Contamination Knowledge Report

Sonotech Soundsafe ultrasonic couplant

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7 October 2014

Summary

Sonotech Soundsafe ultrasonic couplant (NSN 6850-01-157-4348) (aka Dolphin Lube) is used to for the testing of Frangibolts. The presence of hydantoin, which hydrolyzes to glycine, is unacceptable. A different material must be found.

Method

Aliquots of soundsafe was analyzed via GCMS and DART-FTMS. The detection of hydantoin was verified by hydrolysis followed by LCMS amino acid identification. Details follow.

Results:

From GCMS

The GCMS analysis showed the main components of the SOUNDSAFE® LUBE to be propylene glycol at 3.58 minutes and glycerin at 7.55 minutes.

Table III. Retention times and Library hits for GCMS analysis of the SOUNDSAFE® LUBE in Methanol

Retention Time	Base Peak (m/z)	Library Hit	20141006_Methanol_dolphinlube_01 Sample Base Peak Area	Blank Base Peak Area
24.81	100	Octadecanoic acid-2,3-dihydroxypropyl ester	476494906	-
7.05	77	Propylene glycol	473837336	-
15.2	93	Glycerin/glycerol	295131301	-
9.58	88	Methyldiethanolamine	194899532	-
17.79	55	Dedecylacrylate	78191867	9.82 min - 2136785
16.25	100	Hexadecanamine	50427541	-
16.25	100	Hydantoin	49926491	-
10.49	90	2-amino-2-ethyl-1,3-propanediol	30969944	-
17.39	44	1-tetradecanamine	17800213	-
18.8	100	Hexadecanamine	10091535	-
16.82	90	Diphenyl sulfide?	7536840	-
16.77	100	Hexadecanamine	7368327	-

19.63	**100**	**Hexadecanamine**	**4511887**	**-**
8.89	**90**	**2-propanol, 1,1-‘oxybis**	**3066493**	**-**
21.23	**106**	**Propanoic acid, 3-mercapto-dodecylester**	**1330253**	**-**

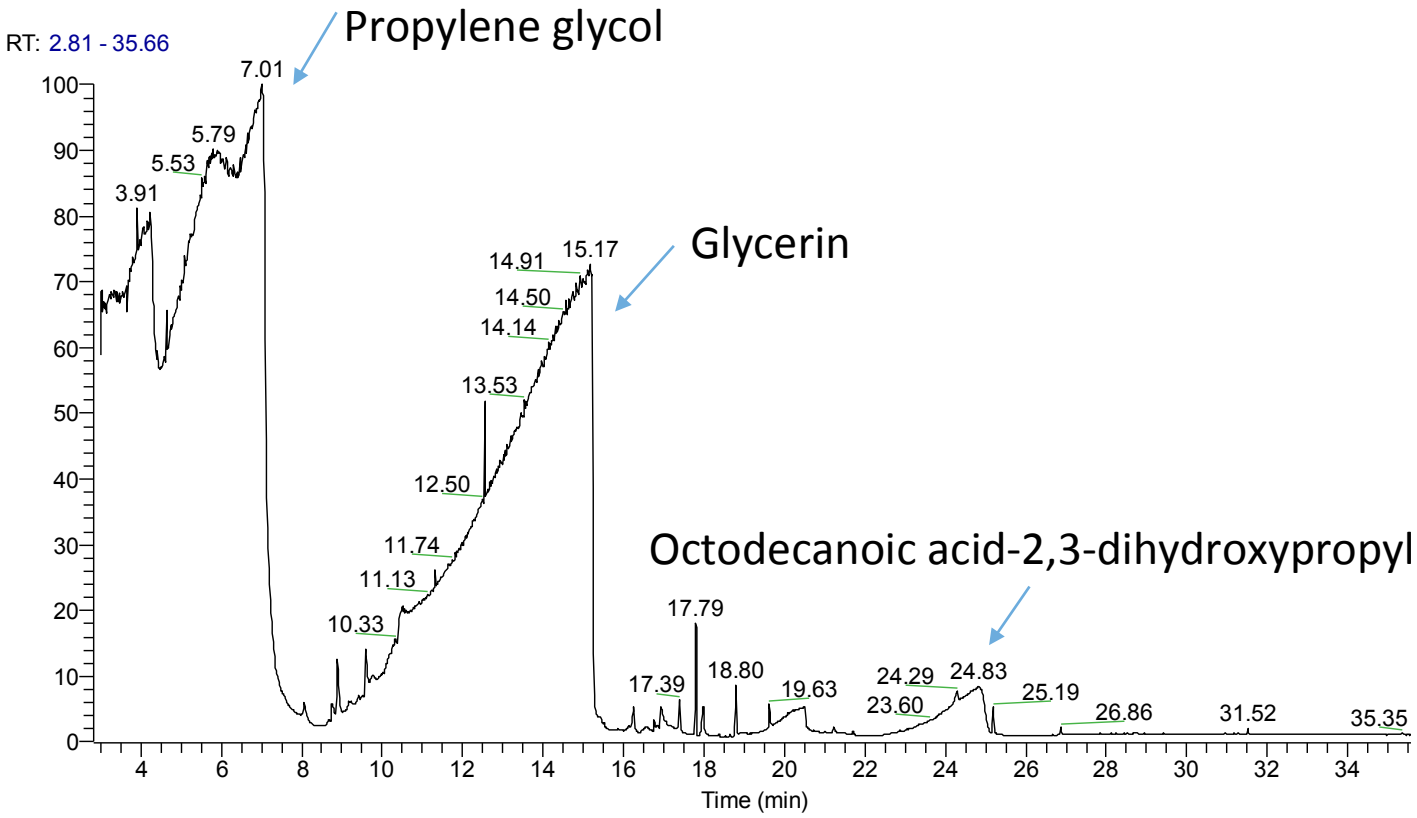


Figure 1. TIC of Dolphin Lube Sample

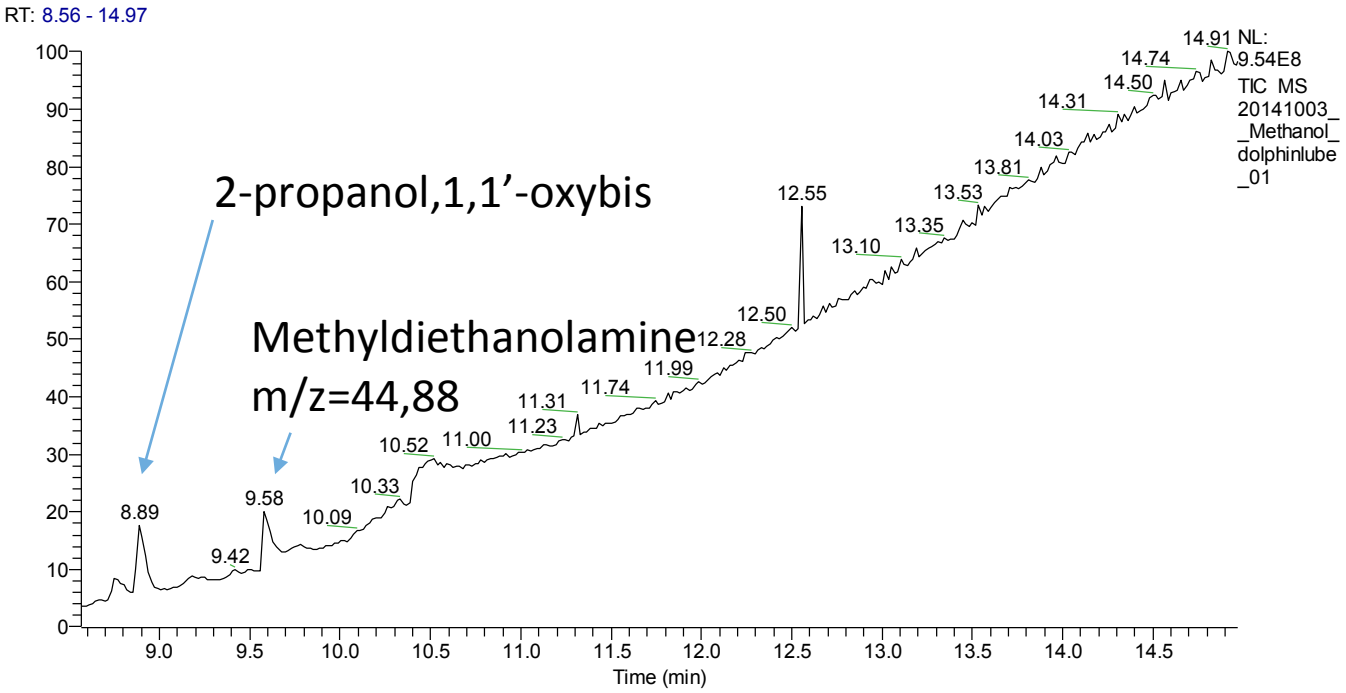


Figure 2. Closeup of 8-15 minutes

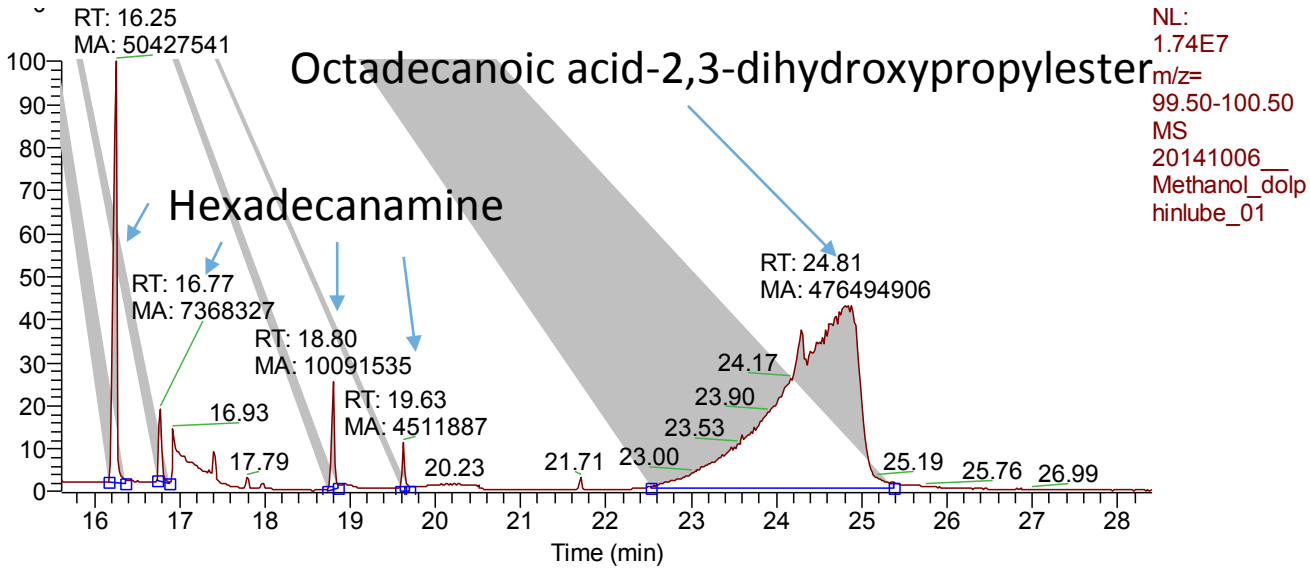


Figure 3. SIC of m/z=99.5-100.5

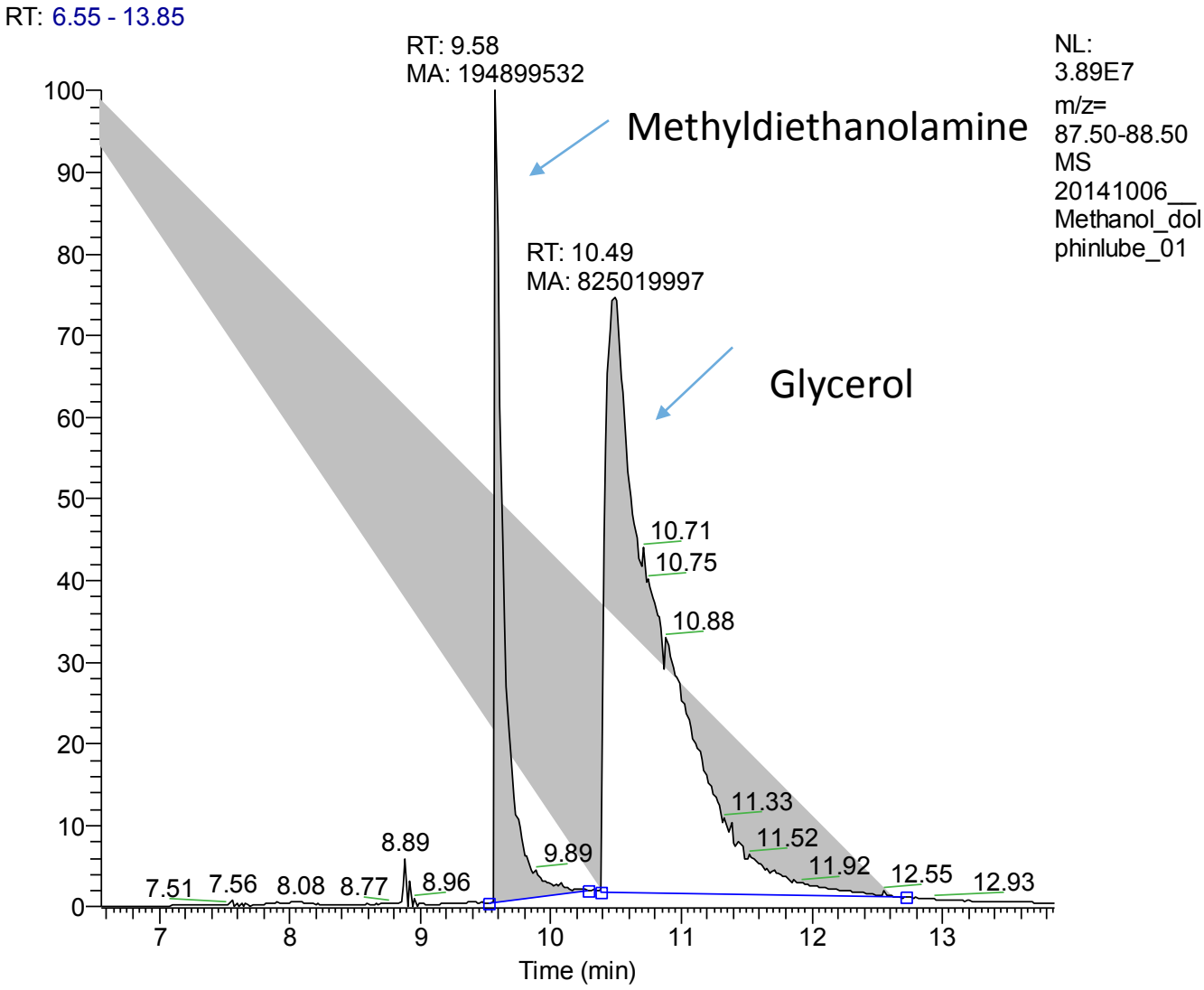
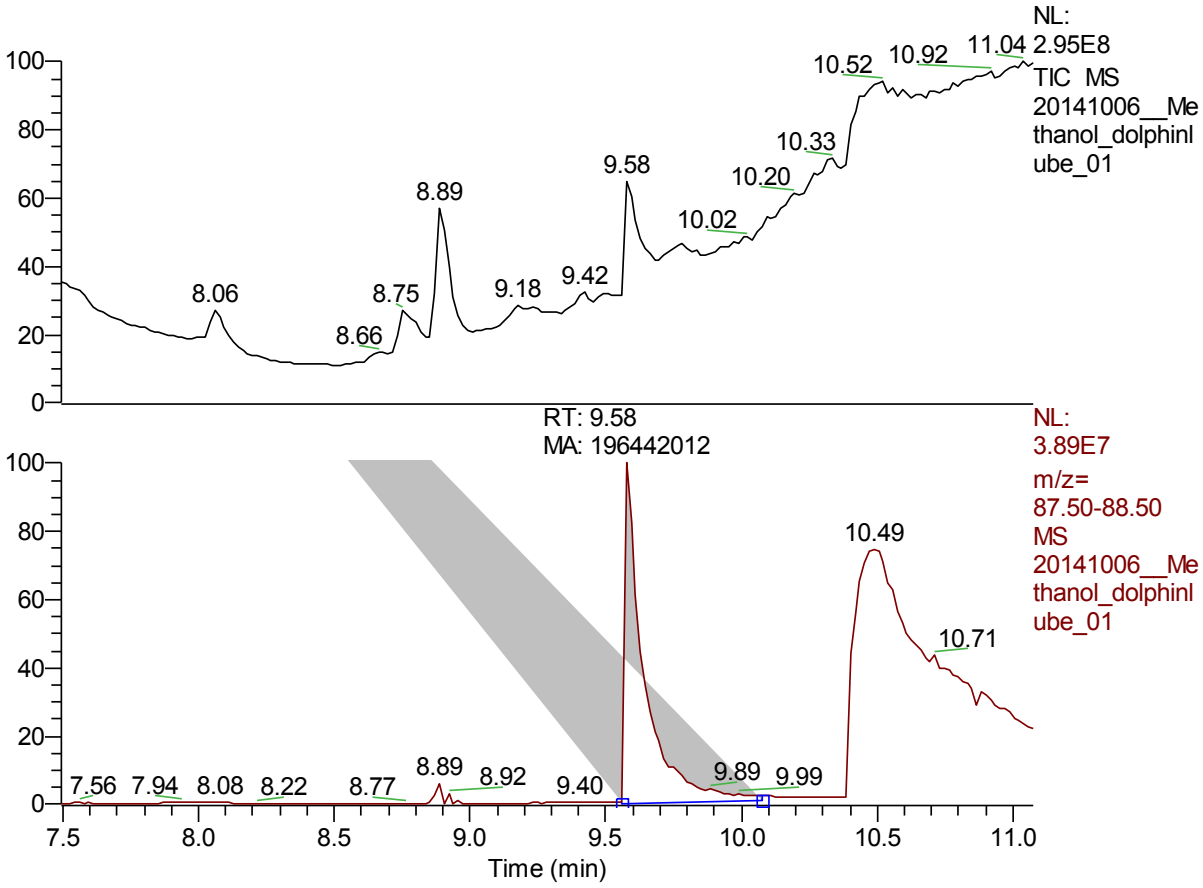


Figure 4. SIC of m/z=87.5-88.5

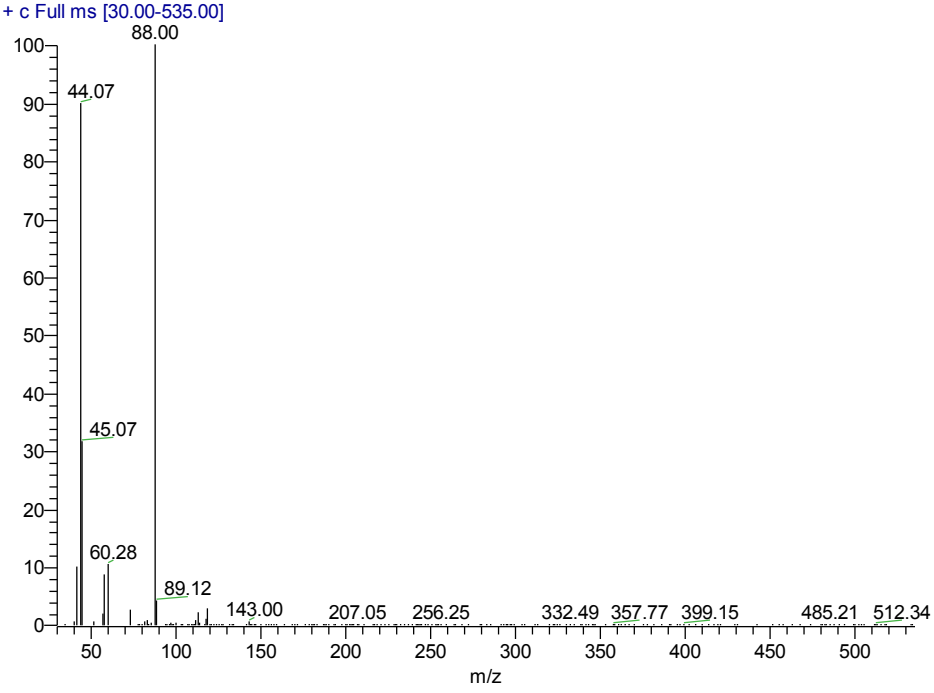
Further Analysis of Methyldiethanolamine

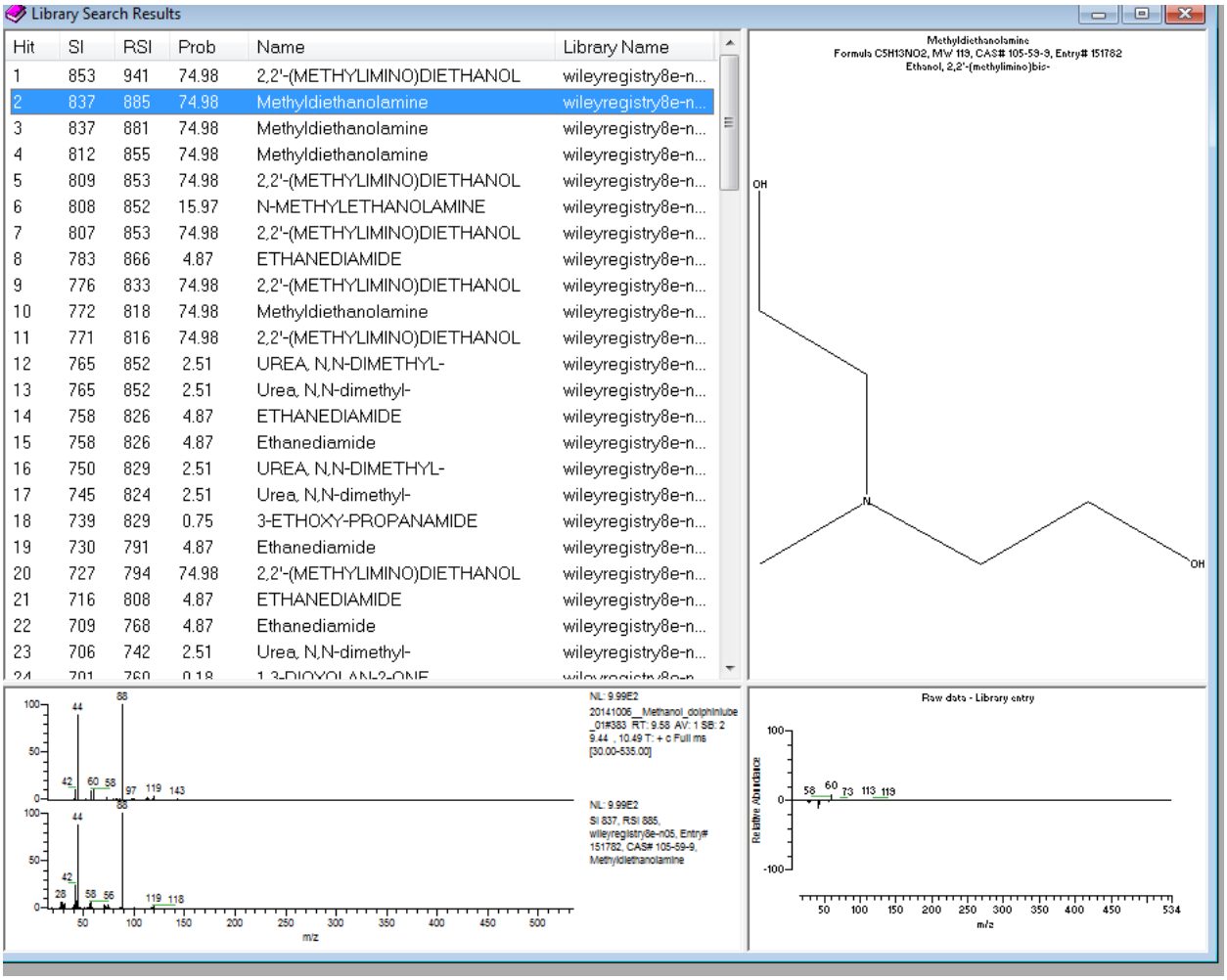
RT: 7.49 - 11.07



20141006__Methanol_dolphinlube_01 #383 RT: 9.56 AV: 1 SB: 2 9.44 , 10.49 NL: 2.43E7

T: + c Full ms [30.00-535.00]





From LCTP

49.727 mg of dolphin lube was weighed out and hydrolyzed with 1 mL of HCL along with a procedural blank.

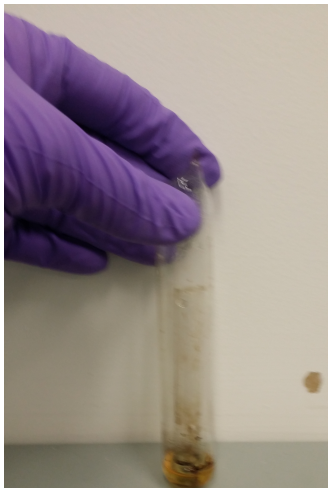


Figure. The picture above shows that some of the dolphin lube migrated into the 20 mm HCl vial during hydrolysis.

The sample was then cracked open and the 10 mm test tube was dried in the centrivap for 20 minutes. The sample was reconstituted in 100 uL of milli-q water and vortexed, the sample was placed in a total recovery vial and two more washes of the 10 mm test tube were performed.

The sample was then dried down in the centrivap.

The figure below shows that there is glycine present in the dolphin lube sample after hydrolysis:

Glycine was seen in levels greater than the blank in the hydolyzed sample. Quantitation is somewhat difficult since I am not sure how much was lost in the hydrolysis.

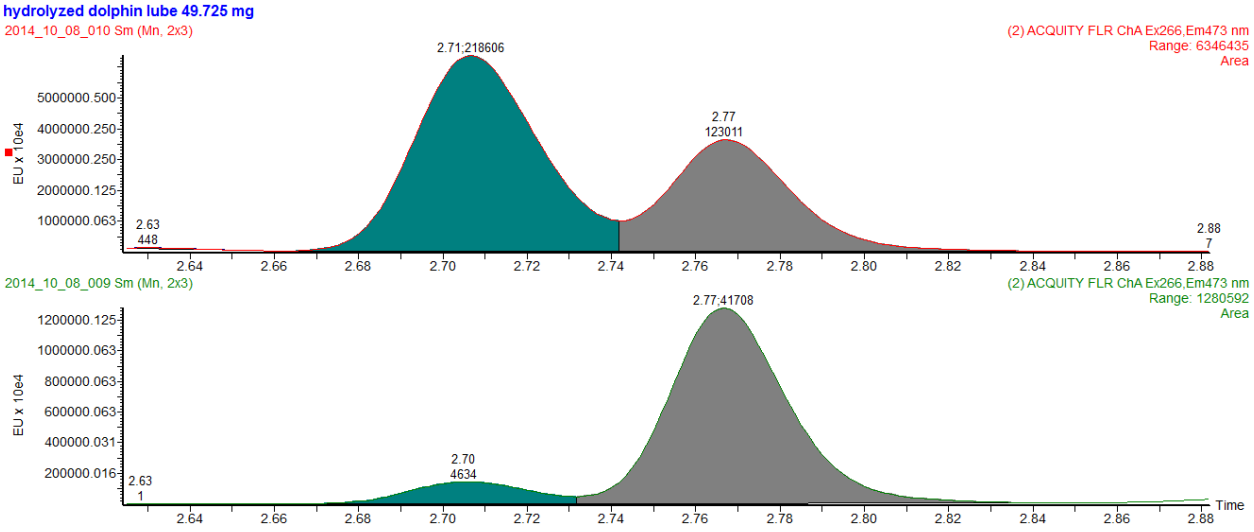


Figure 1. Fluorescence trace of Dolphin Lube (top) and Procedural blank (bottom). The Blue-green peak at 2.71 minutes is glycine.

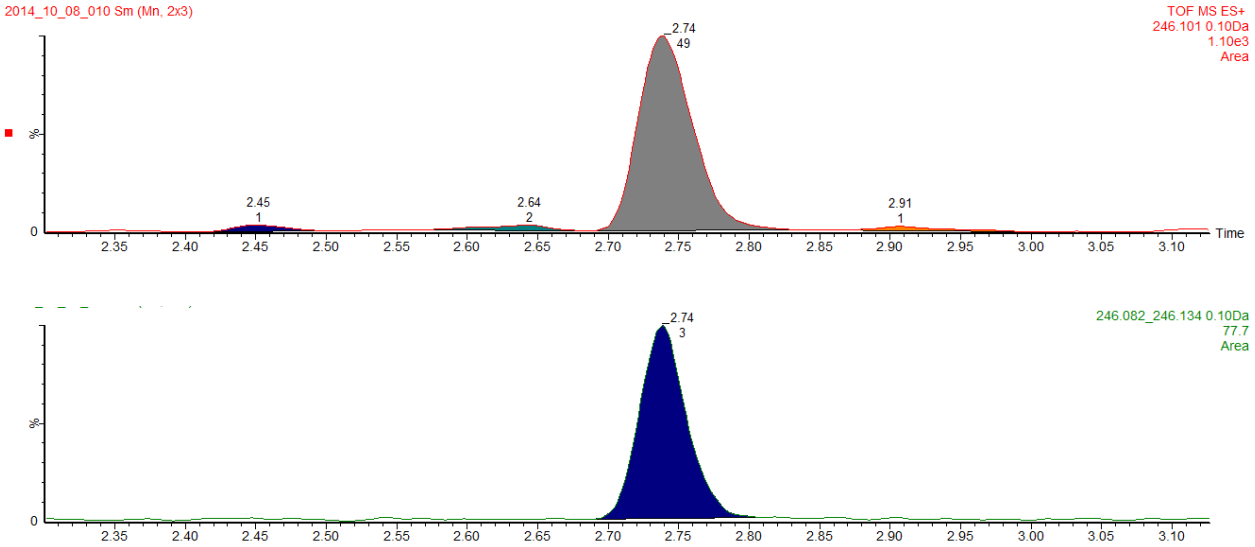
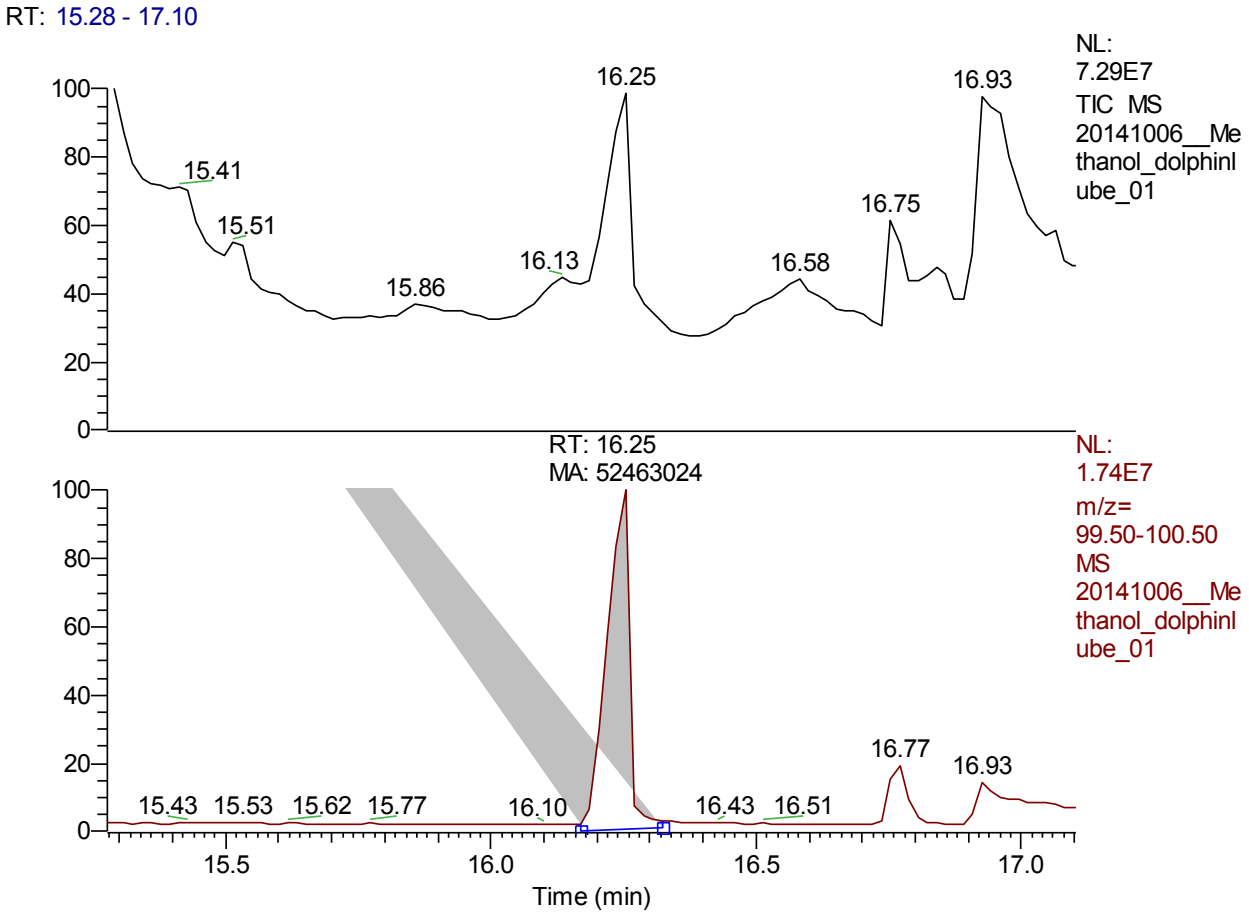
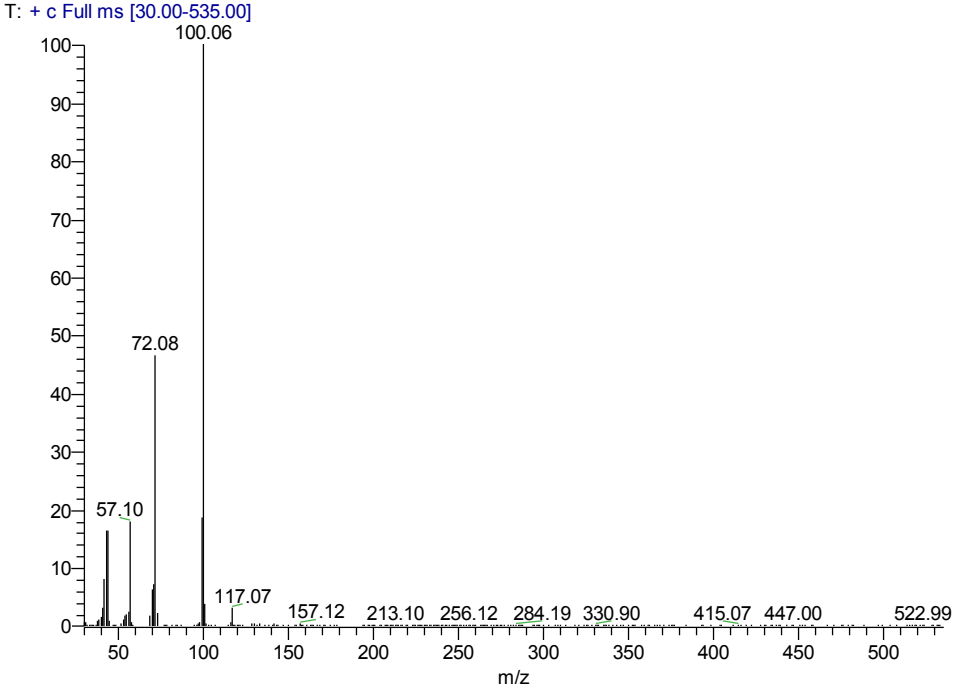


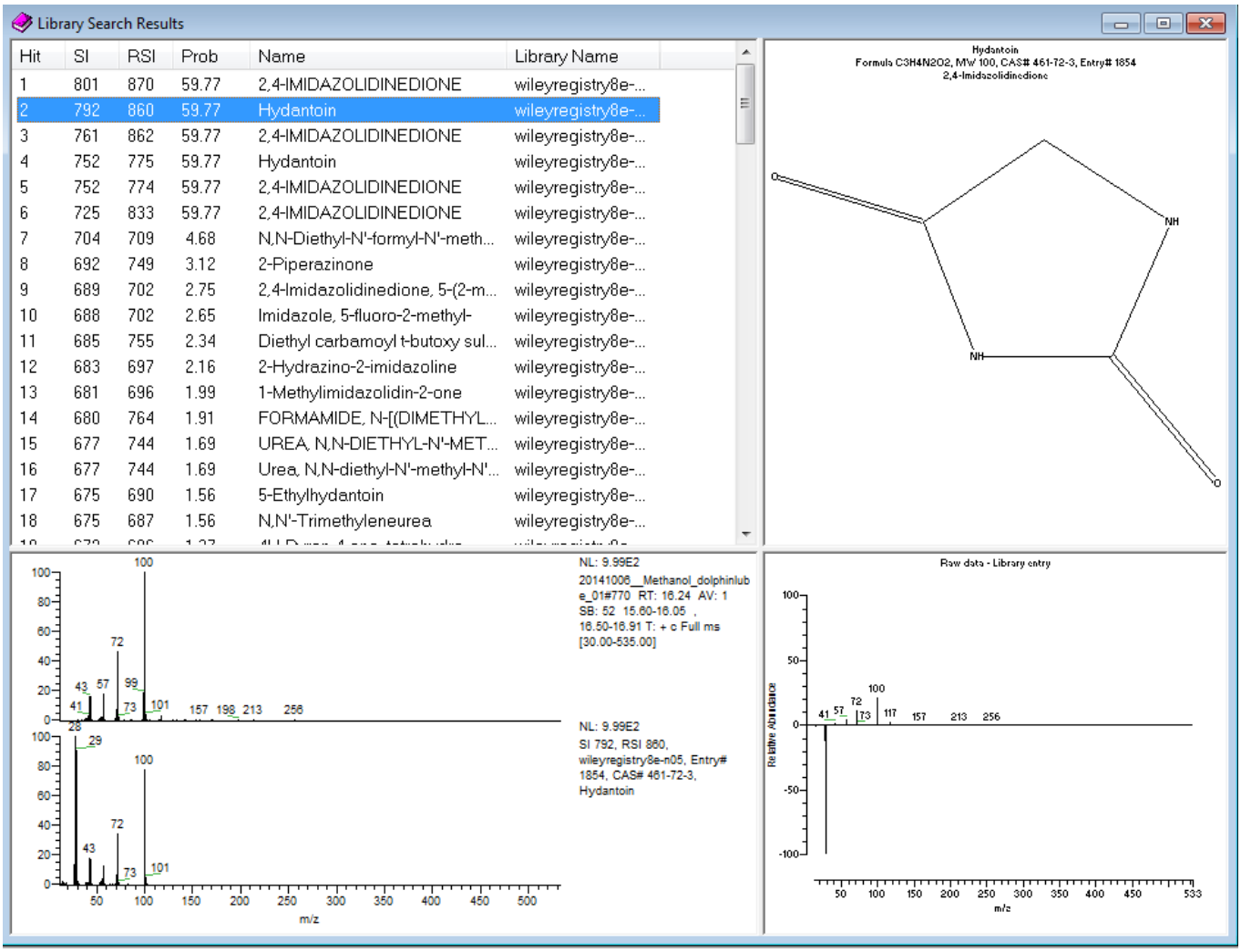
Figure 2. SIC trace of glycine for mass width at half-height of dolphin Lube (top) and Procedural Blank (bottom). The peak at 2.74 min is glycine.

There was not enough glycine in the unhydrolyzed trace to integrate in the Fluorescence or SIC.

Further Analysis of Hydantoin


RT: 15.28 - 17.10
NL:
7.29E7
TIC MS
20141006__Methanol_dolphinlube_01
16.25
16.93
15.41
15.51
16.75
16.13
16.58
15.86
RT: 16.25
MA: 52463024
NL:
1.74E7
m/z=
99.50-100.50
MS
20141006__Methanol_dolphinlube_01
16.77
16.93
15.43
15.53
15.62
15.77
16.10
16.43
16.51
Time (min)


20141006__Methanol_dolphinlube_01 #770 RT: 16.24 AV: 1 SB: 52 15.60-16.05 , 16.50-16.91 NL: 1.40E7
T: + c Full ms [30.00-535.00]
100.06
72.08
57.10
117.07
157.12
213.10
256.12
284.19
330.90
415.07
447.00
522.99
m/z


Library Search Results
Hit SI RSI Prob Name Library Name
1 801 870 59.77 2,4-IMIDAZOLIDINEDIONE wileyregistry8e-...
2 792 860 59.77 Hydantoin wileyregistry8e-...
3 761 862 59.77 2,4-IMIDAZOLIDINEDIONE wileyregistry8e-...
4 752 775 59.77 Hydantoin wileyregistry8e-...
5 752 774 59.77 2,4-IMIDAZOLIDINEDIONE wileyregistry8e-...
6 725 833 59.77 2,4-IMIDAZOLIDINEDIONE wileyregistry8e-...
7 704 709 4.68 N,N-Diethyl-N'-formyl-N'-meth... wileyregistry8e-...
8 692 749 3.12 2-Piperazinone wileyregistry8e-...
9 689 702 2.75 2,4-Imidazolidinedione, 5-(2-m... wileyregistry8e-...
10 688 702 2.65 Imidazole, 5-fluoro-2-methyl- wileyregistry8e-...
11 685 755 2.34 Diethyl carbamoyl t-butoxy sul... wileyregistry8e-...
12 683 697 2.16 2-Hydrazino-2-imidazoline wileyregistry8e-...
13 681 696 1.99 1-Methylimidazolidin-2-one wileyregistry8e-...
14 680 764 1.91 FORMAMIDE, N-[(DIMETHYL... wileyregistry8e-...
15 677 744 1.69 UREA, N,N-DIETHYL-N'-MET... wileyregistry8e-...
16 677 744 1.69 Urea, N,N-diethyl-N'-methyl-N'... wileyregistry8e-...
17 675 690 1.56 5-Ethylhydantoin wileyregistry8e-...
18 675 687 1.56 N,N'-Trimethyleneurea wileyregistry8e-...
Hydantoin
Formula C3H4N2O2, MW 100, CAS# 461-72-3, Entry# 1854
2,4-Imidazolidinedione
NH
NH
O
O
NL: 9.99E2
20141006__Methanol_dolphinlube_01#770 RT: 16.24 AV: 1 SB: 52 15.60-16.05 , 16.50-16.91 T: + c Full ms [30.00-535.00]
NL: 9.99E2
SI 792, RSI 860, wileyregistry8e-n05, Entry# 1854, CAS# 461-72-3, Hydantoin
m/z
Raw data - Library entry
Relative Abundance
m/z

DART-MS Analysis (M. Callahan)

A small amount of SOUNDSAFE® LUBE was smeared on the end of a sterilized glass pipet and was placed between the direct analysis in real-time (DART) ion source (IonSense) and the transfer tube of an LTQ Orbitrap XL mass spectrometer (Thermo Scientific) operated in positive ion mode. The temperature of the DART gas (helium) was set to 350 °C.

LCTP Analysis (H. McLain)

A small amount (28.657 mg) of SOUNDSAFE® LUBE was dissolved in 10 uL milli-Q water, 20 uL Accqtag derivitizing agent, and 70 uL of Borate Buffer. The sample was heated at 55 °C for 10 minutes. The Accqtag LC MS method with Flurorescence and MS analysis was used.

from DART-MS (M. Callahan)

The DART mass spectrum of the SOUNDSAFE® LUBE is shown in Figure 1. Additionally, a table of the 10 most intense mass peaks along with their probable elemental composition (using C, H, O, and N) is shown in Table 1. For the most intense peaks, a quick structure search brings up candidates that contain primary or secondary amines. If this were the case, then these compounds may also be detected using the AccQ-Tag methods and LC-FD/ToF MS. Product ion spectra acquired for m/z 106 and 150 using the HCD cell indicated the presence of a hydroxyl group (rather than an amine group).

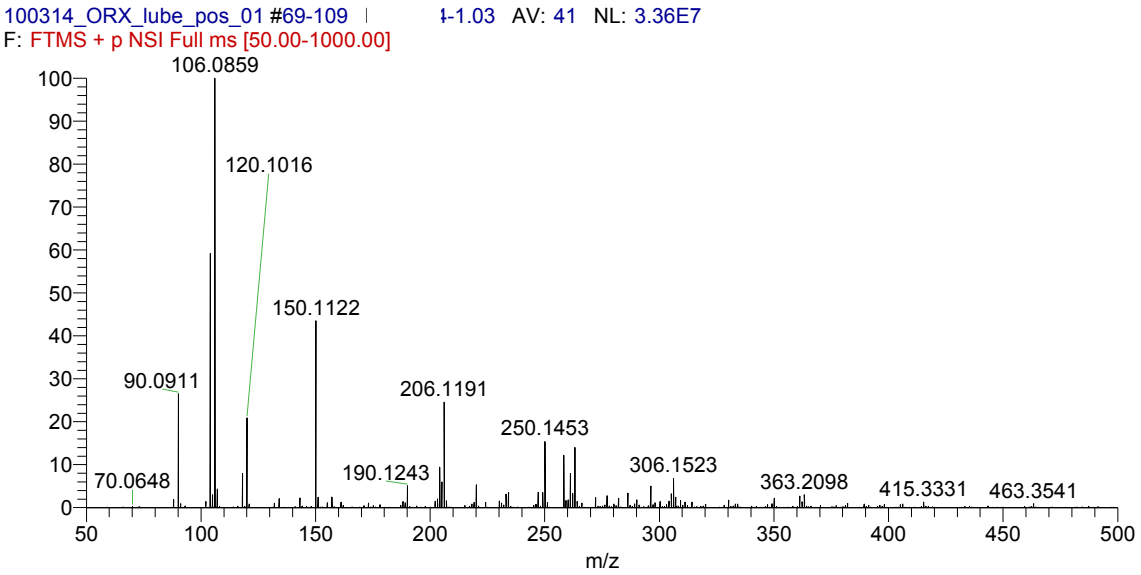


Figure 1: DART mass spectrum of SOUNDSAFE® LUBE.

From LCMS

The fluorescence trace for the SOUNDSAFE® LUBE is shown in Figure 2. Table II lists the most intense peaks along with their mass. Only a few of the peaks seen in the DART-MS analysis showed up in the LCMS study. No known free amino acids were detected in the SOUNDSAFE® LUBE using the Accqtag method with a direct injection of the substance.

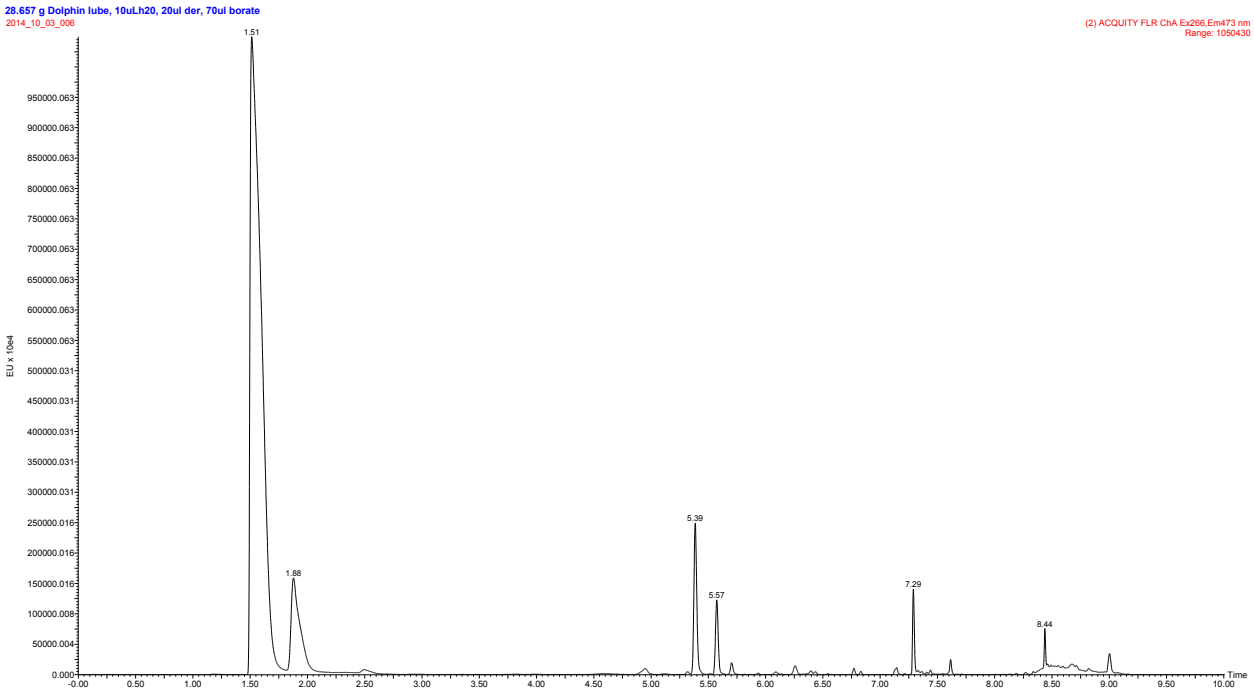


Figure 2. LCMS fluorescence trace of SOUNDSAFE® LUBE

From GCMS

The GCMS analysis showed the main components of the SOUNDSAFE® LUBE to be propylene glycol at 3.58 minutes and glycerin at 7.55 minutes.

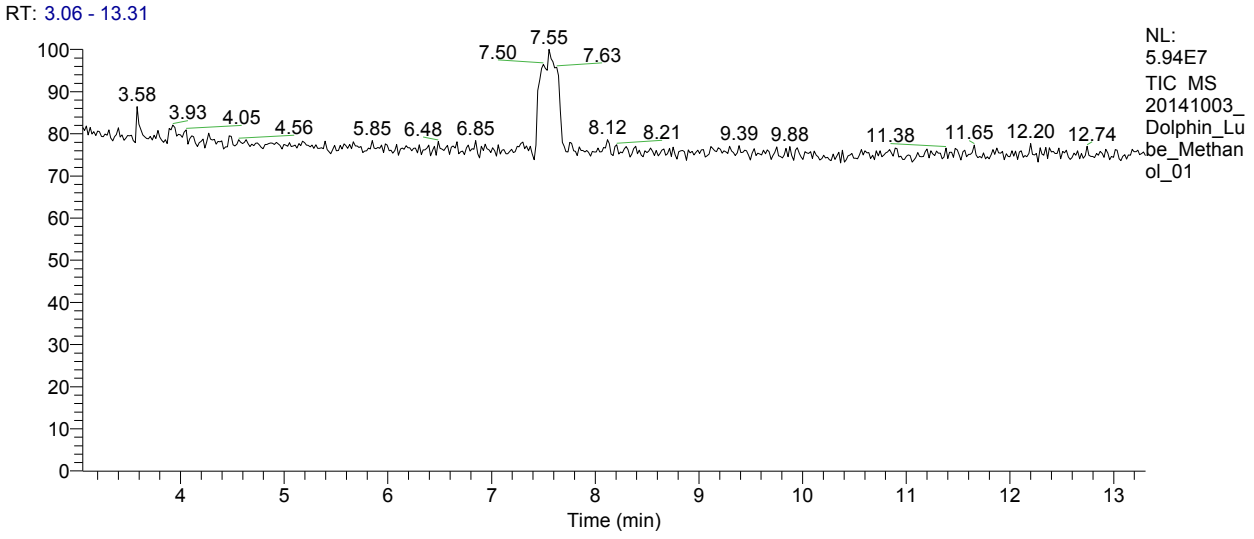


Table 1: Top 10 most intense peaks in the DART mass spectrum.

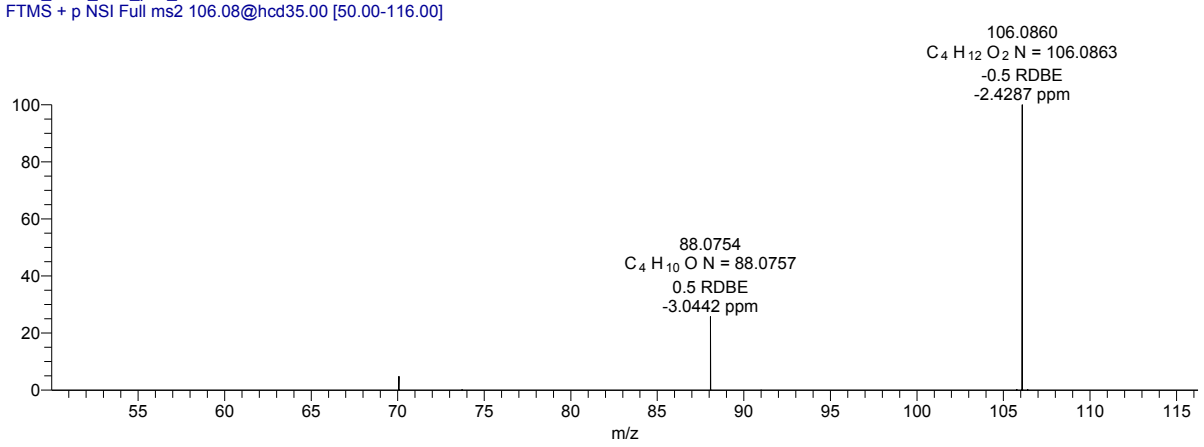
m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
106.0859	34597180	100	106.0863	-2.89	-0.5	C4 H12 O2 N

104.1068	20128186	58.18	104.107	-2.13	-0.5	C5 H14 O N
150.1122	14695151	42.47	150.1125	-1.83	-0.5	C6 H16 O3 N
90.0911	8967100	25.92	90.0913	-3.22	-0.5	C4 H12 O N
206.1191	8446094	24.41				
120.1016	7055051	20.39	120.1019	-2.21	-0.5	C5 H14 O2 N
250.1453	5190730	15	250.1465	-4.41	10	C17 H18 N2
263.1769	4843881	14				
258.2425	4104407	11.86	258.2428	-1.1	0.5	C15 H32 O2 N
204.14	3169623	9.16				

Table II: Most intense LCMS peaks

Fluorescence Retention Time	Selected Ion Retention Time	LCMS m/z	Corresponding Dart MS m/z	Identified Peaks	Guessed Peaks
	6.80	**277.15**	**106.0859**		**Diethanolamine**
	7.66	**321.15**	**150.1122**		**Aminoethoxyethanol, Triethanolamine??**
	8.69	**261.15**	**90.0911**		**Dimethylethanolamine, Diethylhydroxylamine, Aminomethyl propanol**
	9.02	**421.15**	**250.1453**		**Difenzoquat??**
1.88	**1.92**	**188.15**		**Ammonia**	
5.39	**5.42**	**245.15**			
7.29	**7.32**	**315.15**			
7.61	**7.68**	**432.2**			
	7.88	**476.2**			
	8.42	**349.15**			

100314_ORX_lube_pos_04 #32-40 RT: 0 AV: 9 NL: 1.02E7
T: FTMS + p NSI Full ms2 106.08@hcd35.00 [50.00-116.00]



100314_ORX_lube_pos_04 #75-91 RT: 0 AV: 17 NL: 1.44E6
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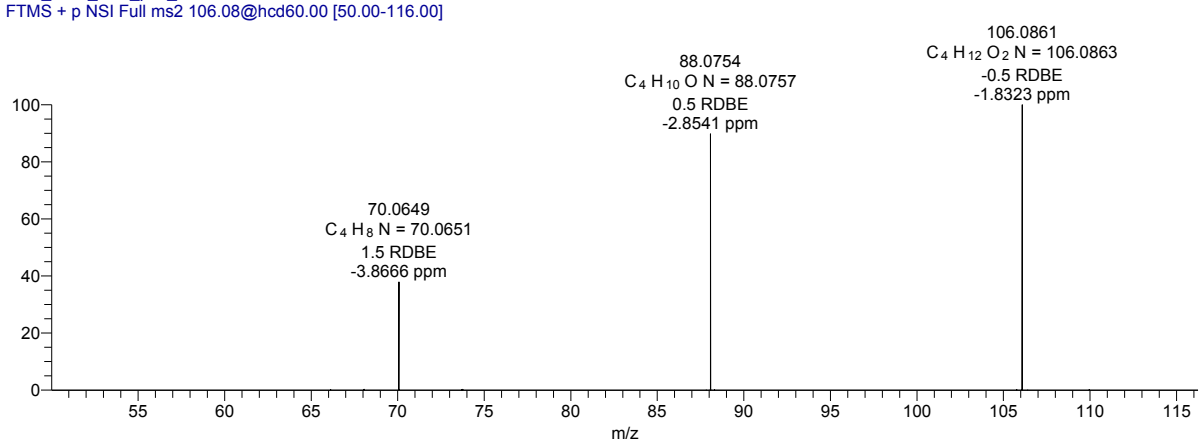

100314_ORX_lube_pos_04 #103-116 RT .52 AV: 14 NL: 8.37E5
T: FTMS + p NSI Full ms2 106.08@hcd100.00 [50.00-116.00]

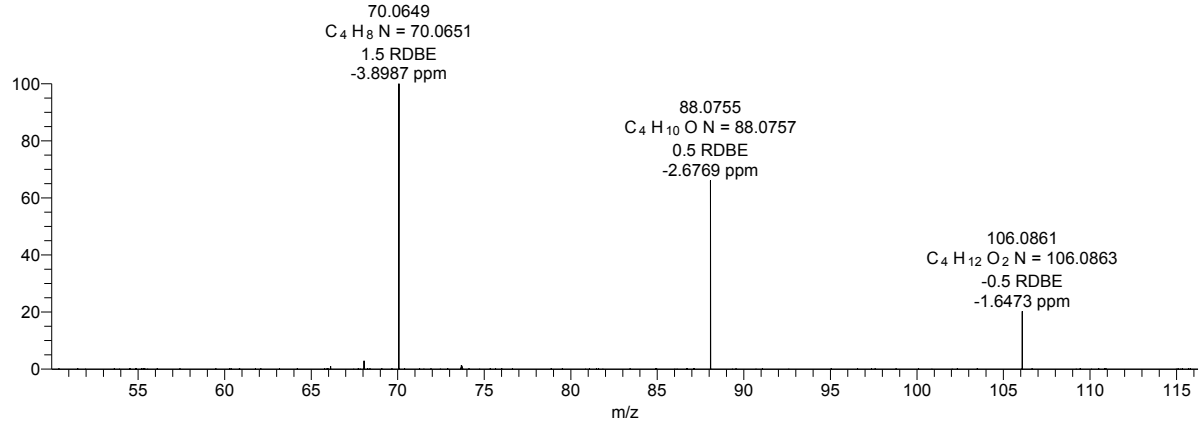

Figure 2: Product ion spectra of m/z 106.08 recorded at HCD 35% (top), 60% (middle), and 100% (bottom).

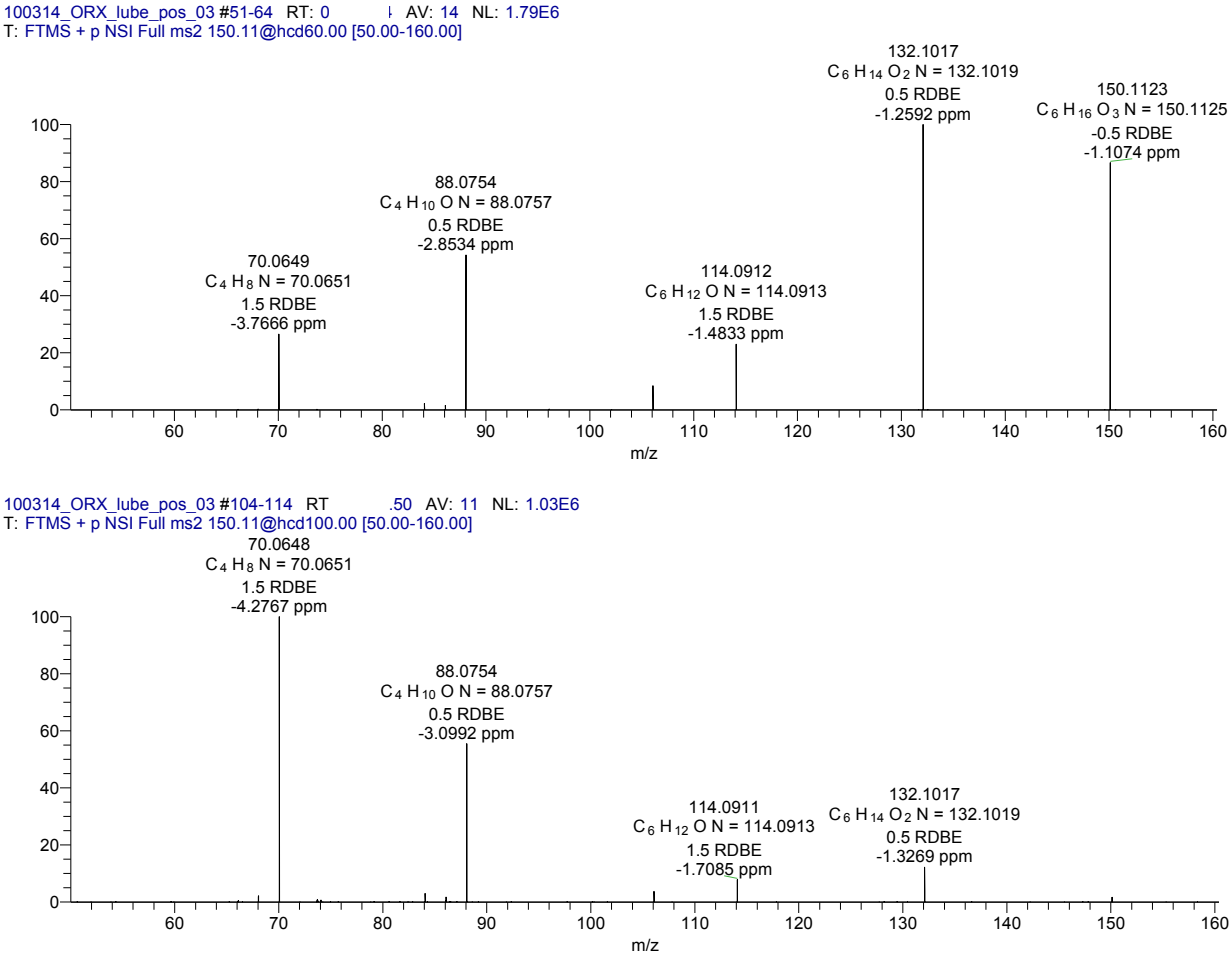

Figure 3: Product ion spectra of m/z 150.11 recorded at HCD 35% (top), 60% (middle), and 100% (bottom).