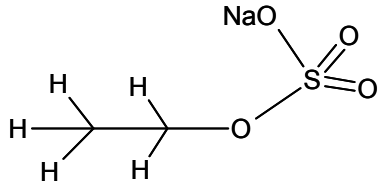
Cerilliant Quality
ISO GUIDE 34
ISO/IEC 17025
ISO 13485
ISO 9001
GMP/GLP

Certificate of Analysis

Ethyl sulfate sodium salt

Sulfuric acid monoethyl ester sodium salt



Catalog Number: E-064
Solution Lot: FN061412-02
Retest Date: October 2016
Solvent: Methanol
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.
Safety: Flammable, Poison

- Retest Date - stability studies ongoing. Certificate of Analysis will be updated upon completion of retest.
- Ampules are overfilled to ensure a minimum 1 mL volume fill. We advise laboratories to use measured volumes of this standard solution before diluting to the desired concentration.

Component	Solution Purity	Certified Concentration
Ethyl sulfate sodium salt	100.0%	1.000 ± 0.006 mg/mL (as ethyl sulfate)

- 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 purity, residual water, residual solvent, and residual inorganics by quantitative ^{1}H-NMR.

Solution Standard Verification and Homogeneity

Standard Solution	Lot Number	Verified Concentration (mg/mL) Actual Results	Verified Concentration (mg/mL) Acceptance Criteria	%RSD - Homogeneity Actual Results	%RSD - Homogeneity Acceptance Criteria
New Lot	FN061412-02	0.999	± 3%	0.4	≤ 3%
Previous Lot	FN111610-02	1.021	± 3%	0.8	≤ 3%

- 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.
- The % RSD of the Previous Lot represents variability of the analysis performed at the time of release.

Traceability

- 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.

ACCLASS ACCREDITED REFERENCE MATERIAL PRODUCER

Darron Ellsworth, Quality Assurance Manager

March 12, 2014
Date

Standard Solution Assay Parameters

Analysis Method: LC/MS/MS
Column: Luna C18, 3 µm, 2 x 100 mm
Mobile Phase: 0.1% Formic acid in Water::0.1% Formic acid in Methanol (95::5)
Flow Rate: 0.3 mL/min
Scan Type: MRM, Negative mode

Calibration Curve

Calibration Curve: Linear Regression
Number of Points: 4
Linearity (r): 1.000

Neat Material Data

Compound Name: Ethyl sulfate sodium salt
Compound Lot: PN061812-01

Chemical Formula: $C_2H_5NaO_4S$
CAS Number: 546-74-7
Molecular Weight: 148.11

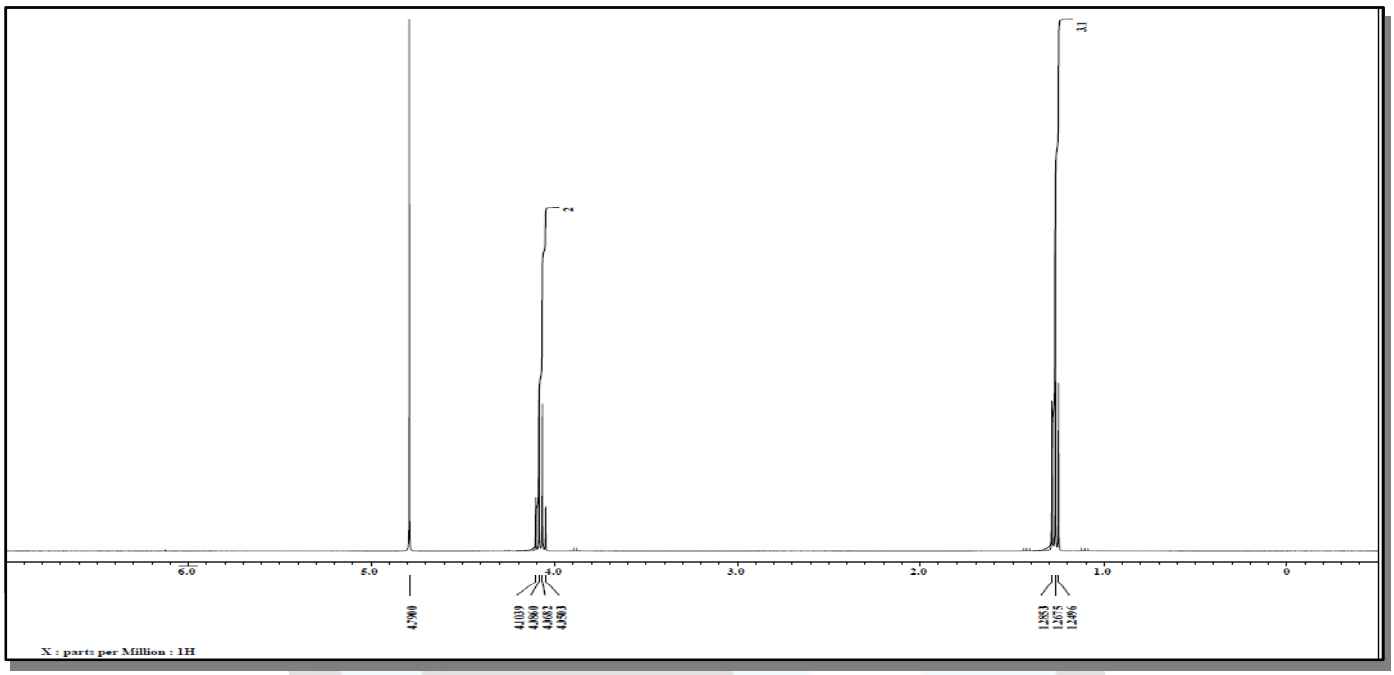
Neat Material Characterization Summary

Analytical Test	Method	Results		
Identity by LC/MS Analysis	SP10-0107	Consistent with Structure		
Identity by ^{1}H-NMR Analysis	USP <761>, SP10-0116	Consistent with Structure		
Identity by ^{13}C-NMR Analysis	USP <761>, SP10-0116	Consistent with Structure		
Residual Water Analysis by Karl Fischer Coulometry	USP <921>, SP10-0103	1.46%		
Elemental Analysis	SP10-0117		**Calculated**	**Analyzed**
		C	15.91%	16.00%
		H	3.37%	3.15%
		S	21.65%	20.30%
Sodium Composition by ICP-MS	Outsourced	15.18%		
Assay by Quantitative NMR Analysis	USP <761>, SP10-0116	98.90%		

Spectral and Physical Data

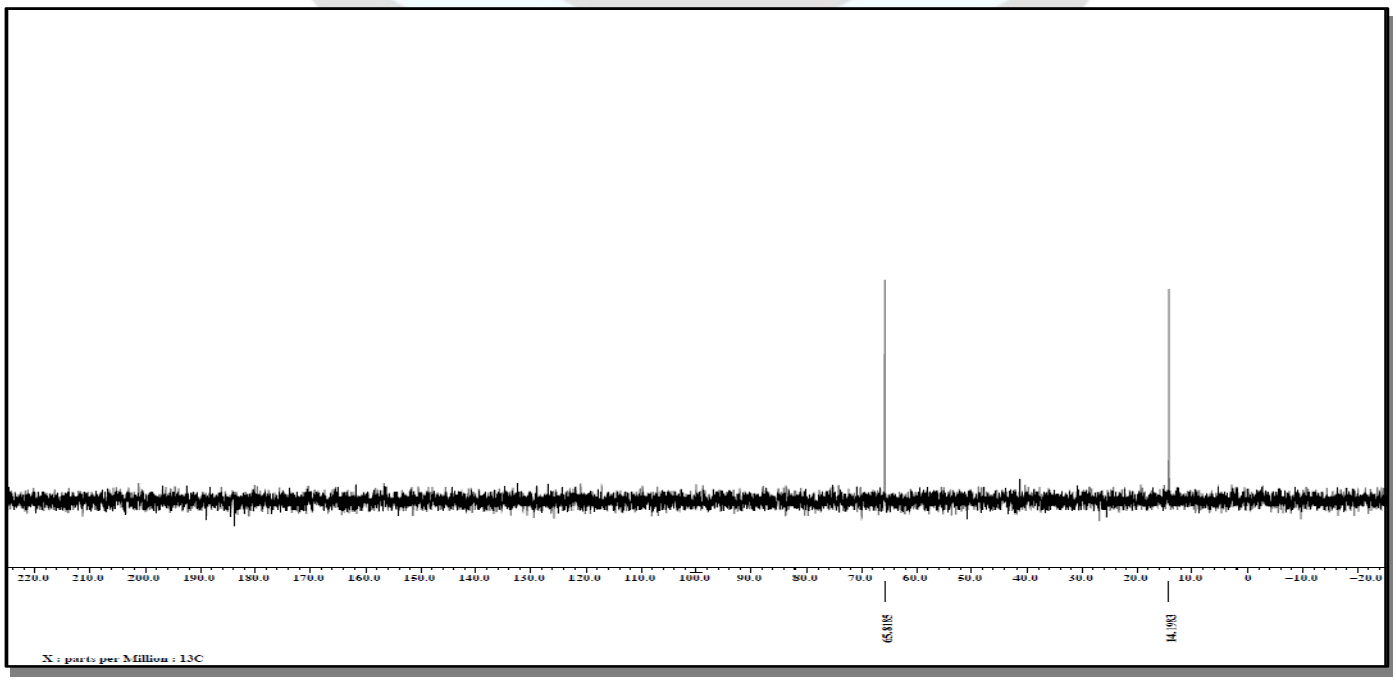
^{1}H NMR

Instrument: JEOL ECS 400
Solvent: D_2O



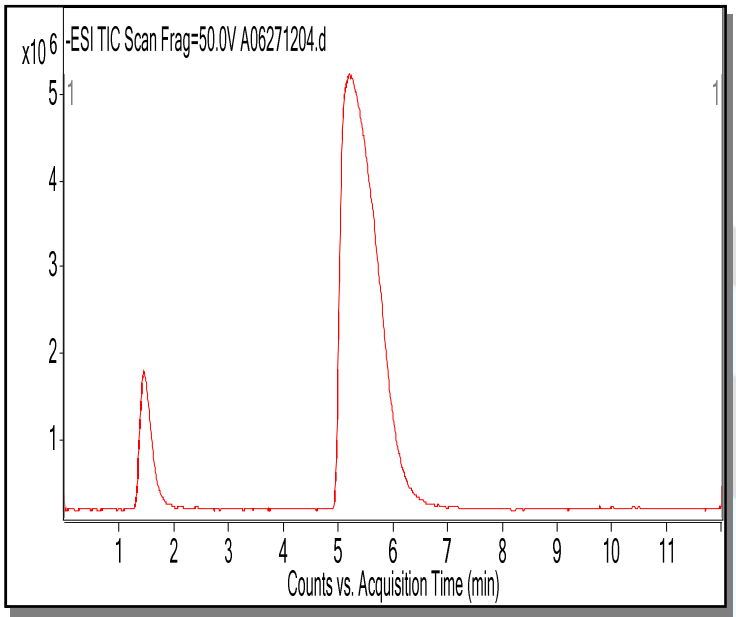
^{13}C NMR

Instrument: JEOL ECS 400
Solvent: D_2O

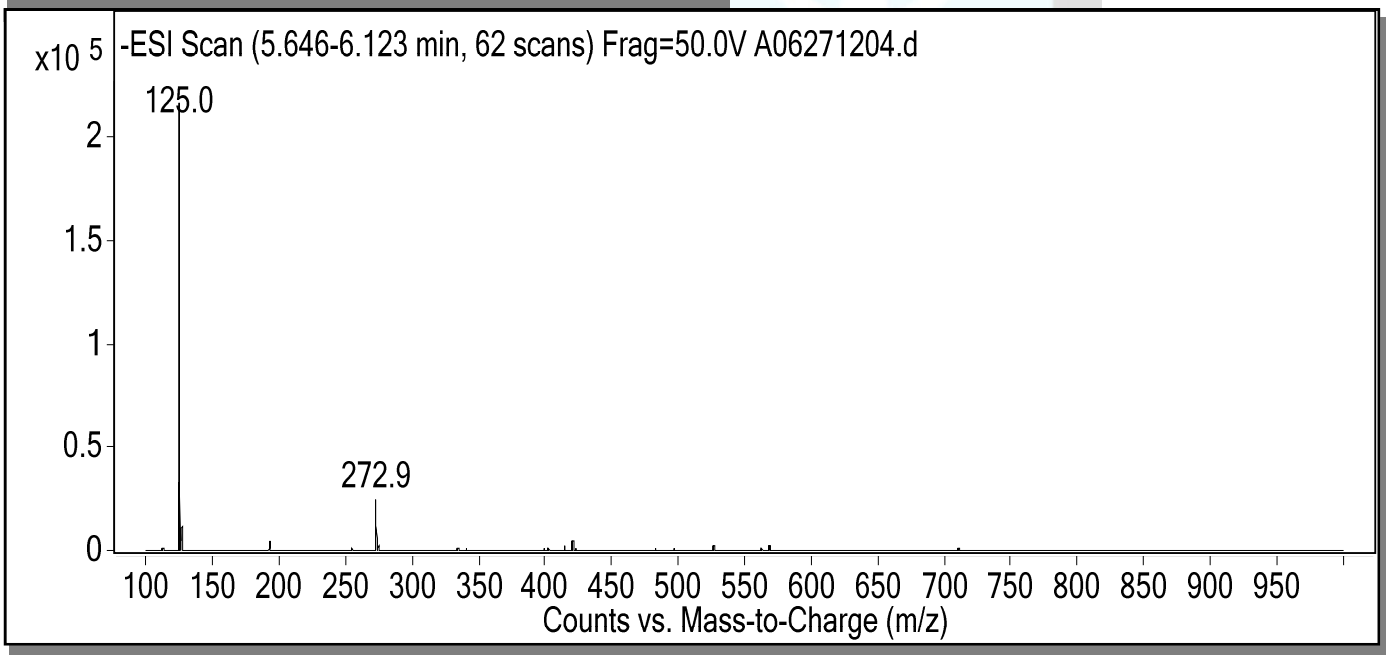


Spectral and Physical Data (cont.)

LC/MS



Column:	Luna C18 (2) 3µ, 2.0 x 100 mm
Mobile Phase:	0.1% Formic acid in Water::0.1% Formic acid in Methanol (99::1 to 95::5 at 3 mins, to 99::1 at 4.1 mins, hold 7.9 mins)
Flow Rate:	0.2 mL/min
Scan Range:	100-1000 amu
Ionization:	Electrospray, Negative Ion
Data File Name:	A06271204.d
Instrument:	LC/MS/MS
Sample Name:	PN061812-01
Acquired:	June 27, 2012

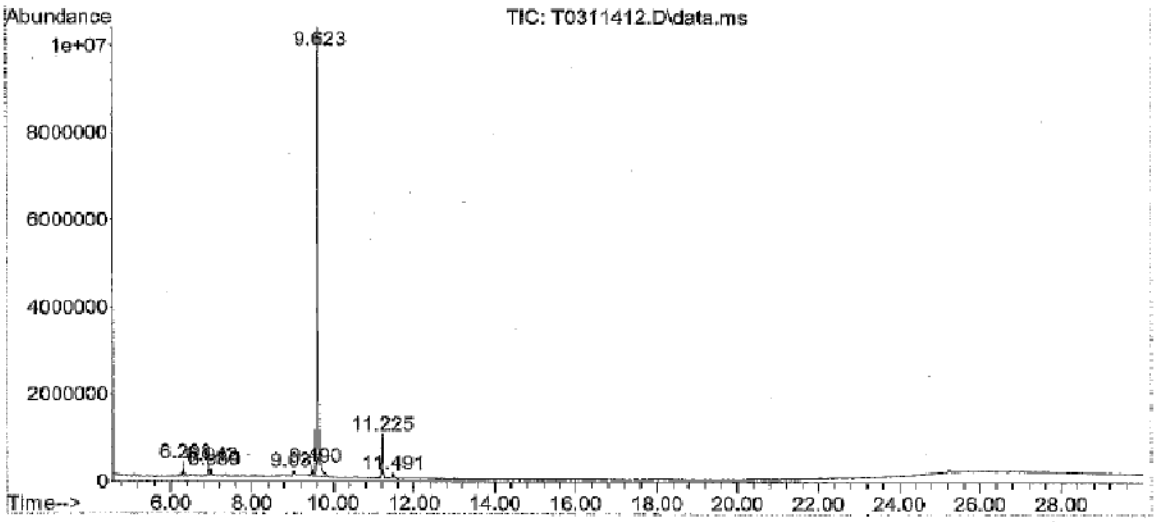


Spectral and Physical Data (cont.)

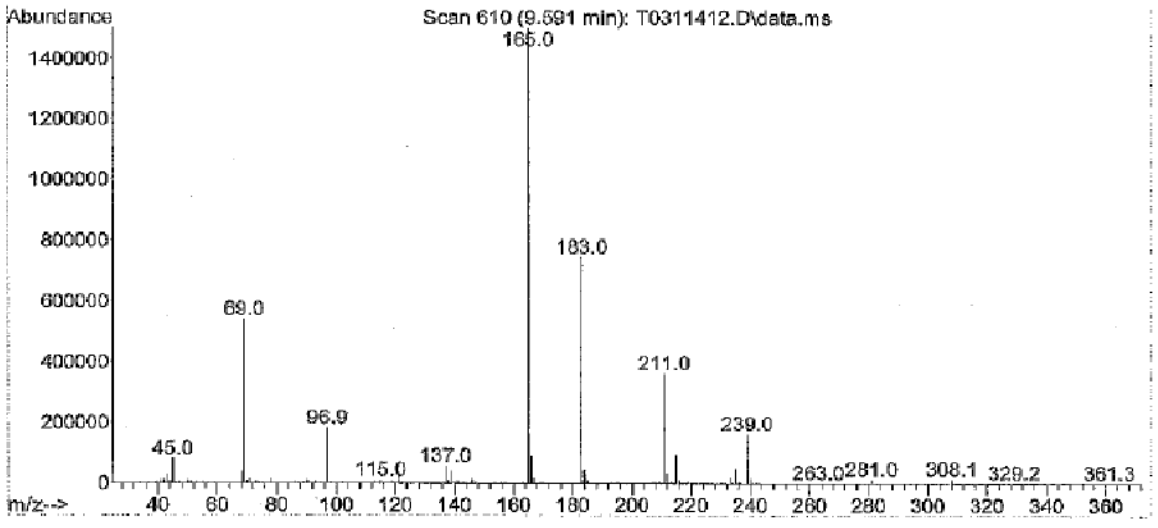
GC/MS

Compound Name	:	Ethyl sulfate
Lot Number	:	FN061412-02
Instrument	:	Agilent GCMS
Operator	:	ECM(SAMUEL_GIUFFRE)
Date Reported	:	Tue Mar 11 16:26:16 2014
Column Type	:	DB-5ms, 30m x 0.25mm ID, 0.25um film thickness
Temp. Program	:	50°C to 300°C @ 10°C/min (hold for 5 min)
Injector Temp.	:	Cool on-column
Carrier Gas	:	Helium
Flow Rate (mL\min)	:	0.80 mL/min
Transfer Line Temp.	:	280°C
Scan Range	:	35-400

Total Ion Chromatogram



Mass Spectrum



Stability

***Short Term Stability** : A summary of accelerated stability findings for this product is listed below.*		
Storage Condition	**Mean Kinetic Temperature (MKT)**	**Time Period**
Freezer	-15°C	No decrease in purity was noted after four weeks.
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 for no more than 3 months at Refrigerate conditions.*		

COA Revision History

Revision No.	Date	Reason for Revision
00	August 28, 2012	Initial version
01	September 13, 2012	Added Stability Section.
02	December 5, 2012	Revised Stability Section to reflect four-week study.
03	March 19, 2013	Revised Retest Date from November 2015 to October 2016.
04	March 12, 2014	Added GC/MS chromatogram and spectrum.