

MILLENNIUM APPLICATION

BRIEF

TOPIC: Capillary Ion Analysis of Chromate in Wastewater from Plating Operations

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Introduction:

Analysis of hexavalent chromium (in the form of chromate) is of importance in the plating industry for monitoring of both plating operations as well as waste produced from these operations. CIA offers an attractive means of analyzing for chromate in these matrices. Using a phosphate and OFM electrolyte with direct UV detection at 185nm, analysis of chromate as well as other anions can be done. In the case of wastewaters which may contain a high level of chloride or nitrate which may mask the presense of small amounts of chromate selective detection at 365nm can be used. Figure 1 is an electropherogram of a chromate and chloride standard at 1 ppm each. As can be seen a run time of under 5.0 minutes is possible. Figure 2 is an electropherogram of a sample containing nitrate as well with fairly good resolution of both nitrate as well as chromate with detection at 185nm. In the case of high levels of chloride and nitrate, chromate may not be resolved as is the case demonstrated in figure 3 which is a wastewater sample containing high levels of chloride, sulfate, phosphate and nitrate as well as a spiked amount of chromate (1 ppm). In figure 3a (top) chromate is not easily seen due to the high levels of chloride and nitrate present. Dilution will not help here since the chromate levels are already low. By monitoring at 365nm you eliminate detection of all anions except chromate as shown in figure 3b (bottom) which is the same sample except analyzed at 365nm. Detection limits (3X SN) for chromate using either wavelength are listed below.

Detection Limits (3X) (hydrostatic)

	<u>185nm (ppb)</u>	<u>365nm (ppb)</u>
Chromate	83	520

Figure 4 shows a calibration curve ($r^2=0.998$) for chromate using this method. As shown in the previous examples, chromate monitoring can be easily done by CIA in less than 5.0 minutes by using either direct UV detection at 185nm or 365nm.

EXPERIMENTAL:

Anion Analysis

System:	Q4000E
Electrolyte:	Phosphate/OFM
Capillary:	75um X 60cm
Injection:	Hydrostatic, 30 seconds
Run Voltage:	-15kV
Detection:	Direct UV at 185 nm or 365 nm
Data:	Millennium 2010 Chromatography Manager, Ver. 2.1 with CIA Option

FIGURES:

Figure 1: Electropherogram of 1 ppm chloride and chromate

Figure 2: Electropherogram of chloride, nitrate and chromate standard

Figure 3: Electropherogram of wastewater sample monitored at 185nm (top) and 365nm (bottom)

Figure 4: Calibration curve for chromate (duplicate injections)



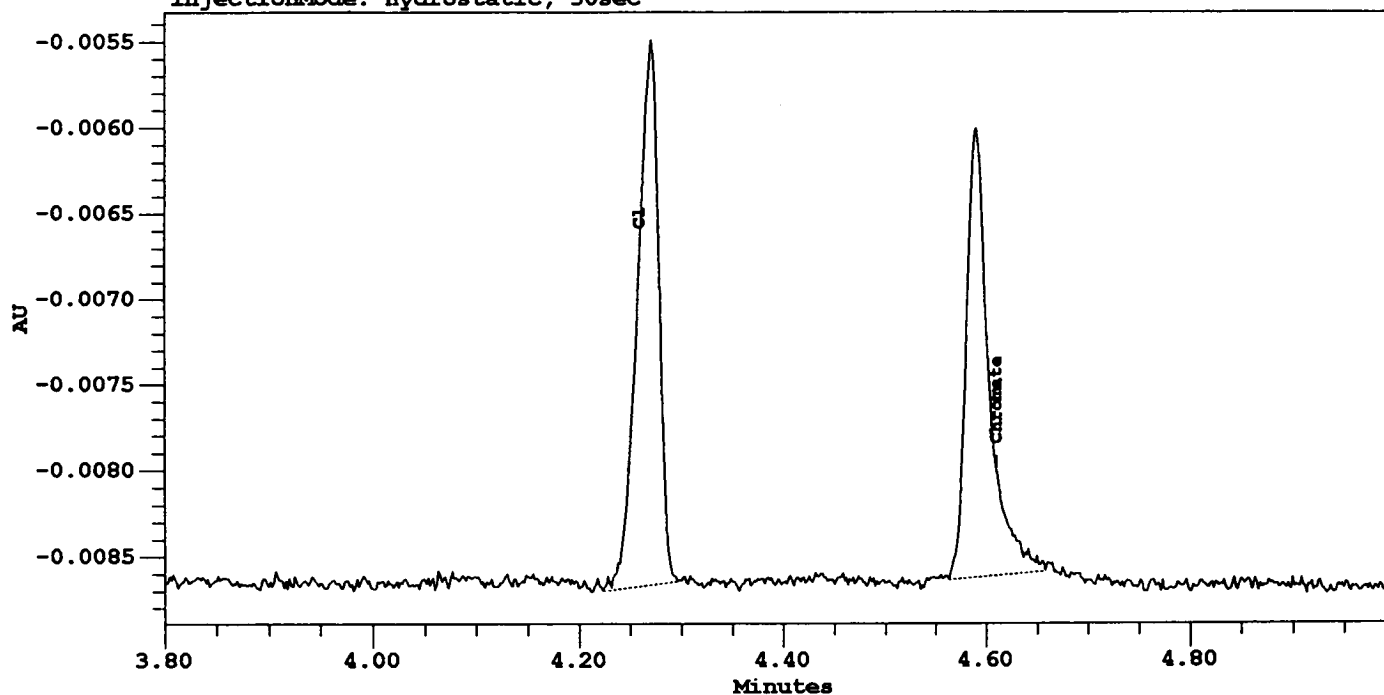
Northern Kentucky Laboratory

Sample Information

Project Name: CIA_Electronics03
SampleName: 1 ppm Chromate
Vial: 3
Injection: 2
Channel: SATIN
Date Acquired: 07/19/95 04:43:22 PM
Scale Factor: 1.00
Acq Meth Set: Phosphate_OFM
Processing Method: Chromate_185nm

Sample Type: Hydrostatic Unknown
Volume: 0.00
Run Time: 5.0 min
Date Processed: 07/20/95 09:44:20 AM
Dilution: 1.00000

SampleName: 1 ppm Chromate Detection: Direct 185nm, 0.3 TC
InjectionMode: Hydrostatic, 30sec



Peak Results

#	Name	Migration Time (min)	Area (uV*sec)	Time Corr. Area	Height (uV)
1	Cl	4.260	4622	1084.96	3169
2	Chromate	4.611	4098	888.67	2615



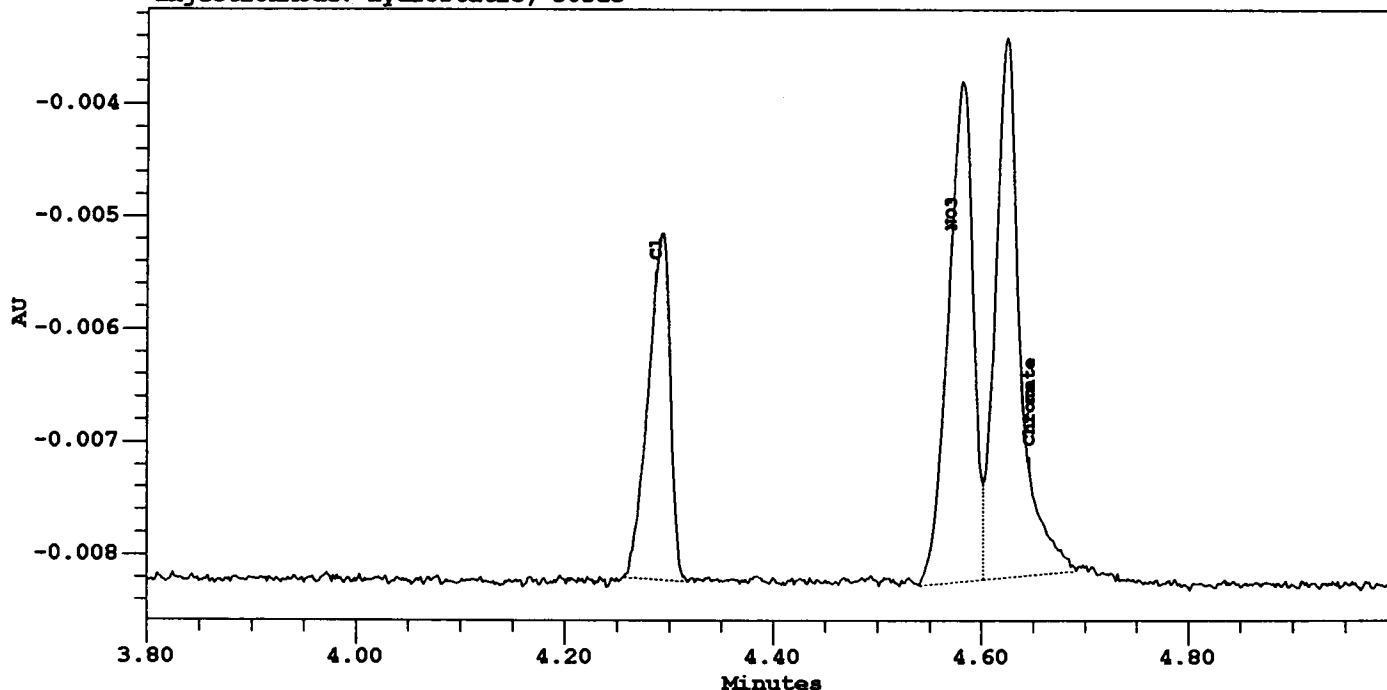
Northern Kentucky Laboratory

Sample Information

Project Name: CIA_Electronics03
SampleName: Cl, NO3, SO4, Chromate
Vial: 2
Injection: 2
Channel: SATIN
Date Acquired: 07/19/95 04:26:04 PM
Scale Factor: 1.00
Acq Meth Set: Phosphate OFM
Processing Method: Chromate_185nm

Sample Type: Hydrostatic Unknown
Volume: 0.00
Run Time: 5.0 min
Date Processed: 07/20/95 09:43:44 AM
Dilution: 1.00000

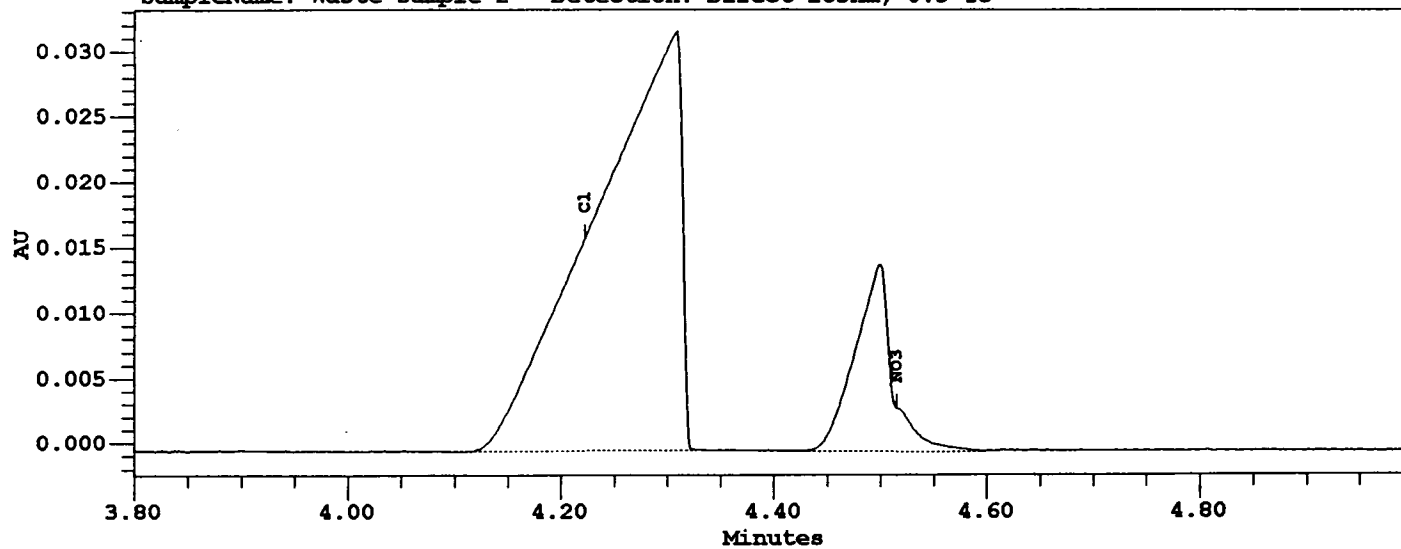
SampleName: Cl, NO3, SO4, Chromate Detection: Direct 185nm, 0.3 TC
InjectionMode: Hydrostatic, 30sec



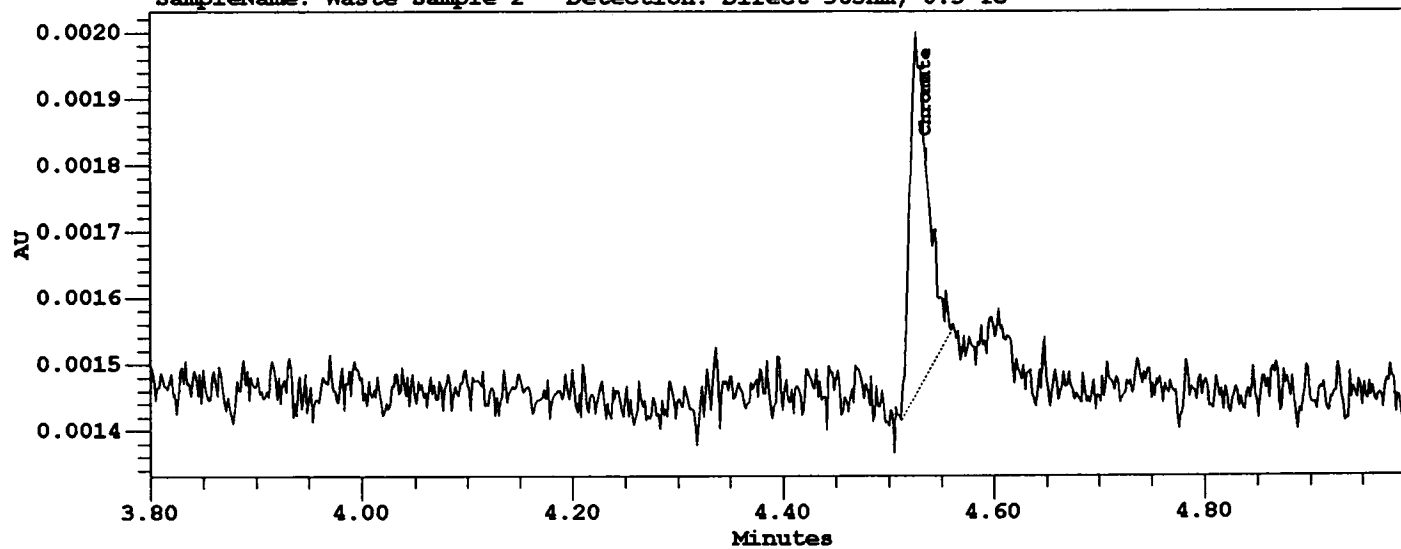
Peak Results

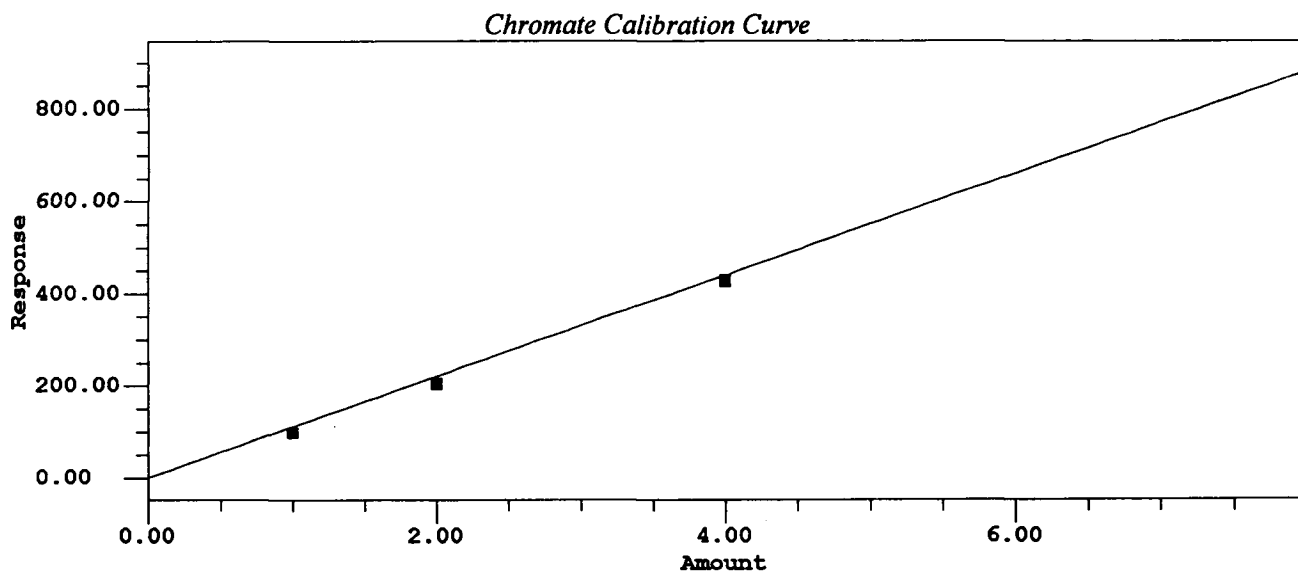
#	Name	Migration Time (min)	Area (uV*sec)	Time Corr. Area	Height (uV)
1	Cl	4.287	4517	1053.50	3075
2	NO3	4.571	7415	1622.25	4436
3	Chromate	4.647	7993	1720.23	4787

SampleName: Waste Sample 2 Detection: Direct 185nm, 0.3 TC



SampleName: Waste Sample 2 Detection: Direct 365nm, 0.3 TC





Chromate Calibration Information

Processing Method	Chromate_365nm	System	Q4000e
Channel	SATIN	Date	20-JUL-95
Type	LC	Name	Chromate
Retention Time	4.644 min	Order	1
A	0.000000	B	109.936128
C	0.000000	D	0.000000
E	0.000000	F	0.000000
R	0.999000	R ²	0.998001
Standard Error	14.555006		

Chromate Point Table

#	Amount	Response	Calc. Amount	% Deviation	Manual	Ignore?
1	1.000000	97.189099	0.884051	-11.595	No	No
2	1.000000	97.576254	0.887572	-11.243	No	No
3	2.000000	202.892372	1.845548	-7.723	No	No
4	2.000000	206.476343	1.878148	-6.093	No	No
5	4.000000	427.087882	3.884873	-2.878	No	No
6	4.000000	430.660245	3.917368	-2.066	No	No
7	8.000000	879.710618	8.002016	0.025	No	No
8	8.000000	900.870182	8.194487	2.431	No	No

Table 'Chromate Average Table' contains no data.