APPENDIX 5

FOOD MICROBIOLOGY AND FOOD CHEMISTRY SINCE 1967

ABC Research Corporation
(Originally American Bacteriological & Chemical Research Corporation)

3437 S.W. 24th Avenue
Gainesville, Florida 32607

(352) 372-0436
Fax (352) 378-6483
e-mail: info@abcr.com

Determination of Chlorite, Chloride and Chlorate in Waste Stream Waters

Objective:

This evaluation was conducted on behalf of Alcide Corporation, Redmond, WA. The purpose of the study was to evaluate the waste streams from four commercial poultry processing facilities for the presence of oxy-chlorine residues. Alcide Corporation's Sanova (acidified sodium chlorite) antimicrobial intervention process had previously been installed at all four facilities and was fully operational at the time of sample collection. Spent solutions from the Sanova process were therefore entering into the total wastewater stream at each facility.

Samples:

The wastewater samples received were designated as follows:

Sample Number	Location
69242-1	Townsend's Inc., Pittsboro, NC
69242-3	Townsend's Inc., Siler City, NC
69242-4	Perdue Farms, Georgetown, DE
69242-5	Gold Kist, Inc. Boaz, AL

An aliquot of Sample 69242-1 was set aside for another test, and this aliquot was designated Sample 69242-2. The anticipated additional test on this aliquot was not performed, so Sample 69242-2 does not appear in this report.

Samples were not stabilized at the time of collection.

The pH's of Samples 1, 3, 4 and 5 were 6.3, 6.9, 7.04 and 6.3, respectively. The pH measurements were made three (Sample 4), six (Samples 1 and 3), or 18 (Sample 5) days after receipt.

Dates:

Sample Collection:

Sample 1: 5/23/00
Sample 3: 5/23/00
Sample 4: 5/26/00
Sample 5: Not reported

Sample Receipt:

Sample 1: 5/25/00
Sample 3: 5/25/00
Sample 4: 5/28/00
Sample 5: 6/8/00

Sample Analysis Initiation:

Sample 1: 5/31/00
Sample 3: 5/31/00
Sample 4: 5/31/00
Sample 5: 6/15/00

Final Report: 6/26/00

Methodology:

All analyses were done with a Dionex Model 4500i ion chromatograph using a 4-mm Dionex IonPac A59-HC column and a 12mM carbonate/5mM bicarbonate mobile phase. The injection volume was 25 µL.

Injection of chlorite, chloride and chlorate standards establish retention times of 5.5 min, 7.2 min and 13.1 min, respectively, for these three ions. These retention times were verified by standard injection prior to each new set of wastewater injections.

A standard curve was prepared using standard solutions with the following concentrations. The lowest concentration that produced a measurable signal was designated the limit of quantitation (LOQ).

Chlorite: 10 ppm
1ppm
0.1 ppm

Chloride: 10ppm
1ppm
0.1 ppm
0.01 ppm

Chlorate: 10 ppm
1ppm
0.1 ppm

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Wastewater samples were analyzed as follows:

Chlorite and chlorate: A five-milliliter aliquot of wastewater was removed from the container in which it was received and filtered to avoid fouling of the chromatograph. Twenty-five microliters of the filtrate was injected into the chromatographic column.

Chloride: Treatment of samples as described in the preceding paragraph produced chromatograms in which the chloride signal was off scale and thus unmeasurable. For analysis of chloride, therefore, a 1-mL aliquot was diluted to 20 mL with distilled water. The resulting solution was sufficiently clear that it was not filtered before making the 25-µL injection.

Results:

The resulting concentrations (ppm) of the three ions in the four samples were as follows:

Sample Number	Chlorite	Chloride	Chlorate
69242-1	ND	176	ND
69242-3	ND	149	ND
69242-4	ND	172	ND
69242-5	ND	103	ND

Where ND = Not detected (no peak seen at the appropriate retention time).
The chloride values were calculated using the standard curves and correcting for the 20-fold dilution.

The raw data (chromatograms) and figures of merit for the chloride standard curve are attached.

Conclusion:

Chromatograms of the four wastewater samples indicate high concentrations of chloride, some unknowns responses at <5 min, ca. 11 min and > 14 min, but no chlorite or chlorate.

Respectfully submitted by:

John P. Toth, Ph.D.
Vice President, Chemistry
johnt@abcr.com

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[redacted]

TEST: Chloride
DATE: 6/23/00
ANALYST: Diane Northcott

SAMPLE	WGHT g	VOL ml.	AREA	CONC ppm
Blank	1.00	1.00	0.00000	0.00
69242-1	1.00	20.00	442679660	176.35
69242-3	1.00	20.00	375225478	149.39
69242-4	1.00	20.00	431305619	171.74
69242-5	1.00	20.00	259403251	103.22

STANDARDS (ppm)

CONC	AREA
10.00	502226416
1.00	50333067
0.10	5769138

Regression Output:

Constant	470142.61
Std Err of Y Est	419744.96
R Squared	0.9999988
No. of Obeservations	3
Degrees of Freedom	2
X Coefficient(s)	50172810
Std Err of Coef.	54215.956

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[redacted]

Sample	Chloride ppm	Chlorate ppm	Chlorite ppm
69242-1	176	<0.1	<0.1
69242-3	149	<0.1	<0.1
69242-4	171	<0.1	<0.1
69242-5	103	<0.1	<0.1
LOQ	0.01	0.10	0.10

Sample Name : 10ide/ate
Date Time Updated : 6/23/00 10:25:46 AM
Calibration Date : 6/12/00 8:10:02 AM
Injection Number : 24
Vial ID :
Data Points Collected : 6000
Data Collection Rate : 5.00 Hz

System Name : DIONEX ION CHROMATOGRAPH
System Operator : diane northcott
Column Type :
Detector Name :
Module Name :
Module ID : 50 07 de
Moduleware Version : 01.09

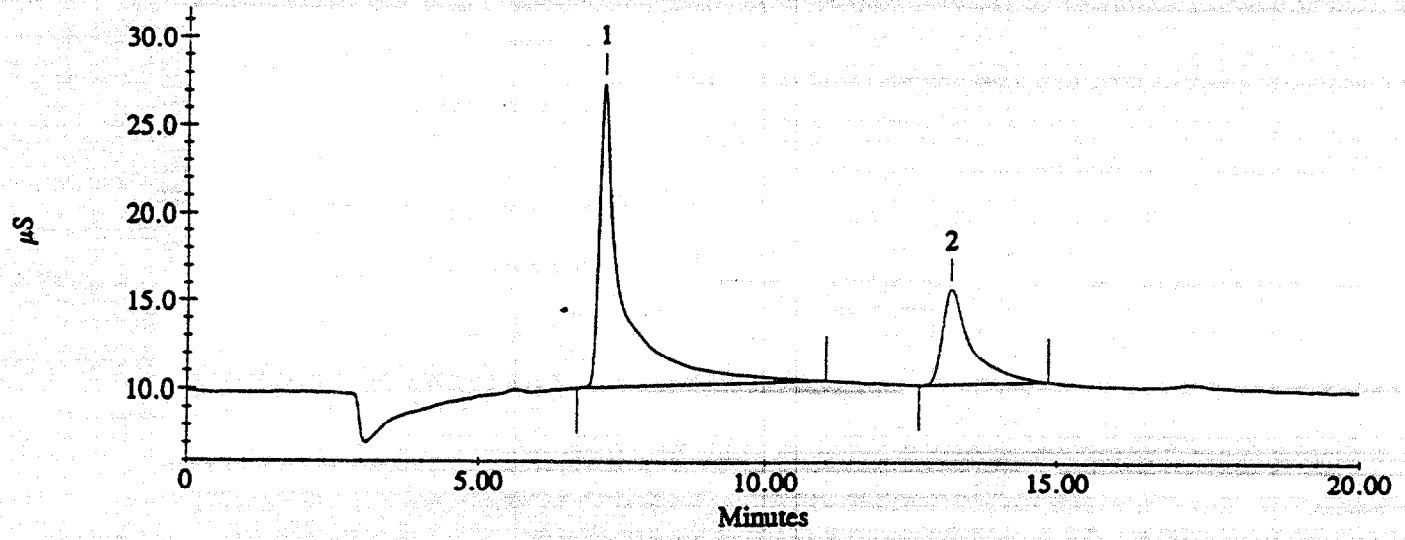
Data File Name : C:\PEAKNET\DATA\062200_024.DXD
Calibration Type : EXTERNAL
Injection Volume : 1.00
Internal Standard Amount : 1.00
Dilution Factor : 1.00

Method File Name : c:\peaknet\method\anions i.met
Test Collection Period :
Area Reject Value : 1000.00
Starting Peak Width : 6.00
Starting Peak Threshold : 1000.00

Peak Information : All Peaks

Pk. Num	Ret Time	Component Name	Concentration	Height	Area	Bl. Code	%Delta
1	7.20	CL	89.65	17329895	502226416	1	0.84
2	13.14	ClO3	2.90	5449977	193142466	1	0.31
		—total(s)—	92.55	22779872	695368883		

10ide/ate



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Sample Name : 1ide/ate
Date Time Updated : 6/23/00 10:16:35 AM
Calibration Date : 6/12/00 8:10:02 AM
Injection Number : 25
Vial ID :
Data Points Collected : 6000
Data Collection Rate : 5.00 Hz

System Name : DIONEX ION CHROMATOGRAPH
System Operator : diane northcott
Column Type :
Detector Name :
Module Name :
Module ID : 50 07 de
Moduleware Version : 01.09

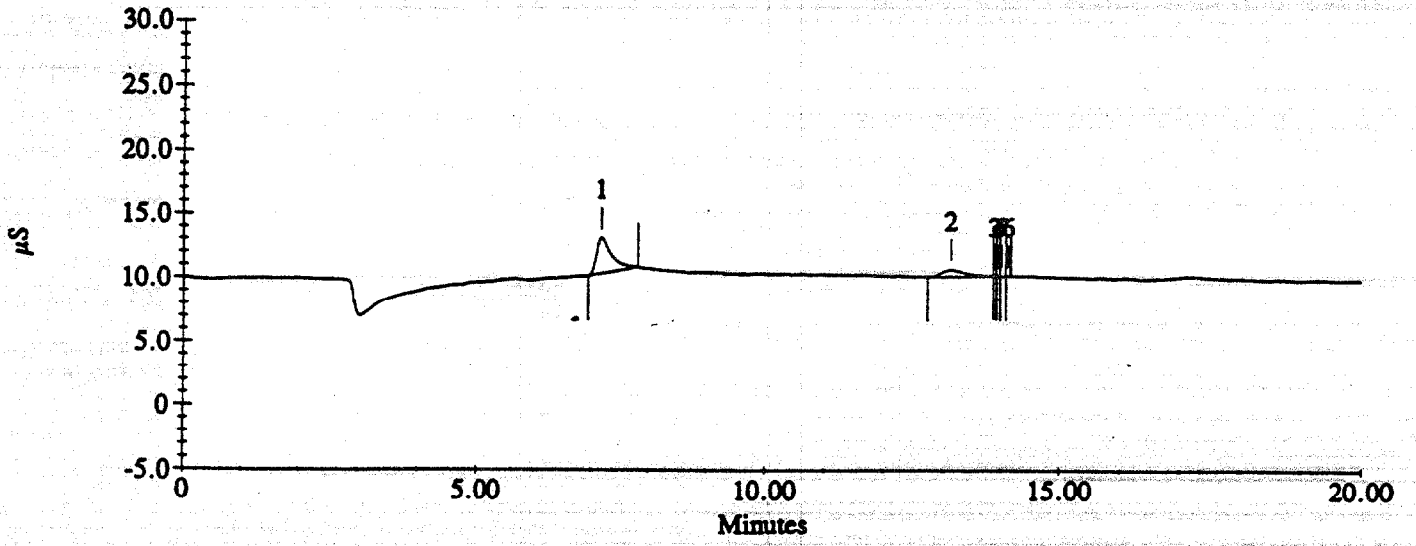
Data File Name : C:\PEAKNET\DATA\062200_025.DXD
Calibration Type : EXTERNAL
Injection Volume : 1.00
Internal Standard Amount : 1.00
Dilution Factor : 1.00

Method File Name : C:\peaknet\method\anions i.met
Test Collection Period :
Area Reject Value : 1000.00
Starting Peak Width : 6.00
Starting Peak Threshold : 1000.00

Peak Information : All Peaks

Pk. Num	Ret Time	Component Name	Concentration	Height	Area	Bl. Code	%Delta
1	7.16	CL	9.33	2771844	50333067	1	0.28
2	13.13	ClO3	3.72	556717	17427441	3	0.20
3	13.88		0.00	413	193	4	
4	13.95		0.00	3780	4381	4	
5	14.01		0.00	8907	26979	4	
6	14.12		0.00	8291	19959	4	
		---total(s)---	13.05	3349953	67812020		

1ide/ate



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Sample Name : 0.1ide ate
Date Time Updated : 6/23/00 10:24:33 AM
Calibration Date : 6/12/00 8:10:02 AM
Injection Number : 26
Vial ID :
Data Points Collected : 6000
Data Collection Rate : 5.00 Hz

System Name : DIONEX ION CHROMATOGRAPH
System Operator : diane northcott
Column Type :
Detector Name :
Module Name :
Module ID : 50 07 de
Moduleware Version : 01.09

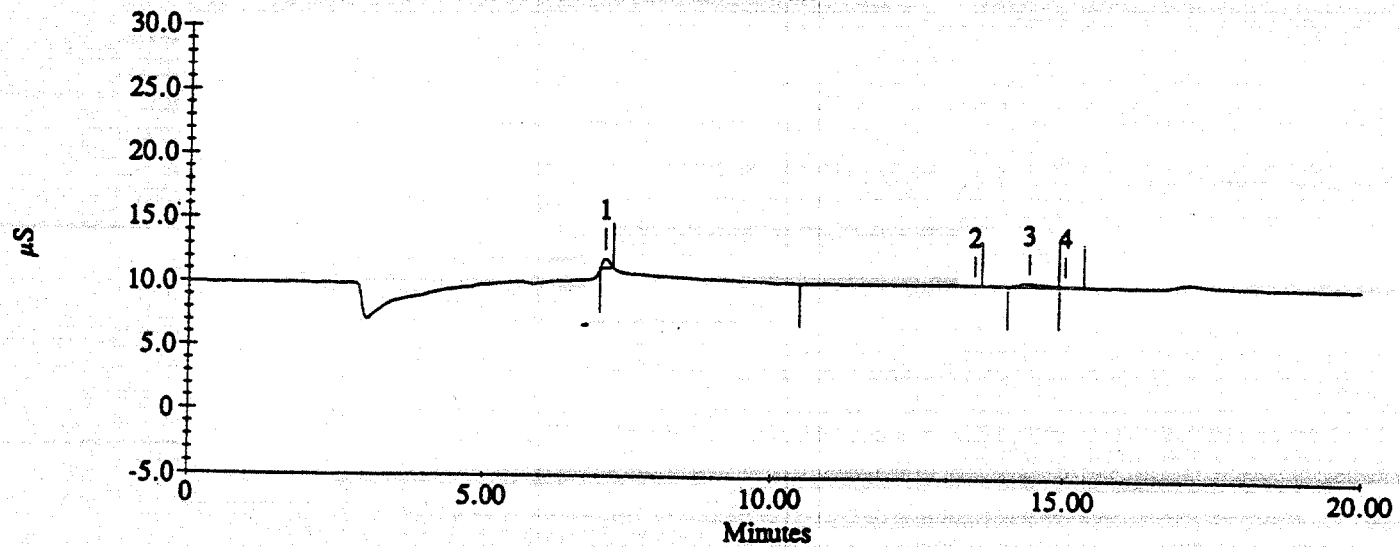
Data File Name : C:\PEAKNET\DATA\062200_026.DXD
Calibration Type : EXTERNAL
Injection Volume : 1.00
Internal Standard Amount : 1.00
Dilution Factor : 1.00

Method File Name : c:\peaknet\method\anions i.met
Test Collection Period :
Area Reject Value : 1000.00
Starting Peak Width : 6.00
Starting Peak Threshold : 1000.00

Peak Information : All Peaks

Pk. Num	Ret Time	Component Name	Concentration	Height	Area	Bl. Code	%Delta
1	7.11	CL	1.41	742314	5769138	1	-0.47
2	13.45	ClO3	3.79	20173	1393574	1	2.65
3	14.39		0.00	249088	7202436	2	
4	15.01		0.00	67644	991070	2	
		---total(s)---	5.20	1079218	15356217		

0.1ide ate



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Software Revision : PeakNet 5.01

Current Date : 06/23/00

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Sample Name : 0.01 ide
Date Time Updated : 6/23/00 1:53:57 PM
Calibration Date : 6/12/00 8:10:02 AM
Injection Number : 52
Vial ID :
Data Points Collected : 6000
Data Collection Rate : 5.00 Hz

System Name : DIONEX ION CHROMATOGRAPH
System Operator : diane northcott
Column Type :
Detector Name :
Module Name :
Module ID : 50 07 de
Moduleware Version : 01.09

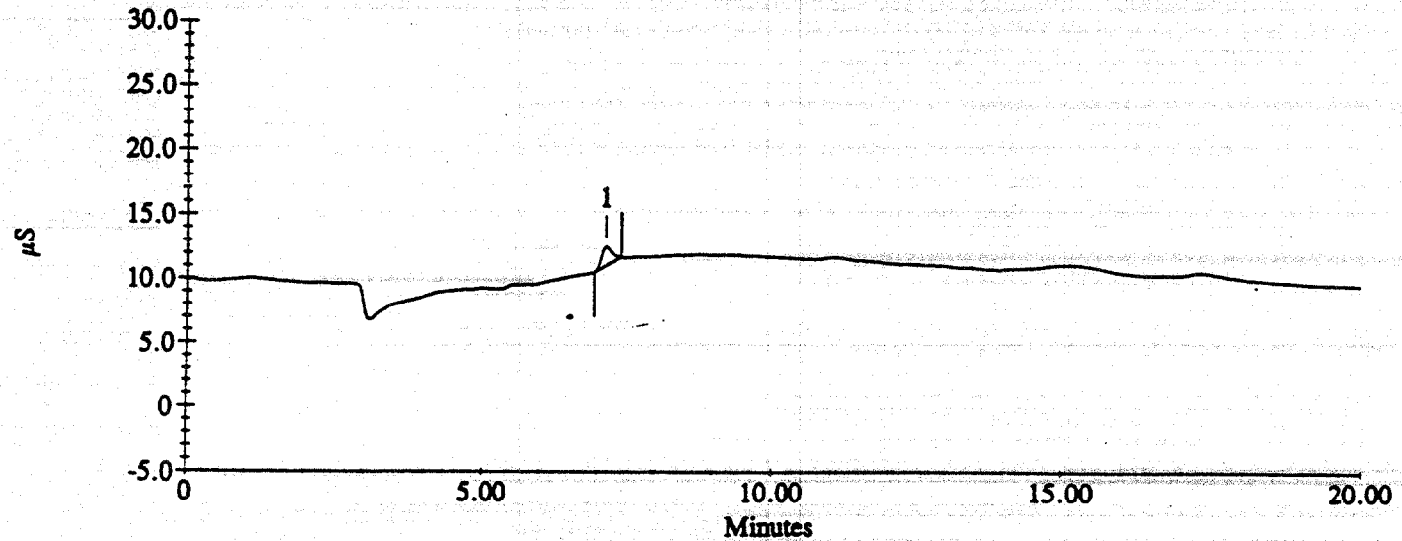
Data File Name : C:\PEAKNET\DATA\062200_052.DXD
Calibration Type : EXTERNAL
Injection Volume : 1.00
Internal Standard Amount : 1.00
Dilution Factor : 1.00

Method File Name : C:\peaknet\method\anions i.met
Test Collection Period :
Area Reject Value : 1000.00
Starting Peak Width : 6.00
Starting Peak Threshold : 1000.00

Peak Information : All Peaks

Pk. Num	Ret Time	Component Name	Concentration	Height	Area	Bl. Code	%Delta
1	7.15	CL	3.75	1496158	18941391	1	0.09
		---total(s)---	3.75	1496158	18941391		

0.01 ide



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Sample Name : 10ite
Date Time Updated : 6/23/00 11:35:12 AM
Calibration Date : 6/12/00 8:10:02 AM
Injection Number : 23
Vial ID :
Data Points Collected : 6000
Data Collection Rate : 5.00 Hz

System Name : DIONEX ION CHROMATOGRAPH
System Operator : diane northcott
Column Type :
Detector Name :
Module Name :
Module ID : 50 07 de
Moduleware Version : 01.09

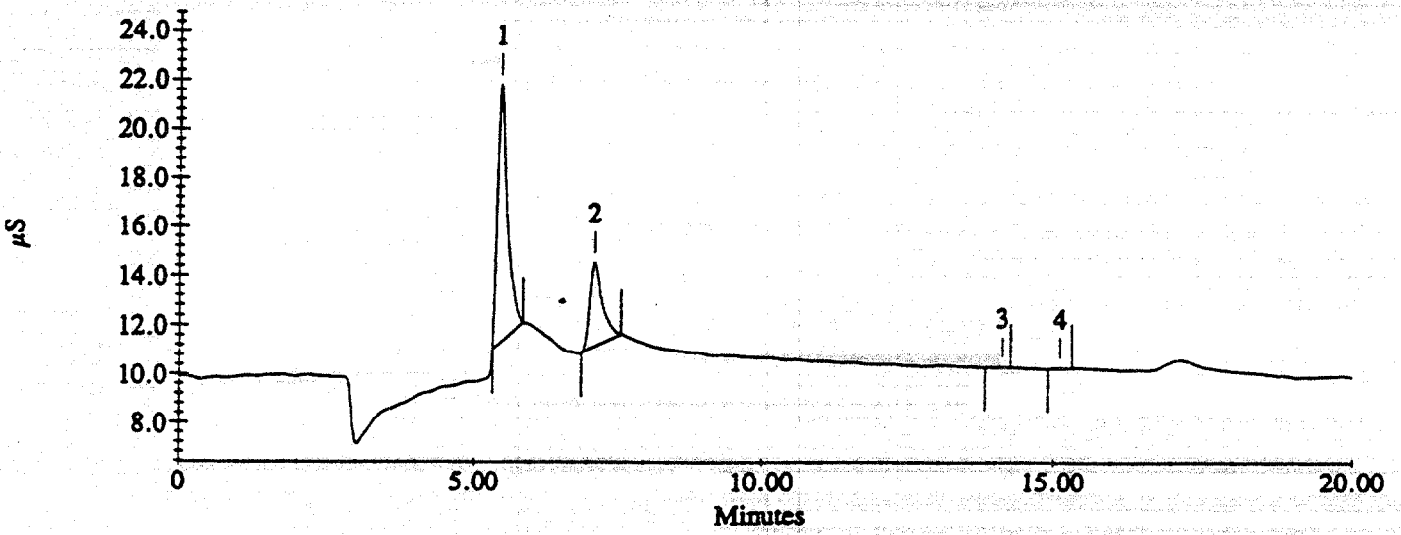
Data File Name : C:\PEAKNET\DATA\062200_023.DXD
Calibration Type : EXTERNAL
Injection Volume : 1.00
Internal Standard Amount : 1.00
Dilution Factor : 1.00

Method File Name : c:\peaknet\method\anions i.met
Test Collection Period :
Area Reject Value : 1000.00
Starting Peak Width : 6.00
Starting Peak Threshold : 1000.00

Peak Information : All Peaks

Pk. Num	Ret Time	Component Name	Concentration	Height	Area	Bl. Code	%Delta
1	5.44	ClO2	2.76	10545257	129991887	1	0.74
2	7.13	CL	9.74	3421622	52660222	1	-0.19
3	14.13		0.00	26142	345135	1	
4	15.11		0.00	25191	307180	1	
		---total(s)---	12.51	14018212	183304423		

10ite



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Software Revision : PeakNet 5.01

Current Date : 06/23/00

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Sample Name : 1ite
Date Time Updated : 6/23/00 11:34:17 AM
Calibration Date : 6/12/00 8:10:02 AM
Injection Number : 22
Vial ID :
Data Points Collected : 6000
Data Collection Rate : 5.00 Hz

System Name : DIONEX ION CHROMATOGRAPH
System Operator : diane northcott
Column Type :
Detector Name :
Module Name :
Module ID : 50 07 de
Moduleware Version : 01.09

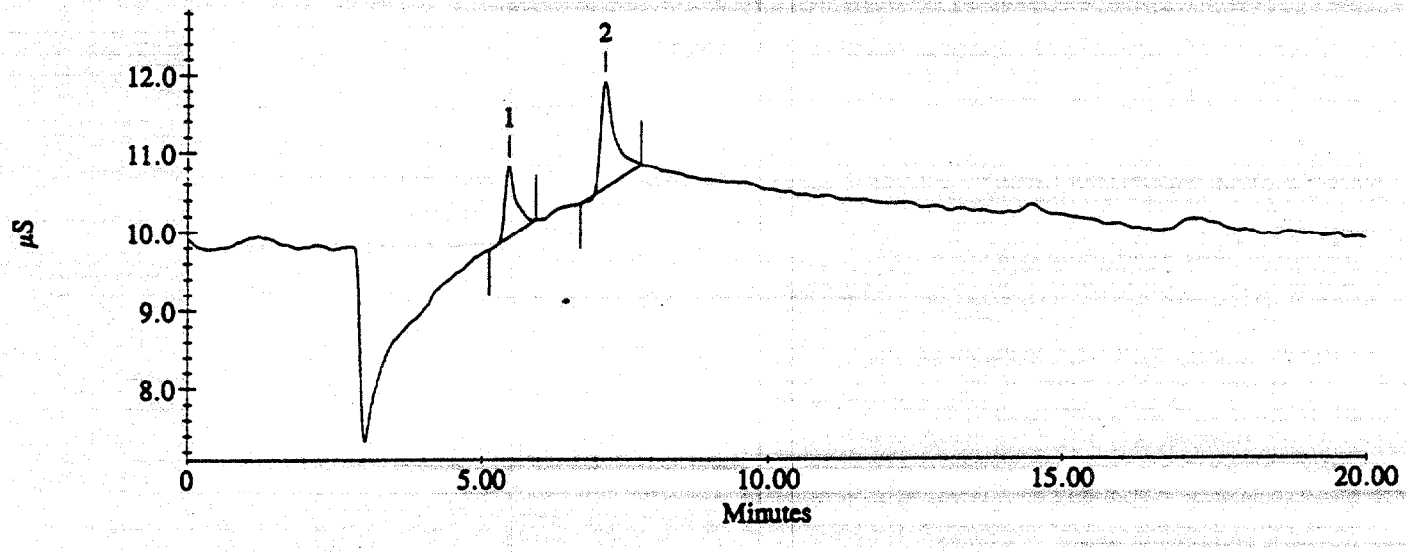
Data File Name : C:\PEAKNET\DATA\062200_022.DXD
Calibration Type : EXTERNAL
Injection Volume : 1.00
Internal Standard Amount : 1.00
Dilution Factor : 1.00

Method File Name : c:\peaknet\method\anions i.met
Test Collection Period :
Area Reject Value : 1000.00
Starting Peak Width : 6.00
Starting Peak Threshold : 1000.00

Peak Information : All Peaks

Pk. Num	Ret Time	Component Name	Concentration	Height	Area	Bl. Code	%Delta
1	5.47	ClO2	3.34	898968	12591145	1	1.23
2	7.15	CL	4.57	1371811	23552980	1	0.19
		—total(s)—	7.90	2270779	36144124		

1ite



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Software Revision : PeakNet 5.01

Current Date : 06/23/00

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Sample Name : 0.1ite
Date Time Updated : 6/23/00 11:33:01 AM
Calibration Date : 6/12/00 8:10:02 AM
Injection Number : 21
Vial ID :
Data Points Collected : 6000
Data Collection Rate : 5.00 Hz

System Name : DIONEX ION CHROMATOGRAPH
System Operator : diane northcott
Column Type :
Detector Name :
Module Name :
Module ID : 50 07 de
Moduleware Version : 01.09

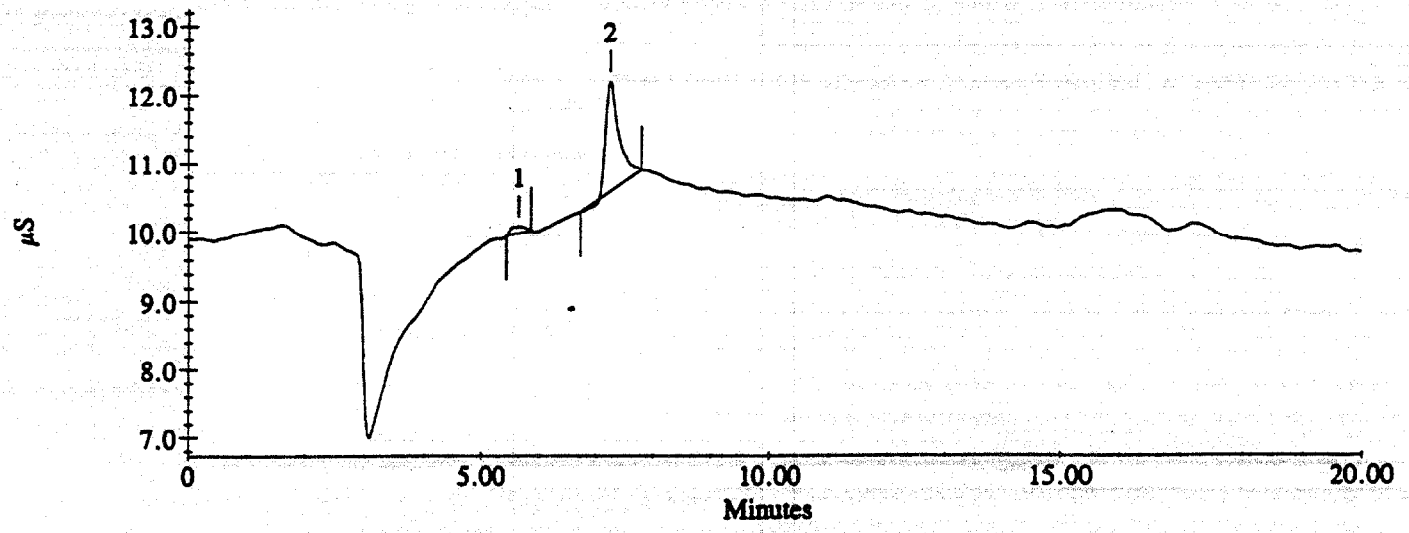
Data File Name : C:\PEAKNET\DATA\062200_021.DXD
Calibration Type : EXTERNAL
Injection Volume : 1.00
Internal Standard Amount : 1.00
Dilution Factor : 1.00

Method File Name : c:\peaknet\method\anions i.met
Test Collection Period :
Area Reject Value : 1000.00
Starting Peak Width : 6.00
Starting Peak Threshold : 1000.00

Peak Information : All Peaks

Pk. Num	Ret Time	Component Name	Concentration	Height	Area	Bl. Code	%Delta
1	5.65	ClO2	3.39	99942	1764573	1	4.69
2	7.23	CL	5.02	1620419	26109473	1	1.31
		---total(s)---	8.41	1720361	27874046		

0.1ite



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Sample Name : ~~69282-1~~ 69242-1 JPT 7/25/00
Date Time Updated : 5/31/00 2:38:53 PM
Calibration Date : 12/10/98 9:13:05 AM
Injection Number : 16
Vial ID :
Data Points Collected : 6000
Data Collection Rate : 5.00 Hz

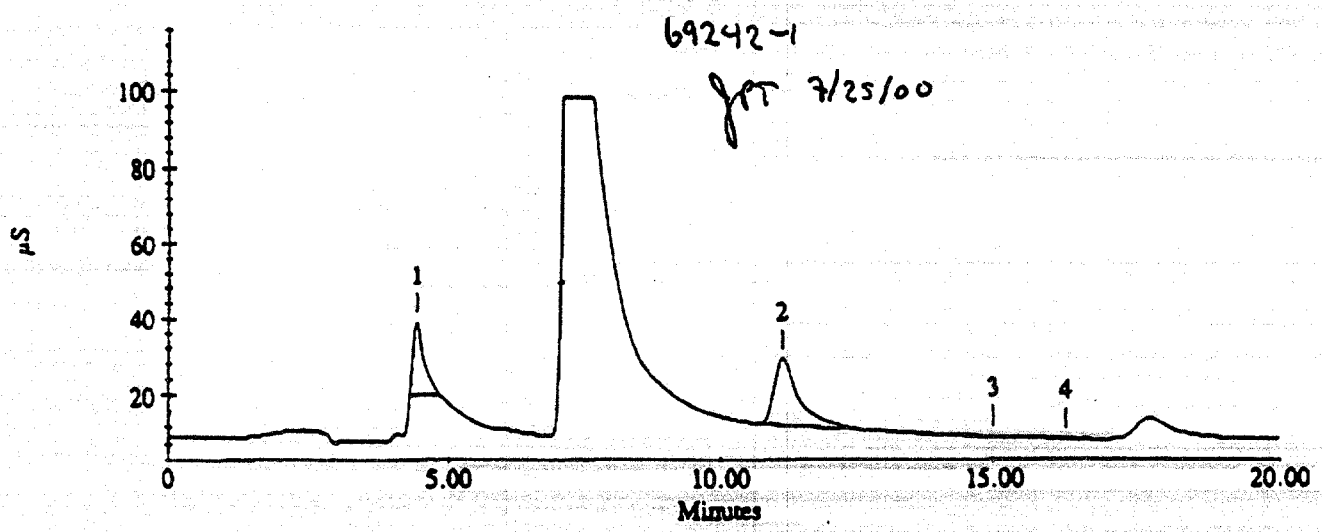
System Name : DIONEX ION CHROMATOGRAPH
System Operator : diane northcott
Column Type :
Detector Name :
Module Name :
Module ID : 50 07 de
Moduleware Version : 01.09

Data File Name : C:\PEAKNET\DATA\0531_016.DXD
Calibration Type : EXTERNAL
Injection Volume : 1.00
Internal Standard Amount : 1.00
Dilution Factor : 1.00

Method File Name : c:\peaknet\method\anions i.met
Test Collection Period :
Area Reject Value : 1000.00
Starting Peak Width : 6.00
Starting Peak Threshold : 1000.00

Peak Information : All Peaks

Pk. Num	Ret Time	Component Name	Concentration	Height	Area	Bl. Code	%Delta
1	4.43		358.48	19051006	254800740	I	-3.91
2	11.11		389.93	16972647	500902188	I	2.90
3	14.99		0.00	42186	1630705	I	
4	16.26		0.00	36020	461723	I	
		---total(s)---	748.41	36101859	757795356		



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Software Revision : PeakNet 5.01

Current Date : 05/31/00

Sample Name : ~~69282-4~~ 69242-4 JPT 7/25/00
Date Time Updated : 5/31/00 3:01:22 PM
Calibration Date : 12/10/98 9:13:05 AM
Injection Number : 17
Vial ID :
Data Points Collected : 6000
Data Collection Rate : 5.00 Hz

System Name : DIONEX ION CHROMATOGRAPH
System Operator : diane northcott
Column Type :
Detector Name :
Module Name :
Module ID : 50 07 de
Moduleware Version : 01.09

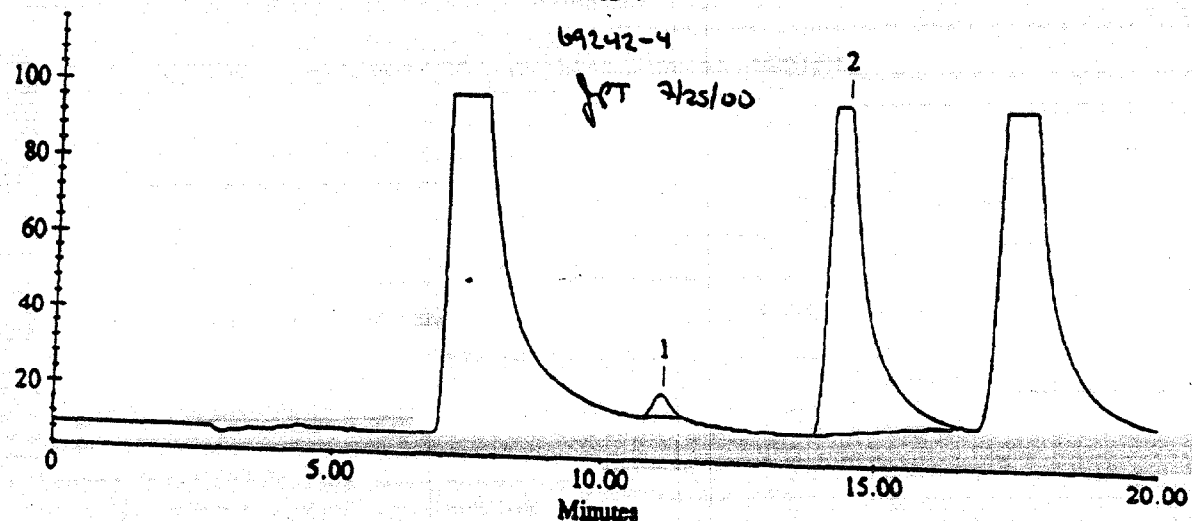
Data File Name : C:\PEAKNET\DATA\0531_017.DXD
Calibration Type : EXTERNAL
Injection Volume : 1.00
Internal Standard Amount : 1.00
Dilution Factor : 1.00

Method File Name : c:\peaknet\method\anions i.met
Test Collection Period :
Area Reject Value : 1000.00
Starting Peak Width : 6.00
Starting Peak Threshold : 1000.00

Peak Information : All Peaks

Pk. Num	Ret Time	Component Name	Concentration	Height	Area	Bl. Code	%Delta
1	11.11		97.63	5576306	124826943	1	2.90
2	14.38		0.00	86609868	4266013111	1	
---total(s)---			97.63	92186175	4390840054		

~~69282-4~~
69242-4
JPT 7/25/00



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Software Revision : PeakNet 5.01

Current Date : 05/31/00

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