



Atlas V 411 (AV-067) Fairing 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

One sample and blank (1 ft² each) plates were sent intact to GSFC. The foils were torn from the tape edges and separated from the backing in an ISO 5 flow bench and placed in tubes. Foils for analysis were sandwiched between top and bottom clean foils. 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.

Table I. Mass of LC Foils

	Mass of Test Tube (g)	Mass after foil added (g)	Mass (g)
OSIRIS-REx Fairing 1	5.2310	5.2975	0.0665
OSIRIS-REx Fairing 2	5.1978	5.2727	0.0749
OSIRIS-REx Fairing 3	5.2139	5.2677	0.0538
Blank Foil 1	5.2380	5.3308	0.0928
Blank Foil 2	5.2547	5.3570	0.1023
Blank Foil 3	5.2339	5.2971	0.0632

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 μL

syringe, 50 μL loop, and 30 μL needle were used. The total injection volume was 1 μL . A set of 9 calibrators of proteinogenic amino acids (0.25 to 250 μM) 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 cm^2 of aluminum foil a factor of 97.2 cm^2/g . The amount of amino acids in these samples is well below the 180 ng/cm^2 limit.

Table II. Amino acid content on Aluminum witness plates in ng/cm^2

Average	Glycine	Glycine Blank Average Subtracted	Glycine Blank Subtracted Average
OSIRIS-REx Fairing 1	0.51	0.49	0.48 \pm 0.04
OSIRIS-REx Fairing 1	0.45	0.43	
OSIRIS-REx Fairing 1	0.54	0.51	
OSIRIS-REx Fairing 2	0.21	0.18	0.17 \pm 0.02
OSIRIS-REx Fairing 2	0.17	0.14	
OSIRIS-REx Fairing 2	0.22	0.19	
OSIRIS-REx Fairing 3	0.10	0.07	0.05 \pm 0.02
OSIRIS-REx Fairing 3	0.06	0.03	
OSIRIS-REx Fairing 3	0.09	0.06	
Blank Foil 1	0.01		
Blank Foil 1	0.04		
Blank Foil 1	0.00		
Blank Foil 2	0.06		
Blank Foil 2	0.07		
Blank Foil 2	0.07		
Blank Foil 3	\leq 0.01		
Blank Foil 3	\leq 0.01		
Blank Foil 3	\leq 0.01		

Pyrolysis GCMS Analysis:

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

Table IV: Mass of foils used for pyrolysis

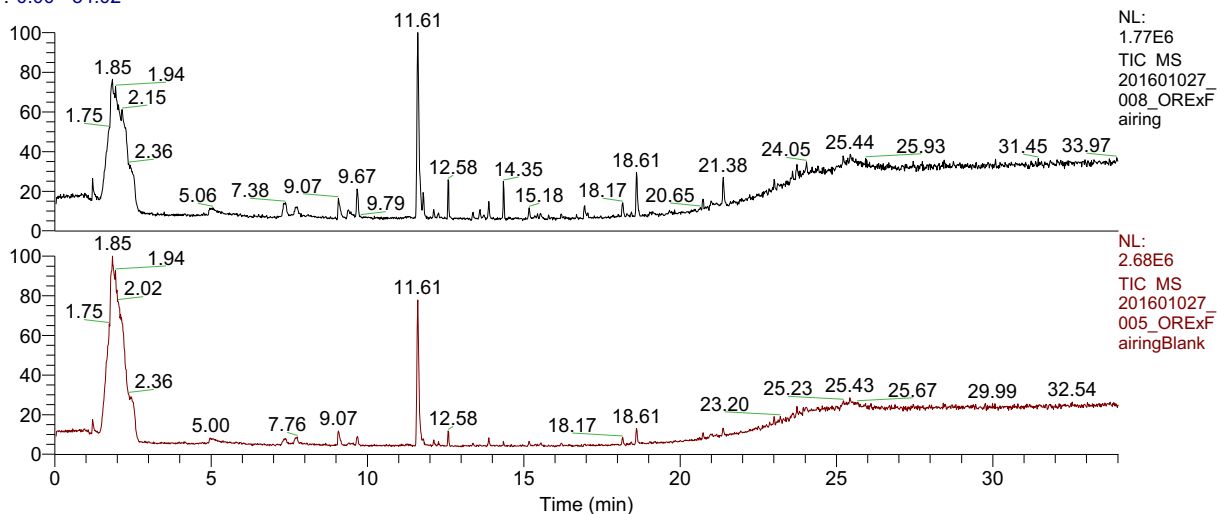
Pyrolysis	Mass (g)
Fairing	0.0062
Fairing Blank	0.0092

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

Retention Time	Base Peak	Analyte	Fairing	Fairing Blank
9.07	31	Methyl Alcohol	16.7	14.9
9.67	44	Acetaldehyde	45.6	14.6
11.61	55	1-Butene	0	0.0
13.88	58	Acrolein	13.4	5.7
13.88	43	Acetone	32.9	13.6
14.35	53	Propenenitrile	39.6	1.4
15.16	70	Cyclopentane	2.6	0.9
18.17	84	1-hexene	3.1	1.6
18.61	78	Benzene	63.7	23.7
20.73	98	1-heptene	1.7	1.1
		Total	219.4	77.5

Pyrolysis Figure 1. (Top) OSIRIS-REx Pyrolysis Fairing (bottom) OSIRIS-REx Blank Foil. Please note the peak at 14.35 that has a library hit with a 93.9% probability of being 2-propenenitrile

RT: 0.00 - 34.02



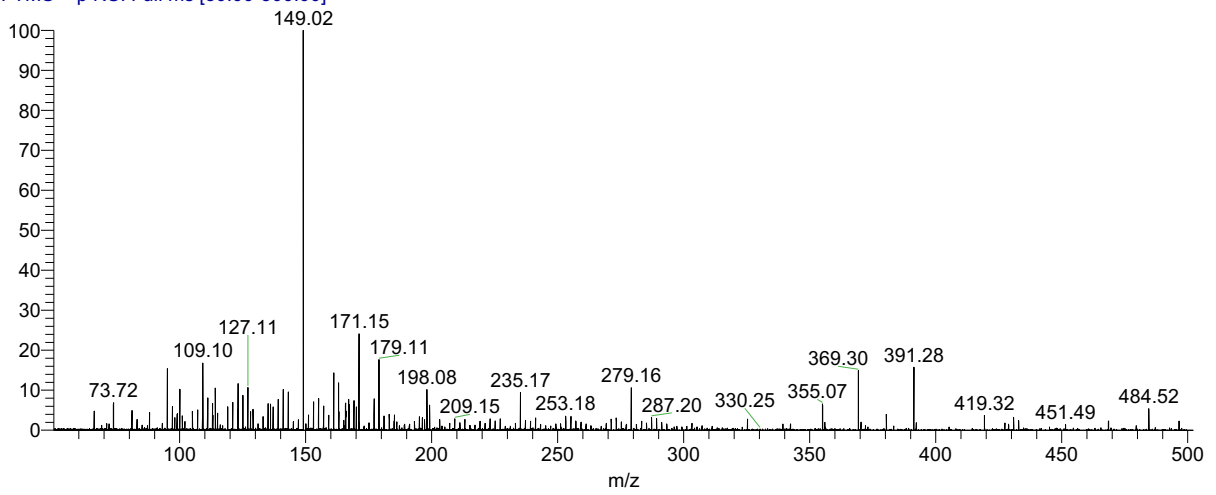
DART-MS Analysis:

Sample extracts were spotted (5 μ 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 $^{\circ}$ 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 Fairing

20161031_ORX_006 #18-41 RT: 0.2' AV: 24 NL: 2.79E5
T: FTMS + p NSI Full ms [50.00-500.00]



DART Figure 2. OSIRIS-REx Blank foil

20161031_ORX_003 #20-41 RT: 0.2' AV: 22 NL: 2.33E5
T: FTMS + p NSI Full ms [50.00-500.00]

