Storage: Stability data supports short term storage up to 1 year at Refrigerate conditions.

