

## รายการอ้างอิง

### ภาษาไทย

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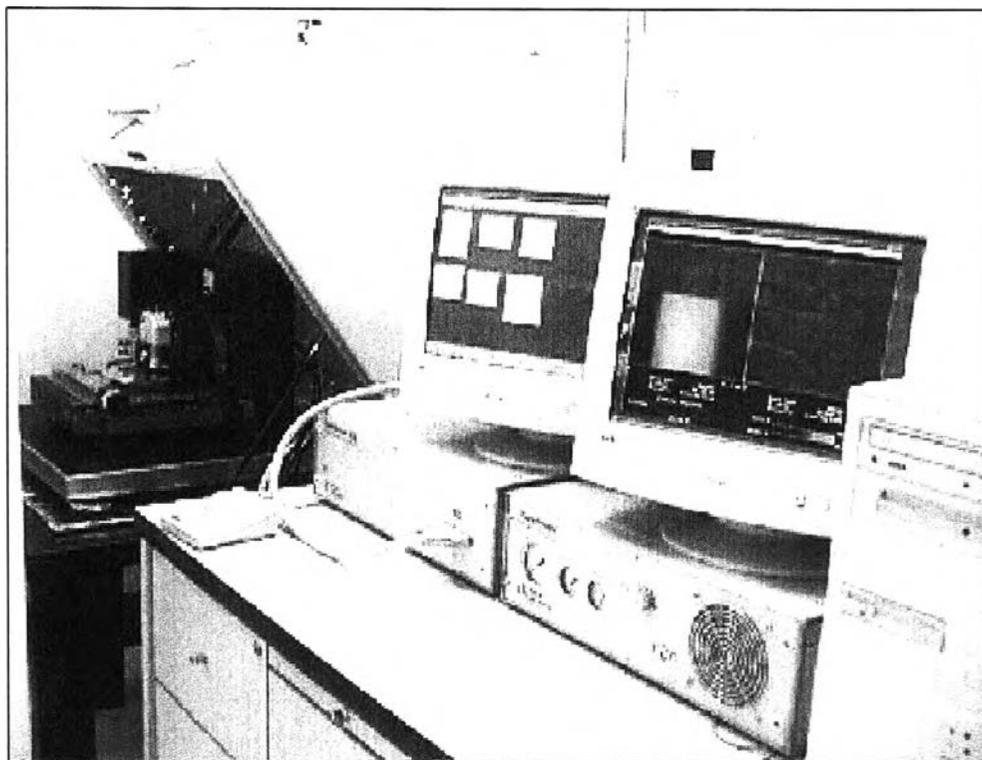


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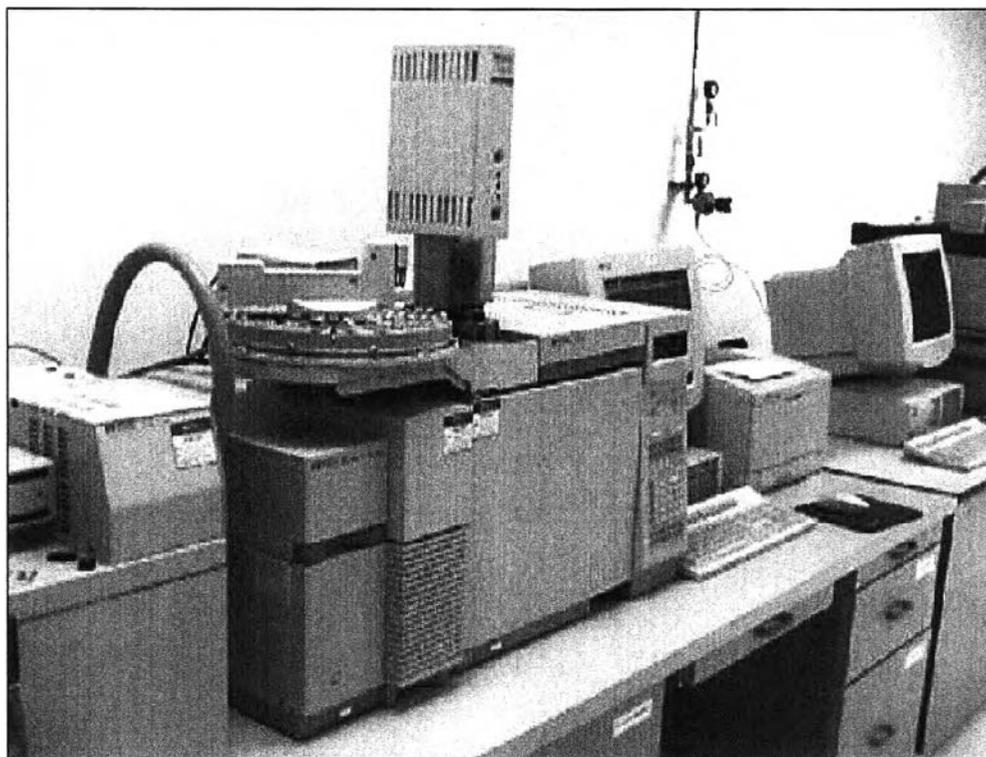
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และชุดประกอบหัวอ่าน - เขียน

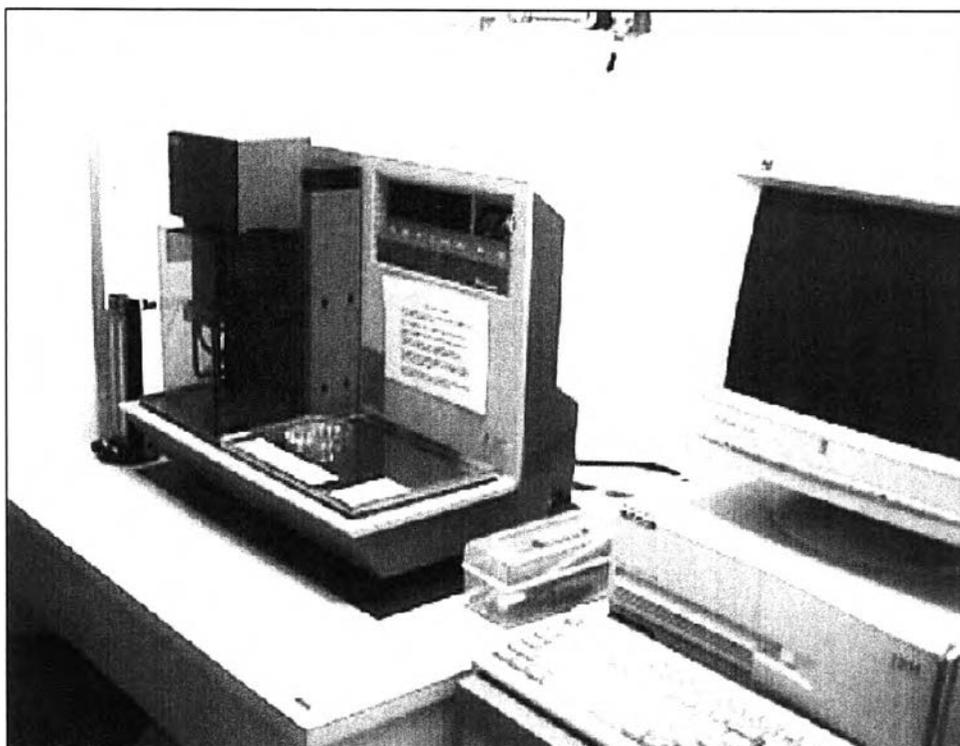
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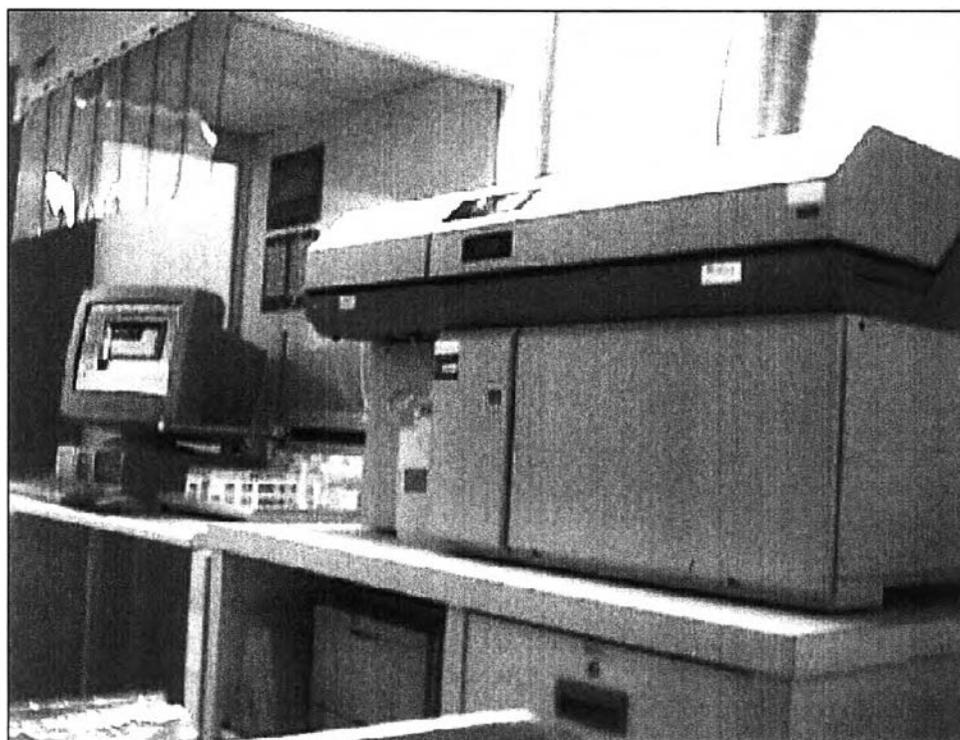
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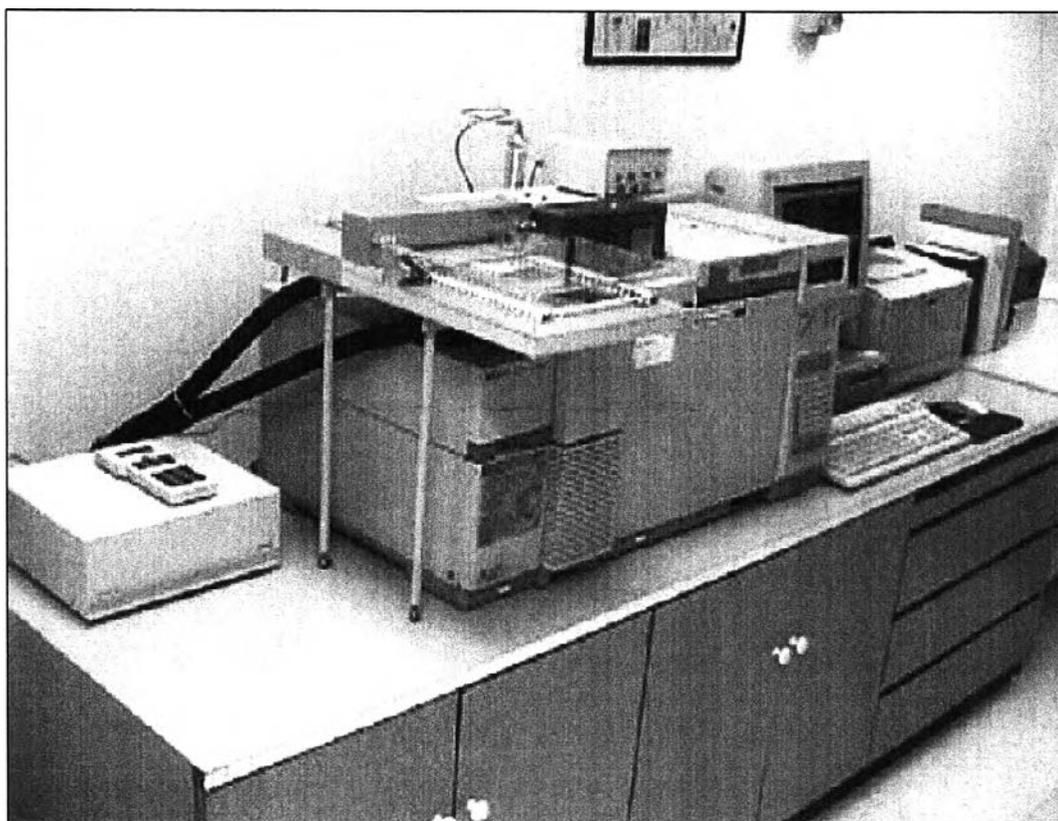
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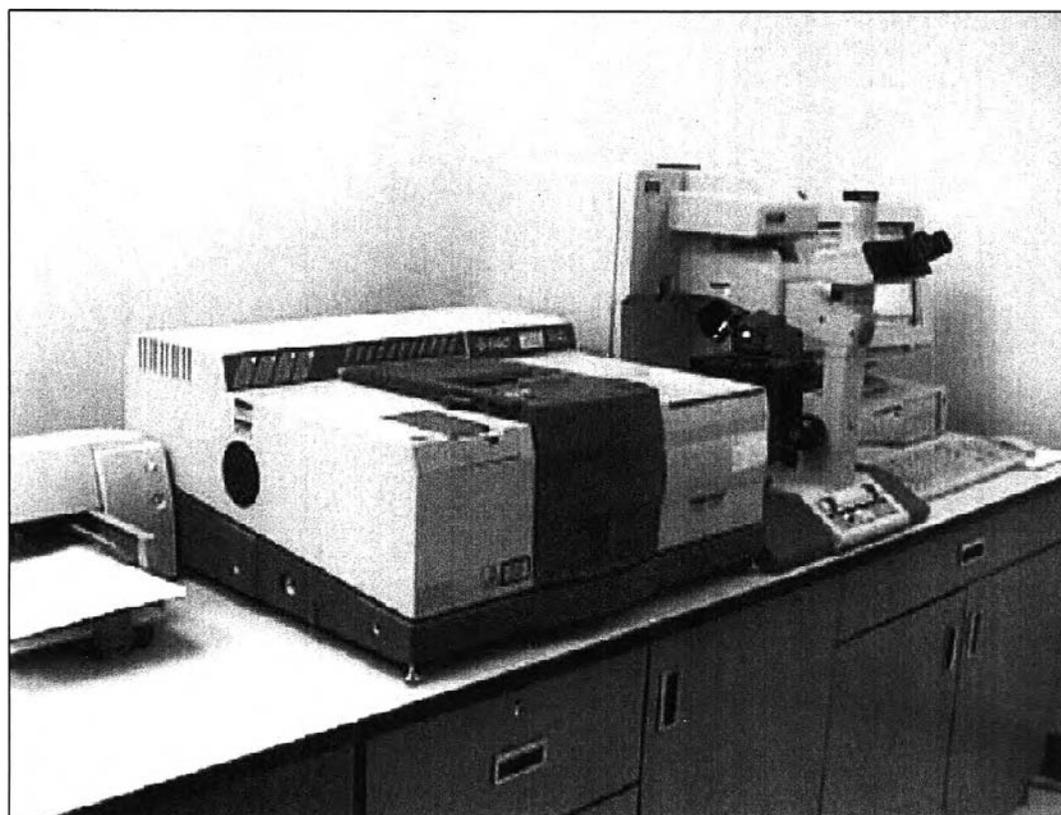
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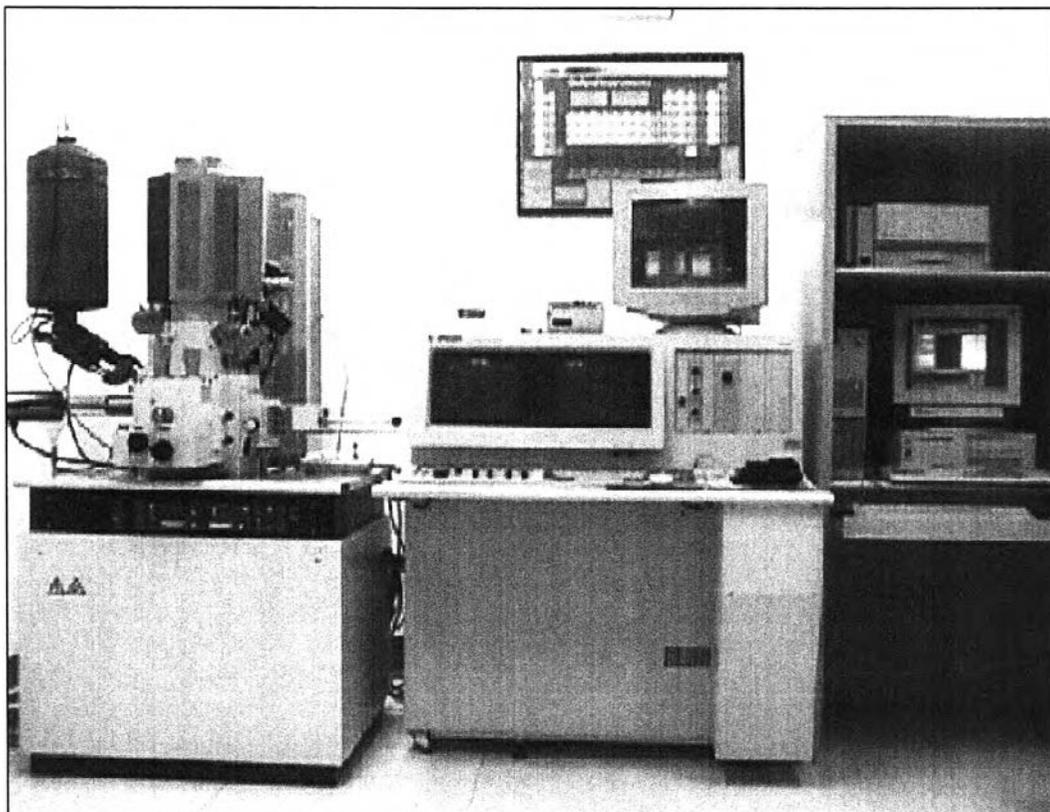
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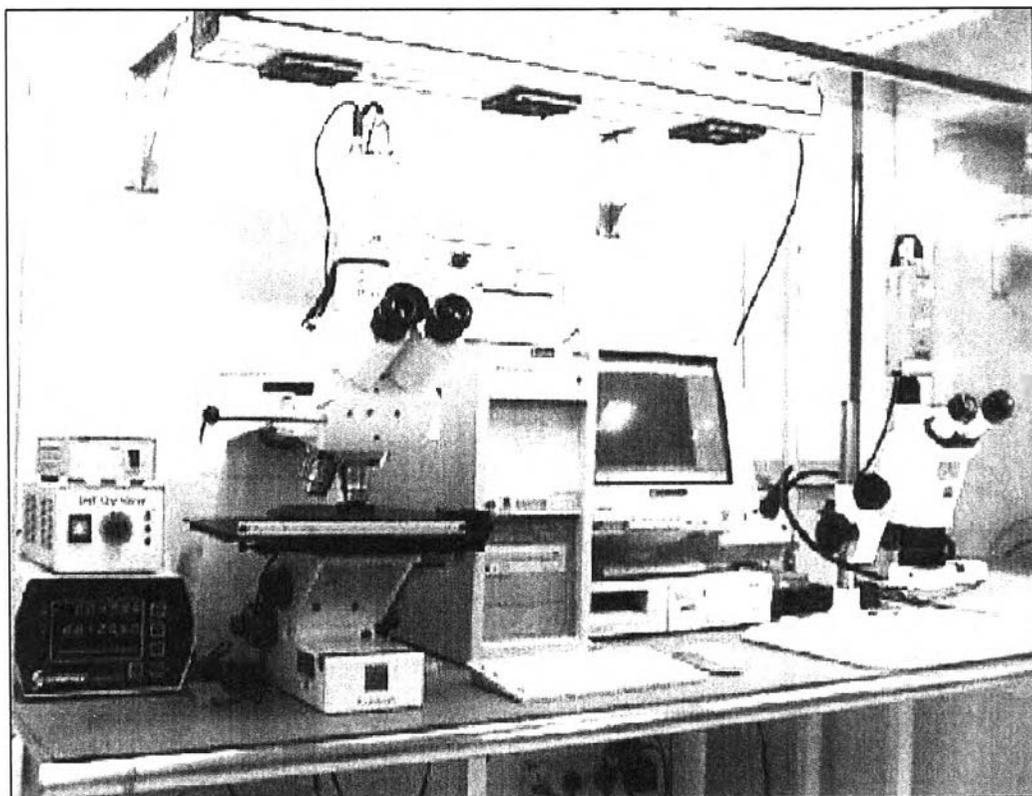
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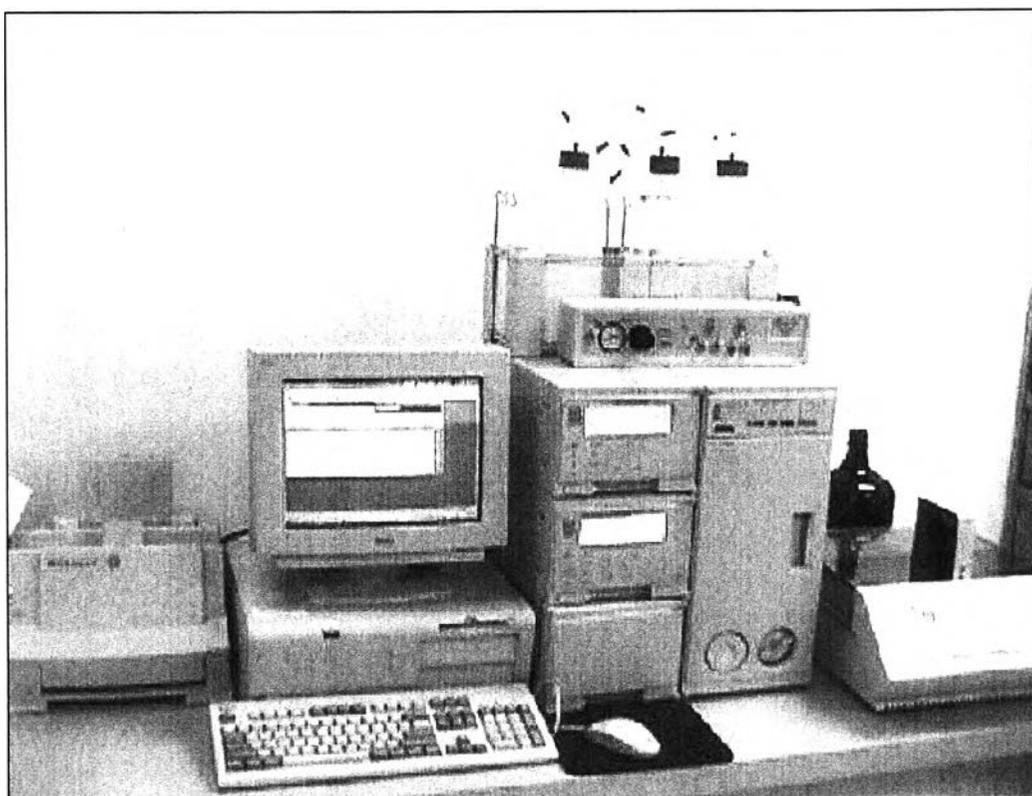
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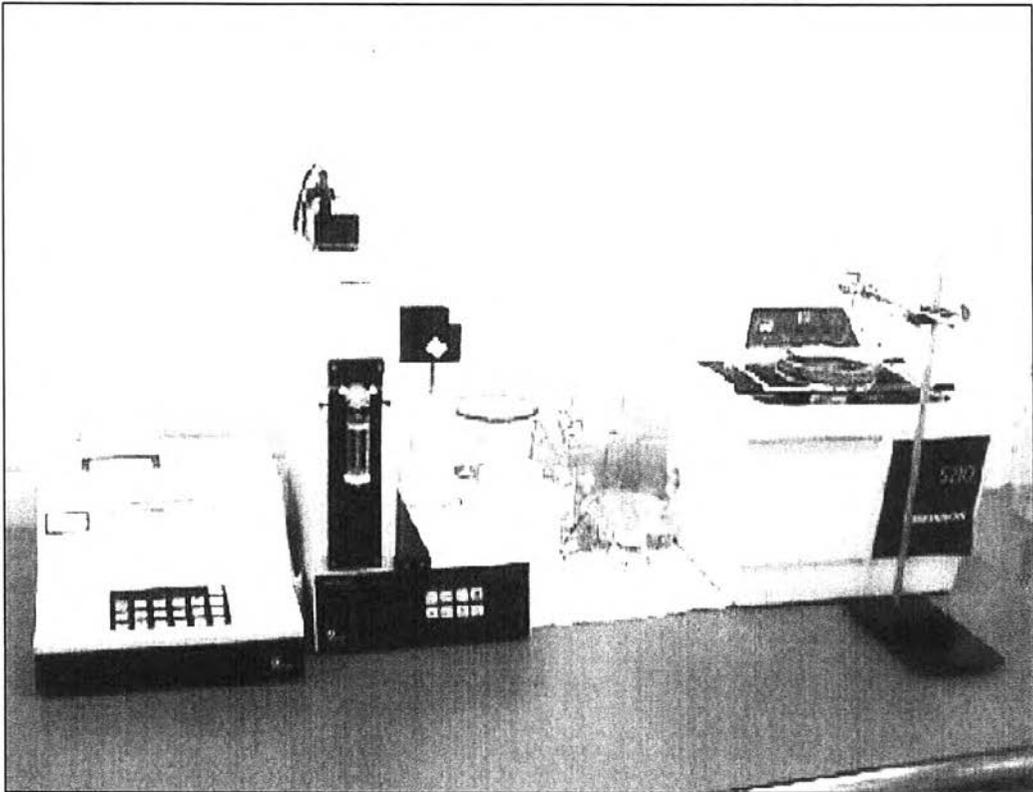
ภาคผนวก ก. 8 Optical Microscope



ภาคผนวก ก. 9 Ion Chromatography ( IC )



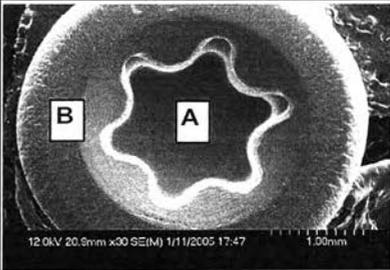
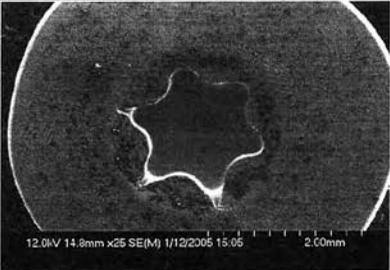
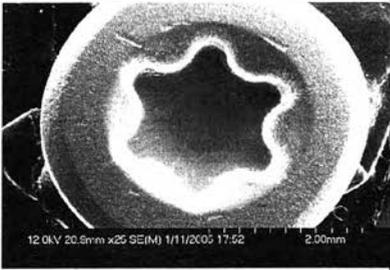
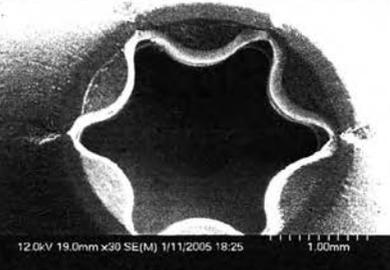
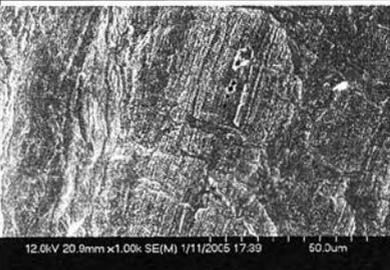
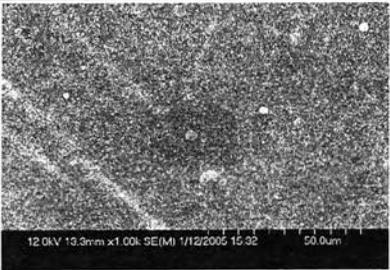
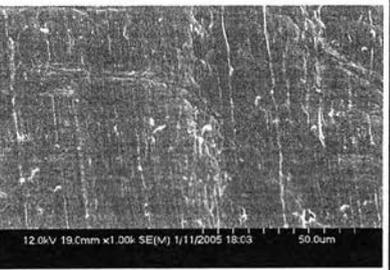
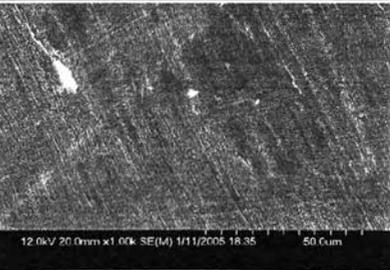
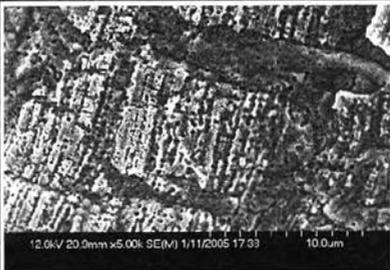
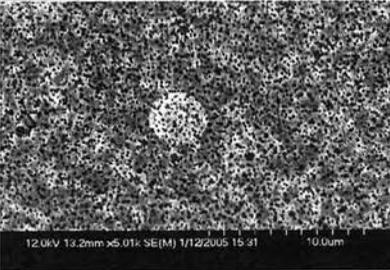
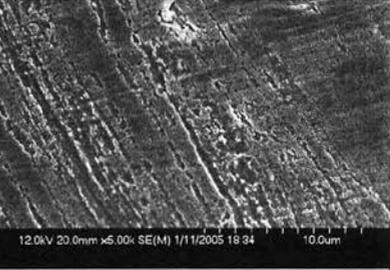
ภาคผนวก ก. Liquid Particle Counter ( LPC )



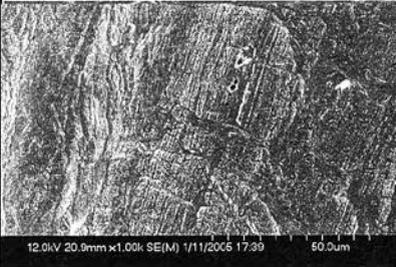
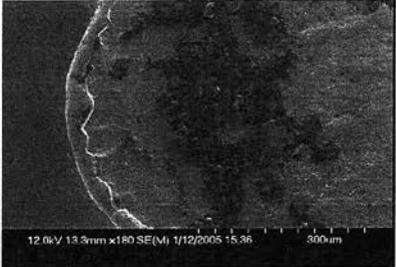
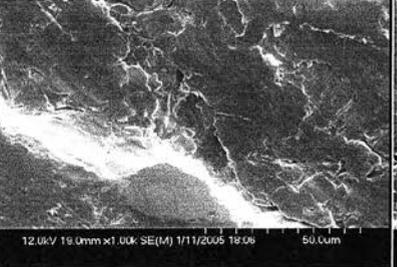
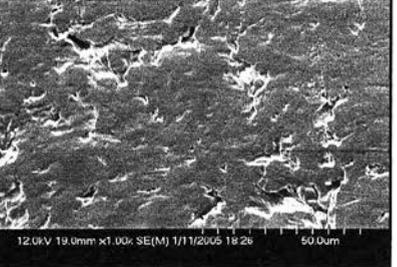
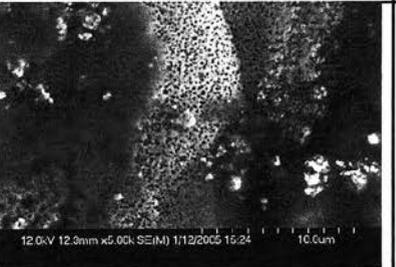
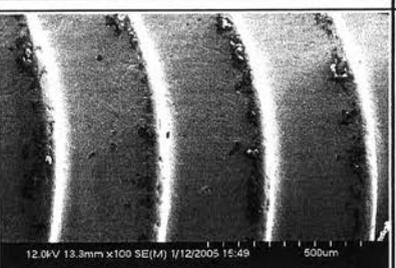
ภาคผนวก ข.

ตัวอย่างการหาค่า LPC และถ่ายรูปวิเคราะห์ด้วยเทคนิค SEM  
บนชิ้นส่วน Screw

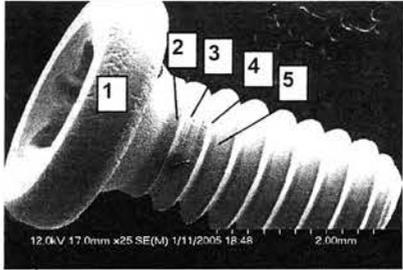
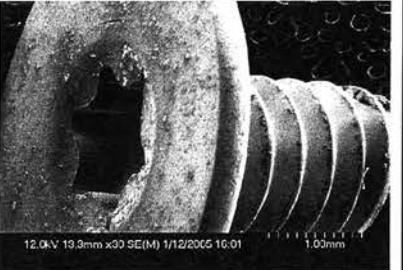
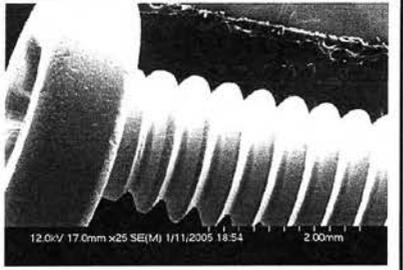
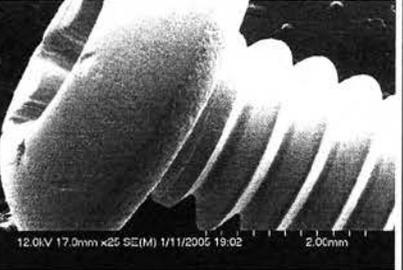
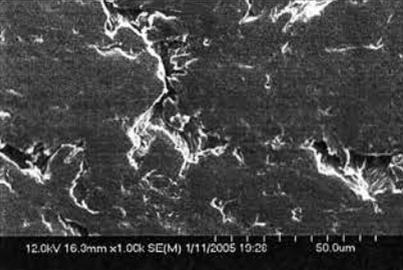
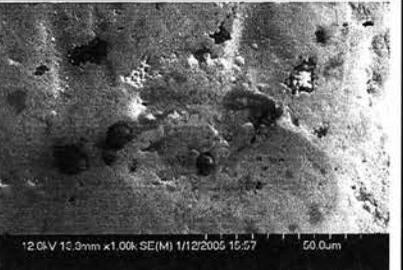
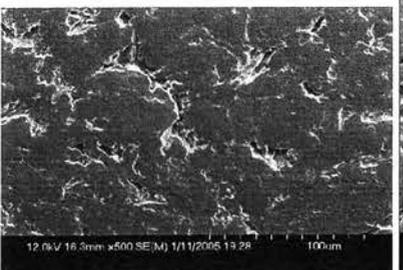
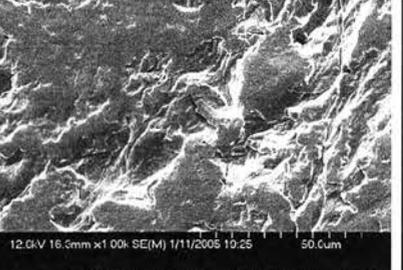
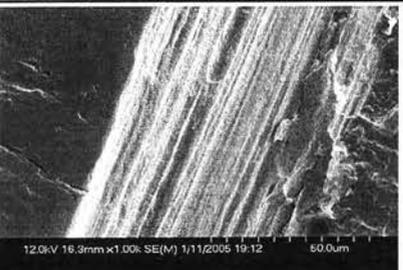
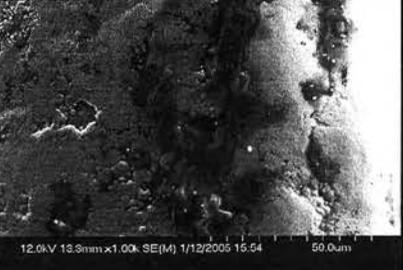
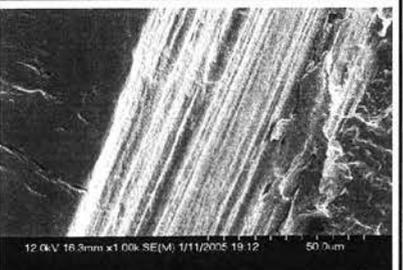
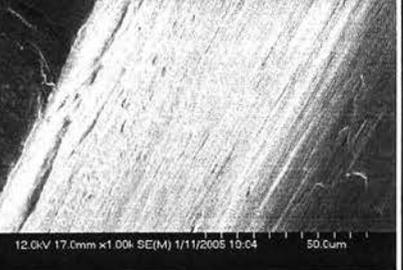
ภาคผนวก ข. 1 การวิเคราะห์หาค่า LPC และถ่ายรูปด้วยเทคนิค SEM บน Screw ( ต่อ)

ชนิดของ Screw	Scorpio 1233 040	Scorpio 1247 (EN-PTFE)	3.5" Clamp Screw 1233 000	3.5" Top cover Screw 4020
ค่า LPC ( อนุภาค / ตารางเซนติเมตร@0.5um)	280,521	474,519	335,229	58,787
Screw Head (from top)				
<b>A</b> Screw Cavity (1000 x)				
<b>A</b> Screw Cavity (5000 x)				

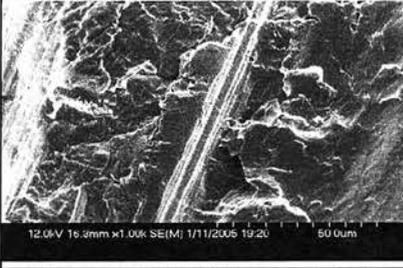
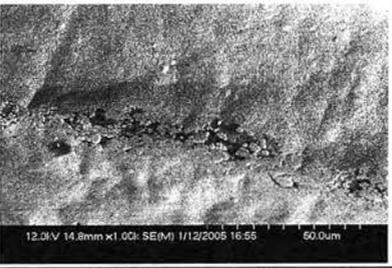
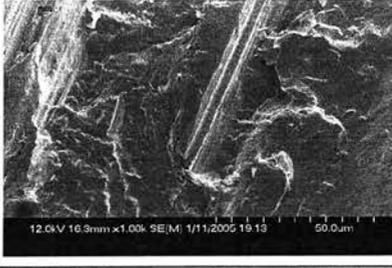
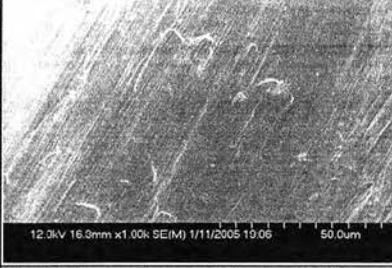
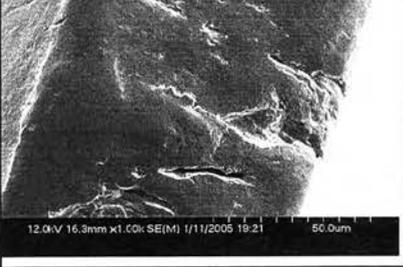
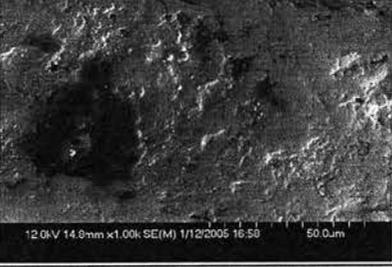
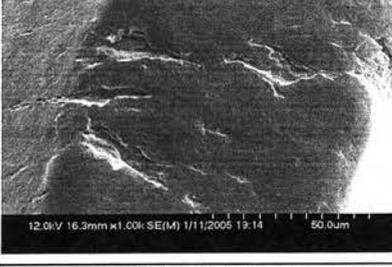
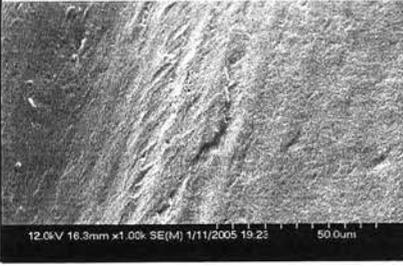
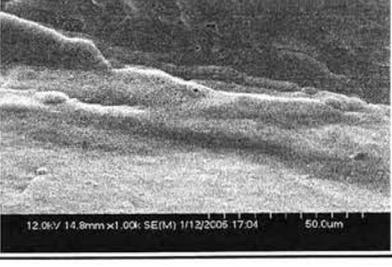
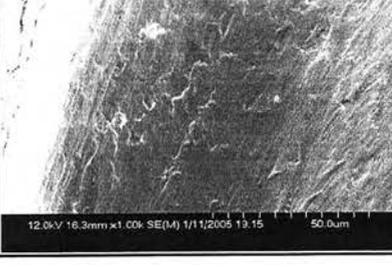
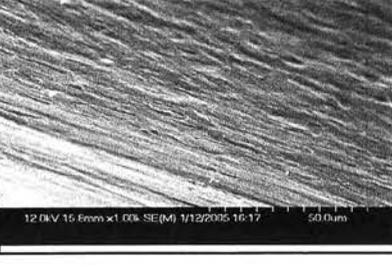
ภาคผนวก ข. 1 การวิเคราะห์หาค่า LPC และถ่ายรูปด้วยเทคนิค SEM บน Screw ( ต่อ)

ชนิดของ Screw	Scorpio 1233 040	Scorpio 1247 (EN-PTFE)	3.5" Clamp Screw 1233 000	3.5" Top cover Screw 4020
ค่า LPC ( อนุภาค / ตาราง เซนติเมตร@0.5um)	280,521	474,519	335,229	58,787
<b>B</b> 1000x				
Additional Photos on 1247 Scorpio Screw: Ni-P & PTFE particulating on the top surface (5000x)				
Additional Photos on 1247 Scorpio Screw: Shadings of PTFE & Ni-P on screw treads (100x)				

ภาคผนวก ข. 1 การวิเคราะห์หาค่า LPC และถ่ายรูปด้วยเทคนิค SEM บน Screw

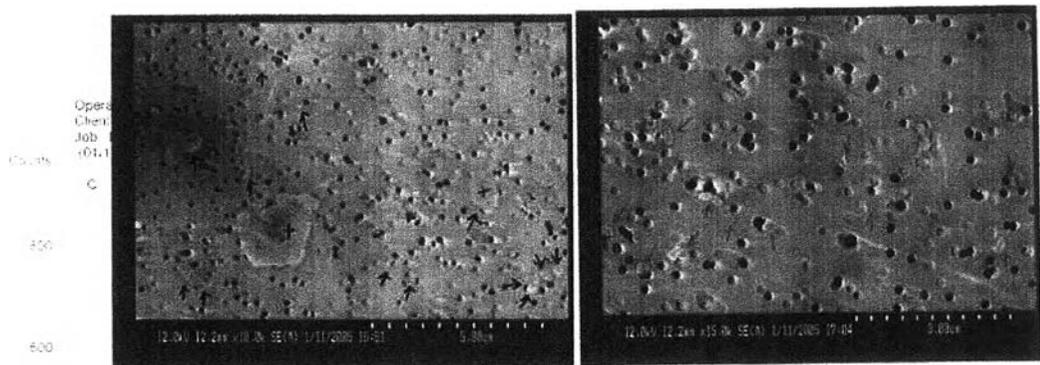
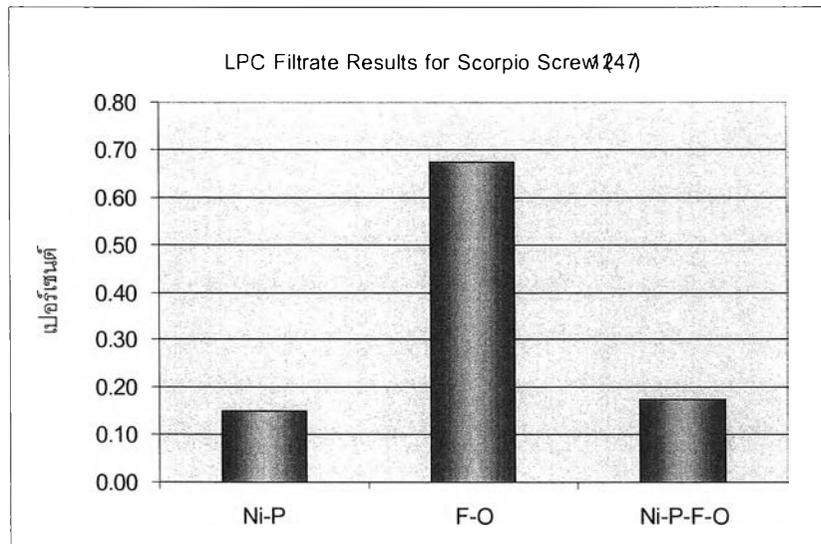
ชนิดของ Screw	Scorpio 1233 040	Scorpio 1247 (EN-PTFE)	3.5" Clamp Screw 1233 000	3.5" Top cover Screw 4020
ค่า LPC ( อนุภาค / ตาราง เซนติเมตร@0.5um)	280,521	474,519	335,229	58,787
Side view @ 30x				
1 1000x				
2 1000X				

ภาคผนวก ข. 1 การวิเคราะห์หาค่า LPC และถ่ายรูปด้วยเทคนิค SEM บน Screw ( ต่อ)

ชนิดของ Screw	Scorpio 1233 040	Scorpio 1247 (EN-PTFE)	3.5" Clamp Screw 1233 000	3.5" Top cover Screw 4020
ค่า LPC ( อนุภาค / ตาราง เซนติเมตร@0.5um)	280,521	474,519	335,229	58,787
<b>3</b> 1000x				
<b>4</b> 1000x				
<b>5</b> 1000x				

ภาคผนวก ข. 2 การวิเคราะห์หาส่วนประกอบของอนุภาคหลังจากหาค่า LPC ของ Screw ( 1247 ) สำหรับผลิตภัณฑ์ Scorpio

อนุภาค	จำนวน	เปอร์เซ็นต์
Ni-P	6	0.15
F-O	27	0.68
Ni-P-F-O	7	0.18



ภาคผนวก ค

COMPONENT CLEANLINESS TEST PROCEDURE

## PARTICLE MEASUREMENT BY LIQUID PARTICLE COUNT TECHNIQUE

### 1. PURPOSE

This document describes the procedure for the measurement of particle counts of HDA components by ultrasonic extraction and Liquid Particle Count (LPC).

### 2. SCOPE

This procedure applies to all Liquid Particle Count analyses performed at factory case study and supplier laboratories. Factory case study Analytical Laboratories reserve the right to revise this document as required.

### 3. EQUIPMENT NEEDED

Factory case study approved Ultrasonic tank that meets the frequency and power requirements in Appendix 1

Factory case study approved Liquid Particle Counter to meet the requirements in Appendix 1

2000 mL, 1000mL or 500mL Kimax glass beakers as required in Appendix 1

500 ml high-density wide mouth Nalgene polyethylene bottle (if beaker does not fit under sampler)

Class 100 Laminar Flow Hood or WD approved environment

Analytical stand with base and rod (See photo 1)

8" clamp with 3 fingers (See photo 1)

6" piece stainless steel rod, 1/8"diameter, last inch curled (See photo 1)

3" long red solderable polyurethane nylon (SPN) motor stator wire size #28 gauge, curled in circle loop (See photo 1) or WD approved equivalent.

Fixtures for suspending multiple parts, as required

Plastic tweezers (non-metal)

DI water, 18 MOhm, filtered to at least 0.1 um if sensor range is <0.5, at least 0.2 um if sensor range is 0.5 and above; Flow rate: 1GPM

Beaker fixture for the ultrasonic tank

IPA ChromAR Grade or WDSJ approved

#### 4. COMPONENTS FOR MEASUREMENTS

For components not cleaned at factory case study, the components will be submitted for testing in their original shipping containers to minimize handling contamination. Components after cleaning at factory case study must be submitted for testing in approved Clean Room bags. The components must be doubled bagged in a Class 100 Clean Room environment. There is no minimum surface requirement for the LPC testing. Parts that are handled incorrectly or have been exposed will not be tested.

#### 5. TEST SET-UP

5.1 Perform any required steps for the respective LPC that is necessary for that LPC to run properly. For instance, the sampler must be turned on before the counter and wait one half hour before use in order to warm up the electronics. Dead volume within the sampler must be taken into account, and the sampling must be done to ensure that no carryover from the previous sample is counted in the next sample. This may be done by running 2 "clean" cycles before counting for each sample.

5.2 The LPC sensor must be calibrated by an authorized vendor on an annual or semiannual frequency. For internal quality control, the performance of the sensor should be assessed by using a particle count standard and particle size standard.

5.3 Fill the ultrasonic tank with water to the operating level. This level must be maintained, and the volume of water in the tank at this level should be on record and degas the water for 1 minute before use.

- 5.4 Place the tank fixture for the beaker into the ultrasonic tank so that the beaker is in the same position in the tank during every sampling. The fixture should be made to allow different sizes of beakers to be used (see appendix). The beaker should not touch the bottom of the tank.
- 5.5 New glass beakers will need to be cleaned as normal glassware, then fill the beaker with DI water to the rim and ultrasonic for 10 minutes. Repeat this step again as needed
- 5.6 Set the following bin sizes at the counter: For sensor range of 0.2-2.0, use bin sizes of 0.2, 0.3, 0.5, 0.7, 1.0, and 2.0  $\mu\text{m}$ . For sensor range of 0.5-5.0, use bin sizes of 0.5, 0.7, 1.0, 2.0 and 5.0  $\mu\text{m}$ . Please refer to Appendix 1 for the required sensor range.

## 6. PROCEDURE

### 6.1 Blanks

- 6.1.1 A blank must be performed before each sample is analyzed. Rinse the beaker with clean DI water. Fill the beaker with the appropriate amount of fresh DI water for the blank. Avoid touching the interior of the beaker with gloves when rinsing and testing.
- 6.1.2 Place the beaker in the fixture of the ultrasonic tank. Lower the stator wire or multi-part fixture into the water, insuring the stainless steel hook does not touch the water. The wire should be submerged at a level that will comply to requirement in section 6.2.5 when analyzing the sample. Sonicate for 2 minutes.
- 6.1.3 When the ultrasonic extraction is complete, raise the stator wire out of the water. Place the beaker on the sample platform. If the beaker will not fit, decant 500 mL of the sample fluid into a clean, blanked polyethylene bottle. Set the LPC sampling probe to the middle of the water level. Set the LPC to sample 50 mL

- 6.1.4 Sample with the LPC until the particle counts are at the normal blank range. A normal blank range will depend on factors such as room cleanliness and the quality of DI water. The normal blank range for the LPC is less than 100 particles per 50 mL at 0.5  $\mu\text{m}$  (differential). An acceptable blank range will be less than 10% of the Liquid Particle Count of the component being tested but not to exceed 1000 particle counts per 50 mL at 0.5 $\mu\text{m}$  differential range.
- 6.1.5 If the particle counts do not end up at the normal blank range repeat steps 6.1.1 through 6.1.4. This may need to be performed many times before the blank can be used. Once the blank is in the normal range, record 2 readings and calculate an average of the 2 readings.
- 6.1.6 If an acceptable blank cannot be achieved within 4 hours, the following must be checked: stator wire, the beaker, the filters on DI water source, and the HEPA filters of the laminar flow hood.

## 6.2 Test Method

- 6.2.1 When the blank is complete, add clean DI water to the beaker to the mark on the beaker. Place the beaker back in the fixture of the ultrasonic tank.
- 6.2.2 With clean tweezers, remove the component that will be sampled from its packaging. Place the component on the stator wire hook.
- 6.2.3 Light and plastic components will tend to float in the water. To insure that the component does not float off the stator wire, the stator wire can be squeeze together around the part with a pair of clean tweezers. Only squeeze enough to hold the part in place, any additional pressure will generate more particles.

- 6.2.4 Place the component into the beaker so that the component is completely immersed in the water without it touching the sides and the bottom of the beaker. The component should be in the center of the beaker.
- 6.2.5 For small components, the stator wire will hang half of the way in the water of the beaker. Large components, like bases and top covers, will need to be adjusted so that the component does not touch the sides and bottom of the beaker. The stainless steel rod should never be immersed in the sample water.
- 6.2.6 Sonicate for 2 minutes. After 2 minutes, raise the component out of the water. Place the beaker on the sample platform. If the beaker will not fit, decant 500 mL of the sample fluid into a clean polyethylene bottle.
- 6.2.7 Record all particle counts from the LPC. Sample 2 times with the LPC and record an average. Ensure the variation of the 2 runs at every bin size are within 10%, otherwise, resample the extraction.
- 6.2.8 Repeat the blank, step 6.1.1 through 6.1.4. The LPC must be rinsed and blanked between each component so that the sensor is cleaned back to the original blank range.

### 6.3 Calculation of Particle Counts

6.3.1 Calculations use differential particle counts.

6.3.2 Total Particles per Component

$$\text{Particles per part} = \frac{(\text{Sample counts} - \text{blank counts})(\text{total water used in mL})}{(50)(\# \text{ parts used})}$$

to determine the total particle count in the entire part since the counted volume is 50 mls.

### 6.3.3 Particle Counts per Surface Area

$$\text{Particle counts/cm}^2 = \frac{\text{Particles per part}}{\text{Surface area in cm}^2}$$

## 7. TEST SHUT-DOWN

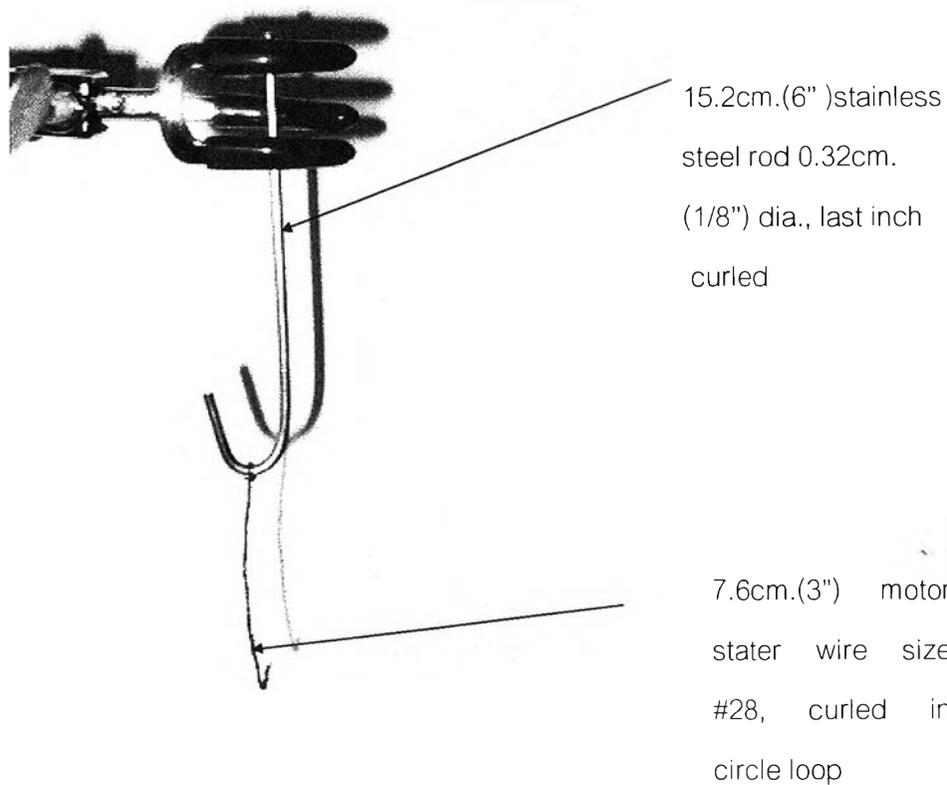
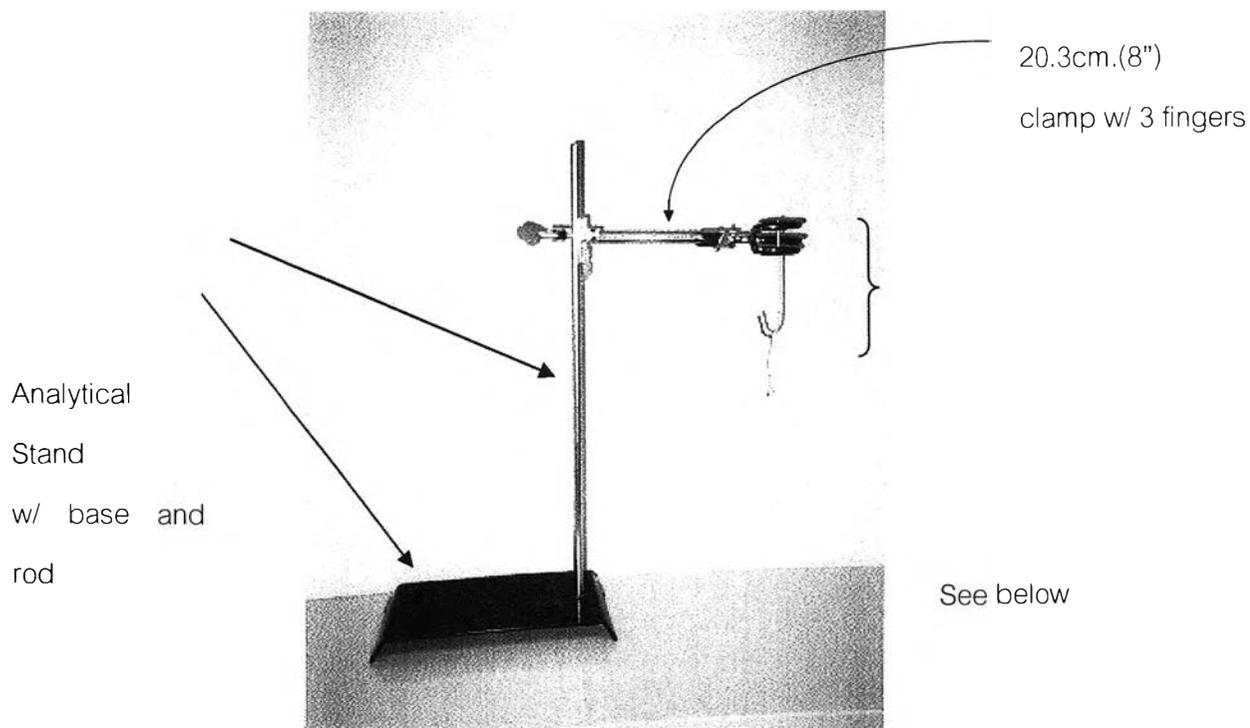
- 7.1 When testing is complete, sample clean DI water through the sensor of the LPC until the particle counts are in the original clean range for the LPC. Never leave the LPC sensor dirty. Allow the LPC to completely drain all remaining water from the system. Before turning off the LPC, sample IPA through the unit and collect waste. The LPC may then be turned off.
- 7.2 Whenever the beaker is not in use, it must be filled with clean DI water and covered with a watch glass until the next testing period.

## ภาคผนวก ค. 1 Appendix 1

Components	Sensor Range um	Ultrasonic Frequency kHz	Ultrasonic Power Watts/liter	Parts per Extraction	Volume of Water per Test mL
HSA	0.5-5.0	68/72	4.8	1	2000
HGA	0.2-2.0	68/72	4.8	5	500
Slider	0.2-2.0	68/72	4.8	20	500
Suspension	0.2-2.0	68/72	4.8	5	500
Suspension Base Plate	0.2-2.0	68/72	4.8	5	500
Suspension Load Beam	0.2-2.0	68/72	4.8	5	500
Suspension TSA	0.2-2.0	68/72	4.8	5	500
APFA	0.5-5.0	68/72	4.8	1	2000
AFA	0.5-5.0	68/72	4.8	1	2000
ACA	0.5-5.0	68/72	4.8	1	2000
Arm Block	0.5-5.0	68/72	4.8	1	2000
Overmold	0.5-5.0	68/72	4.8	1	2000
Coil	0.5-5.0	68/72	4.8	1	2000
Tang	0.5-5.0	68/72	4.8	20	500
FCBA	0.5-5.0	68/72	4.8	1	2000
Flex Circuit	0.5-5.0	68/72	4.8	1	2000
Flex Clip	0.5-5.0	68/72	4.8	5	500
Connector	0.5-5.0	68/72	4.8	1	2000
PreAmp	0.5-5.0	68/72	4.8	1	2000
Flip-Chip on Flex	0.5-5.0	68/72	4.8	1	2000
Flex Bracket	0.5-5.0	68/72	4.8	1	2000
Flex Circuit Gasket	0.5-5.0	68/72	4.8	1	2000
Shipping Comb	0.5-5.0	68/72	4.8	1	2000

Pivot	0.5-5.0	68/72	4.8	1	2000
Retaining Ring	0.5-5.0	68/72	4.8	1	2000
Media	0.2-2.0	40-72	4.8-20	3	1000
Base	0.5-5.0	68/72	4.8	1	2000
Cover	0.5-5.0	68/72	4.8	1	2000
Clamp	0.5-5.0	68/72	4.8	1	500
Spacer	0.5-5.0	68/72	4.8	1	500
Screw	0.5-5.0	68/72	4.8	5	2000
VCM	0.5-5.0	68/72	4.8	1	500
Latch	0.5-5.0	68/72	4.8	1	500
Motor	0.5-5.0	68/72	4.8	1	2000
Filter	0.5-5.0	40-72	4.8	1	2000
Shroud	0.5-5.0	68/72	4.8	1	500
Suppressor Comb	0.5-5.0	68/72	4.8	1	2000
Suppressor Arm	0.5-5.0	68/72	4.8	1	500

ภาคผนวก ค. 2 อุปกรณ์ในการวัดค่า LPC



**Addendum:**General component handling during LPC testing

- Component should be centered in the beaker without touching any part of the glass sides
- Add water to ultrasonic tank if water is not at the operating level
- Gloves should not come in contact with component during handling and testing

Media and Head Stack Assemblies are not cleaned prior to testing and are tested "As Received"**Media:**

Since media has low particle counts, it will be unacceptable to have blanks over 100 particle per 50 mL at the 0.5 um differential.

- Carefully raise disk with media tool and place securely on stator wire multipart fixture
- Gently put the lid on the cassette to cover remaining media but do not snap closed
- Snap the cassette shut only when testing is completed for the shift

**HSA:**

- Pivots must be removed before submission for LPC testing.
- Remove comb, shunting bar and flex gasket prior to testing
  - Use clean tweezers to remove flex gasket while holding the comb in place
  - Then rotate comb and slip out of place while holding the HSA with clean tweezers

**Motors, Pivots:**

- Epoxy or other adhesive can be used to seal the shaft end and electric contact end. Ensure the epoxy is cured properly and the contribution of the cured adhesive to the sample count is minimal (<10%)

**Screws:**

- Using polymer tweezers, place 5 screws, one at a time, into the beaker per extraction
- Since stator wire is not required, blanks and sample LPC tests should be performed without the stator wire apparatus

Bases, covers, spacers, and clamps and other components:

- Adjust stator wire with polymer tweezers in circle loop so component can be securely suspended on wire
- Wrap stator wire around stainless steel rod at least two times when testing heavy components like bases and top covers
- Components that are not cleaned must be submitted for testing in original "as received" packaging

After aqueous cleaning, the components must be

- Handled w/ gloves only when packaged
- Individually packaged in a Cleanroom bag
- Heat sealed for protection
- Double bagged in a larger polyethylene bag to enclose individually packed components

For smaller components, one nylon bag can be used to package several components.

For LPC testing aqueous cleaned components

- Tweezers should not be used
- Slide component out of bag
- Place securely on stator wire
- Glove should not come in contact with cleaned parts

**LPC Statistical Calculations:**

The surface area of the component can be found on the Mechanical Engineering Drawings.

After formatting the raw data into particles/cm<sup>2</sup>, the following should be calculated:

$$a. \text{ Average} = \bar{y} = \sum y/n$$

where

n= number of samples

y= observation

b. **Standard deviation (Sample) =**

$$s = \left[ \sum (y - \bar{y})^2 / (n - 1) \right]^{1/2}$$

where,

n = number of samples

y = observation

y bar = average

ภาคผนวก ค. 3 Required LPC Data format when submitting to service laboratory

(For all components that required sensor sensitivity of 0.5  $\mu\text{m}$ )

Component		Vendor		Date	
Surface Area ( $\text{cm}^2$ )		Ref. Doc of Surface Area		PROGRAM	
Extraction Vol. (mL)		Sample Vol. (mL)	50	Operator	
Number of components/ extraction		Volume of water in tank			
Sonication frequency (kHz)		Ultrasonic power (W/L)			
Particle Size ( $\mu\text{m}$ )					
Specification					
Comments	Passed/ Failed				
	( $\mu\text{m}$ )	Blank	Sample	Counts/part	counts/ $\text{cm}^2$
Sample 1	0.5				
	1.0				
	2.0				
	5.0				
Sample 2	0.5				
	1.0				
	2.0				
	5.0				
Sample 3	0.5				
	1.0				
	2.0				
	5.0				

Sample 4	0.5				
	1.0				
	2.0				
	5.0				
Sample 5	0.5				
	1.0				
	2.0				
	5.0				
Average	0.5				
	1.0				
	2.0				
	5.0				
Std. Dev.	0.5				
	1.0				
	2.0				
	5.0				

ภาคผนวก ค. 4 Required LPC Data format when submitting to service laboratory

(For all components that required sensor sensitivity of 0.2  $\mu\text{m}$ )

Component		Vendor		Date	
Surface Area ( $\text{cm}^2$ )		Ref. Doc of Surface Area		Program	
Extraction Vol. (mL)		Sample Vol. (mL)	50	Operator	
Particle Size ( $\mu\text{m}$ )					
Specification					
Comments	Passed/ Failed				
	( $\mu\text{m}$ )	Blank	Sample	Counts/part	counts/ $\text{cm}^2$
Sample 1	0.2				
	0.5				
	1.0				
	2.0				
Sample 2	0.2				
	0.5				
	1.0				
	2.0				
Sample 3	0.2				
	0.5				
	1.0				
	2.0				
Sample 4	0.2				
	0.5				
	1.0				
	2.0				

Sample 5	0.2				
	0.5				
	1.0				
	2.0				
Average	0.2				
	0.5				
	1.0				
	2.0				
Std. Dev.	0.2				
	0.5				
	1.0				
	2.0				

## MEASUREMENT OF IONIC CONTAMINATION / IONIC CHROMATOGRAPHY

### 1. PURPOSE

This document outlines the methods for determining the concentrations of ionic contaminants on disk drive components.

### 2. SCOPE

Capability for the measurement of fluoride, chloride, nitrate, phosphate, and sulfate is required. For specific ions to be measured, consult the appropriate Drive Program Cleanliness Specification. Detection limits depend on the purity of the water used for sample and standard preparation, the size sample used, instrument set-up, as well as operator skill and laboratory cleanliness. Facilities may deviate from this procedure as long as equivalency is shown and acceptable detection limits are obtained. Deviations must be documented and approved by factory case study. Procedures are included for the optional measurement of cations (lithium, sodium, ammonium, potassium, magnesium and calcium).

### 3. TERMS/DEFINITIONS

IC	Ion Chromatography
DI Water	Distilled Deionized Water

### 4. APPLICABLE DOCUMENTS

96-4575	HDA Component Cleanliness Specification
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### 5. EQUIPMENT

- 5.1 Ion Chromatograph: Dionex DX500 system or equivalent
- 5.2 Volumetric flasks, sizes to include 25, 50, 100, 250, 500, 1000 ml
- 5.3 Volumetric pipettors: 100 ul, 1000 ul, 5000 ul
- 5.4 Graduated cylinders: 10 ml, 25 ml, 100 ml

- 5.5  $\geq 18$  mOhm DI water
  - 5.6 Anion stock solution concentrate containing  $F^-$ ,  $Cl^-$ ,  $NO_3^-$ ,  $HPO_4^{2-}$ ,  $SO_4^{2-}$   
(such as Alltech Mix 5 or Dionex Combined 5 Anion standard)
  - 5.7 Evaporating dishes to soak small parts
  - 5.8 Disposable glass pipettes and dispensing bulb
  - 5.9 Hotplate for heating water to  $80^\circ C$
  - 5.10 2-250 ml beakers or two beakers of the same size for preheating water
  - 5.11 Thermometer for measuring water temperature
  - 5.12 Plastic or metal tweezers for handling parts
  - 5.13 Disposable syringe for IC injection
  - 5.14 0.5M  $Na_2CO_3$  concentrate from Dionex
  - 5.15 0.5M  $NaHCO_3$  concentrate from Dionex
  - 5.16 Clean plastic beakers 20 ml
  - 5.17 Polypropylene or Teflon beakers 1000 ml and 600 ml capacity
  - 5.18 Ultrasonic cleaner for disk extraction
  - 5.19 Polypropylene wide mouth bottles with caps: 30 ml, 50 ml, 125 ml, 250 ml.
  - 5.20 Plastic trays 7  $\frac{3}{4}$ " x 6" x 1  $\frac{1}{2}$ " for 3  $\frac{1}{2}$ " base or cover extraction
  - 5.21 PVC or nitrile gloves (not latex)
  - 5.22 Polyvials (5-ml size) and plain caps from Dionex
  - 5.23 \*Cation stock solution concentrate containing  $Li^+$ ,  $Na^+$ ,  $NH_4^+$ ,  $K^+$ ,  $Mg^{+2}$ ,  
 $Ca^{+2}$  (such as Dionex Combined 6 Cation Standard II)
  - 5.24 \* 95-98%  $H_2SO_4$
- \*Optional—for cation measurement

## 6. REQUIREMENTS FOR SAMPLE PREPARATION

- 6.1 For testing ionic contamination on disk drive components, select the parts in the condition they would be in on the drive production line. Parts, which are not cleaned at Western Digital, should have been stored in standard packaging for a minimum of 24 hours. Select a quantity of parts which gives a minimum surface area of  $10 \text{ cm}^2$ .
- 6.2 For failure analysis and engineering studies, it may not be possible to obtain enough parts to have the required total surface area of  $10 \text{ cm}^2$ . In this case use the largest surface area possible and the smallest volume of water for extraction to maximize the final anion concentration.
- 6.3 The ion chromatograph must be properly calibrated.
- 6.4 Glassware, plasticware, and other supplies must be clean.
- 6.5 Gloves must be worn during sample preparation.

## 7. SET-UP

### 7.1 Eluent

**7.1.1 Prepare eluent for anion system:** If using Dionex AS12A columns, prepare a 2.7mM  $\text{Na}_2\text{CO}_3$ / 0.3mM  $\text{NaHCO}_3$  solution by adding 1.2 ml 0.5 M  $\text{NaHCO}_3$  and 10.4 ml 0.5 M  $\text{Na}_2\text{CO}_3$  to a 2.0 L flask and diluting to the 2.0 L mark with  $\geq 18$  MOhm DI water. The preparation must be documented if it is different than above.

**7.1.2 \*Prepare eluent for cation system:** If using Dionex CS12A columns, prepare a 20mN  $\text{H}_2\text{SO}_4$  by putting 40ml 1N  $\text{H}_2\text{SO}_4$  into a 2.0 L flask and diluting to the 2.0 L mark with  $\geq 18$  MOhm DI water. To make 1N  $\text{H}_2\text{SO}_4$ , the exact concentration of the  $\text{H}_2\text{SO}_4$  should be used in the sample calculation below:

$$\text{FW of } \text{H}_2\text{SO}_4 = 98.08 \text{ g}$$

$\text{H}_2\text{SO}_4$  concentration = 98% (example, use whatever percentage your acid actually is)

$$1 \text{ liter} \times \frac{98.08\text{g}}{1 \text{ mole}} \times \frac{1 \text{ mole}}{2 \text{ eq}} \times \frac{1 \text{ mole}}{\text{liter}} \times \frac{100\text{g}}{98\text{g}} = 50.04\text{g}$$

Therefore, to make 1N  $\text{H}_2\text{SO}_4$  with 98%  $\text{H}_2\text{SO}_4$ , weigh out 50.04 g 98%  $\text{H}_2\text{SO}_4$  in 20 ml plastic beaker and transfer to a 1.0 L flask and dilute to the 1.0 L mark with  $\geq 18$  MOhm DI water.)

## 7.2 Water For Extraction

- 7.2.1 On a hot plate, heat  $\geq 18$  MOhm DI water in a beaker to  $80^\circ\text{C}$ .
- 7.2.2 To monitor the water temperature, place a thermometer in a second beaker of the same size. Fill with the same quantity of water at the same temperature as the water in the first beaker. Heat both beakers simultaneously. Do not use the water from the beaker with the thermometer for extractions.
- 7.2.3 Run a water blank to ensure no ionic contaminants are present (see criteria in 9.7).

## 7.3 Extraction Supplies

- 7.3.1 If using an auto sampler, all vials and caps must be soaked in DI water, preferably overnight, prior to use. The caps and vials have trace levels of contamination which must be removed by washing.
- 7.3.2 If using a syringe, rinse the syringe at least 5-10 times with DI water between injections to avoid cross contamination or use a new syringe for every injection. New syringes must be rinsed prior to use.
- 7.3.3 All glassware, plasticware, tweezers, etc. used in preparation of standards and samples have to be thoroughly cleaned.

7.3.3.1 Detergent is not recommended for cleaning. IPA can be used to remove oily films on plasticware.

7.3.3.2 Supplies used for IC should be isolated for IC use only.

7.3.3.3 When possible, all glassware, plasticware, tweezers, etc. should be left soaking in DI water whenever not in use.

The water should be changed daily.

7.3.4. Prepare and analyze by IC a blank on glassware and other equipment to be used for the extraction.

7.3.4.1 Use 25 mls of water total to rinse all extraction supplies - transfer the same water sample from one container to the next. This way, each container does not have to be checked one at a time.

7.3.4.2 Test the collected sample on the ion chromatograph to ensure that any ionic contaminants present do not exceed the criteria in 9.7. If contamination exceeds the criteria, re-clean all the supplies and run another method blank until acceptable.

#### 7.4 Calibration Standards

7.4.1 Combined Anion Standards containing fluoride, chloride, nitrate, phosphate, and sulfate may be purchased (\*Combined Cation Standards containing lithium, sodium, ammonium, potassium, magnesium, calcium may also be purchased) through Dionex or Alltech. All stock solutions which are purchased must be traceable to a certified reference such as NIST. Alltech or Dionex are two recommended suppliers. If standards are prepared from dried salts, the procedure must be documented.

7.4.2 Standards should be prepared regularly.

- 7.4.3 All standard solutions containing less than 100 ppb of any ion must be prepared fresh the day of use.
- 7.4.4 All standard solutions which contain more than 100 ppb of each anion should be replaced once a month.
- 7.4.5 Standards should be stored in air tight containers in a refrigerator.
- 7.4.6 The calibration standards should be prepared by pipetting the required amount of stock standard into a volumetric flask, and diluting to the mark with DI water.

Example Calculation:  $C (cs) = \frac{C (ss) \times V (ss)}{V (cs)}$

where  $C (cs)$  = Concentration of calibration standard in ng/ml

$C (ss)$  = Concentration of stock solution in ng/ml

$V (cs)$  = Final volume of calibration standard in ml

$V (ss)$  = Volume of stock solution used in ml

- 7.4.7 Suggested Calibration Standard Preparation for Anion
- 7.4.7.1 Preparation of calibration standards using Alltech anion concentrate stock is shown here. Any calibration must conform to the items in section 8.0. Media suppliers must use a lower calibration standard (than that listed below) in order to conform to section 8.3
- 7.4.7.2 Use Alltech Mix 5, which contains 25 ppm fluoride, 50 ppm chloride, 50 ppm nitrite, 50 ppm bromide, 50 ppm nitrate, 50 ppm phosphate, and 50 ppm sulfate.

7.4.7.3 Prepare the calibration standards by diluting the stock as shown in **table 2** below. Place the indicated volume of stock solution into a volumetric flask (size shown under "final volume" column) using a 100  $\mu$ L pipettor. Dilute to the mark with DI water.

Table 1

<u>Level</u>	<u>Anion Concentration</u>	<u>ul stock</u>	<u>Final volume</u>
(1)	2.5 ppb F <sup>-</sup> , 5.0 ppb others	100 ul	1000 ml
(2)	5.0 ppb F <sup>-</sup> , 10.0 ppb others	100 ul	500 ml
(3)	10.0 ppb F <sup>-</sup> , 20.0 ppb others	100 ul	250 ml
(4)	25.0 ppb F <sup>-</sup> , 50.0 ppb others	100 ul	100 ml

7.4.8 \*Suggested Calibration Standard Preparation for Cation

7.4.8.1 Use Dionex cation stock solution, which contains 50 ppm lithium, 200 ppm sodium, 250 ppm ammonium, 500 ppm potassium, 250 ppm magnesium, and 500 ppm calcium.

7.4.8.2 Prepare the calibration standards by diluting the stock as shown in Table 2 below. Place the indicated volume of stock solution into a volumetric flask (size shown under "final volume" column) using a 100 uL pipettor. Dilute to the mark with DI water.

Table 2

<u>Level</u>	<u>Cation Concentration</u>	<u>ul stock</u>	<u>Final volume</u>
(1)	5 ppb Li <sup>+</sup> , 20ppb Na <sup>+</sup> , 25ppb NH <sub>4</sub> <sup>+</sup> , 50ppb K <sup>+</sup> , 25ppb Mg <sup>+2</sup> , 50ppb Ca <sup>+2</sup>	100 ul	1000 ml (1L)
(2)	10 ppb Li <sup>+</sup> , 40ppb Na <sup>+</sup> , 50ppb NH <sub>4</sub> <sup>+</sup> , 100ppb K <sup>+</sup> , 50ppb Mg <sup>+2</sup> , 100ppb Ca <sup>+2</sup>	100 ul	500 ml
(3)	10 ppb Li <sup>+</sup> , 40ppb Na <sup>+</sup> , 50ppb NH <sub>4</sub> <sup>+</sup> , 100ppb K <sup>+</sup> , 50ppb Mg <sup>+2</sup> , 100ppb Ca <sup>+2</sup>	100 ul	250 ml

## 8. CALIBRATION

- 8.1 Calibration is required every time the eluent is replenished.
- 8.2 The calibration range should bracket the expected concentration range of the samples.
- 8.3 At a minimum, all instruments must be capable of accurately (within  $\pm$  20% or 1.0 ppb, whichever is greater) measuring an anion peak at a level corresponding to 1/5<sup>th</sup> of the spec limit applicable to any sample being tested at the low end of the calibration scale being used. See Appendix B (16.0) for an example of this calculation.
- 8.4 Linearity over the selected range should be established. The calibration curve for each ion should contain at least three points (from 3 standards) if it extends over one order of magnitude, and at least five points if it covers two orders of magnitude.

8.5 The Dionex DX 500 has linear regression software that automatically calculates the peak area for each anion (\*cation) detected and updates the calibration curve. Check the correlation coefficient of the curves after each calibration. The correlation coefficient should be equal to or greater than 0.990 for fluoride, and greater than 0.995 for the other ions. (\*The correlation coefficient should be equal to or greater than 0.995 for all cations.)

8.6 If not, re-calibrate the equipment using new standards and/or eluent.

## 9. QUALITY CONTROL

9.1 A calibration check is required every time the instrument is calibrated. A calibration check standard should be made using an Anion (\*Cation) Standard stock concentrate from a different source than that used for calibration. A bottle from a different lot from the same vendor is acceptable.

9.2 A calibration verification standard should be run at the beginning and end of every schedule, and after every 15 samples. This standard should have anion concentrations in the middle of the calibration curves. For the recommended standards in 7.4.7 for anion, this would be level (4). (\*For cation, this would be level (2).)

9.3 The analyzed concentrations of the chloride, nitrate, phosphate, and sulfate peaks (\*lithium, sodium, ammonium, potassium, magnesium, and calcium) in the check and verification standards must be  $\pm 5\%$  of the theoretical values. The concentration of the fluoride must be  $\pm 10\%$  of the theoretical value. If not, the problem must be resolved and corrected before any more samples are run.

9.4 For low calibration standards, the verification standards are permissible to be as stated in 9.3, OR  $\pm 1.0$  ppb, whichever is greater.

9.5 If any samples have ion concentrations 25% or greater than the highest calibration standard, the sample should be diluted and rerun.

9.6 Water Blanks and Method Blanks must be prepared and analyzed for every batch of samples. The water blank should consist of a sample of the DI water used for extraction (7.2.3). The method blank should consist of a sample which has undergone all the same extraction steps as a component sample, without the component.

9.7 For components which have specified maximum acceptable limits, (spec limits), the levels of any ion in the blanks must not exceed the concentration of that ion which corresponds to 10% of the specified limit for that ion. See Appendix C for examples of this calculation.

## 10. SAMPLE INJECTION

10.1 Run the baseline for 1 hour prior to use, or until steady.

10.2 If performing manual injections, inject a quantity of the sample that is three times the size of the sample loop.

10.3 If using an auto sampler, prepare schedule and name the file with the run date.

10.4 The first two samples are to be water blanks, then a calibration standard, then 15 samples, then a calibration standard, then another 15 samples, etc.

## 11. SAMPLE EXTRACTIONS

### 11.1 Small Component Extraction

11.1.1 Table 3 lists the guidelines for the minimum number of some typical components, for every 25 ml of water, required for extraction in wide-mouthed bottles. Surface area guidelines are stated in sections 6.1 and 6.2. Components from different drives products may have different surface areas, so this is only a guideline. Consult Western Digital for the exact surface area of each component. The number of components and the amount of extraction water used should be consistent with each instruments calibration range, detection limits, and any applicable specification limits.

Table 3

<u>Component</u>	<u># of parts</u>	<u>Amount of water (ml)</u>
HSA/APFA*	1	25
Actuator	1	25
Disk Spacer	2	15
Disk Clamp	2	15
VCM	1	25
Crashstop	3	15
Shipping Comb	2	15

\*remove shorting block, gasket, and shipping comb, then separate heads

11.1.2 Use clean tweezers to transfer samples into clean 250 ml or 150 ml polypropylene, wide-mouth bottles.

11.1.3 Use graduated cylinder to measure the known volume of DI water which has been pre-heated to 80<sup>0</sup>C. CAUTION: WATER IS HOT!

11.1.4 Pour water into wide mouth bottle and cap the bottle immediately.

11.1.5 Shake the bottle gently for 15 minutes. The sample and bottle geometry should maximize the exposure of the sample to the water

11.1.6 Remove the component from the container using clean tweezers.

11.1.7 Inject the water extract into the IC.

11.1.8 If the anion peaks from the sample are more than 25% higher than the most concentrated calibration standard, dilute the sample with DI water so that the sample concentration falls within the standard concentration range.

## 11.2 Entire Cover and Base Casting Extraction

11.2.1 Table 4 below suggests tray sizes and amounts of water for base and cover extraction.

Table 4

<u>Drive/Component</u>	<u>Tray size</u>	<u>ml of water</u>
Base Casting	7 3/4" x 6" x 1 1/2"	200 ml
Top Cover	7 3/4" x 6" x 1 1/2"	75 ml

11.2.2 Place base casting or top cover into the appropriate sized plastic tray.

11.2.3 Using a graduated cylinder, measure the corresponding amount of DI water. The DI water is to be heated to 80°C.  
CAUTION: WATER IS HOT!

11.2.4 Pour water over component.

11.2.5 Gently agitate the tray for 7 minute.

11.2.6 Using clean tweezers, flip the component over.

11.2.7 Agitate the tray for 8 minute. The entire part should now be washed with hot DI water for a total of 15 minutes.

11.2.8 Remove the component from the tray using clean tweezers.

11.2.9 Inject the water extract into the IC.

11.2.10 If the ion peaks from the sample are more than 25% higher than the most concentrated calibration standard, dilute the sample with DI water so that the sample concentration falls within the standard concentration range.

11.2.11 When the ionic content of the inside of the base casting or top cover is of interest, use a localized extraction method as listed in 11.6.

### 11.3 Media Extraction

11.3.1 Place disk into 1000 ml polypropylene or Teflon beaker.

11.3.2 Using a graduated cylinder, measure 15 ml/disk of DI water. The DI water is to be heated to 80°C. CAUTION: WATER IS HOT! Note, the water level should completely immerse the disk. The amount of water used can be adjusted in order to completely cover the disk. A note should be included with the results if a volume other than 15 ml is used.

11.3.3 Cover beaker with lid.

11.3.4 Agitate the beaker for 30 seconds. The agitation will sufficiently allow the water to contact all exposed disk surfaces.

11.3.5 Place beaker in an ultrasonic bath beaker for 15 minutes.

11.3.6 At the 5 and 10 minute intervals, agitate the beaker for 10 seconds.

11.3.7 After sonication, agitate the beaker for another 30 seconds.

11.3.8 Inject the extraction water into the IC.

11.3.9 If the anion peaks from the sample are more than 25% higher than the most concentrated calibration standard, dilute the sample with DI water so that the sample concentration falls within the standard concentration range.

#### 11.4 Localized Sample Extraction

11.4.1 Recommended amount of water per component extraction is listed in table 5 below.

Table 5

<u>Component</u>	<u># parts / #ml of water</u>
Motor mounting flange	1/10
Base casting (inside only)	1/25
Top cover (inside only)	1/25

11.4.2 Using clean tweezers, hold the sample over an evaporating dish.

11.4.3 With a pipette and dispensing bulb, rinse the area of interest with 80<sup>0</sup>C DI water. CAUTION: WATER IS HOT!

11.4.4 Repeat the rinsing step for a total of five times.

11.4.5 Inject the water extract into the IC.

11.4.6 If the anion peaks from the sample are more than 25% higher than the most concentrated calibration standard, dilute the sample with DI water so that the sample concentration falls within the standard concentration range.

## 12. CALCULATIONS

12.1 Use the linear regression equation to calculate the sample's anion (\*cation) concentration in ppb. Record this value as A.

12.2 The final result reported should be in ng/cm<sup>2</sup>. The calculation is as follows:

$$C = \frac{(A - B) * V}{N * S}$$

C = result in ng/cm<sup>2</sup>

A = anion concentration of the sample in ppb (ng/ml)

B = anion concentration of the blank in ppb (ng/ml)

V = volume of water used for extraction in ml

N = number of parts extracted

S = surface area in  $\text{cm}^2$

### 13. REPORTING METRICS

13.1 All QC results should be archived. All the items below should be reported with the results. Other items may also be added to the report at the analysts or requesters discretion.

13.2 Minimum reporting limits for each anion quantified

13.3 All blank values (water and method).

13.4 Number of parts, surface area, and volume of water used for samples.

### APPENDIX A

To convert  $\text{ng}/\text{cm}^2$  to  $\text{mg}/\text{m}^2$ , a conversion factor of "1.0  $\text{ng}/\text{cm}^2$  equals 0.01  $\text{mg}/\text{m}^2$ " may be used.

Example: To convert 20  $\text{ng}/\text{cm}^2$  to  $\text{mg}/\text{m}^2$ :

$$20 \text{ ng/cm}^2 \times \frac{0.01 \text{ mg/m}^2}{1.0 \text{ ng/cm}^2} = 0.2 \text{ mg/m}^2$$

### APPENDIX B

At a minimum, all instruments must be capable of accurately (within  $\pm 20\%$  or 1.0 ppb, whichever is greater) measuring an anion peak at a level corresponding to 1/5<sup>th</sup> of the spec limit applicable to any sample being tested at the low end of the calibration scale being used. Below are two examples of this:

## Example 1

One component, surface area  $100 \text{ cm}^2$ , is extracted in 25 ml of water. It is specified that the component is not allowed to exceed  $50 \text{ ng/cm}^2$  of chloride by IC testing. The amount of chloride in the extract water that corresponds to  $50 \text{ ng/cm}^2$  is

$$\text{ng/ml (ppb)} = \frac{(50 \text{ ng/cm}^2) \times (100 \text{ cm}^2) \times (1 \text{ part})}{25 \text{ ml}} = 200 \text{ ppb}$$

Thus, the instrument must be capable of measuring  $1/5^{\text{th}}$  of 200 ppb, which equals 40 ppb, with an accuracy of  $\pm 20\%$  ( $40 \text{ ppb} \pm 8 \text{ ppb}$ ) or better.

## Example 2

One component, surface area  $10 \text{ cm}^2$ , is extracted in 25 ml of water. It is specified that the component is not allowed to exceed  $10 \text{ ng/cm}^2$  of chloride by IC testing. The amount of chloride in the extract water that corresponds to  $10 \text{ ng/cm}^2$  is

$$\text{ng/ml (ppb)} = \frac{(10 \text{ ng/cm}^2) \times (10 \text{ cm}^2) \times (1 \text{ part})}{25 \text{ ml}} = 4 \text{ ppb}$$

Since  $1/5^{\text{th}}$  of 4 ppb equals 0.8 ppb, and 1 ppb is greater than this, the instrument must be capable of measuring 1 ppb with an accuracy of  $\pm 20\%$  or better.

## APPENDIX C

For components which have specified maximum acceptable limits, (spec limits), the levels of any ion in the blanks must not exceed the concentration of that ion which corresponds to 10% of the specified limit for that ion. Below is an example of how to determine this:

One component, surface area  $100 \text{ cm}^2$ , is extracted in 25 ml of water. It is specified that the component is not allowed to exceed  $50 \text{ ng/cm}^2$  of chloride by IC testing. The amount of chloride in the extract water that corresponds to  $50 \text{ ng/cm}^2$  is

$$\text{ng/ml (ppb)} = \frac{(50 \text{ ng/cm}^2) \times (100 \text{ cm}^2) \times (1 \text{ part})}{25 \text{ ml}} = 200 \text{ ppb}$$

**Thus, the blank cannot contain more than 10% of 200 ppb, which equals 20 ppb.**

## DYNAMIC HEADSPACE OUTGASSING PROCEDURE

### 1. PURPOSE

This document describes a general procedure for dynamic headspace analysis (DHS) for use by in-house and external laboratories of factory case study

### 2. SCOPE

This procedure can be applied to quantitatively and qualitatively measure the outgassing material from disk drive piece parts and/or sub-assemblies, as well as raw materials undergoing evaluation for use in the disk drive.

This test is not designed to sample all of the material that could outgas from a particular part at a given temperature, but rather to provide a snapshot of the outgassing performance of a material under certain conditions. Multiple dynamic outgassing tests, using various conditions of time, temperature, flow rate, etc. would need to be carried out to fully characterize the outgassing behavior of a material or part.

Compounds studied can encompass a range of volatilities depending on the adsorbent system used, but the most common solid-phase adsorbents work best in the range of C<sub>8</sub> to C<sub>25</sub>. Compounds more volatile than C<sub>8</sub> may not be adequately trapped, and the species with volatilities less than that of C<sub>25</sub> may not be completely desorbed without going to higher desorption temperatures than those recommended in this document.

This procedure uses as standard conditions sample heating for 3 hours at 85°C with outgassed materials absorbed onto an adsorbent tube, GC-MS as a detection method and semi-quantitative analysis using an internal standard. Other temperatures and times may be used for materials characterization studies or as specified in the HDA Component Cleanliness Specification.

Dynamic Headspace Outgassing instrumentation is available which utilizes cryogenic trapping of outgassed materials in place of adsorbent tube trapping. If such instrumentation is used, the portions of this procedure which describes the

adsorbent tube is not applicable and appropriate modifications of the procedure are required.

### 3. TERM / DEFINITIONS

DHS	Dynamic Headspace
GC-MS	Gas Chromatography-Mass Spectrometry
In-house laboratory	Laboratory owned and operated by WDC
External laboratory	Laboratory owned and operated by vendor or provider of analytical laboratory services.
Carbotrap 300	Mixed-bed adsorbent trap, filled with a mixture of Carbotrap B Carbotrap C and Carbosieve S III.

### 4. APPLICABLE DOCUMENT

- 4.1 96-004575 Desktop HDA Component Cleanliness Specification
- 4.2 NIOSH Manual of Analytical Methods (NMAM) 4th Edition,, Introduction Sections A - E (This is a valuable reference for the definition of terms and also offers guidance for method development in Sections D and E).
- 4.3 IDEMA M11-99General Outgas Test Procedure by Dynamic Headspace Analysis

### 5. RECOMMENDED EQUIPMENT AND MATERIALS

#### 5.1 Equipment

- 5.1.1 Sample Chamber --The chamber should be inert, large enough to house the part(s) being tested and leak proof. The chamber should also allow for the intake and exit of the extraction gas and provisions for adequate mixing of the gas should be taken.

- 5.1.2 Adsorbent Tubes, filled with Carbotrap 300 Tenax TA, or other suitable materials
- 5.1.3 Temperature-controlled heating apparatus (forced-flow laboratory oven or equivalent).
- 5.1.4 Flowmeter, Humonics Veri-Flow 500 or equivalent, calibrated
- 5.1.5 Hydrocarbon trap for purge gas
- 5.1.6 Regulator, dual stage, 0 - 30 psi
- 5.1.7 Gas Chromatograph equipped with Mass Spectrometer (HP 6890 GC with HP 5973 MSD or equivalent).
- 5.1.8 Thermal desorption system (Perkin-Elmer ATD 400 or equivalent)
- 5.1.9 GC-Column, Fused Silica Coated w/ poly (5%-diphenyl-95%-dimethylsiloxane) w/0.10 to 0.25 micron film thickness, length of 10 to 35 meters, o.d. of 0.25 mm, or approved equivalent
- 5.1.10 Flow controller
- 5.1.11 Thermocouple, calibrated
- 5.2 Materials
  - 5.2.1 Compressed nitrogen or helium, 99.99% or better
  - 5.2.2 Aluminum Foil, food grade, cut into 3 cm x 3 cm squares
  - 5.2.3 Surrogate Standard- 200 mg/L Anthracene d-10 solution, in methylene chloride (prepared by weighing 20 mg of anthracene d-10 into a 100 ml volumetric flask, diluting to the mark with solvent and inverting the flask repeatedly to mix).
  - 5.2.4 Internal Standard—100 mg/L Hexadecane-d34 solution, in hexane (prepared by weighing 10 mg of hexadecane d-34 into a 100 ml volumetric flask, diluting to the mark with

solvent and inverting the flask repeatedly to mix). If hexadecane d-34 is not available, hexadecane may be substituted, but there is a risk that the samples tested may outgas hexadecane.

**5.2.5 Hydrocarbon Standard—** A hydrocarbon standard containing C<sub>16</sub> to C<sub>20</sub> n-hydrocarbons (even carbon numbers, equal amounts of each) to be used to assess and monitor system performance

5.2.6 Adsorbents

## 6. PROCEDURE

### 6.1 Sample Collection

6.1.1 Pack the adsorption tubes with the appropriate type and amount of adsorbent. The adsorbent should not extend into regions of the tube that are not heated. Alternatively, commercially prepared adsorbent tubes can be used. The adsorbent should be chosen so that it adsorbs the compound(s) of interest and also desorbs the compound(s) of interest at reasonable desorption temperatures. Care must be taken that the adsorbent design has the appropriate breakthrough volume (refer to NIOSH Manual of Analytical Methods Section E (Development and Evaluation of Methods) for the compounds of interest. The adsorbent should be chosen such that the requirements of Section 6.4.4 are met.

6.1.2 Condition the adsorption tubes by heating them 25 to 50 degrees Celsius above the sample desorption temperature, while purging with nitrogen or helium gas.

6.1.3 Heat chamber to 85 degrees Celsius

6.1.4 Install the adsorption tube at the flow exit of the chamber but outside the heated zone.

- 6.1.5 Prepare the sample and place the sample in the sample chamber. For HDA components and assemblies, the entire assembly/component that is used in the drive is sampled. For piece parts and subassemblies, one component per container is sampled. For raw materials, packaging, etc, samples are weighed (into aluminum pan if liquid) or cut and placed in the chamber
- 6.1.6 Prepare a method blank (empty sampling chamber) at the same time as the sample and run the method blank in the same set of runs to check the cleanliness of the sampling system.
- 6.1.7 Spike each sample by injecting 5  $\mu$ l of 200 mg/L anthracene d-10 onto an aluminum pan and placing one pan in each chamber to act as a surrogate sample.
- 6.1.8 Use a calibrated flow rate meter and a flow controller to set the flow rate in the range 50 to 75 ml/minute. Take measurement at exit of adsorption tube. Record flow rate used.
- 6.1.9 Remove the adsorption tube after three hours.
- 6.1.10 Load 1000 nanograms of hexadecane internal standard into all tubes by adding 10 microliters of a 100 mg/L solution into the front end (the end first contacting the incoming gas during sampling) of the adsorption tube.
- 6.1.11 Load 1000 nanograms of anthracene d-10 standard into an extra tube for recovery calculation by adding 5 microliters of a 200 mg/L solution into the front end (the end first contacting the incoming gas during sampling) of the adsorption tube.
- 6.1.12 Immediately after a standard has been injected into a adsorbent tube, run clean (99.99% or better) nitrogen into

the tube by connecting a nitrogen output into side of the tube closest to where the standard was added (the front of the tube) at 50 ml/minute for 10 minutes. The direction of gas flow is from the front of the tube to the back so that the standard is carried into the tube. Store at 0°C if not to be analyzed within 4 hours.

6.1.13 Remove parts from sample chamber.

## 6.2 Operating Conditions for the Thermal Desorption Unit

6.2.1 Consult instrument operation manual to ensure that compounds of interest are desorbed from the tube and deposited on the GC/MS column.

6.2.2 Apply general good chromatography practices so as to avoid co-elution of compounds of interest and obtain proper peak shapes etc. Adjust instrument split ratios to avoid overloading column.

6.2.3 Do not exceed the maximum temperature recommended for the adsorbents during the desorption step.

## 6.3 Gas Chromatography-Mass Spectrometry (GC-MS) Based Quantitation

### 6.3.1 Typical Mass Spectrometer Settings

Electron Energy	70eV
Mass Range	33-550 amu
Scan Time	1 second or less

### 6.3.2 Mass Spectrometer Performance Check

At regular intervals (at least weekly), perform tune of the mass spectrometer according to the manufacturer's recommendations to verify performance. Obtain a mass spectrum of perfluorotributylamine (PFTBA) and document that previously established criteria are achieved. If they cannot, instrument maintenance must be performed.

### 6.3.3 Typical Gas Chromatograph-Mass Spectrometry (GC-MS)

Instrument Parameters (deviations are acceptable)

Prepare instrument by setting the following parameters:

injector temperature (if applicable): 265°C

initial column temperature: 40°C

initial time: 1 min.

temperature ramp: 15 degree/min.

final temperature: 260°C

Total run time: approximately 30 min.

GC Transfer line temperature: 280°C

Column flow: approximately 1 ml/min.

solvent delay: 3.0 minutes (may vary with sampling requirements)

### 6.3.4 Establish instrument sensitivity by detection of the 1,000

ng of hexadecane that is added as an internal standard with each tube. The instrument must be capable of clear detection of this quantity and with reasonable peak shape.

Note the retention time, the signal intensity and the peak height of the standard for QC records. Signal intensity refers to integrated area under the peak and not peak height. Calculate signal to noise ratio by dividing peak height by noise level.

### 6.3.5 Use the historical value obtained for the signal intensity of

the internal standard for a particular instrument to establish a benchmark for that particular instrument. Large variations in the integrated intensity suggests the instrument is operating in a questionable manner and that

any data obtained while in that condition is unsuitable for use.

6.3.6 When in question always inject a fresh sample to establish instrument sensitivity, column condition and general condition of the instrument and compare that to historical results.

6.3.7 Note and record the percent recovery of anthracene d-10 added to each sampling chamber.

$$\% \text{ Recovery} = \frac{\text{Intensity of anthracene d-10 in chamber sample}}{\text{intensity of anthracene d-10 in tube sample}} \times 100$$

#### 6.4 System Performance Criteria

6.4.1 Since DHS systems used by various laboratories will have different sampling containers, DHS units, GC/MS instruments and other parameters, the following criteria are established to enable commonality of DHS results.

##### 6.4.2 Mass spectrometer Performance

The mass spectrum obtained from perfluorotributylamine (PFTBA) should conform to the following criteria:

<u>Mass</u>	<u>m/z abundance criteria (%)</u>
69	100
70	0.5-1.5
219	>40 and <70
220	2-8
502	>2
503	5-15% of 502

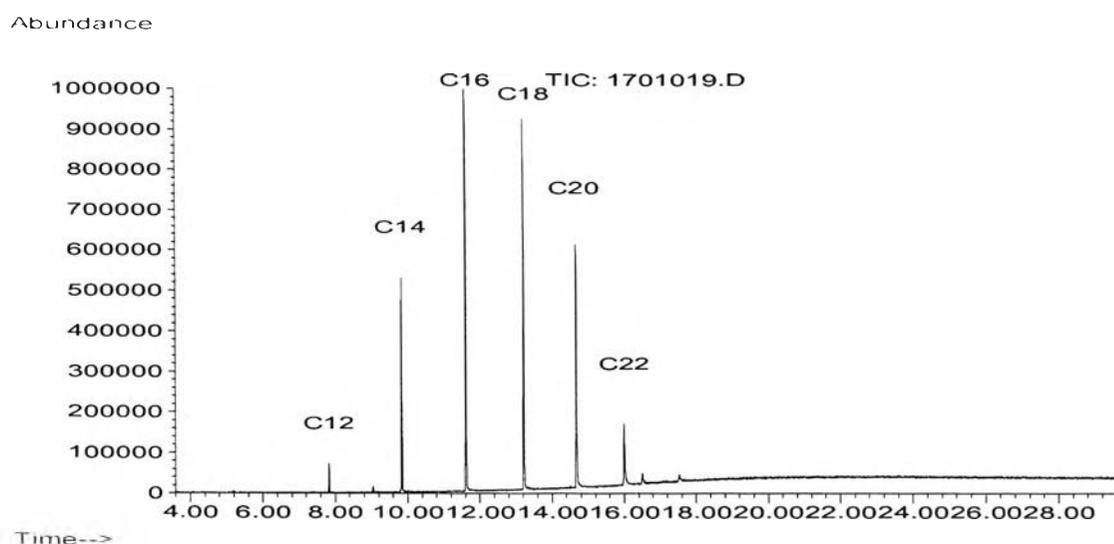
#### 6.4.3 Signal to Noise Ratio of Hexadecane d-34

The height of the hexadecane d-34 peak must be at least 200 times the height of the absolute value of the base line noise, measured near the end of the chromatographic run

#### 6.4.4 Recovery of Hydrocarbon Standard from Jar

A hydrocarbon standard containing C<sub>16</sub> to C<sub>20</sub> n-hydrocarbons should be periodically analyzed as a sample. For equivalent amounts, the recovery of C<sub>20</sub> to C<sub>16</sub> should be at least 50%. The chromatogram should be similar to that below.

ภาคผนวก ค. 5 The Chromatogram



#### 6.4.5 Anthracene d10 Recovery from the Sample Chamber

The percent recovery of anthracene d10 calculated in Section 6.3.7 should not be less than 50% unless exceptions are granted by factory case study

## 7. CALCULATIONS

### 7.1 Calculating Values for Semi-Quantitative Analyses

7.1.1 Record the total integrated ion counts for each compound of interest.

7.1.2 Calculate the response factor for the internal standard:

$$\text{Response Factor} = \frac{\text{Total ion count of internal standard}}{\text{ng of internal standard}}$$

7.1.3 Calculate the semi-quantitative amount collected in the adsorbent by dividing the total ion counts for the compound or compounds by the response factor for the internal standard:

$$\text{Amount (ng)} = \frac{\text{Total Ion Counts of Compound (area counts)}}{\text{Response Factor}}$$

7.1.4 Typical final result expressions can be as follows:

7.1.4.1 Assemblies and General Piece Parts - nanograms per part

7.1.4.2 Pressure Sensitive Adhesives - micrograms per gram of adhesive or nanograms per centimeter squared

7.1.4.3 Packaging and Cured Adhesives - nanograms per gram, micrograms per gram.

## 8. REPORTING OF RESULTS

### 8.1 State the Test Conditions

8.1.1 Amount of Sample (mass and volume)

8.1.2 Sample Preparation (if applicable)

8.1.3 Volume of Sample Chamber

8.1.4 Flow Rate, Type of Extraction Gas

8.1.5 Temperature

8.1.6 Test Duration

8.1.7 Surrogate Standard Used and Internal Standard

8.1.8 Quantification Method (semi-quantitative)

8.1.9 Detection Limit (per analyte) and method used to determine detection limit

8.1.10 Recovery of surrogate sample, expressed as a percentage versus the external d-10 standard.

8.2 Report in metric units of mass per unit or mass per sample mass, mass per sample area.

8.3 Report the semi-quantitative amount on individual compounds or compound classes as required by the Program Specific Component Cleanliness Specification. Outgassing compounds are identified by standard mass spectrometer interpretation procedures, computer library searches and comparison to standard materials. Typically, several compound classes are quantitated:

8.3.1 Total Acrylates/Methacrylates – esters of 2-propenoic acid or 2-methyl-2-propenoic acid .

8.3.2 Total Initiators – 2,2-Dimethoxy-2-phenylacetophenone (DMP), 1-

Hydroxycyclohexylphenyl ketone (CHAP) or similar compounds.

8.3.3 Total Antioxidants – BHT, Ionol-2 or similar compounds

8.3.4 Total Hydrocarbons – Aliphatic and aromatic hydrocarbons

8.3.5 Total Outgassing – All outgassing compounds

8.3.6 Other part specific compounds or compound classes may also be requested to be quantitated.

Table 1 can be used as a guideline for compounds identified and quantitated and classifications used for reporting purposes.

## 9. ACCURACY, PRECISION, RECOVERY AND DETECTION LIMITS

9.1 The measured values obtained from known amounts of target compound injected into an empty sample chamber can be used to assess the accuracy, precision and detection limits of the desorption and measurement system.

9.2 Run the necessary repeatability, reproducibility, recovery and detection limit experiments to assess the efficacy of the method for critical target compounds. This may include, but is not limited to, sample container spikes to establish theoretical recoveries of typical compounds measured, and/or the analysis of samples that have been well characterized by other techniques.

ภาคผนวก ค. 6 TABLE 1

COMPOUNDS (ng/part)	#1	#2	#3	#4
<b>Methacrylates and Acrylates (total)</b>				
Hydroxy Ethyl Acrylate (HEA)				
Hydroxy Ethyl Methacrylate (HEMA)				
Hydroxy Propyl Acrylate (HPA)				
Hydroxy Propyl Methacrylate (HPMA)				
2-Ethylhexyl Acrylate				
2-Ethylhexyl Methacrylate				
Isobornyl Methacrylate (IBM)				
Phenoxy Ethyl Methacrylate				
Diethyleneglycol Dimethacrylate(DGDM)				
Triethyleneglycol Dimethacrylate(TGDM)				
Dodecyl Acrylate				
<b>Initiators(total)</b>				
2,2-Dimethoxy-2-phenylacetophenone				
1-Hydroxycyclohexylphenylketone				
Methylphenylglyoxalate				
Benzaldehyde				
Acetophenone				
Benzophenone				
<b>Antioxidants (total)</b>				
Di-t-butylphenol				
Di-t-butylmethylphenol (BHT)				
Di-t-butyl-ethylphenol (IonoI-2)				
<b>Phenols(total)</b>				
Phenol				
Cresols				
<b>Sulfur Compounds (total)</b>				
<b>Total Siloxane</b>				
<b>Total Phthalate</b>				
Diethyl Phthalate				
Dibutyl Phthalate				
Butyl Benzyl Phthalate				
Di(2-ethylhexyl) Phthalate				
<b>Hydrocarbons (total)</b>				
<b>Aromatic Hydrocarbon (total)</b>				
<b>Aliphatic Hydrocarbon (total)</b>				
<b>Carbon Range (Cx-Cy)</b>				
<b>Unknown and Others (total)</b>				
a, a-Dimethylbenzenemethanol				
Benzenemethanol				
Benzaldehyde				
Acetophenone				
Benzophenone				
Benzoic Acid				
2-Ethyl Hexanoic Acid				
2-Ethyl Hexanol				
Ethylene Glycol				
Caprolactam				
Tetrahydrofurfuryl Alcohol				
Unknown				

## NON-VOLATILE RESIDUE OF DISK DRIVE COMPONENTS

### 1. SCOPE

This document describes the procedure for Non-Volatile Residue (NVR) analysis of components used inside disc drives. Except for those base castings monitored for surfactant, the gc/ms portion of this document is not implemented.

### 2. REFERENCE DOCUMENTS

2.1 WI-HM721 Head Media Work Instructions, "Procedures and Operation of Fourier Transform Infrared

2.2 Disk Extraction Procedure

### 3. EQUIPMENT NEEDED

3.1 Microbalance with an accuracy of 0.1  $\mu\text{g}$

3.2 Evaporating dishes

3.3 Microbalance weighing pans with 2ml liquid capacity

3.4 Hexane (HPLC grade)

3.5 Methylene chloride (HPLC grade)

3.6 Clean graduated cylinders, 10ml and 100ml sizes

3.7 Glass pipettes and dispensing bulb

3.8 Hexane

3.9 Disposable test tube

3.10 Disposable weighing pans for microbalance

3.11 1 ml, 100  $\mu\text{l}$ , 25  $\mu\text{l}$ , 10  $\mu\text{l}$  syringes

- 3.12 Fourier Transform Infrared spectrometer (FTIR) with Mercury Cadmium Telluride (MCT) detector or equivalent with  $\mu\text{g}$  sensitivities to silicone, hydrocarbon, and plasticizer.
- 3.13 Potassium Bromide (KBr) crystals or equivalent for FTIR analysis
- 3.14 HP 5890 Gas Chromatograph and HP 5970 Mass Selective Detector (GC/MS) or equivalent with HP-5 capillary column (25 m X 0.2 mm X 0.5  $\mu\text{m}$ )
- 3.15 Kimble Ekonal autosampler vials, 11mm, 0.1 ml
- 3.16 Hewlett-Packard 2 ml autosampler vials, 32 mm x 11 mm or equivalent
- 3.17 Hewlett-Packard vial caps or equivalent
- 3.18 Vial cap crimp seal tool, 11 mm or equivalent
- 3.19 ml volumetric flask
- 3.20 2ppm anthracene-d10 solution

#### 4. PARTS FOR MEASUREMENTS

- 4.1 For parts normally cleaned in-house at WD and not cleaned at WD, the parts are to be submitted to the lab in the original shipping containers to minimize handling contamination.
- 4.2 Parts after cleaning at WD should be submitted to the lab in clean analytical lab approved packaging.
- 4.3 The number of parts collected for NVR will be determined by calculating the exposed surface area to be tested. Sufficient numbers of parts should be collected so that a minimum surface area of  $0.01 \text{ m}^2$  ( $100 \text{ cm}^2$ ) will be tested.
- 4.4 The types of parts will be divided into separate groups:

- 4.4.1 Metal parts: Methylene chloride will be used to rinse the residue for analysis.
- 4.4.2 Plastic parts and those with organic coatings (such as E-coat) or adhesive: Freon TF or Hexane will be used to rinse the residue for analysis.

## 5. TEST SET-UP

- 5.1 Carefully clean the evaporating dishes before the test. The sample dish and the blank dish should be washed together using the same cleaning method.
- 5.2 Verify evaporating dishes are silicone-free. In addition, each lot of the disposable test tubes should be tested for silicone.
- 5.3 Immediately before the NVR test begins, both dishes should be thoroughly rinsed with the HPLC grade methylene chloride. Disposable test tubes should also be rinsed with methylene chloride prior to testing. Periodically, lots of the disposable test tubes should be checked for cleanliness..
- 5.4 The solvent washing steps should be carried out under a fume hood by personnel trained in handling organic chemicals.
- 5.5 To make a 2ppm solution of anthracene-d10, add 20 mg of anthracene to a 100 ml volumetric flask, fill to volume with methylene chloride, and mix thoroughly.

## 6. PROCEDURE

- 6.1 Test method for metal and plastic parts
  - 6.1.1 Minimum volume requirement for extraction for soaking parts required to completely submerge all the parts to cover the minimum surface area of 0.01 square meter.
  - 6.1.2 For washing components the minimum amount of solvent required is 30 ml.

- 6.1.3 Pre-weight a microbalance weighing pans.
- 6.1.4 Using clean tweezers, transfer the samples for NVR analysis to a clean evaporating dish. Pour the appropriate solvent over the parts.
- 6.1.5 Swirl the samples and soak for 5 minutes.
- 6.1.6 Draw up clean solvent into a pipette. Remove the samples from the evaporating dish and rinse the samples over the dish.
- 6.1.7 For parts too large to fit into an evaporating dish, use clean tweezers to hold the parts over the dish. Pipette the appropriate solvent over the parts so that all of the solvent washes directly into the evaporating dish. Each area of the part should be rinsed twice with the solvent.
- 6.1.8 For HSA
  - 6.1.8.1 Remove flex gasket, and head comb, shorting block and pivot before washing and separate the heads
  - 6.1.8.2 Do not wash the connector, flex clamp, flex gasket, and other areas where there are adhesives or labels w/adhesives.
  - 6.1.8.3 Everything else should be washed
- 6.1.9 Record the total volume of solvent used as "V".
- 6.1.10 Place the evaporating dish at an angle so that the solvent and the residue will evaporate and collect in a localized area. Note the area of evaporation. (See Figure 1).
- 6.1.11 When a few drops of solvent are remaining in the localized area, pour the solvent into a clean test tube.

- 6.1.12 Rinse the evaporating dish with additional solvent and pour the solvent into the test tube. The evaporating dish should be rinsed a minimum of three times. In addition, rinse the wall of the test tube, but make sure, the amount of solvent use for the total rinse process does not exceed 75% of total volume of the test tube. 3 to 4 ml should be the approximate total volume used for the rinse process. (See Figure 2).
- 6.1.13 evaporate the solvent from the test tube with one of the following methods.
- 6.1.13.1 Evaporate test tube under a heat lamp, inside a fume hood. Be sure the temperature does not exceed 35°C
- 6.1.13.2 Evaporate at room temperature, inside a fume hood.
- 6.1.13.3 Evaporate by purging test tube with nitrogen gas. The flow rate should not exceed a rate to cause solvent turbulence or splashing.  
(Nitrogen gas tip should NOT contact the solvent. Clean the purging needle prior to use.)
- 6.1.14 When all the solvent has completely evaporated, add 500 microliters of 2 ppm D10 to the bottom of the test tube.
- 6.1.15 Remove 50 microliters of the sample for GC/MS analysis.
- 6.1.15.1 Use following GC/MS instrument parameters listed in the Disk Extraction Procedure.

- 6.1.15.2 Establish instrument sensitivity by injecting 3  $\mu\text{l}$  of 2 PPM anthracene-d10. The instrument must be capable of clearly detecting anthracene-d10 and with reasonable peak shape. The signal to noise ratio must be at least 50:1.
- 6.1.16 Again, evaporate to dryness. Then add 90 microliters of methylene chloride.
- 6.1.16.1 For NVR, use a 50  $\mu\text{l}$  syringe to transfer 50  $\mu\text{l}$  of the 100  $\mu\text{l}$  diluted sample to the tared weighing pan.
- 6.1.16.1.1 Allow the solvent to evaporate from the weighing pan and weigh the residue.
- 6.1.16.1.2 Record the weight in  $\mu\text{g}$ .
- 6.1.16.1.3 Since half of the sample was used for weighing, multiply the weight by two to obtain the total weight. Record the "Sample Residue Weight" in micrograms.
- 6.1.16.2 For micro-FTIR analysis, transfer 25  $\mu\text{l}$  of the diluted sample to a KBr crystal for analysis.
- 6.1.16.2.1 Carry out the appropriate bench FTIR analysis to quantify silicone and hydrocarbons in the residue. Record amount in  $\mu\text{g}$ .
- 6.1.16.2.2 The related document, WI-HM721, is one possible method for setting up a Nicolet FTIR for quantitative analysis.

## 7. CALCULATION

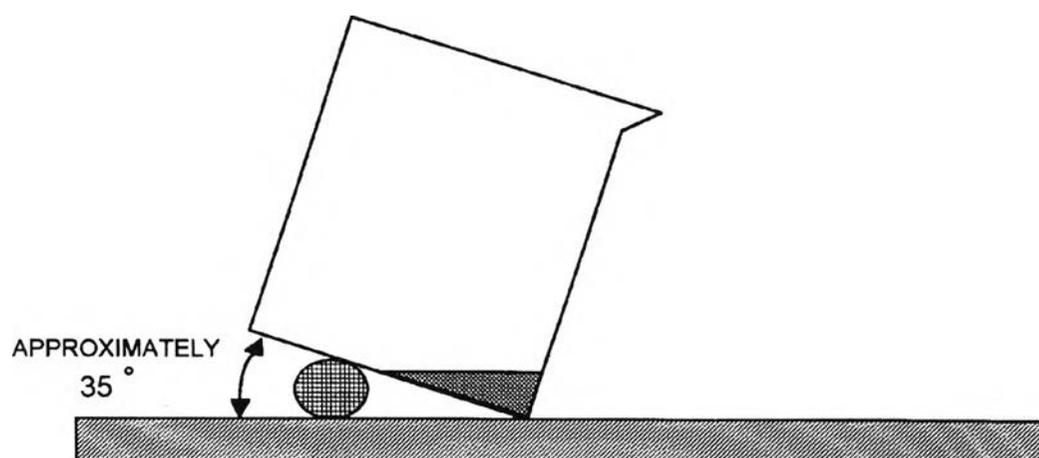
- 7.1 Non-Volatile Residue NVR ( $\text{mg}/\text{m}^2$ ) =  $[(2 \times \text{Sample Residue Weight in } \mu\text{g}) - (\text{Total Blank residue weight in } \mu\text{g})] / (1000 \times \text{Total Surface Area Washed in } \text{m}^2)$
- 7.2 Silicone ( $\text{mg}/\text{m}^2$ ) =  $(4 \times \text{Silicone Weight in } \mu\text{g}) / (1000 \times \text{Total Surface Area Washed in } \text{m}^2)$
- 7.3 Hydrocarbon ( $\text{mg}/\text{m}^2$ ) =  $(4 \times \text{Hydrocarbon Weight in } \mu\text{g}) / (1000 \times \text{Total Surface Area Washed in } \text{m}^2)$
- 7.4 GC/MS Quantification (relative to anthracene-d10 internal standard) :
- 7.5 Compound X (ng) =  $(\text{integrated value of compound X}) / [(\text{integrated value of anthracene d-10}) \times (1000 \text{ ng d10})]$

## 8. PASS/FAIL CRITERIA

- 8.1 All components internal to the HDA shall have NVR level must not exceed 10 mg/square meter with no silicone level greater than 0.5mg/square meter.
- 8.2 Parts **Prior** to in-house cleaning at WD shall have NVR level must not exceed 15 mg/square meter with no silicone level greater than 0.5 mg/square meter. In addition, if the parts **Prior** to in-house cleaning at WD have NVR level greater than 10mg/square meter, vendor shall be notified and corrective action from the vendor is then required.
- 8.3 By GC/MS, total surfactant level must not exceed 100 ng. Contaminants other than those found during the qualifying process are also not allowed.

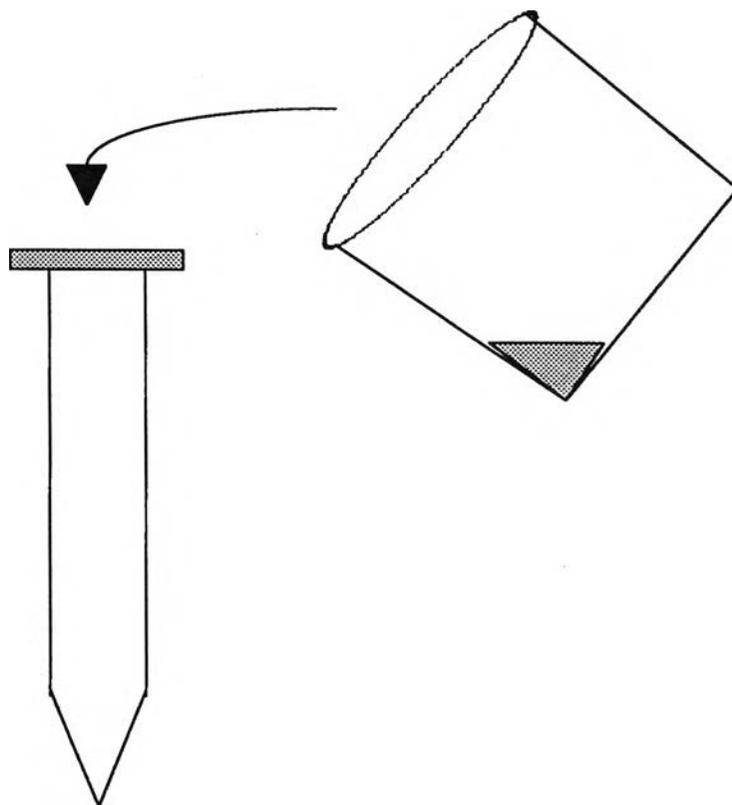
ALLOW THE METHYLENE CHLORIDE TO EVAPORATE AT ROOM TEMPERATURE  
IN ORDER TO CONCENTRATE OUTGASSING RESIDUE IN THE CORNER OF THE  
BEAKER

ภาคผนวก ค. 7 FIGURE 1

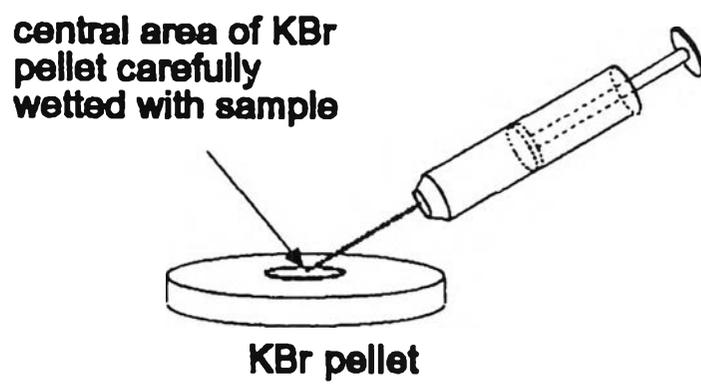


RINSE EVAPORATING DISH WITH SOLVENT AND POUR SOLVENT INTO TEST TUBE. THE EVAPORATING DISH SHOULD BE RINSED A MINIMUM OF THREE TIMES. SOLVENT SHOULD NOT EXCEED 75% OF TOTAL VOLUME OF THE TEST TUBE.

ภาคผนวก ค. 8 FIGURE 2



ภาคผนวก ค. 9 FIGURE 3



ภาคผนวก ค 10 FIGURE 4

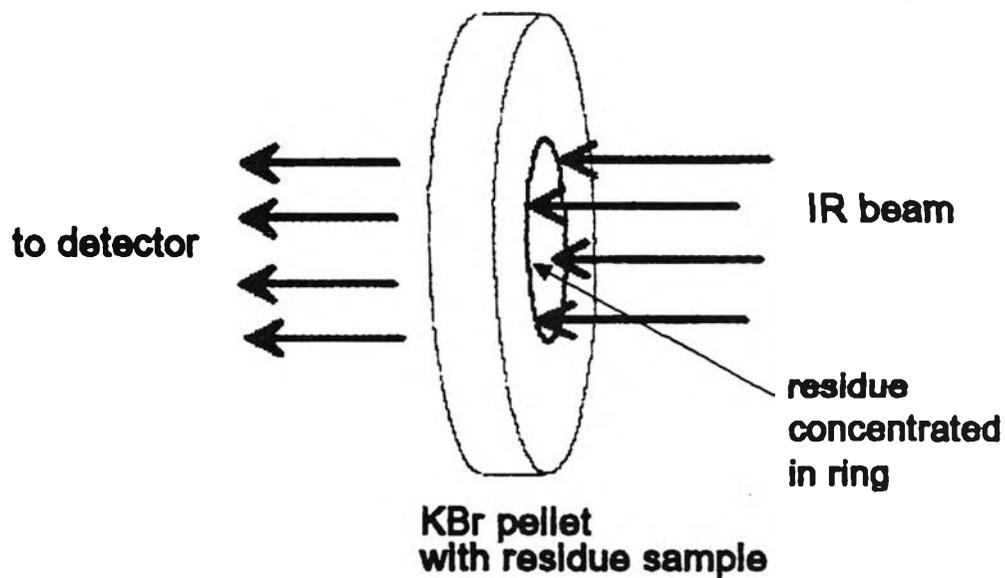
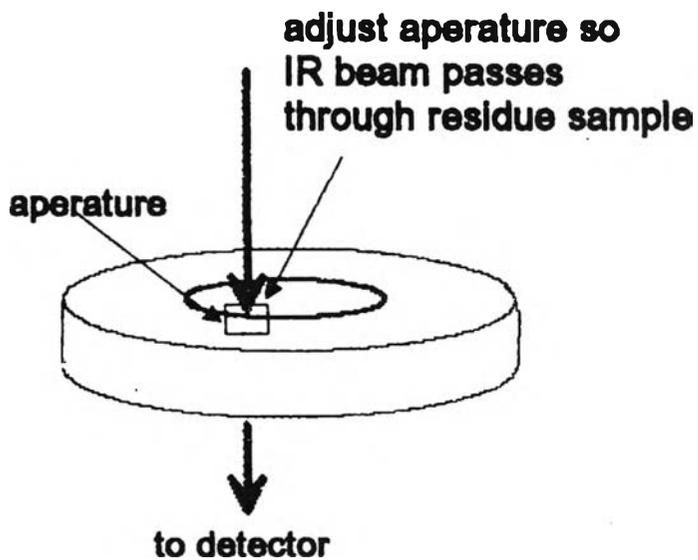


Figure 7a, Bench FTIR set-up



ต้นฉบับ หน้าขาดหาย

ภาคผนวก ง

CLEANROOM CONSUMABLE TEST METHOD & SPECIFICATION

## CLEANROOM CONSUMABLE TEST METHOD & SPECIFICATION

### 1. PURPOSE

To provide guidelines for analyses required in qualification of cleanroom consumable materials including sample preparation technique of each analysis.

Analyses required : The analyses required for qualification of each cleanroom consumable material are summarized as a guidelines as follow :

#### ภาคผนวก ง. Analysis Required

Cleanroom Consumable Materials	Analyses Required
Gloves, ambidextrous or nitride	IC, NVR, FT-IR, LPC and % talc, Tape test by SEM/EDX
Wipers	IC, NVR, FT-IR, LPC and APC
Swabs	IC, NVR, FT-IR and LPC
Bags	IC, NVR, FT-IR, LPC and Outgassing
Labels, Stickers or tapes	IC, NVR, FT-IR, Outgassing, 85/85 corrosion test
Cleanroom papers	IC, FT-IR, APC
Tweezers	IC, NVR, FT-IR, LPC
Plastics parts, all	IC, NVR, FT-IR, LPC, Outgassing, 85/85 corrosion test
Epoxy Dispensing Components	IC, NVR, FT-IR
Plastic wrap	IC, NVR, FT-IR, Outgassing and LPC
Pen or marker pen ink	IC, FT-IR, 85/85 corrosion test
Wrist strap	FT-IR
Cleanroom garment	IC, NVR, FT-IR, APC and ASTM

In additional materials which are introduced to MR manufacturing lines are required ESD property testing

## 2. SCOPE

For First Article Approval qualification and incoming qualification of Cleanroom Consumable Materials

## 3. APPLICABLE FORMS

N/A

## 4. APPLICABLE DOCUMENT

- 4.1 Micro balance instruction manual
- 4.2 DX – 100 Ion Chromatograph with SRS control operator's manual
- 4.3 AI – 450 Chromatography software user's guide
- 4.4 IR data manager reference manual
- 4.5 System 2000 FT-IR user's manual
- 4.6 Manually operated hydraulic presses P/N 15.011 and 25.011
- 4.7 Instructions JSM – 5800 LV scanning microscope
- 4.8 Operating manual SC7640 sputter coater
- 4.9 Instruction manual RV3, RV5, RV8 and RV12 Rotary Vane pumps
- 4.10 Document number 80 - 508107-000 : FT-IR Spectrometer operations
- 4.11 Document number 80 – 508109 – 000 : SEM/EDX operation, JSM – 5800LV with link ISIS
- 4.12 Document number 80 – 508110 – 000 : Operating procedure of DX – 100 Ion chromatography
- 4.13 Document number 80 – 500744 – 000 : PORM ( Parametric Ongoing Reliability Monitoring ) test
- 4.14 Document number 80 – 500714 – 000 : Non – Volatile Residue ( NVR ) Analysis
- 4.15 Document number 2092 – 001026 : Dynamic Headspace Outgassing procedure

## 5. RESPONSIBILITY

5.1 M&P Lab is responsible for ensuring that all testing is performed following this document.

5.2 Contamination Control Group is responsible for maintaining this document and all changes must be approved by Contamination Control Group or M&P Lab.

5.3 Others : SQE, Product or Process Engineer who will submit the sample to be tested is ensuring all samples is enough for the test. In addition, the samples must be submitted in original packaging or in the packaging that is approved by M&P Lab.

## 6. TERMS AND DEFINITION

IC	;	Ion Chromatography Analysis
NVR	;	Non – volatile residue determination
FTIR	:	Fourier Transformed Infrared Spectrometry
APC	:	Air particle count determination ( in drum tumbler )
LPC	:	Liquid particle count determination
SEM/EDX	:	Scanning electron microscope equipped with Energy dispersive X – Ray spectrometer

## 7. TOOLING / EQUIPMENT

### 7.1 Ionic Determination

7.1.1 Laminar flow hood class 100 or better

7.1.2 Oven with temperature control

7.1.3 Wide mouth glass or polyethylene bottles with caps

7.1.4 Graduated cylinder

- 7.1.5 Tweezers
  - 7.1.6 Desiccators with silica gel
  - 7.1.7 Pipette bulb
  - 7.1.8 Pipette
  - 7.1.9 Dionex Ion Chromatography
  - 7.1.10 Scissors or razor blade
  - 7.1.11 Auto sampler
- 7.2 No – Volatile Residue ( NVR ) Determination :
- 7.2.1 Laminar flow hood class 100 or better
  - 7.2.2 Chemical fume hood
  - 7.2.3 Analytical grade balance. 1.0 ug readability
  - 7.2.4 Oven with temperature control
  - 7.2.5 Hot plate
  - 7.2.6 Wide mouth glass bottles with caps
  - 7.2.7 .Graduated cylinder
  - 7.2.8 Tweezers
  - 7.2.9 Deciccators with silica gel
  - 7.2.10 Safety glasses
  - 7.2.11 Glass micro – syringe
  - 7.2.12 Fourier Transformed Infrared Spectrometer
  - 7.2.13 Scissors or razor blade
  - 7.2.14 Analytical grade balance, 0.1 mg readability or equivalent
- 7.3 Fourier Transformed Infrared Spectrometric Analysis :
- 7.3.1 Fourier Transformed Infrared Spectrometer
  - 7.3.2 Qwik Handi – press body
  - 7.3.3 Die set
  - 7.3.4 KBr pellet holder
  - 7.3.5 Spectro grade KBr powder
  - 7.3.6 Presslok demountable cell

- 7.3.7 KBr, NaCl crystal or equivalent
- 7.3.8 Microscope
- 7.3.9 10 ul or 50 ul glass syringe
- 7.3.10 Chemical fume hood
- 7.3.11 ATR crystal
  
- 7.4 SEM/EDX Analysis :
  - 7.4.1 Scanning Electron Microscope equipped with energy dispersive X – ray spectrometer
  - 7.4.2 Ultrasonic tank
  - 7.4.3 Vacuum pump
  
- 7.5 Particle Count Determination :
  - 7.5.1 Laser liquid particle counter
  - 7.5.2 Laminar flow hood
  - 7.5.3 Orbital shaker at 150rpm
  - 7.5.4 Tumbler drum
  - 7.5.5 Laser air particle counter
  - 7.5.6 Beaker for liquid particle counting
  - 7.5.7 Cleanroom garment
  - 7.5.8 Scissors or razor blade
  
- 7.6 Outgassing Analysis
  - 7.6.1 Gas chromatograph equipped with Mass Selective Detector and Headspace Sampler
  - 7.6.2 Capillary column
  - 7.6.3 Bubble flow meter and adapter
  - 7.6.4 Clamp
  
- 7.7 85/85 Corrosion test

7.7.1 Microscope with 1000X magnification minimum

7.7.2 Environmental chamber with temperature and humidity control

7.8 Scanning Electron Microscope with Energy Dispersive x- ray attachment

## 8. MATERIAL

### 8.1 Anion Determination :

8.1.1 Deionized water,  $\geq$  18 mega – ohms

8.1.2 Low residue, low chloride laboratory detergent

8.1.3 20 ml borosilicate glass scintillation vials with Teflon lined caps

8.1.4 Disposable plastic syringe

8.1.5 Pipette

8.1.6 Glove, nitrile

8.1.7 Whatman # 54 filter paper

8.1.8 Sodium Carbonate, AR grade or better

8.1.9 Sodium Bicarbonate, AR grade or better

8.1.10 Standard anion solution P/N : 56933 from Dionex or equivalent

### 8.2 Non – Volatile Residue ( NVR ) Determination :

8.2.1 Deionized water,  $\geq$  18 mega – ohms

8.2.2 Hexane, reagent or HPLC grade, or better

8.2.3 Isopropyl alcohol ( IPA ), reagent grade or better

8.2.4 Low residue, low chloride laboratory detergent

8.2.5 Glove, nitrile

8.2.6 Weighing pans

8.2.7 Evaporating dishes

### 8.3 Fourier Transformed Infrared Spectrometric Analysis and SEM/EDX Analysis

8.3.1 Deionized water,  $\geq$  18 mega – ohms

- 8.3.2 Hexane, reagent or HPLC grade or better
- 8.3.3 Low residue, low chloride laboratory detergent
- 8.3.4 Capillary tubes
- 8.3.5 Glove, nitrile
- 8.3.6 Membrane filter, 25 mm diameter with 0.20 micron pore size
- 8.3.7 Glass slides
- 8.3.8 Pasteur pipette
- 8.3.9 Evaporating dished

#### 8.4 Particle Count Determination :

- 8.4.1 Glove, nitrile
- 8.4.2 Head cover
- 8.4.3 Face mask
- 8.4.4 Deionized water ,  $\geq 18$  mega – ohms
- 8.4.5 Low residue, low chloride laboratory detergent

#### 8.5 Outgassing analysis

- 8.5.1 Glove, nitrile
- 8.5.2 Deionized water,  $\geq 18$  mega – ohms
- 8.5.3 Glass vials with cap
- 8.5.4 Septa
- 8.5.5 Helium gas ( 99.999% purity )
- 8.5.6 Isopropyl Alcohol, Chromatography grade
- 8.5.7 n – Hexane , Chromatography grade

### 9. SAFETY

- 9.1 In the event of an emergency, employee are directed to contact the site emergency number immediately.
- 9.2 Issued protective equipment shall be worn at all times in the work area.
- 9.3 Employees must notify their supervisor and site Medical Department immediately in the event of an accident.

## 10. GENERAL / OTHERS

### 10.1 Sample Preparation Procedures

10.1.1 Qualified Cleanroom gloves must be worn at all times ( Put on gloves, wash in DI water and air dry wipe dry with Alpha Wipe ( or cleanroom approved wipers ). Open the package containing test samples in a clean laminar flow hood. All solvents must be handled in a chemical fume hood.

10.1.2 Prepare required volume of IPA / Hexane solution for the consumable item to be tested.

10.1.3 Label prepared containers as sample or blank, if needed.

10.1.4 Place appropriate number of sample into a clean, dry sample bottle, preparing gloves, swabs, wipes and bags as items below

10.1.4.1 GLOVES – Cut slits with a clean razor blade along the fingers and gloves length.

10.1.4.2 SWABS – If the swabs are double ended, break it in half and count as 2.

10.1.4.3 WIPER – Fold and place whole wipes into the container. Do not cut wipers. For larger wipes use 1 whole wipe and adjust the volume of the solvent to maintain the same weight / volume ratio. Record sample weight.

10.1.4.4 EPOXY DISPENSING COMPONENTS – Cut larger epoxy dispensing components as needed to fit in the extraction container.

10.1.5 Carefully add the specified amount of DI water, IPA, IPA/Hexane

(50/50 vol% ) to the appropriate sample or blank container.  
Record the volume used as  $V_1$

## 10.2 Equipment preparation procedure

### 10.2.1 Ionic Determination

#### 10.2.1.1 Containers

- a) Detergent is not recommended for cleaning IPA can be used to remove oily films on containers,
- b) All containers ( extraction bottles, Glassware, plastic ware, pipette, etc. ) used for Ionic Chromatograph Analysis only.
- c) Rinse the interior of all containers that are to be used with DI water for at least 3 times.
- d) If possible, all containers should be left soaking in DI water whenever not in use.
- e) Rinse all containers and equipment to be used with DI water ( transfer the same water sample from one container to the next ). Test the collected sample on the Ion Chromatograph to ensure that no ionic contaminant is present. If contaminant is present, re-clean all the containers and equipment and run another blank until no ionic contaminants is detected.

#### 10.2.1.2 Eluent Preparation

- a) Dissolve 2.86 g. sodium carbonate and 0.25 g. sodium bicarbonate with 50 ml of  $\geq 18$  mega – ohms water in the clean beaker. Then dilute to 100 ml in clean volumetric flask. Cap and shake vigorously to mix well. Store in clean – labeled plastic bottle.
- b) Pipette 10 ml of solution from (1) into a Clean 1000 ml volumetric flask.
- c) Fill to the mark with  $\geq 18$  mega – ohms water. Cap and shake vigorously to mix well. The solution will be 2.7 mM sodium carbonate and 0.3 mM sodium bicarbonate.
- d) Degas the solution. Then pour the solution into eluent reservoir.

10.2.1.3 Standard Solution Preparation ( Refer Doc # 20-16077-000 )

10.2.1.4 Operating Procedure

- a) Set up the Ionic Chromatograph in accordance with the manufacturer's instruction.
- b) Equilibrate the system until a stable baseline is obtained.
- c) Calibrate daily using diluted standard.
- d) Dionex has linear regression software that automatically calculates the peak area for each anion detected. The correlation coefficient

should be greater than 0.95. If not, re – calibrate using new standards and / or event until correlation coefficient  $\geq 0.95$  is achieved.

- e) The first two samples are to be water blanks.
- f) Run a mid – range calibration standard every 15 injections and at the end of the run to check the drift.

#### 10.2.1.5 Calculation

$$C = \frac{(S - B) \times V}{A}$$

Where :

C = Concentration in  $\mu\text{g} / \text{cm}^2$

S = Anion concentration of the sample in ppm

B = Anion concentration of the water blank in ppm

V = Volume of water used for extraction in ml

A = Total surface area of the sample under test in  $\text{cm}^2$

Or

$$C = \frac{(S - B) \times V}{A}$$

Where

C = Concentration in  $\mu\text{g} / \text{pc}$

S = Anion concentration of the sample in ppm

B = Anion concentration of the water blank in ppm

V = Volume of water used for extraction in ml

N = Total sample under test in piece

10.2.2 Non – Volatile Residue ( NVR ) Determination

10.2.2.1 Container and Equipment

- a) Place beaker filled with n – Hexane on hot plate. Heat the solvent to 40 °C
- b) Thoroughly rinse all weighing pans with pre-heated Hexane. Bake pan in a 105 ± 5 °C oven for 30 minutes. Remove from the oven and cool to ambient temperature in the desiccator for 1 hour. Obtain the weight of weighing pans.
- c) Immediately before the NVR test begins, all beakers and containers to be used must be thoroughly rinsed with pre-heated Hexane.

10.2.2.2 Sample Extraction

- a) IPA, n – Hexane or IPA / n – Hexane will be used as an extraction solvent individually.
- b) The sample to be tested will be soaked in the appropriate amount of each individual extraction solvent for 1 hour at ambient temperature. Be sure that the solvent level should cover all sample surfaces and then close the sample container cap during extraction period. After extraction, remove the tested samples using clean tweezers and rinse

the sample over the container. Do the gravimetric analysis with analytical balance. The n – Hexane extracted residue of sample can later be transferred to KBr or NaCl pellet ( or crystal ) for FTIR analysis after completion of NVR analysis.

#### 10.2.2.3 Gravimetric Analysis

- a) Set up analytical balance in accordance with the manufacturer's manual.
- b) Weight the clean weighing pans. Record one for each sample as " S<sub>1</sub> " and a blank as " B<sub>1</sub> "
- c) Place the container from ( b2 ) and ( b3 ) on a hot plate. Allow the solvent to evaporate until the amount of solvent remaining is 1 – 2 ml. Do not allow the solvent to boil as bumping can occur, resulting in loss of extract.
- d) Transfer the solvent into a clean and pre – weigh weighing pan.
- e) Rinse all surface of the inside of the container with three 1 ml portion of additional solvent and pour the rinse into the weighing pan. Allow the solvent to evaporate until no visible solvent remains.
- f) Place the weighing pan with residue in  $105 \pm 5$  °C oven for 30 minutes.

- g) Remove the weighing pan with residue from oven and place in desiccators for 1 hour.
- h) Weight the weighing pan with residue. Record the reading " S<sub>1</sub> " for sample and " B<sub>1</sub> " for blank.
- i) Retain the NVR for further analysis by FTIR.

#### 10.2.2.4 Calculation

$$\text{NVR, } \mu\text{g / cm}^2 = \frac{( S_2 - S_1 ) - ( B_2 - B_1 ) \times 1,000,000 \times F}{A}$$

Where :

S<sub>2</sub> = Mass of sample weighing pan plus residue, g

S<sub>1</sub> = Mass of sample weighing pan, g

B<sub>2</sub> = Mass of blank weighing pan plus residue, g

B<sub>1</sub> = Mass of blank weighing pan, g

A = Total surface area of sample under test, cm

Or

$$\text{NVR, } \mu\text{g / pc} = \frac{( S_2 - S_1 ) - ( B_2 - B_1 ) \times 1,000,000}{N}$$

Where

S<sub>2</sub> = Mass of sample weighing pan plus residue, g

S<sub>1</sub> = Mass of sample weighing pan, g

B<sub>2</sub> = Mass of blank weighing pan plus residue, g

B<sub>1</sub> = Mass of blank weighing pan, g

N = Total sample under test, pc

F = Multiply Factor = Total extraction volume ( ml )

Evaporate volume ( ml )

### 10.2.3 Fourier Transformed Infrared Spectrometric Analysis

#### 10.2.3.1 Container

- a) Place beaker filled with n – Hexane on hot plate. Heat the solvent to 40 °C
- b) Immediately before the test begins, all beakers and containers to be used must be thoroughly rinsed with pre – heated Hexane.

#### 10.2.3.2 Sample Extraction

- a) Soak the sample to be tested in Hexane ( or IPA / Hexane ) solution for 1 hour at ambient temperature in an appropriate container. For parts with large surface area that could not be soaked in hexane solution properly ( such as box, HGA or HS A trays or equivalent ), use hexane solution rinse technique or hexane vapor rinse technique for better extraction.
- b) Place the container on the hot plate. Allow the solvent to evaporate until no visible solvent remains. Do not allow the solvent to boil as bumping can occur, resulting in loss of extract.

Note : The residue in the weighing pan after NVR analysis ( 10.1.2 ) can be retained for further FTIR analysis.

#### 10.2.3.3 Operation

- a) Set up FTIR in accordance with the manufacturer's instruction
- b) Rinse thoroughly the inside of the container with a few drop of Hexane.
- c) Use capillary tube to transfer the Hexane extract from the sample onto the KBr or NaCl pellet ( crystal ) one drop at a time allowing each drop to evaporate until all solvent is transferred.

( Using clean disposable pipettes, transfer less than one millimeter of Hexane from the beaker to the aluminum pan containing the sample residue from above. Swirl the Hexane in the pan and allow the solution to air evaporate to 10 – 50 ml. Transfer the solution with a clean syringe to an ATR crystal. Allow the solvent to evaporate. )

- d) Obtain the spectrum.
- e) Clean the pellet by rinsing with Hexane between each sample. The infrared reference spectrum of silicone oil, amide slipping agent and DI – isooctyl phthalate ester

10.2.4 SEM/EDX Analysis of %Magnesium Silicate Distribution  
( %TALC ) on gloves :

10.2.4.1 Sample preparation

10.2.4.2 Test to be conducted in class 100 laminar flow bench. Used the residue solution from LPC of gloves by filtration. Filter test solution using a 25 mm size of 0.20 micron membrane filter. Allow membrane filter to thoroughly dry in vacuum extractor prior to removing for SEM/EDX analysis. Attach membrane filter to SEM stub ( using colloidal graphite paste or carbon tape ) and bisect the surface area until 6 sectors of equal or approximately equal area are obtained. Perform a palladium coating and transfer the stub for SEM/EDX analysis.

10.2.4.3 Analysis technique

Perform SEM/EDX analysis on 5 Randomly selected particles for incoming and first article samples ( 4 sectors at 8 particles per sector, together with 9 particles on two remaining sectors ( in the opposite side ). Using 15 kV accelerating voltage, sigma to noise ration can be optimized by utilizing 1022 – 2000 count per second with a 20 – 30 % dead time and an acquisition time of 15 seconds minimum. Repeat above test two times to insure statistical uniformity.

## 10.2.5 Air Particle Count Determination

10.2.5.1 Sample preparation for Drum Tumbler test Wear full set of cleanroom garments ( shoe covers optional dependent upon existing requirement in area being used for test ). Set particle counter to cumulative mode, with range 0.3, 0.5, 0.7, 1.0, 2.0, 10.0. Turn on printer. For drum tumbler, set RPM control knob to 10 and confirm by manually counting revolutions per minute. Connect filtered air to feeder pipe of tumbler and set regulator at 10 Psi. Place an isokinetic probe  $0.25 \pm 0.25$  inch from the drum opening. Be sure airflow detector is positioned over the probe to deflect clean air from laminar flow bench, which could influence results.

### 10.2.5.2 Operating procedure

- a) Turn on the tumbler and filtered air
- b) Turn on counter
- c) Operate counter until 5 minutes cumulative count is under 100 particle per cu.ft. Log in baseline particle count.
- d) Stop counter and turn off tumbler and air
- e) Cut outer – protective bag containing samples with clean sclape or razor blade.
- f) Place appropriate number of samples to be tested into drum. ( Note : Garments

should be in snapped or zipped closed condition during test ).

- g) Turn on tumbler and air supply. Operate particle counter for 5 minutes ( auto run ) and record the counts.
- h) Turn off particle counter, tumbler, and, air supply.
- i) Record highest one minute reading during 5 minutes of testing. Calculate particle count ( cumulative ) by the following formula.

Particle count = ( Sample Count – Baseline Count ) / number of pieces tested.

## 10.2.6 Liquid Particle Count Determination

### 10.2.6.1 Operation procedure

- a) Turn on particle counter and batch sampler.
- b) Adjust size ranges to 0.5, 0.6, 0.7, 0.8, 0.9, 1.0, 1.5, 2.0, 3.0, 5.0, 10.0, and > 20.0 microns. Threshold values ( in mV ) indicated in operating manual must match the serial number of particle counter being used.
- c) Set the flow rate of batch sampler at 80 ml/min. Test to be conducted in Class 100 laminar flow bench.
- d) Fill LPC beaker with desired amount of DI water.

- e) Rinse beaker with DI water. Beaker should now be clean enough to continue tests. Clean beaker should have a baseline count of less than 10% of sample.

#### 10.2.7 85/85 Environment Testing Method

- 10.2.7.1 Preparation of samples for 85/85 Test Chamber
- 10.2.7.2 To hold samples in the chamber, use clean glass petri dish.
- 10.2.7.3 Need to do control petri dish by using 5 slider place in clean glass petri dish and put in the chamber every sample testing. Chamber operation, slider inspection criteria and SEM/EDX testing of optical rejects is detailed in Doc# 80-500744-000.

ภาคผนวก จ.

ค่า LPC ของแต่ละทางเลือจาก Analytical Service Lab

## XXX DIGITAL (BangPa-In) AS Lab Report

REQUEST NUMBER: 05-2307A

**Date Completed:** February 18' 2005  
**Requester:** Jakkrod N. / Contamination, Ext.6217  
**Date received:** February 11' 2005  
**Samples:** To do LPC test and Filter after LPC extraction on Scorpio HSA3x for WW#34'05 by use current cleaning process

### 1. Background:

Scorpio HSA3x P/N# 2063-702583-1F3 samples for LPC test from current cleaning process were submitted to monitor LPC test and Filter after LPC extraction.

Sample	Product	Surface area (cm <sup>2</sup> )
HSA 3X	2.5 inch	HSA 3X = 43.78 HSA 3X w/o bearing and flex gasket = 41.96

Sample	HSA Subcomponents information		
	Flex	Suspension	CFA
Scorpio HSA 3X	Sumitomo	NHK	Min Aik

### 2. Techniques of analysis:

- 2.1 The samples were analysis for "*Particle Measurement by Liquid Particle Count*" in accordance to Document No# 92-004230, Rev. AD.
- 2.2 Followed "*XXX HDA Component Cleanliness Specification*" Doc. No. 96-004575, Rev. AF and prelim AG (Feb 01'05).
- 2.3 SEM/EDX analysis (using accelerate voltage 15 kV) for Filter test  
Pd coating technique using  $1 \times 10^{-3}$  mbar  
Note: SEM/EDX analysis was randomly analyzed 50 particles on each sample.

### 3. Analysis results:

#### Table #1: LPC results:

Scorpio HSA3x test for current cleaning process WW#34-05

Particle Size (um)	Count/cm2			Average	Spec. Rev. AF and AG (Prelim)	Status
	Trial#1	Trial#2	Trial#3			
0.5	7,660	7,372	7,864	7.632	6,000	Failed
1	3,353	1,442	1,828	2,208	NA	Passed
2	1,291	315	530	712	NA	Passed

Denote: \*NA = Not Available

**Table #2: SEM/EDX results**

Elemental composition	Particle size (mm)			
	Scorpio HSA3x test WW34			
	<1um	1-5um	5-10um	>10um
C based	-	-	-	-
C/F	-	-	-	-
Mg/Si/O	-	-	-	-
S	-	-	-	-
Fe/Cr (SST400s)	-	-	-	-
Fe/Cr/Ni (SST300s)	-	-	-	-
Al/Si	-	-	-	-
Al/Si + other element	-	-	-	-
Al/Si/O	-	1	-	-
Si/Ca/Al/O	-	1	-	-
Al/O	-	-	-	-
Al + other element	-	2	-	-
Si/O	-	7	-	-
Si/O + other element	-	14	-	-
Si/K/O	-	-	-	-
Sn/O	-	13	-	-
S/O	-	-	-	-
Ca + other element	-	1	-	-
Cr/O	-	1	-	-
Ca/S	-	-	-	-
Cl/S	-	-	-	-
Cu/Zn (Al)	-	1	-	-
Fe + other element	-	2	-	-
Other	-	7	-	-
<b>Total particle count</b>	<b>50</b>			

**Conclusion:**

The submitted Scorpio HSA3x samples test WW#34'05 failed LPC test at 0.5um size per specification limit for revision AF and prelim AG (Feb 01'05).

Filter after LPC extraction by SEM/EDX analysis found major particles elemental were Si/O + other element and Sn/O.

Analyzed by: Prasert P.  
(Prasert P.)

Approved by: Prasert P. (For)  
(Yongyut W.)

## XXX DIGITAL (BangPa-In) AS Lab Report

REQUEST NUMBER: 05-2308A

**Date Completed:** February 18' 2005  
**Requester:** Jakkrod N. / Contamination, Ext.6217  
**Date received:** February 11' 2005  
**Samples:** To do LPC test and Filter after LPC extraction on Scorpio HSA3x for WW#34'05 by use alternative a1 for cleaning process

### 1. Background:

Scorpio HSA3x P/N# 2063-702583-1F3 samples for LPC test from alternative a1 for cleaning process ( components cleaning by aqueous or solvent system ) were submitted to monitor LPC test and Filter after LPC extraction.

Sample	Product	Surface area (cm <sup>2</sup> )
HSA 3X	2.5 inch	HSA 3X = 43.78 HSA 3X w/o bearing and flex gasket = 41.96

Sample	HSA Subcomponents information		
	Flex	Suspension	CFA
Scorpio HSA 3X	Sumitomo	NHK	Min Aik

### 2. Techniques of analysis:

- 2.1 The samples were analysis for "*Particle Measurement by Liquid Particle Count*" in accordance to Document No# 92-004230, Rev. AD.
- 2.2 Followed "*XXX HDA Component Cleanliness Specification*" Doc. No. 96-004575, Rev. AF and prelim AG (Feb 01'05).
- 2.3 SEM/EDX analysis (using accelerate voltage 15 kV) for Filter test  
Pd coating technique using  $1 \times 10^{-3}$  mbar  
Note: SEM/EDX analysis was randomly analyzed 50 particles on each sample.

### 3. Analysis results:

#### Table #1: LPC results:

Scorpio HSA3x test for alternative a1 WW#34-05

Particle Size (um)	Count/cm <sup>2</sup>			Average	Spec. Rev. AF and AG (Prelim)	Status
	Trial#1	Trial#2	Trial#3			
0.5	5,324	5,130	5,236	5,230	6,000	Passed
1	2,124	2,351	2,243	2,239	NA	Passed
2	986	854	882	907	NA	Passed

Denote: \*NA = Not Available

**Table #2: SEM/EDX results**

Elemental composition	Particle size (mm)			
	Scorpio HSA3x test a1 WW34			
	<1um	1-5um	5-10um	>10um
C based	-	-	-	-
C/F	-	-	-	-
Mg/Si/O	-	-	-	-
S	-	-	-	-
Fe/Cr (SST400s)	-	-	-	-
Fe/Cr/Ni (SST300s)	-	-	-	-
Al/Si	-	-	-	-
Al/Si + other element	-	-	-	-
Al/Si/O	-	1	-	-
Si/Ca/Al/O	-	2	-	-
Al/O	-	-	-	-
Al + other element	-	1	-	-
Si/O	-	5	-	-
Si/O + other element	-	10	-	-
Si/K/O	-	-	-	-
Sn/O	-	8	-	-
S/O	-	-	-	-
Ca + other element	-	1	-	-
Cr/O	-	1	-	-
Ca/S	-	-	-	-
Cl/S	-	-	-	-
Cu/Zn (Al)	-	-	-	-
Fe + other element	-	1	-	-
Other	-	5	-	-
<b>Total particle count</b>	<b>35</b>			

**Conclusion:**

The submitted Scorpio HSA3x samples test a1 ( component cleaning by aqueous or solvent system ) WW#34'05 Passed LPC test at 0.5um size per specification limit for revision AF and prelim AG (Feb 01'05).

Filter after LPC extraction by SEM/EDX analysis found major particles elemental were Si/O + other element and Sn/O.

Analyzed by: Prasert P.  
(Prasert P.)

Approved by: Prasert P. (For)  
(Yongyut W.)

## XXX DIGITAL (BangPa-In) AS Lab Report

REQUEST NUMBER: 05-2309A

**Date Completed:** February 18' 2005  
**Requester:** Jakkrod N. / Contamination, Ext.6217  
**Date received:** February 11' 2005  
**Samples:** To do LPC test and Filter after LPC extraction on Scorpio HSA3x for WW#34'05 by use alternative a2 for cleaning process

### 1. Background:

Scorpio HSA3x P/N# 2063-702583-1F3 samples for LPC test from alternative a2 for cleaning process ( component cleaning by solvent only ) were submitted to monitor LPC test and Filter after LPC extraction.

Sample	Product	Surface area (cm <sup>2</sup> )
HSA 3X	2.5 inch	HSA 3X = 43.78 HSA 3X w/o bearing and flex gasket = 41.96

Sample	HSA Subcomponents information		
	Flex	Suspension	CFA
Scorpio HSA 3X	Sumitomo	NHK	Min Aik

### 2. Techniques of analysis:

- 2.1 The samples were analysis for "*Particle Measurement by Liquid Particle Count*" in accordance to Document No# 92-004230, Rev. AD.
- 2.2 Followed "*XXX HDA Component Cleanliness Specification*" Doc. No. 96-004575, Rev. AF and prelim AG (Feb 01'05).
- 2.3 SEM/EDX analysis (using accelerate voltage 15 kV) for Filter test  
Pd coating technique using  $1 \times 10^{-3}$  mbar  
Note: SEM/EDX analysis was randomly analyzed 50 particles on each sample.

### 3. Analysis results:

#### Table #1: LPC results:

Scorpio HSA3x test for alternative a2 WW#34-05

Particle Size (um)	Count/cm2			Average	Spec. Rev. AF and AG (Prelim)	Status
	Trial#1	Trial#2	Trial#3			
0.5	4,235	4,680	4,775	4,563	6,000	Passed
1	2,168	2,025	1,963	2,052	NA	Passed
2	762	698	724	728	NA	Passed

**Denote:** \*NA = Not Available

**Table #2: SEM/EDX results**

Elemental composition	Particle size (mm)			
	Scorpio HSA3x test WW34			
	<1um	1-5um	5-10um	>10um
C based	-	-	-	-
C/F	-	-	-	-
Mg/Si/O	-	-	-	-
S	-	-	-	-
Fe/Cr (SST400s)	-	-	-	-
Fe/Cr/Ni (SST300s)	-	-	-	-
Al/Si	-	-	-	-
Al/Si + other element	-	-	-	-
Al/Si/O	-	1	-	-
Si/Ca/Al/O	-	1	-	-
Al/O	-	-	-	-
Al + other element	-	2	-	-
Si/O	-	5	-	-
Si/O + other element	-	8	-	-
Si/K/O	-	-	-	-
Sn/O	-	7	-	-
S/O	-	-	-	-
Ca + other element	-	1	-	-
Cr/O	-	1	-	-
Ca/S	-	-	-	-
Cl/S	-	-	-	-
Cu/Zn (Al)	-	1	-	-
Fe + other element	-	2	-	-
Other	-	3	-	-
<b>Total particle count</b>	<b>32</b>			

**Conclusion:**

The submitted Scorpio HSA3x samples test ( component cleaning by solvent system only ) WW#34'05 Passed LPC test at 0.5um size per specification limit for revision AF and prelim AG (Feb 01'05).

Filter after LPC extraction by SEM/EDX analysis found major particles elemental were Si/O + Sn/O and other element.

Analyzed by: Prasert P.  
(Prasert P.)

Approved by: Prasert P. (For)  
(Yongyut W.)

**XXX DIGITAL (BangPa-In)**  
**AS Lab Report**

REQUEST NUMBER: 05-2310A

**Date Completed:** February 18' 2005  
**Requester:** Jakkrod N. / Contamination, Ext.6217  
**Date received:** February 11' 2005  
**Samples:** To do LPC test and Filter after LPC extraction on Scorpio HSA3x for WW#34'05 by use alternative a3 for cleaning process

**1. Background:**

Scorpio HSA3x P/N# 2063-702583-1F3 samples for LPC test from alternative a3 cleaning process ( component cleaning by suppliers ) were submitted to monitor LPC test and Filter after LPC extraction.

Sample	Product	Surface area (cm <sup>2</sup> )
HSA 3X	2.5 inch	HSA 3X = 43.78 HSA 3X w/o bearing and flex gasket = 41.96

Sample	HSA Subcomponents information		
	Flex	Suspension	CFA
Scorpio HSA 3X	Sumitomo	NHK	Min Aik

**2. Techniques of analysis:**

- 2.1 The samples were analysis for "*Particle Measurement by Liquid Particle Count*" in accordance to Document No# 92-004230, Rev. AD.
- 2.2 Followed "*XXX HDA Component Cleanliness Specification*" Doc. No. 96-004575, Rev. AF and prelim AG (Feb 01'05).
- 2.3 SEM/EDX analysis (using accelerate voltage 15 kV) for Filter test  
Pd coating technique using  $1 \times 10^{-3}$  mbar  
Note: SEM/EDX analysis was randomly analyzed 50 particles on each sample.

**3. Analysis results:**

**Table #1: LPC results:**

Scorpio HSA3x test for alternative a3 WW#34-05

Particle Size (um)	Count/cm <sup>2</sup>			Average	Spec. Rev. AF and AG (Prelim)	Status
	Trial#1	Trial#2	Trial#3			
0.5	5,859	5,998	6,034	5,964	6,000	Passed
1	2,578	2,432	2,216	2,049	NA	Passed
2	1,221	1,009	987	1,072	NA	Passed

**Denote:** \*NA = Not Available

**Table #2: SEM/EDX results**

Elemental composition	Particle size (mm)			
	Scorpio HSA3x test WW34			
	<1um	1-5um	5-10um	>10um
C based	-	-	-	-
C/F	-	-	-	-
Mg/Si/O	-	-	-	-
S	-	-	-	-
Fe/Cr (SST400s)	-	-	-	-
Fe/Cr/Ni (SST300s)	-	-	-	-
Al/Si	-	-	-	-
Al/Si + other element	-	-	-	-
Al/Si/O	-	1	-	-
Si/Ca/Al/O	-	1	-	-
Al/O	-	-	-	-
Al + other element	-	2	-	-
Si/O	-	5	-	-
Si/O + other element	-	18	-	-
Si/K/O	-	-	-	-
Sn/O	-	11	-	-
S/O	-	-	-	-
Ca + other element	-	1	-	-
Cr/O	-	1	-	-
Ca/S	-	-	-	-
Cl/S	-	-	-	-
Cu/Zn (Al)	-	1	-	-
Fe + other element	-	2	-	-
Other	-	5	-	-
<b>Total particle count</b>	<b>48</b>			

**Conclusion:**

The submitted Scorpio HSA3x samples test ( component cleaning by suppliers ) WW#34'05 Passed LPC test at 0.5um size per specification limit for revision AF and prelim AG (Feb 01'05).

Filter after LPC extraction by SEM/EDX analysis found major particles elemental were Si/O + Sn/O and other element.

Analyzed by: Prasert P.  
(Prasert P.)

Approved by: Prasert P. (For)  
(Yongyut W.)

### ประวัติผู้เขียนวิทยานิพนธ์

นางสาวเกสัช ชัยพงษ์ เกิดเมื่อวันที่ 13 มกราคม 2518 ที่จังหวัดชัยภูมิ สำเร็จ การศึกษาระดับปริญญาวิศวกรรมศาสตรบัณฑิต จากภาควิชาวิศวกรรมเคมี คณะ วิศวกรรมศาสตร์ มหาวิทยาลัยเทคโนโลยีสุรนารี เมื่อปี 2540 และได้เข้าศึกษาต่อในระดับ ปริญญาโท สาขาวิชาวิศวกรรมอุตสาหการ คณะวิศวกรรมศาสตร์ จุฬาลงกรณ์ มหาวิทยาลัย เมื่อปี 2546

