



# GAKMAPS Foils 14 -16 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

Sample	Date	Description
OR-CKP-14-1-A, O	4/27-6/17	LM (SARA deployment/flight head test) / ship / PHSF (s/c spin/balance, battery install, instrument inspections)
OR-CKP-15-1-A, O	6/17-7/14	PHSF (MLI installations, OLA and OTES cleaning and inspection)
OR-CKP-16-1-A, O	7/14-8/26	PHSF (solar array install., OVIRS inspection, contam. cleaning and sampling, encapsulation)

## LCMS Analysis:

### Procedure

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

Sample	Mass (g)
OR-CKP-14-1-A, O	0.0232
OR-CKP-15-1-A, O	0.0240
OR-CKP-16-1-A, O	0.0220

The extract was removed and sample tubes rinsed with 1000μL 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 100μL water. Of that 50 μL was dried in a total recovery vial and reconstituted in 10 μL of water, 20 μL of Waters AccQ•Tag derivatizing agent, and 70 μL 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/Δm but without external mass accuracy calibration. Sample was

introduced via a Waters Acquity UPLC with fluorescence detector. For UPLC analysis a 250  $\mu\text{L}$  syringe, 50  $\mu\text{L}$  loop, and 30  $\mu\text{L}$  needle were used. The total injection volume was 1  $\mu\text{L}$ . A set of 9 calibrators of proteinogenic amino acids (0.25 to 250  $\mu\text{M}$ ) 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  $\text{cm}^2$  of aluminum foil a factor of 97.2  $\text{cm}^2/\text{g}$ . The amount of amino acids in these samples is well below the 180  $\text{ng}/\text{cm}^2$  limit.

It is unexpected that the abundances and diversity of amino acids in OR-CKP-15-1-A, O is as large as it is. Contamination control samples covering a nearly identical time, though on a much larger surface with a different exposure angle to the PHSF highbay show nearly blank levels of amino acids, only glycine was observed (Table 2). This is nearly 300x lower than observed in OR-CKP-15-1-A, O.

**Table 1.** Amino acid content on Aluminum witness plates in  $\text{ng}/\text{cm}^2$

Average	OR-CKP-14-1-A, O	OR-CKP-15-1-A, O	OR-CKP-16-1-A, O
His	$\leq 0.1$	$\leq 0.1$	$\leq 0.1$
Ser	$0.06 \pm 0.17$	$2.85 \pm 0.54$	$0.55 \pm 0.19$
Arg	$\leq 0.1$	$1.42 \pm 0.50$	$\leq 0.1$
Gly	$0.79 \pm 0.17$	$2.86 \pm 0.62$	$2.50 \pm 0.40$
Asp	$\leq 0.1$	$\leq 0.1$	$\leq 0.1$
Glu	$\leq 0.1$	$2.33 \pm 0.66$	$\leq 0.1$
Thr	$\leq 0.1$	$1.14 \pm 0.19$	$\leq 0.1$
Ala	$0.05 \pm 0.07$	$1.49 \pm 0.29$	$0.35 \pm 0.16$
Pro	$\leq 0.1$	$2.29 \pm 0.60$	$\leq 0.1$
Tyr	$\leq 0.1$	$\leq 0.1$	$\leq 0.1$
Cys	$\leq 0.1$	$\leq 0.1$	$\leq 0.1$
Lys	$\leq 0.1$	$\leq 0.1$	$\leq 0.1$
Met	$\leq 0.1$	$\leq 0.1$	$\leq 0.1$
Val	$\leq 0.1$	$1.63 \pm 0.19$	$\leq 0.1$
Leu	$\leq 0.1$	$0.82 \pm 0.34$	$\leq 0.1$
Ile	$\leq 0.1$	$1.54 \pm 0.45$	$\leq 0.1$
Phe	$\leq 0.1$	$0.41 \pm 0.36$	$\leq 0.1$
<b>Total</b>	<b><math>0.90 \pm 0.42</math></b>	<b><math>18.78 \pm 4.73</math></b>	<b><math>3.40 \pm 0.70</math></b>

**Table 2.** Amino acid content (only glycine was detected) on CC witness plates in  $\text{ng}/\text{cm}^2$

Description	Glycine
PHSF Environmental Monitoring - East 6/17-7/13/16	$0.069 \pm 0.008$
PHSF Environmental Monitoring - Blank #1	$0.060 \pm 0.007$

## Pyrolysis GCMS Analysis:

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

Sample	Mass (g)
OR-CKP-14-1-A, O	0.0088
OR-CKP-15-1-A, O	0.0096
OR-CKP-16-1-A, O	0.0090

**Table 3.** Blank subtracted peak area divided by 10<sup>6</sup> and the mass of the foil used

Retention Time	Base Peak	Analyte	OR-CKP-14-1-A, O	OR-CKP-15-1-A, O	OR-CKP-16-1-A, O
9.07	31	Methyl Alcohol	9.5	11.2	10.7
9.67	44	Acetaldehyde	53.6	26.5	41.5
11.61	55	1-Butene	6.3	0.0	14.6
13.88	58	Acrolein	12.3	9.0	17.4
13.88	43	Acetone	33.3	22.8	48.6
15.16	70	Cyclopentane	0.9	5.2	1.0
18.17	84	1-Hexene	2.6	7.7	3.8
18.61	78	Benzene	59.3	91.7	76.1
20.73	98	1-Heptene	1.8	4.1	2.5
		<b>Total</b>	<b>179.6</b>	<b>178.2</b>	<b>216.2</b>

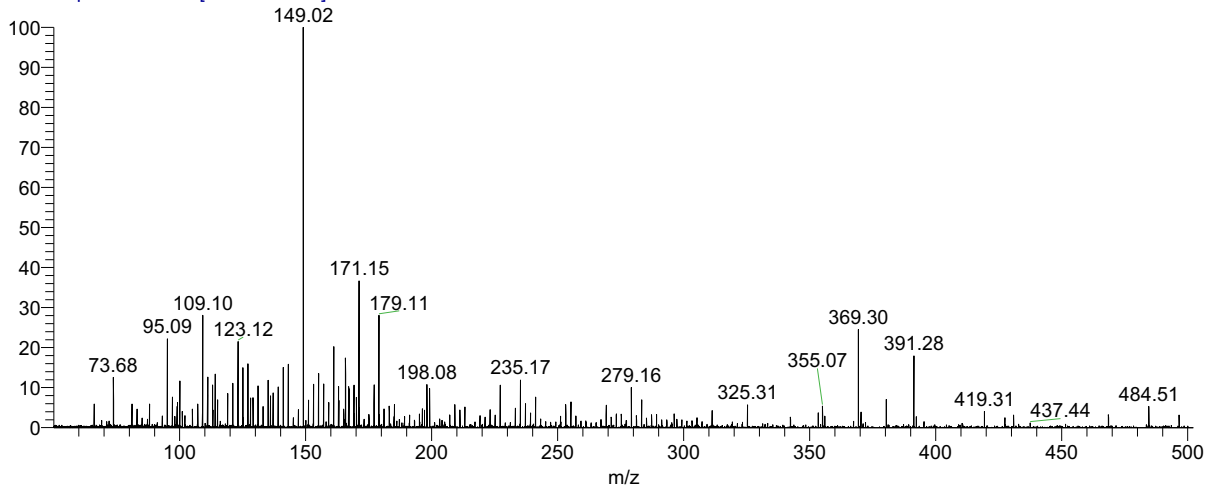
## DART-MS Analysis:

Sample extracts were spotted (5  $\mu$ L) 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.

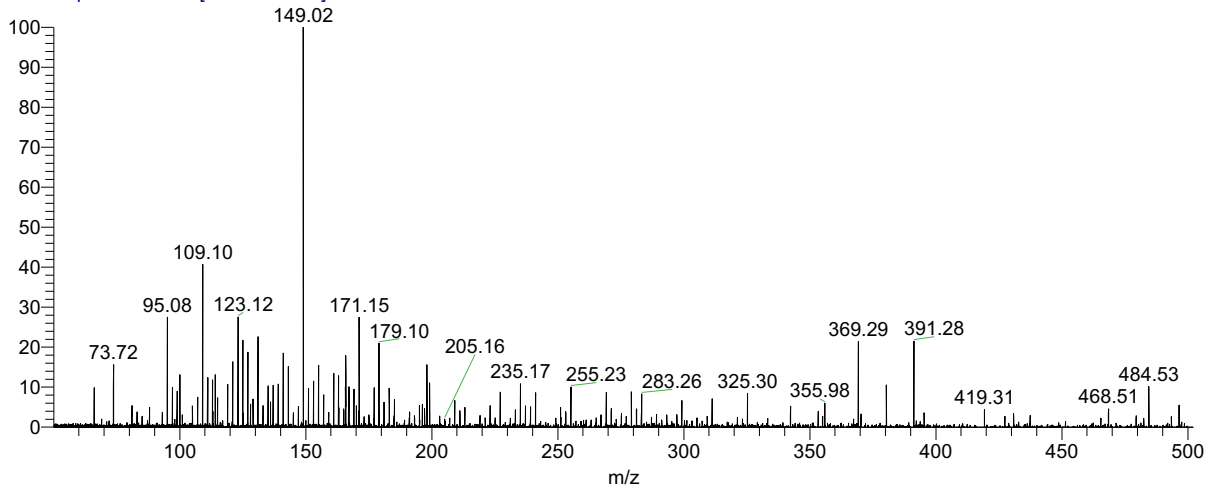
### DART Figure 1. Procedural blank

20161031\_ORX\_0011 #22-41 RT: 0.4 AV: 20 NL: 2.13E5  
T: FTMS + p NSI Full ms [50.00-500.00]



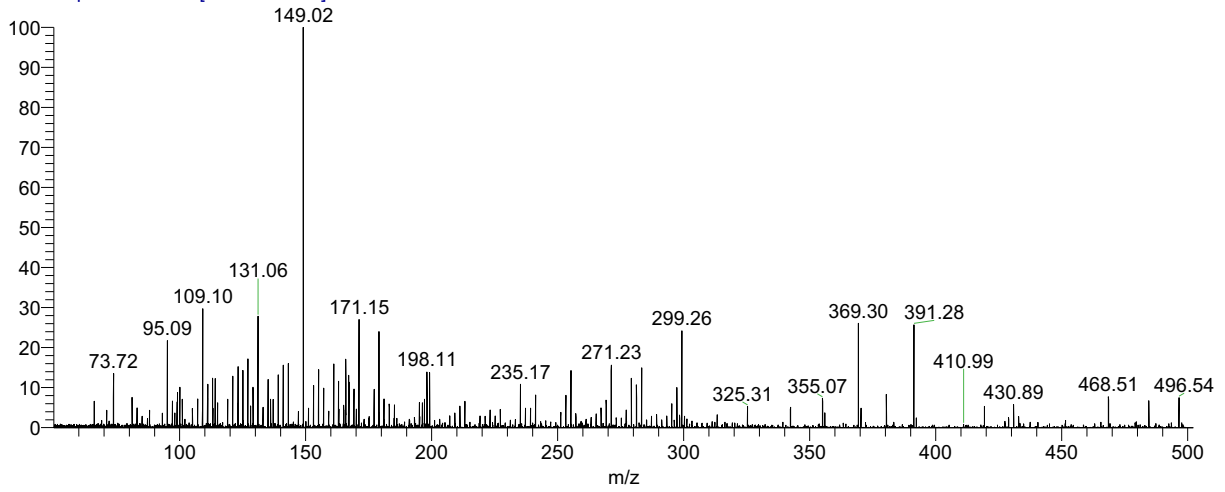
### DART Figure 2. Contamination Knowledge Foil #14

20161031\_ORX\_0013 #21-42 RT: 0.4 AV: 22 NL: 2.16E5  
T: FTMS + p NSI Full ms [50.00-500.00]



### DART Figure 3. OR-CKP-15-1-A, O

20161031\_ORX\_0015 #16-35 RT: 0. AV: 20 NL: 2.07E5  
T: FTMS + p NSI Full ms [50.00-500.00]



### DART Figure 5. OR-CKP-16-1-A, O

20161031\_ORX\_0017 #22-42 RT: 0. AV: 21 NL: 1.84E5  
T: FTMS + p NSI Full ms [50.00-500.00]

