LABORATORY NOTEBOOK

WA 1-49: PCB Project//NASA Method Notebook

ARCADIS GERAGHTY & MILLER

Project No. _____
Book No. _____

E NASA Extraction Procedure

Page No. ____

Measure out desired amount of substrate (eg. paste, paint, caulk or concrete)

Add substrate to 20 mL scintillation vial.

Add 10 mL of 1:1 Ethanol: DI water

Add RCS (typically 100 mL)

Sonicate for 30 mins.

Decant solution into new vial. [Skip this step for any Blanks]

Add 10 mL hexane.

Sonicate 30 mins.

Remove hexane (top) layer for analysis.

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nessed & Understood by me,	Date	Invented by:	Date
		Recorded by:	

Project No. _____
Book No. _____ TITLE AMTS Paste

n Page No. ____

-14/10

AMTS Paste Formulation 7/14/2010 by Corey Mocka

Steps	Component	Desired		Measured	
		Weight (g)	Volume (mL)	Weight (g)	Volume (mL)
1	Magnesium	20		20.05	
2	Glycerin (glycerol)	20		20.00	
3	Glycerin (glycerol)	20		20.00	
4	Manually stir and mix well				
5	Ethanol		154.035		154.0
5	Limonene		17.145		17.1
5	Glacial acetic acid		3.55		3.5
6	Manually stir, mix well, then add stir bar				
7	Glycerin (glycerol)	20		20.01	
8	Carbomax PEG 8000	10		10.03	
9	Calcium Stearate	20		19.95	
10	Sodium Polyacrylate(5'100)	20		20.05	

Notes		
Start Stirring:	10:23	7/14/2010
End Stirring:	15:00	7/14/2010
Covered With Al foil and let sit over night		
Start Stirring:	8:30	7/15/2010
End Stirring:	13:00	7/15/2010
Left uncovered over night		
Manually stirred 3x 7/16/2010		
Left covered for the weekend		
Added ethanol and put in jar 7/19/2010		

Corey Mocka

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Recorded by: CM

TITLE NASA-ST-02 Paste Applications

Project No. _____
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Panel	pTRACK ID	Apply Date	Apply Time	Remove Date	Remove Time
2118	S09701	8/6/10	10:40	8/7/10	10:47
2117	S09702	8/6/10	10:42	8/9/10	10:38
2116	S09703	8/6/10	10:55	8/13/10	12:50
2115	S09704	8/6/10	10:58	8/13/10	13:02
2114	S09705	8/6/10	11:00	8/20/10	10:55
2					

EXTRACTIONS

Panel ID	Date of Extraction	Date	Extraction ID
S09701	8/9/10		L0970101
S09702	8/9/10		L0970201
S09703	8/17/10		L0970301
S09704	8/17/10		L0970401
L0970101 ~~L0970201~~	8/17/10		L0970101
L0970201	8/17/10	CM	L0970201

OMIT! These two extractions did not separate correctly CM 8/17/10

Panel	Amt of Paste for Extraction
S09701	0.5079 g
S09702	0.5059 g
S09703	0.4973 g
S09704	0.5423 g
S09705	

Extraction procedure
1. Measure ~0.50 g aliquot of paste
2. 5 mL EtOH, 5 mL H_2O
3. Sonicate 30 mins
4. Remove top layer to new vial
5. Add 10 mL Hexane
6. Sonicate 30 mins
7. Remove top layer for analysis

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Witnessed & Understood by me, | Date | Invented by: | Date 8/6/10
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Project No. _____
Book No. _____ TITLE NASA-ST-02 QC Samples

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8/13/10

Copy

NASA-ST-02 QC Samples

QC Wipe Samples				
Panel ID	Panel Description	Source	Panel Wipe Date	Wipe ID
S09706	NASA-ST-02-PCB-FB1	S09700	8/13/2010	L0970602

Lab & Field Blank QC Samples		
Sample ID	Description	Date
Q10081701	Field Blank Extract	8/13/2010
Q10081702	NASA Paste Lab Blank Extract	8/13/2010

S09706 → Field Blank panel - roller wipe method.

Q10081701 → Field Blank Extract

Same vial:

1. 5 mL EtOH, 5 mL H_2O
2. Sonicate 30 mins
3. Add 10 mL hexane
4. Sonicate 30 mins
5. remove top hexane layer for analysis

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LE NASA-ST-02 Narrative Project No. _____ Book No. _____

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arrative 7/14/10 - 8/13/10

/2010
S Paste made

010
e applied to 5 Q Panels that had Aroclor 1254 spiked primer (S09700)
ed with artists brush
to maintain uniform paste thickness for all panels
ed once with sealant. Touched up corners after a few mins
ls stored under plexiglass cube. No fan. No ventillation to prevent sovlvent evaporation

010
e removed from panel S09701 and stored in the fridge. Panel wrapped in Al Foil and put in desiccator
ed that top coat was not thick enough or dry enough to be peeled off. Went back and recoated remaining panels.
pears that the EtOH dissolves the paint very easily. Also, the paint is often peeled from the panel when removing the paste.

010
e removed from panel S09702 and stored in the fridge. Panel left out on lab bench so remaining paste would flake off.
0201 had coloration from top coat. Seemed more dry than the 24 hr sample.

ots of paste from L0970101 and L0970201 were extracted
oth extracts the hexane layer had trouble separating. Formed an emulsion. It took a long time to separate. Three layers.
/17/2010 after extracting three more samples, decided to discard the extracts from 8/9/10 and start over. (See Below on 8/17/2010)

/2010
e removed from panels S09703 and S09704. Stored in fridge.
e samples taken of panels S09701, S09702, S09703, S09704, S09706.

Corey M

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essed & Understood by me,	Date	Invented by:	Date
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Project No. _____
Book No. _____ TITLE NASA-ST-02 Narrative
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Narrative 8/17/10 – End of Test

CWW

17/2010
ıiuots of paste samples from S09703 and S09704 extracted.

: Samples Q10081701 and Q10081702 extracted
:10081702- 0.4916g aliquot of paste extracted.
:10081701- 5 mL EtOH and 5 mL water; sonicate 30 mins; add 10 mL hexane; sonicate 30 mins; remove top hexane layer for analysis.

e hexane layer for these samples separated much easier than the ones from 8/9/2010. Only two layers.
cided to extract aliquots of paste samples S09701 and S09702 again.

9/2010
ıan Vial test. Sample S09749. 200 uL of 08062010-167STD00 added to a 1.1275g sample of paste. Start 13:45

0/2010
noved paste from S09705. Stored in Fridge.
·acted aliquot of paste sample S09705.

1:05, added 5 mL Ethanol and 5 mL water to vial test sample S09749. Sonicated 30 mins. Added 10 mL hexane. Sonicated 30 mins.
noved hexane layer for analysis.

3/2010
e sample of S09705.

portant Notes

ıpears that the EtOH dissolves the paint very easily. Also, the paint is often peeled from the panel when removing the paste.
ght of paste sample is not necessarily the same for each sample. Some paste samples retained more solvent.
:xtract the entire paste sample, we would need to develop a modified extraction method. (More solvent needed & blow down most likely)
difficult to pipette off or decant all of the solvent from paste mixture during extraction. For quantitative analysis, it needs to be filtered.
not possible to recover all of the paste applied to a panel- some is left on the panel, some is left on the top coat.
wipe sampling, there is always paste residue left on the panel.

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nessed & Understood by me,	Date	Invented by: CWW	Date 8/23/10
		Recorded by:	

TITLE NASA-ST-02 Wipe Samples

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From Page No. 8/13/10

Source ID: Q118	S09701	Wipe Lab ID: L0970102
		Al Foil Lab ID: N/A
Start Apply IPA:	2:18:45	Sample Notes:
Start Wipe on Panel:	2:20:48	
Start Rolling:	2:21:10	
End Rolling:	2:22:10	
Number of Rolls:	35	
Remove Wipe:	2:22:48	RP wipe

Source ID: Q117	S09702	Wipe Lab ID: L0970202
		Al Foil Lab ID: N/A
Start Apply IPA:	2:10:17	Sample Notes:
Start Wipe on Panel:	2:12:17	
Start Rolling:	2:13:03	
End Rolling:	2:14:03	
Number of Rolls:	31	
Remove Wipe:	2:14:21	RP wipe

Source ID: Q116	S09703	Wipe Lab ID: L0970302
		Al Foil Lab ID: N/A
Start Apply IPA:	2:38:00	Sample Notes:
Start Wipe on Panel:	2:39:31	
Start Rolling:	2:39:52	
End Rolling:	2:40:52	
Number of Rolls:	37	
Remove Wipe:	2:41:15	RP wipe

Source ID: Q115	S09704	Wipe Lab ID: L0970402
		Al Foil Lab ID: N/A
Start Apply IPA:	2:44:51	Sample Notes:
Start Wipe on Panel:	2:46:54	
Start Rolling:	2:47:23	
End Rolling:	2:48:23	
Number of Rolls:	33	
Remove Wipe:	2:48:33	RP wipe

Source ID: Q113	S09706	Wipe Lab ID: L0970601
		Al Foil Lab ID: N/A
Start Apply IPA:	2:27:02	Sample Notes:
Start Wipe on Panel:	2:32:26	Tape on panel
Start Rolling:	2:32:45	QC Sample
End Rolling:	2:33:45	
Number of Rolls:	32	
Remove Wipe:	2:33:57	RP wipe

Source ID:	S09705	Wipe Lab ID: L0970502
		Al Foil Lab ID: N/A
Start Apply IPA:	7:51:40	Sample Notes:
Start Wipe on Panel:	7:52:40	
Start Rolling:	7:53:00	NASI
End Rolling:	7:54:00	14
Number of Rolls:	~~[illegible]~~ 33	day
Remove Wipe:	7:54:24	8/23/10

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Book No. _____ TITLE NASA-ST-02 Vial Test

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A-ST-02 Vial Test

D	Description	PCB Standard	Amt of STD	Start Date	Start Time	End Date	End Time	Elapsed Time, h	Extraction Date	Extraction ID
49	NASA-ST-02-PCB-VialTest-21 hr	08062010-167STD00	200 µL	8/19/2010	13:45	8/20/2010	11:05	21.3	8/20/2010	L0974901

08062010-167STD00 Congeners:
#101
#17
#52
#66
#154
#105
#118
#110
#77
#187

8/19/2010
Began Vial test. Sample S09749. 200 uL of 08062010-167STD00 added to a 1.1275g sample of paste. Start 13:45

8/20/2010
At 11:05, added 5 mL Ethanol and 5 mL water to vial test sample S09749. Sonicated 30 mins. Added 10 mL hexane. Sonicated 30 mins. Removed hexane layer for analysis.

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ssed & Understood by me, | Date | Invented by: | Date 8/20/10
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.E New Paste Recipe

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2/1/10

New Paste made according to recipe below.
The paste made for NASA-02 was done incorrectly. See correspondence between Xoaoyu & NASA.

CM

formulation for a single batch is as follows:

	Total Liquids (mL)	Ethanol (89%) (mL)	Acetic Acid 1% (mL)	Limonene (9%) (mL)	Calcium Stearate (g)	PEG (g)	Glycerol (g)	Sodium Polyacrylate (g)	Mg (g)
batch	175	155.75	1.75	15.75	20	10	40	20	20
batch	87.5	77.875	0.875	7.875	10	5	20	10	2000

is the order to add the ingredients for a 0.5 batch

1. Mix the Ethanol, Limonene, and acetic acid together.
2. Stir the 1% acidified ethanol solution
3. Add the calcium stearate
4. Add the PEG
5. Add the Sodium Polyacrylate
6. Add 10 grams (1/2) of the glycerol to the stirring mixture
7. Allow to stir/set for 12-24 hours
8. Mix the remaining 10 g of glycerol with the 10g of Mg, add to the now set paste mixture (should be the consistency of frosting)
9. Stir in the Mg until homogenized.

s 3-5 can actually be combined into a single step (all the powders can be mixed ther and added to the solution at once).

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nessed & Understood by me,	Date	Invented by: CM	Date 12/1/10
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12/2/10

		PTRACK
20101202-WASAPasteVT-24 hr	1.19 g Paste	513001
20101202-WASAPaste VT-14 day	1.25 g Paste	513002
20101202-NoPaste VT-1	∅ Paste	513004
20101202-WASAPASTEVT-14 days	1.21 g Paste	513003

To each vial 100 µL of 08062010-1675TD00 was added [5 ng/µL PCB Congener Std]

Start: 9:55 on 12/2/10

08062010-1675TD00
100 µL each
PCB Congener
5 ng/µL

9:55

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essed & Understood by me, | Date | Invented by: | Date 12/2/10
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TITLE Vial Tests

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12/3/10

~~Q 10120301~~ CM @ ~~8~~ 9:55

- Extracted S13001 & S13004. according to WASA Method.
- No RCS Added.
- Transferred to Andy for SIM/Scan analysis.

L1300101
L1300401

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12/7/10

Added 100 mL of RCS 11102010-04125TD00 After 10 mL of 1:1 EtOH:H_2O to S13002 & Q10120301.

- Extracted according to NASA method.

- Final Samples:

L1300201 (5 day)

Q10120301 (RES BLK)

~~Q10120801 SLVT BLK MeOH EtOH~~ (cm)

~~Q10120802~~

cm

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Recorded by: cm

TITLE Vial Tests Con't

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12/16/10

- Added 100 µL of RCS1102010-042-STD00 to vial S13003
- Extracted According to NASA method @ 13:32.

↳ L1300301

Q10121601

↳ SIVT B/K

- 10 mL 1:1 EtOH : H_2O
- Sonicate 30 mins
- Add 10 mL Hexane
- Sonicate 30 mins
- Remove hexane layer for analysis.

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rom Page No. ____ Test Plan: NASA Method Evaluation for Panel Preparation

Panel	Tare	Plus tape
NQ1	20.000	21.952
NQ2	19.993	21.832
NQ3	19.921	21.751

iar + lid + paint 503200-5 138.095
weight before second coating 136.915
leight before third coating 134.921

anel wet	1ST coating	2ND coating	3rd coating
NQ1	22.193	22.472	23.030
NQ2	22.081	22.477	22.776
NQ3	21.932	22.278	22.497

nel weight Dry NQ1 22.135
fore 2ND coating NQ2 22.027
NQ3 21.906

anel weight Dry NQ1 22.518
fore 3RD coating NQ2 22.347
NQ3 22.186

nel weight Dry NQ1 22.887
ter 3RD coating NQ2 22.654
NQ3 22.427

+ lid + paint 133.364

Remove tape, Panel + Paint (g)
NQ1 20.930
NQ2 20.802
NQ3 20.581

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nessed & Understood by me, | Date | Invented by: | Date 1/13/16
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TITLE Method Evaluation

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NASA Panel Preparation Method Evaluation
1/19/2011

Panel ID	Panel, g	Panel+Tape, g
NQ1	20.000	21.952
NQ2	19.993	21.832
NQ3	19.921	21.751

Panel ID	Panel+Tape+WetPaint Coat 1	Wet Paint Applied Coat 1	Panel+Tape+DryPaint Before Coat 2
NQ1	22.193	0.241	22.135
NQ2	22.081	0.249	22.027
NQ3	21.932	0.181	21.906

Panel ID	Panel+Tape+WetPaint Coat 2	Wet Paint Applied Coat 2	Panel+Tape+DryPaint Before Coat 3
NQ1	22.672	0.537	22.518
NQ2	22.477	0.450	22.347
NQ3	22.278	0.372	22.186

Panel ID	Panel+Tape+WetPaint Coat 3	Wet Paint Applied Coat 3	Dry Paint+Panel Final
NQ1	23.03	0.512	20.93
NQ2	22.776	0.429	20.802
NQ3	22.497	0.311	20.581

Panel ID	Dry Paint Final	Wet Paint Final, g
NQ1	0.930	1.290
NQ2	0.809	1.128
NQ3	0.660	0.864
Sum	2.399	3.282
Average	0.800	1.094
STDev	0.135	0.215
%RSD	16.91	19.65
	Average+STDEV	1.309
Estimated PCB Paint needed for 7 Panels		9.163

Jar+Lid+Paint Start, g	Jar+Lid+Paint End, g	Paint Consumed, g
138.095	133.364	4.731
Estimated PCB Paint needed for 7 panels		11.039

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		Recorded by:	1/19/11