

# **MILLENNIUM APPLICATION**

## **BRIEF**

### **TOPIC: Low Level Anion Analysis in Disk Drive Heads by CIA**

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**Date: 08/02/95**

#### **Introduction:**

Analysis of low level anionic impurities in high purity water extracts of hard disk drive heads was done using CIA. Analysis of these devices is important in accessing the cleanliness of these heads after cleaning. Levels of the ions (i.e. Cl, SO<sub>4</sub>, etc.) are expected to be low. Figure 1 is an electropherogram of a standard at 2 ppb Cl and 4 ppb SO<sub>4</sub>, NO<sub>3</sub>, Oxalate, and HPO<sub>4</sub>. Samples of these hard disk heads was done using a modified chromate electrolyte. These heads are very small devices (only a few millimeters in size) and were extracted in lots of 10 into 3.0 mL of high purity water. To calculate the amount of contamination present on each head, automatically, custom calculations were used in Millennium. The first custom calculation, **Corrected Amount (ppb)**, subtracts the amount of each ion found in a sample blank (figure 2) from each run using the CCONST1 field in the processing method. This corrects for any contamination from the sample vessels used for the extraction. Next, the **Extraction Volume** and **Number of Parts** fields are used to calculate the **Amount per Part (ng/part)** using the following calculation:

$$\text{Corrected Amount X (Volume extracted/Number of parts)}$$

The analyst simply fills in the **Number of Parts** and **Extraction Volume** fields when loading the samples and Millennium will calculate the amount per part.

Figures 3 and 4 are of two different types of heads. As you can see fairly low levels of chloride and sulfate as well as nitrate and oxalate were found in the heads. Also the corrected amount and amount per part were reported in the reports. Total analysis times of less than 5 minutes was possible.

#### **EXPERIMENTAL:**

##### **Cation Analysis**

System:	<b>Q4000E</b>
Electrolyte:	Modified Chromate/OFM pH=11.2
Capillary:	75um X 60cm
Injection:	Electromigration -5kV, 45 seconds.
Run Voltage:	-15kV
Detection:	Indirect UV at 254nm
Data:	<b>Millennium 2010 Chromatography Manager, Ver. 2.1 with CIA Option *</b>

### **Sample preparation**

For this analysis contamination prevention is important. For this reason non-talc gloves were worn as well as plastic, pre-soaked, volumetric flask used for standard preparations. Samples were extracted in 3.0 mL of high purity water and heated for 1 hour at 80 degrees Celsius per the customer SOP. NaOS was added to all standards and samples as an electromigrative additive. A water and sample blank were run. Standards were run at 4 levels (2-16 ppb) with correlation coefficients ( $r^2$ ) of 0.99x with x being a value of 3 or better. Time corrected peak area was used for calibration and quantitation

### **FIGURES:**

Figure 1: Electropherogram of a low level anion standard

Figure 2: Electropherogram of sample blank

Figure 3: Electropherogram of cleaned heads

Figure 4: Electropherogram of K5 cleaned heads

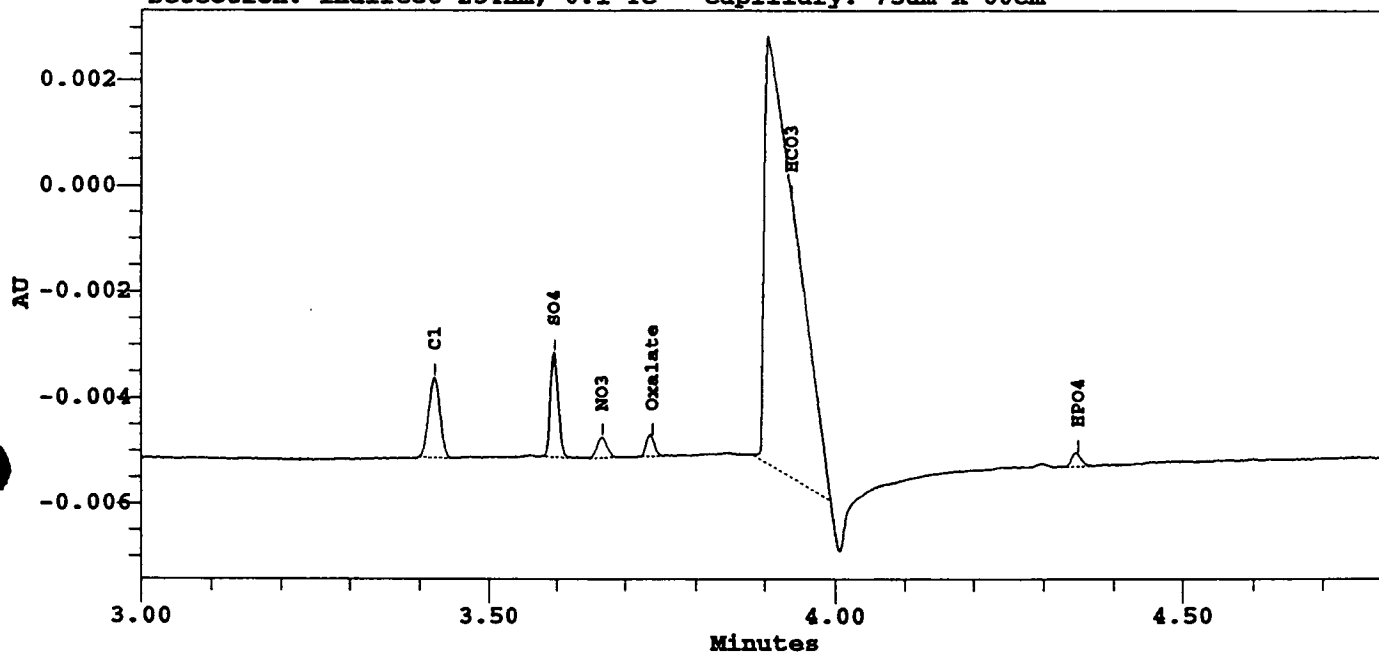


## Northern Kentucky Laboratory

## Sample Information

Project Name: Electronics\_Low01  
SampleName: Std 2  
Vial: 3  
Injection: 1  
Channel: SATIN  
Date Acquired: 06/26/95 04:10:08 PM  
Scale Factor: 1.00  
Acq Meth Set: ppb\_Anions  
Processing Method: ppb\_Anion02b  
Sample Type: Electromigr Standard  
Volume: 0.00  
Run Time: 5.0 min  
Date Processed: 06/28/95 04:21:34 PM  
Dilution: 1.00000

SampleName: Std 2 RunVoltage: -15kV  
InjectionMode: Electromigration 5kV, 45 sec  
Detection: Indirect 254nm, 0.1 TC Capillary: 75um X 60cm



## Peak Results

#	Name	Migration Time (min)	Area (uV*sec)	Time Corr. Area	Height (uV)	Amount (ppb)
1	Cl	3.423	1695	495.13	1517	2.000
2	SO4	3.597	1566	435.40	1985	4.000
3	NO3	3.667	383	104.59	390	4.000
4	Oxalate	3.739	328	87.82	413	4.000
5	HCO3	3.939	25393	6446.32	8081	
6	HPO4	4.350	231	53.16	256	4.000



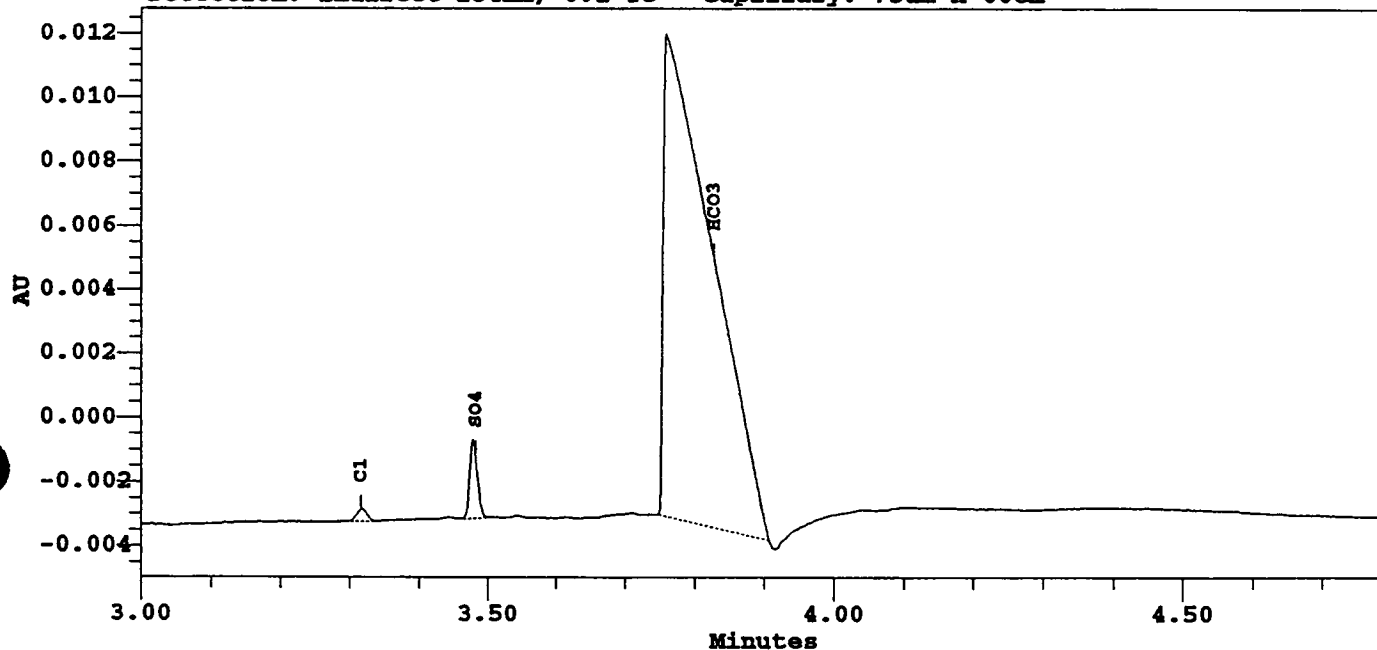
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## Sample Information

Project Name: Electronics\_Low01  
SampleName: Sample Blank  
Vial: 1  
Injection: 1  
Channel: SATIN  
Date Acquired: 06/27/95 05:01:50 PM  
Scale Factor: 1.00  
Acq Meth Set: ppb\_Anions  
Processing Method: ppb\_Anion01

Sample Type: Electromigr Unknown  
Volume: 0.00  
Run Time: 4.8 min  
Date Processed: 07/20/95 12:42:54 PM  
Dilution: 1.00000

SampleName: Sample Blank RunVoltage: -15kV  
InjectionMode: Electromigration, 5 kV, 45 sec  
Detection: Indirect 254nm, 0.1 TC Capillary: 75um X 60cm



## Peak Results

#	Name	Migration Time (min)	Area (uV*sec)	Time Corr. Area	Height (uV)	Amount (ppb)
1	Cl	3.316	371	111.89	375	0.919
2	SO4	3.483	1900	545.48	2456	6.401
3	HCO3	3.827	73438	19186.91	15078	

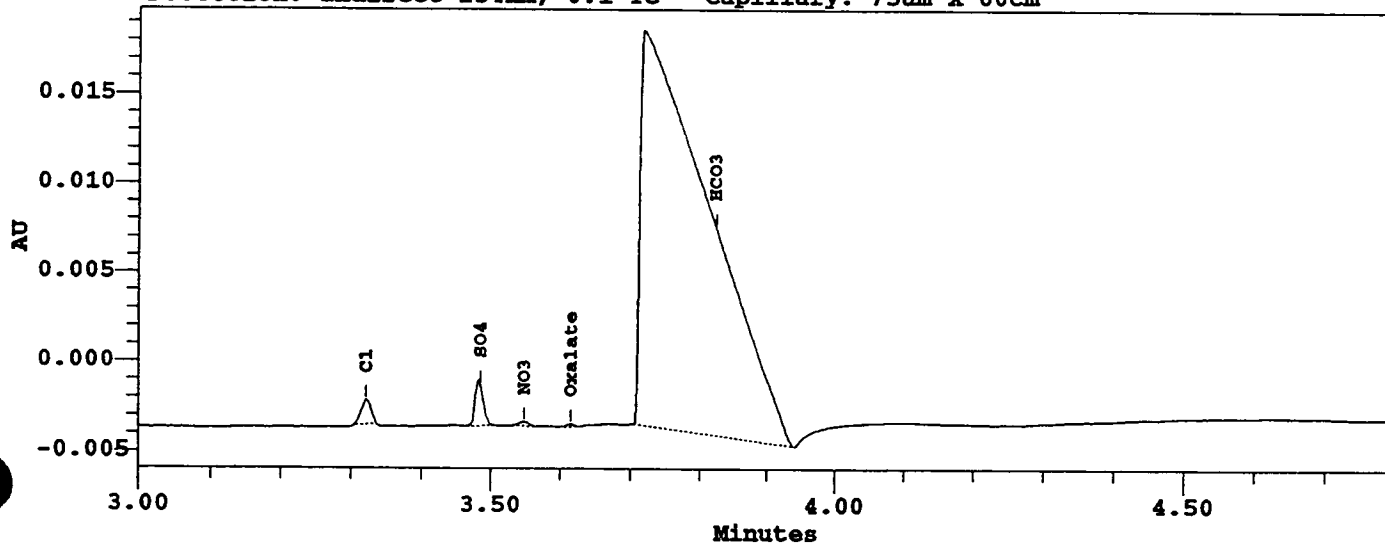


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## Sample Information

Project Name: Electronics\_Low01  
SampleName: 10 Heads-Cleaned-3mL  
Vial: 2  
Injection: 1  
Channel: SATIN  
Date Acquired: 06/27/95 05:09:56 PM  
Scale Factor: 1.00  
Acq Meth Set: ppb\_Anions  
Processing Method: ppb\_Anion01  
Sample Type: Electromigr Unknown  
Volume: 0.00  
Run Time: 4.8 min  
Date Processed: 07/20/95 01:11:04 PM  
Dilution: 1.00000

SampleName: 10 Heads-Cleaned-3mL RunVoltage: -15kV  
InjectionMode: Electromigration, 5 kV, 45 sec  
Detection: Indirect 254nm, 0.1 TC Capillary: 75um X 60cm



## Peak Results

#	Name	Migration Time (min)	Area (uV*sec)	Time Corr. Area	Height (uV)	Amount (ppb)
1	Cl	3.321	1307	393.60	1337	2.758
2	SO4	3.485	1979	567.72	2530	7.002
3	NO3	3.548	214	60.25	223	1.512
4	Oxalate	3.616	84	23.27	142	0.756
5	HCO3	3.824	154990	40533.59	22124	

## Peak Results

#	Corrected Amount (ppb)	Amount per Part (ng/part)
1	1.858	0.557
2	0.602	0.181
3	1.512	0.454
4	0.756	0.227
5		



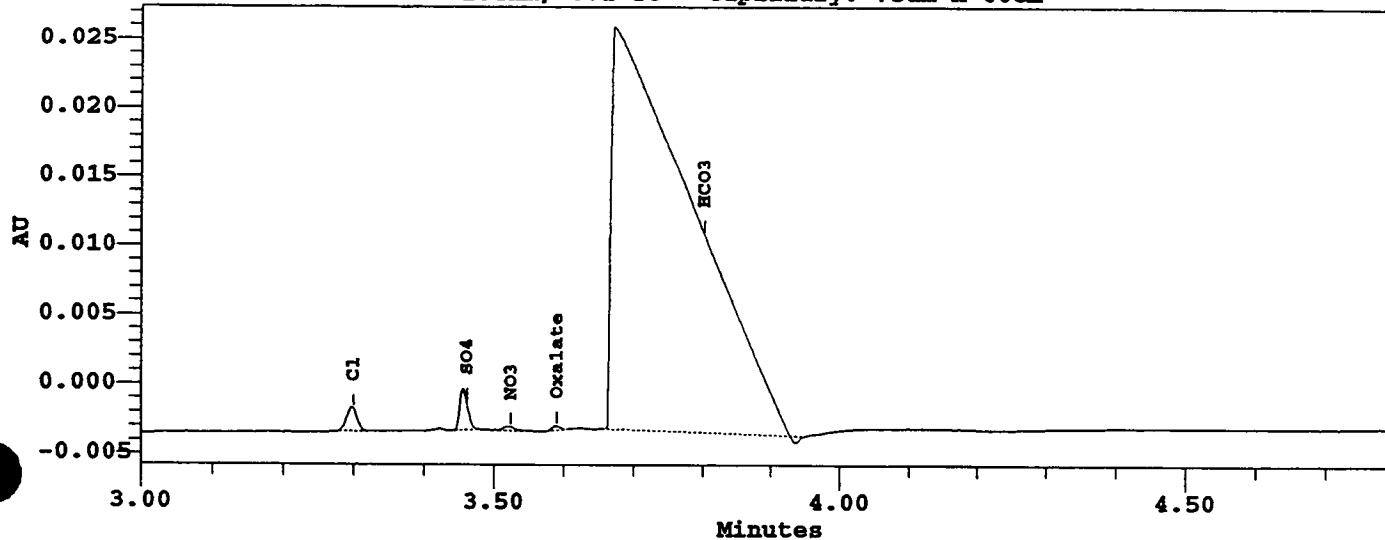
## Northern Kentucky Laboratory

## Sample Information

Project Name: Electronics\_Low01  
SampleName: 10 Heads-K5-3mL  
Vial: 4  
Injection: 1  
Channel: SATIN  
Date Acquired: 06/27/95 05:26:08 PM  
Scale Factor: 1.00  
Acq Meth Set: ppb\_Anions  
Processing Method: ppb\_Anion01

Sample Type: Electromigr Unknown  
Volume: 0.00  
Run Time: 4.8 min  
Date Processed: 07/20/95 01:18:40 PM  
Dilution: 1.00000

SampleName: 10 Heads-K5-3mL RunVoltage: -15kV  
InjectionMode: Electromigration, 5 kV, 45 sec  
Detection: Indirect 254nm, 0.1 TC Capillary: 75um X 60cm



## Peak Results

#	Name	Migration Time (min)	Area (uV*sec)	Time Corr. Area	Height (uV)	Amount (ppb)
1	Cl	3.300	1725	522.72	1691	3.601
2	SO4	3.461	2275	657.28	2951	9.421
3	NO3	3.524	267	75.90	277	2.062
4	Oxalate	3.590	261	72.64	343	2.329
5	HCO3	3.803	235211	61857.01	29121	

## Peak Results

#	Corrected Amount (ppb)	Amount per Part (ng/part)
1	2.701	0.810
2	3.021	0.906
3	2.062	0.619
4	2.329	0.699
5		