

MILLENNIUM APPLICATION

BRIEF

TOPIC: Capillary Ion Analysis (CIA) of Cations in HF based Etchants

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Date: 01/3/95

Introduction:

Analysis of cations in HF based etchants was done. Samples of three HF based etchants were diluted and analyzed using the Q4000e CIA. All samples were diluted (10uL-100mL) with Milli-Q water. Plastic volumetric flask were used for all dilution's and sample prep. Analysis of the these mixes showed varying levels of cations present. Figure 1 is an electropherogram of a cation standard. Figure 2 is an electropherogram of a quartz etchant. The primary composition of it is a mix of HF and NH_4F . As shown in figure 2 the sample contained a high level of NH_4 (as expected) as well as lower levels of potassium, calcium, and sodium. The other two etchants were composed of differing levels of HF and NMP (N-methyl-2-pyrrolidinone). NMP is neutral so it would not be seen using this method. Low levels of NH_4 , K, and Na were found in the samples.

EXPERIMENTAL:

Cation Analysis

System:	Q4000E
Electrolyte:	UV Cat-2 chemistry
Capillary:	75um X 60cm
Injection:	Hydrostatic, 30 seconds
Run Voltage:	+20kV
Detection:	Indirect UV at 185nm
Data:	Millennium 2010 Chromatography Manager, Ver. 2.1 with CIA Option

FIGURES:

Figure 1: Electropherogram of cation standard

Figure 2: Electropherogram of quartz etchant (NH_4 :HF)

Figure 3: Electropherogram of intermediate etchant (NMP:HF)

Figure 4: Electropherogram of final etchant (HF:NMP)

Report Method: Cations_A

Version: 2.10

For Sample: Std 2

Vial: 6

Injection: 1

Channel: SATIN

Proc Chan: SATIN

Processed: 12/28/94 02:31:51 PM

Channel Descr:

Northern Kentucky Laboratory

Sample Information

Project Name: CIA_Electronics_02

SampleName: Std 2

Vial: 6

Sample Type: Standard

Injection: 1

Volume: 30.00

Channel: SATIN

Run Time: 6.0 min

Date Acquired: 12/27/94 10:45:54 AM

Date Processed: 12/28/94 02:31:51 PM

Scale Factor: 1.00

Dilution: 1.00000

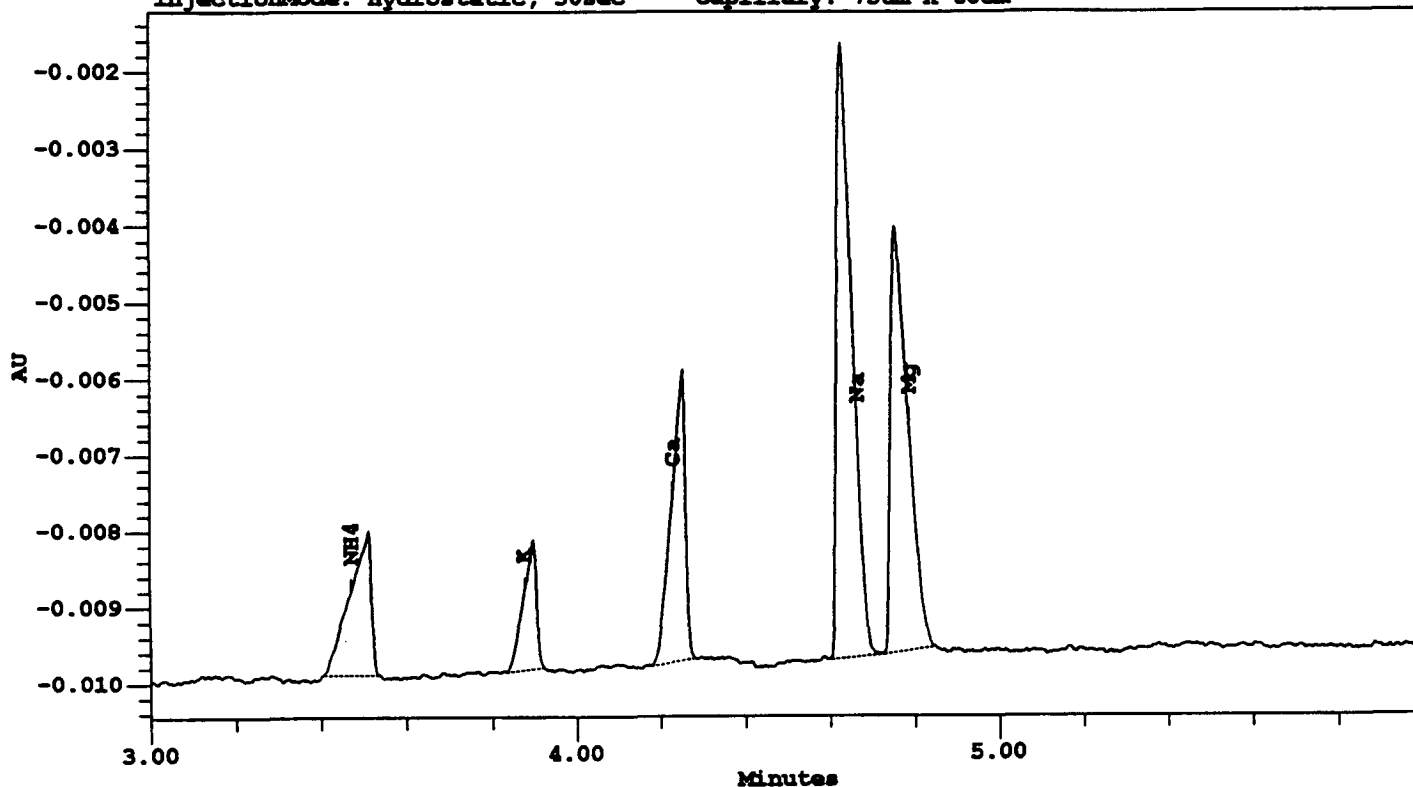
Acq Meth Set: CIA_Cations

Processing Method: Cations_02

SampleName: Std 2 Electrolyte: 1.2mM UV Cat-2/3 mM Tropolone/2 mM Crown

RunVoltage: +20 kV Detection: Indirect 185nm, 0.3 TC

InjectionMode: Hydrostatic, 30sec Capillary: 75um X 60cm



Peak Results

#	Name	Migration Time (min)	Area (uV*sec)	Time Corr. Area	Height (uV)	Amount-ppm
1	NH4	3.472	6598	1900.47	1898	1.000
2	K	3.878	3655	942.42	1693	1.000
3	Ca	4.228	8770	2074.10	3822	1.000
4	Na	4.665	20539	4402.70	8057	2.000
5	Mg	4.787	16198	3383.98	5601	1.000

Report Method: Cations_A

Version: 2.10

For Sample: Quartz Etchant

Vial: 13

Injection: 1

Channel: SATIN

Proc Chan: SATIN

Processed: 12/28/94 02:33:23 PM

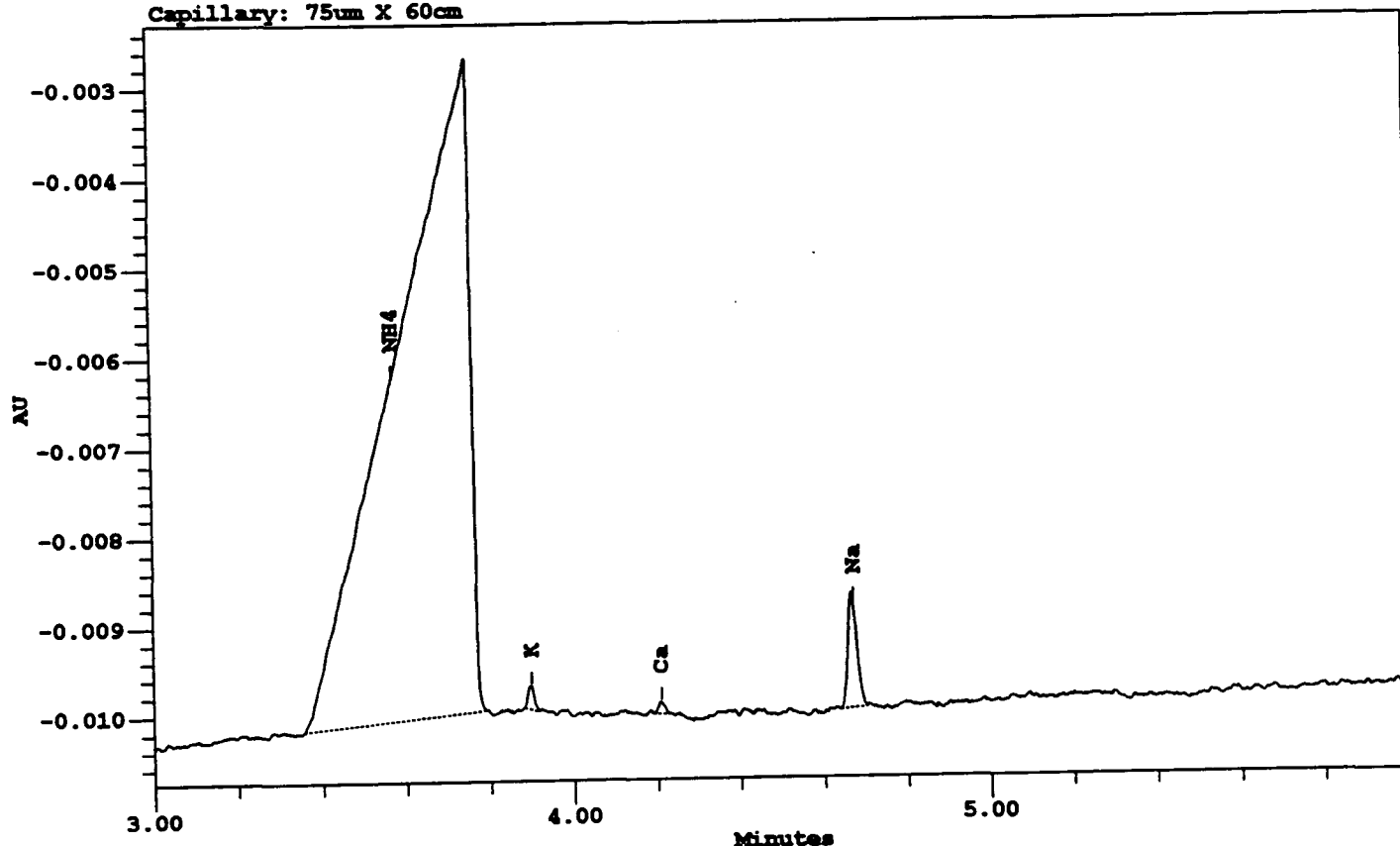
Channel Descr:

Northern Kentucky Laboratory
Sample Information

Project Name: CIA_Electronics_02
SampleName: Quartz Etchant (0.01-100mL)
Vial: 13
Injection: 1
Channel: SATIN
Date Acquired: 12/27/94 11:02:20 AM
Scale Factor: 1.00
Acq Meth Set: CIA_Cations
Processing Method: Cations_02

Sample Type: Unknown
Volume: 30.00
Run Time: 8.0 min
Date Processed: 12/28/94 02:33:23 PM
Dilution: 10000.00000

SampleName: Quartz Etchant (0.01-100mL)
Electrolyte: 1.2mM UV Cat-2/3 mM Tropolone/2 mM Crown RunVoltage: +20 kV
Detection: Indirect 185nm, 0.3 TC InjectionMode: Hydrostatic, 30sec
Capillary: 75um X 60cm



Peak Results

#	Name	Migration Time (min)	Area (uV*sec)	Time Corr. Area	Height (uV)	Amount-ppm
1	NH4	3.573	89647	25087.70	7306	125533.662
2	K	3.900	261	66.87	263	688.025
3	Ca	4.210	134	31.92	126	153.954
4	Na	4.672	1975	422.80	1303	1885.437

Report Method: Cations_A

Version: 2.10

For Sample: Int. Etchant (0 Vial: 14

Injection: 1

Channel: SATIN

roc Chan: SATIN

Processed: 12/28/94 02:32:43 PM

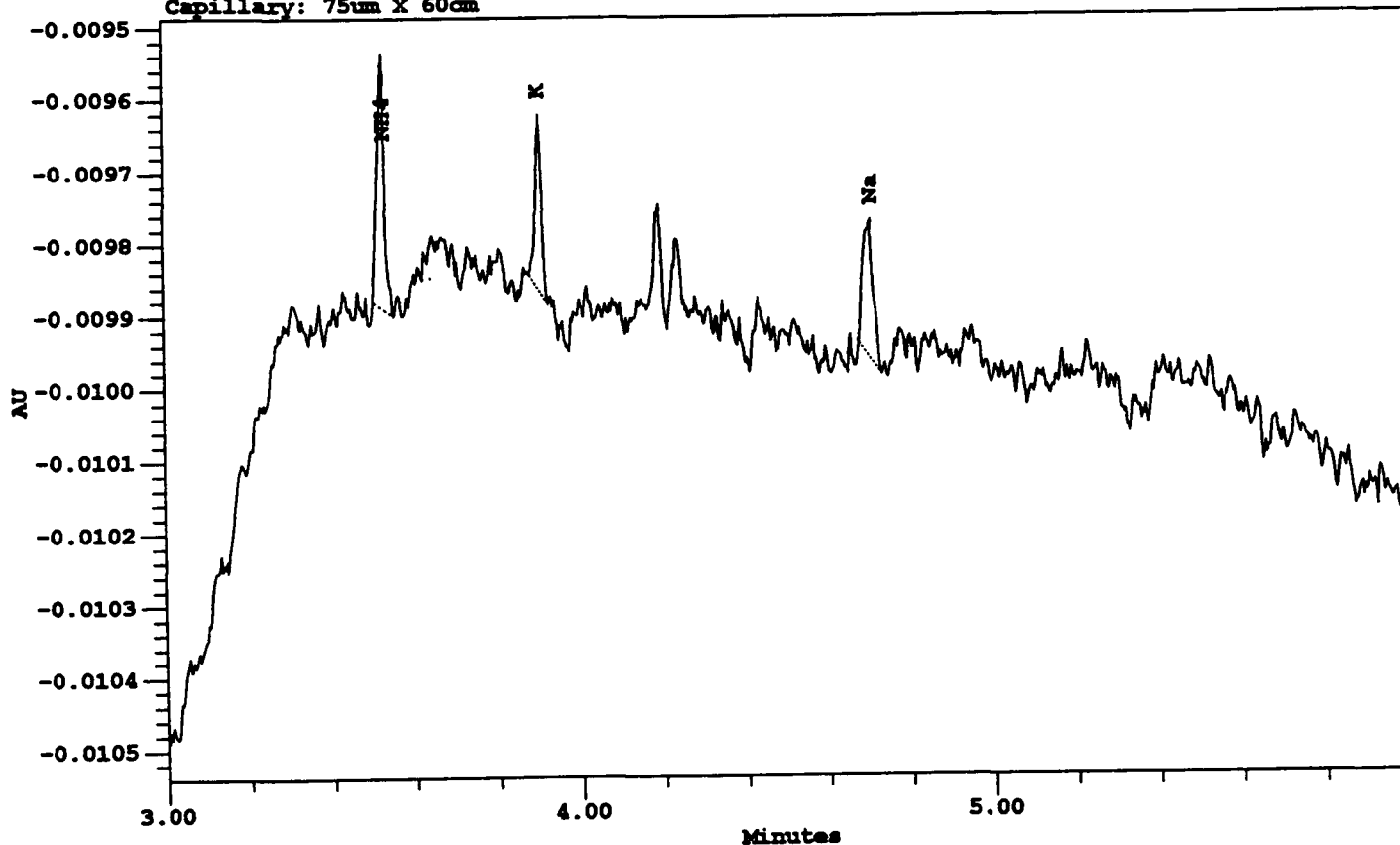
Channel Descr:

Northern Kentucky Laboratory
Sample Information

Project Name: CIA_Electronics_02
SampleName: Int. Etchant (0.1-100mL)
Vial: 14
Injection: 1
Channel: SATIN
Date Acquired: 12/27/94 11:12:33 AM
Scale Factor: 1.00
Acq Meth Set: CIA_Cations
Processing Method: Cations_02

Sample Type: Unknown
Volume: 30.00
Run Time: 8.0 min
Date Processed: 12/28/94 02:32:43 PM
Dilution: 1000.00000

SampleName: Int. Etchant (0.1-100mL)
Electrolyte: 1.2mM UV Cat-2/3 mM Tropolone/2 mM Crown RunVoltage: +20 kV
Detection: Indirect 185nm, 0.3 TC InjectionMode: Hydrostatic, 30sec
Capillary: 75um X 60cm



Peak Results

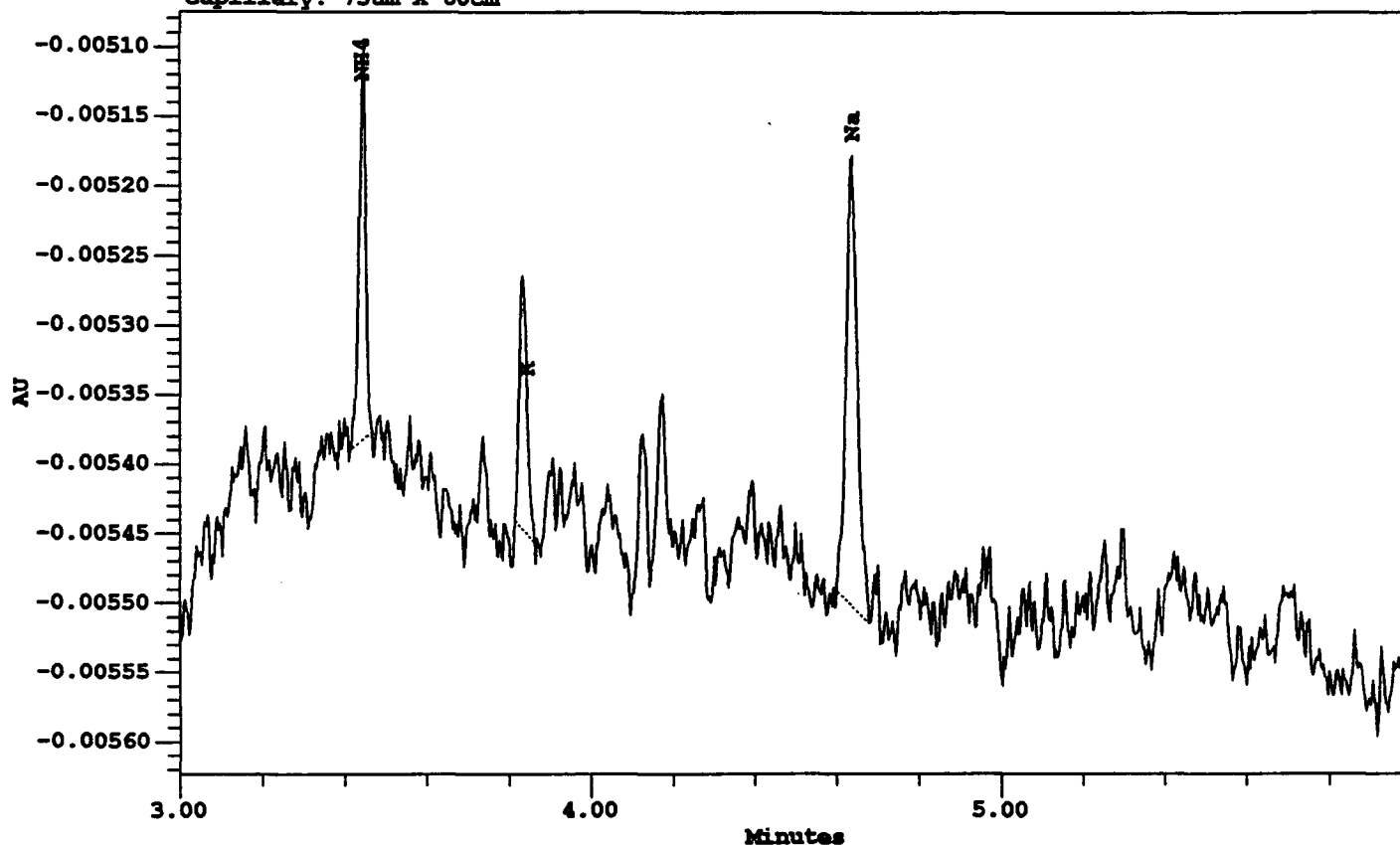
#	Name	Migration Time (min)	Area (uV*sec)	Time Corr. Area	Height (uV)	Amount-ppm
1	NH4	3.528	426	120.74	354	60.414
2	K	3.903	258	66.07	232	67.979
3	Na	4.705	327	69.42	189	30.955

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Report Method: Cations_A	Version: 2.10	
For Sample: Final Etchant (Vial: 15	Injection: 1	Channel: SATIN
roc Chan: SATIN	Processed: 12/28/94 02:32:07 PM	
Channel Descr:		

Northern Kentucky Laboratory
Sample Information

Project Name:	CIA_Electronics_02	Sample Type:	Unknown
SampleName:	Final Etchant (0.1-100mL)	Volume:	30.00
Vial:	15	Run Time:	8.0 min
Injection:	1	Date Processed:	12/28/94 02:32:07 PM
Channel:	SATIN	Dilution:	1000.00000
Date Acquired:	12/27/94 11:33:58 AM		
Scale Factor:	1.00		
Acq Meth Set:	CIA_Cations		
Processing Method:	Cations_02		

SampleName: Final Etchant (0.1-100mL)
 Electrolyte: 1.2mM UV Cat-2/3 mM Tropolone/2 mM Crown RunVoltage: +20 kV
 Detection: Indirect 185nm, 0.3 TC InjectionMode: Hydrostatic, 30sec
 Capillary: 75um X 60cm



Peak Results

#	Name	Migration Time (min)	Area (uV*sec)	Time Corr. Area	Height (uV)	Amount-ppm
1	NH4	3.442	317	91.99	282	46.030
2	K	3.843	247	64.35	183	66.212
3	Na	4.637	627	135.18	321	60.283