



GAKMAPS Foils 10 -13 Contamination Knowledge Report

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Summary

Witness Coupons were analyzed for amino acids for Contamination Knowledge

Introduction

The purpose of this experiment was to test the organic contents of witness foils. The foil was split into three sections (1) one-half was used for hot water extraction and subsequent Dart-MS and LCTP amino acid analysis (2) a small portion was used for pyrolysis GCMS (3) one-quarter was saved for further analyses

LCMS Analysis:

Procedure

The tube with the witness foils were flame sealed with 2 mL of 18.2M Ω 3 ppb TOC Millipore water (hereafter water) and extracted at 100°C for 24 hours.

	Mass (g)
Sample OR-CKP-10-1-A, O	0.0281
Sample OR-CKP-11-1-A, O	0.0208
Sample OR-CKP-12-1-A, O	0.0179
Sample OR-CKP-13-1-A, O	0.0213

The extract was removed and sample tubes rinsed with 1000 μ L of water and dried via centrifugal evaporation. The samples were hydrolyzed over 6M HCl vapor for 3 hours at 150°C. The samples were dried again.

Analysis

The dried sample extract were suspended in 100 μ L water. Of that 50 μ L was dried in a total recovery vial and reconstituted in 10 μ L of water, 20 μ L of Waters AccQ•Tag derivatizing agent, and 70 μ L of borate. Both samples and standards were heated for 10 minutes at 55°C immediately following the addition of the derivatizing agent. The sample was then analyzed via the commercial Waters AccQ•Tag protocol on a Waters LCT Premier time of flight mass spectrometer equipped with an electrospray ionization source (positive ion mode), mass resolution setting of 5,000 m/ Δ m but without external mass accuracy calibration. Sample was introduced via a Waters Acquity UPLC with fluorescence detector. For UPLC analysis a 250 μ L syringe, 50 μ L loop, and 30 μ L needle were used. The total injection volume was 1 μ L. A set of 9 calibrators of proteinogenic amino acids (0.25 to 250 μ M) was prepared in water and analyzed. A linear least-square model was fit to each analyte. Selected ion traces were quantitated. Since two sides of the foil were exposed the final value was halved. A Procedural Blank sample was used to

subtract procedural and laboratory background.

Results

The amino acid levels on the aluminum witness plates are well within the range. To convert from grams of aluminum foil to cm² of aluminum foil a factor of 97.2 cm²/g. The amount of amino acids in these samples is well below the 180 ng/cm² limit.

Table I. Amino acid content on Aluminum witness plates in ng/cm²

	Sample 10	Sample 11	Sample 12	Sample 13
Gly	0.52 ± 0.22	1.23 ± 0.30	≤ 0.95	≤ 1.22

Pyrolysis GCMS Analysis:

Each sample was analyzed using a RT-Q-Bond, 30 meter, 0.25 mm ID, 8um df column to allow for the analysis of small volatile compounds. A blank was ran before each sample.

Pyrolysis	Mass (g)
Sample OR-CKP-10-1-A, O	0.0031
Sample OR-CKP-11-1-A, O	0.0028
Sample OR-CKP-12-1-A, O	0.0039
Sample OR-CKP-13-1-A, O	0.0080

Table 1. Blank subtracted peak area divided by 1e6 and the mass of the foil used

Retention Time	Base Peak	Analyte	JSC 10	JSC 11	JSC 12	JSC 13
8.88	31	Methyl Alcohol	18.8	0.0	0.0	0.0
9.33	44	Acetaldehyde	77.5	51.6	119.8	47.4
11.26	55	1-Butene	9.7	0.0	63.5	29.2
13.57	58	Acrolein	20.5	5.6	24.9	11.0
13.57	43	Acetone	60.7	26.3	58.7	31.1
14.84	70	Cyclopentane	7.7	8.2	11.4	3.5
17.81	84	1-hexene	11.5	10.2	13.9	5.5
18.26	78	Benzene	161.1	205.2	227.4	143.8
20.38	98	1-heptene	7.4	9.5	10.9	2.6
Total			374.9	316.6	530.5	274.1

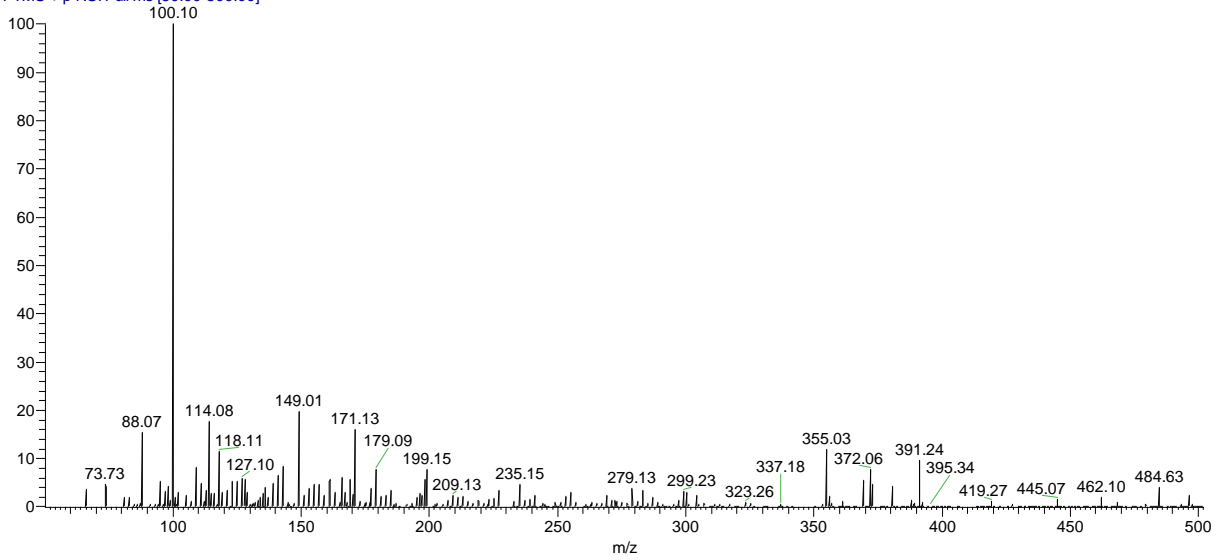
DART-MS Analysis:

Sample extracts were spotted (5 µL) on a steel mesh sampling unit at the inlet of the DART SVP (Direct Analysis in Real Time) source. The DART source (He gas, 350 °C, positive ion mode) was coupled to a LTQ-Orbitrap XL hybrid mass spectrometer with a mass resolution setting 30,000 and a lock mass enabled (on a polysiloxane compound found in air background) for high resolution, accurate mass measurements of low-molecular weight organics.

DART mass spectra of unhydrolyzed and acid hydrolyzed extracts are complex, but strongly resemble the mass spectrum of the stainless steel sampling mesh. No major difference was seen between samples.

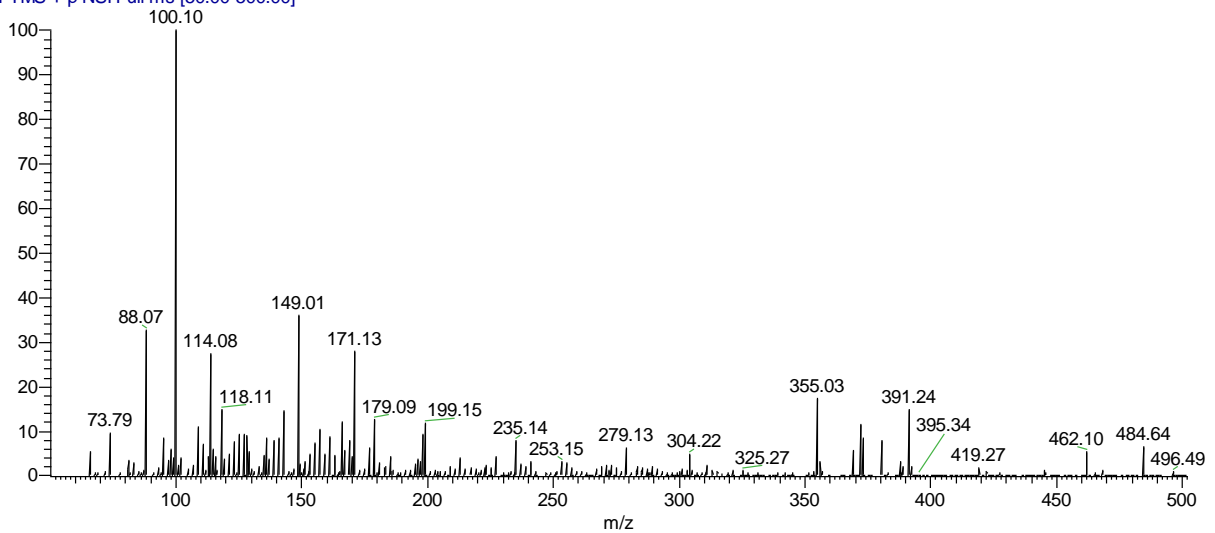
DART Figure 1. OSIRIS-REx JSC Contamination Knowledge Foil #13

20160815_ORX_022 #24-46 RT: 0.27-0.50 AV: 23 NL: 5.33E5
T: FTMS + p NSI Full ms [50.00-500.00]



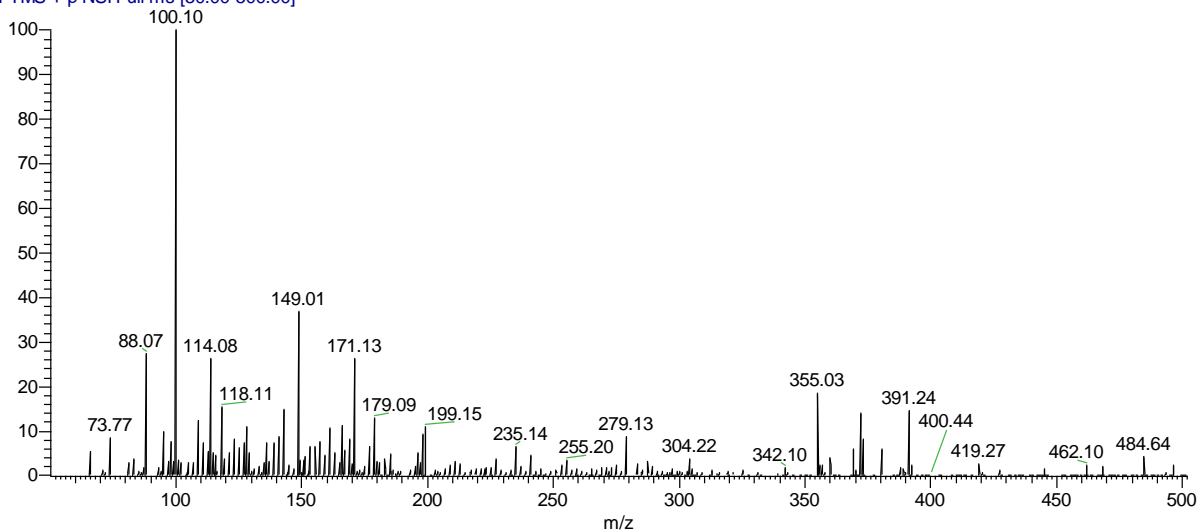
DART Figure 2. OSIRIS-REx JSC Contamination Knowledge Foil PB2

20160815_ORX_020 #29-45 RT: 0.32-0.49 AV: 17 NL: 2.79E5
T: FTMS + p NSI Full ms [50.00-500.00]



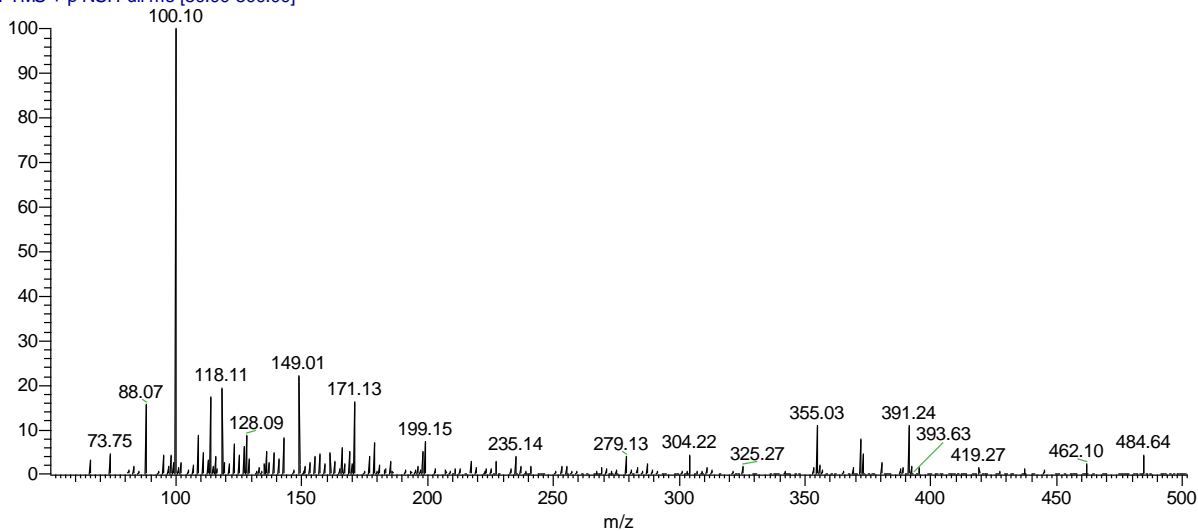
DART Figure 3. OSIRIS-REx JSC Contamination Knowledge Foil #12

20160815_ORX_018 #27-47 RT: 0.30-0.51 AV: 21 NL: 3.18E5
T: FTMS + p NSI Full ms [50.00-500.00]



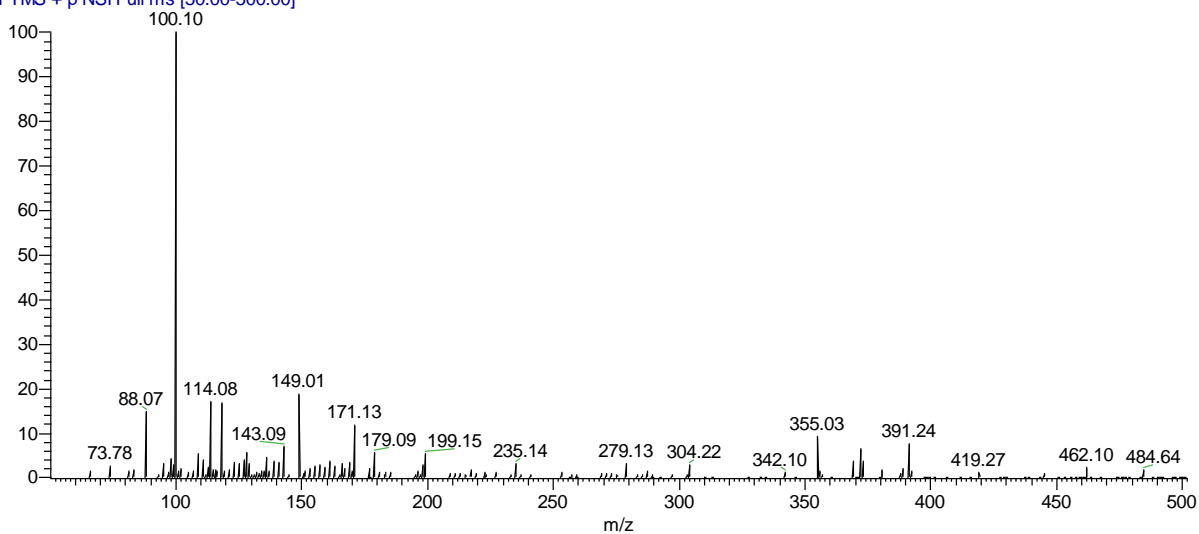
DART Figure 4. OSIRIS-REx JSC Contamination Knowledge Foil PB1

20160815_ORX_016 #29-46 RT: 0.32-0.50 AV: 18 NL: 5.87E5
T: FTMS + p NSI Full ms [50.00-500.00]



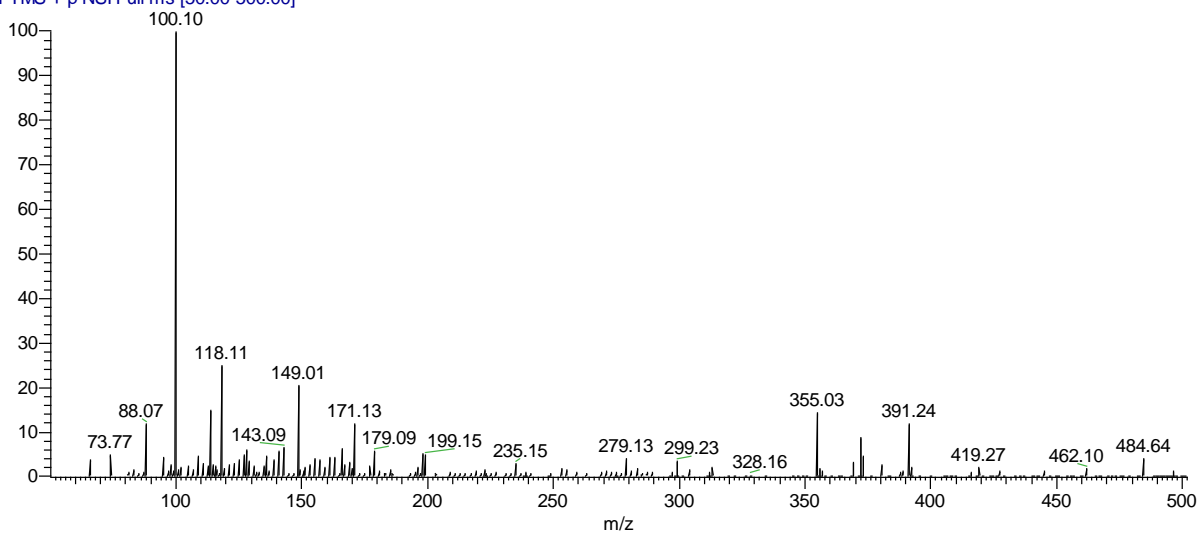
DART Figure 5. OSIRIS-REx JSC Contamination Knowledge Foil #11

20160815_ORX_012 #17-45 RT: 0.19-0.50 AV: 29 NL: 5.44E5
T: FTMS + p NSI Full ms [50.00-500.00]



DART Figure 6. OSIRIS-REx JSC Contamination Knowledge Foil #10

20160815_ORX_010 #19-45 RT: 0.21-0.48 AV: 27 NL: 4.98E5
T: FTMS + p NSI Full ms [50.00-500.00]



DART Figure 7. OSIRIS-REx JSC Contamination Knowledge Foil PB

20160815_ORX_008 #17-41 RT: 0.19-0.45 AV: 25 NL: 2.81E5
T: FTMS + p NSI Full ms [50.00-500.00]

