ANALYSIS OF SOIL AND PLANT EXTRACTS

Denise R. Eubanks and Robert J. Joyce

Dionex Corp., 1228 Titan Way, Sunnyvale, CA 94088-3603

MATERIALS RESEARCH INSTRUMENTS
63, 'B' Wing, Mittal Court,
Nariman Point,
BOMBAY-400 021.

Southern Region Office :
DR. PAUL C. BAYNES, M.Sc., Ph.D.
8, Siddhashram Road,
Off. 17th Cross, Malleswaram
BANGALORE-560 003.

Presented at the 27th Rocky Mountain Conference, July 14-18, 1985
Denver, Colorado (Paper #301)

ANALYSIS OF SOIL AND PLANT EXTRACTS. Denise Eubanks, R.J. Joyce, Dionex Corporation, 1228 Titan Way, Sunnyvale, CA 94088-3603, (408) 737-0700.

To increase crop yield, essential nutrients for plant growth are monitored and replenished through land management programs. Until recently, agriculture has relied solely on wet chemical methods to determine these ionic constituents in soil as well as plant extracts. Steam distillation and reduction followed by colorimetric methods were used to determine such anions as nitrate and sulfate. These methods are complex and subject to many interferences.

Today, scientists in analytical, agronomy and soil labs are becoming more familiar with Ion Chromatography (IC) and its ability to provide such information with less interference and with higher throughput. IC can also be adapted to most of the extraction solutions and procedures currently in use and, in many cases, requires smaller sample amounts. This paper will present ion chromatographic procedures used for the determination of common anions (Cl^-, NO_3^-, HPO_4^{2-}, SO_4^{2-}) and cations (NH_4^+, $K+$, CA^{2+}, Mg^{2+}) in soil and plant extracts.

ANALYSIS OF SOIL AND PLANT EXTRACTS

Denise R. Eubanks and Robert J. Joyce

(1) As you've seen this week, Ion Chromatography is a technique based on liquid chromatographic principles. It is a popular and reliable method for analyzing complex mixture of ions.

Scientists in analytical, agronomy and soil labs are becoming more familiar with Ion Chromatography and it's ability to determine important ionic constituents in soil extracts, plant digests and natural waters.

Unlike the traditional wet chemical methods which are prone to interferences, IC provides a rapid and reliable means of analyzing a variety of constituents associated with efficient land management.

This paper will present ion chromatographic procedures used for the determination of common anions and cations in soil and plant extracts.

(2) Typical applications in the agricultural industry would include monitoring several areas .....

of course soil and plant analaysis are high on the list, but the determination of essential nutrients provided by such sources as fertilizers, irrigation waters and even acid rain are all important in plant growth.

(3) Ions of interest would include nitrogen and sulfur levels in fertilizers as well as exchangeable bases in soils. And such things as Bromide from ethylene dibromide application as a preservative can also be monitored.

(4) Traditional methods used for chloride are colorimetric or titrimetric procedures for nitrate colorimetric or ion electrode and for sulfate turbidimetric analysis.

Most commonly used methods for the alkali metals would be flame photometric and alkaline earths, atomic absorption.

(5) These methods are subject to interferences from a variety of ions and can be very complicated. Usually requiring a high level of analytical skill.

(6) Ion chromatographic methods provide reliable data and simple operation with fewer interferences and higher throughput.

Dr. Tabatabai at Iowa State has found that a single operator can perform 40 analysis of monovalent or divalent bases in a normal working day.

(7) The operating conditions for the determination of anions

would be performed on the AS4A analytical column and MPIC guard column to trap large hydrophobic ions. The mobile phase or eluant used is a 2mM Na_2CO_3/.75mM $NaHCO_3$.

This column configuration allowed the analysis of several soil samples without interferences from humic acids, which are large organic acids, that typically foul conventional HPLC analytical columns.

(8) The determination of NO_3^-,HPO_4^{2-} and SO_4^{2-} was completed in 9 min. in a 10mM KCl extraction solution. For cation analysis a NH_4 Acetate solution is used. Sample preparation was filtration before injection.

(9) IC can also be adapted to most of the extraction solutions and procedures currently in use, and in many cases require smaller sample amounts then traditional methods.

Some of the extractions procedures yield sample volumns in the uL range. IC injection volumns are anywhere from 10-50 uL.

(10) The uptake of nutrients from the soil and any additives to the soil can be monitored through determination of ionic constituents in the plant tissue itself.

(11) The sample prep involves aqueous maceration of the plant and analyzing the supernatent. Several research groups are using IC to make recommendation of nutrients to be added or replenished in the soil over a period of time.

(12) IC can also be used to monitor the quality of fertilizer being administered.

The determination of anions for

(13) monovalent cations
(14) and divalent cations can assist with recommended amounts of fertilizer application.

(15) IC is not limited to only soil and plant extracts, a more complete ecological picture can be obtained on the ionic level. Here, well water was analyzed and found to be high in nitrate, most likely due to fertilizer in run off water. The method is documented in EPA method 300.0.

(16) Other analysis performed routinely involve looking at anions in acid rain where young plant growth or more established areas such as forest can be affected.

(17) and irrigation waters.

(18) The determination of selenium and arsenic is accomplished using a HPIC-AG5 and HPIC-AS4A in series. The eluant is a 1.0mM Na_2CO_3/.75mM $NaHCO_3$.

(19) All of these aspects contribute to a more efficient means of land management. IC provides an important and vital function in determining essential nutrients for increased crop yields and control.

(20) The rapid determination of multiple ions increased sample throughput.

IC is not prone to common interferences associated with traditional wet chemical methods and is adaptable to most extraction procedures. The saml1 sample volumns obtained with extraction procedures does not pose a problem because 10-50 uL of sample is all that is required for analaysis.

And IC has minimized errors associated with sample handling and multiple analytical steps unlike traditional methods.

Future work will include evaluating Ion Chromatographic analysis of transition metals and amino acids in soil and plant extracts and digest.

Analysis of Soil and Plant Extracts

Application of IC in Agriculture

- Soils
- Fertilizers
- Crops
- Plant extracts
- Research
- Preservatives
- Subsurface waters/leachates
- Ground/surface waters
- Acid rain

Typical IC Analyses

- Soils (NO_3^-, PO_4^{3-}, SO_4^{2-}, Na^+, NH_4^+, K^+, Ca^{2+}, Mg^{2+})
- Fertilizers (NO_3^-, PO_4^{3-}, SO_4^{2-}, Na^+, NH_4^+, K^+, Ca^{2+}, Mg^{2+})
- Crops (F^-, Cl^-, NO_2^-, PO_4^{3-}, NO_3^-, SO_4^{2-}, $C_2O_4^{2-}$, Na^+, NH_4^+, K^+, organic acids)
- Research: Nutrient uptake
- Preservatives: Br^-, SO_2

Traditional Analytical Methods

• Anions	
Chloride	Colorimetric, titrimetric
Phosphate	Colorimetric
Nitrate	Colorimetric, electrode
Sulfate	Reduction-distillation colorimetric, turbidimetric
• Alkali metals	
Sodium, Potassium	Flame photometric
• Alkaline earth metals	
Calcium, Magnesium	Atomic absorption or flame photometric

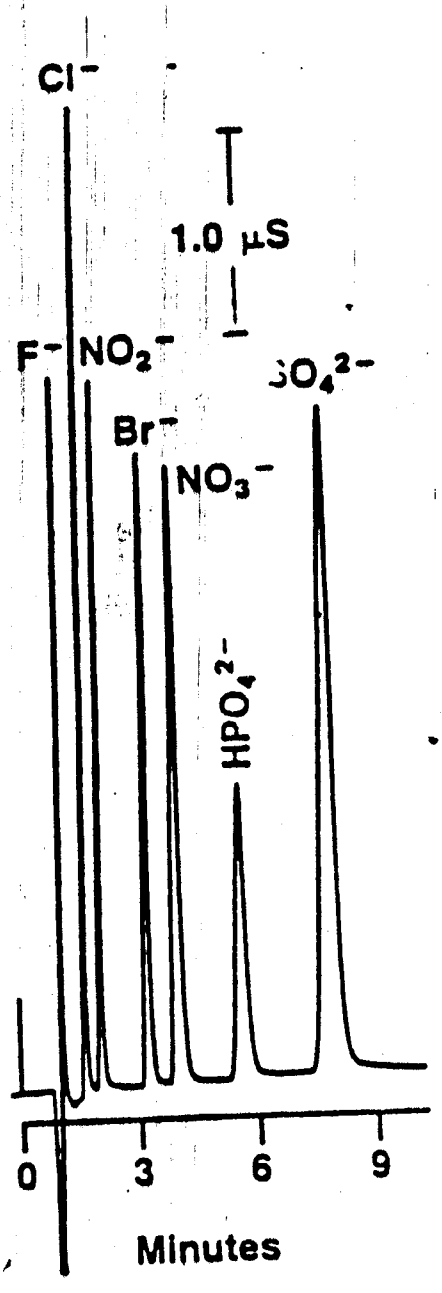
Disadvantages of Traditional Methods

- Labor Intensive
- Extensive sample handling
- Complicated
- Interferences
- Error prone
- Requires analytical skill

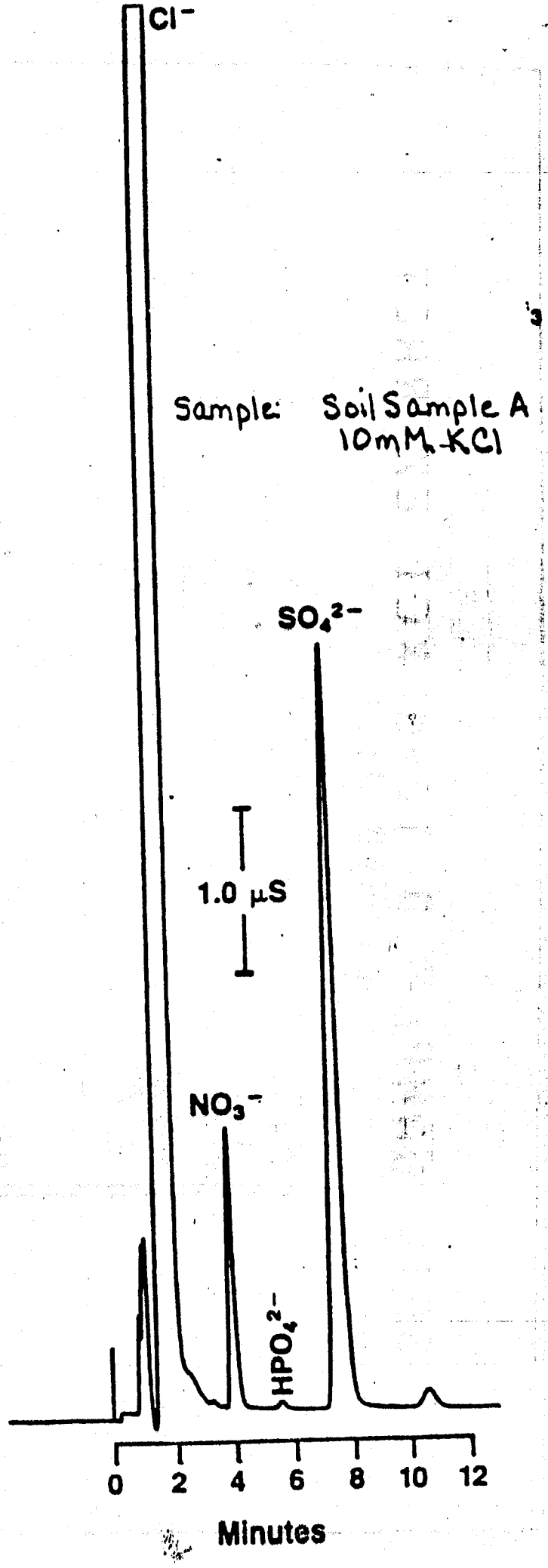
Advantages of Ion Chromatography

- Fast, reliable
- Simplified sample handling
- One step, multi-ion determination
- Reduced interferences
- Small sample volumes
- Cost effective

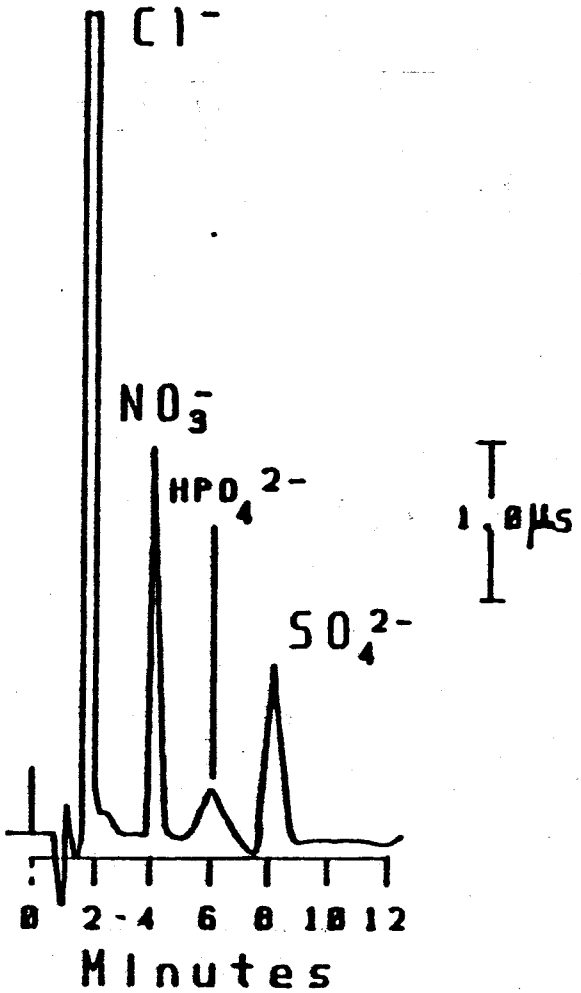
ANION STANDARD

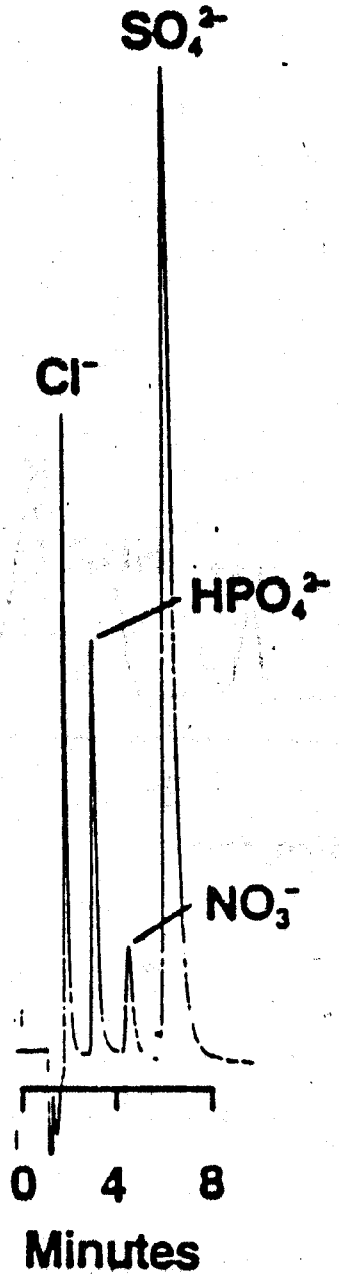


F^-	.5 ppm
Cl^-	1.5 ppm
NO_2^-	2.5 ppm
Br^-	5.0 ppm
NO_3^-	5.0 ppm
$HPO_4^=$	7.5 ppm
$SO_4^=$	7.5 ppm

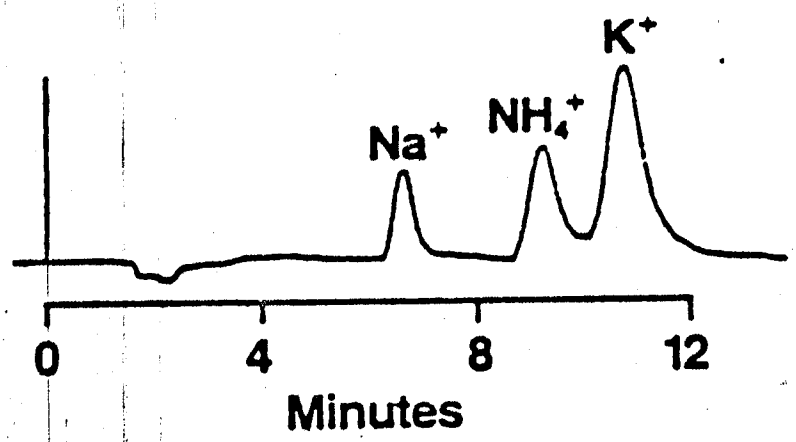

Cl⁻
Sample: Soil Sample A
10mM KCl
SO₄²⁻
1.0 µS
NO₃⁻
HPO₄²⁻
0
2
4
6
8
10
12
Minutes

SOIL SAMPLE B-10mM KCl EXTRACT
(1:500 dil)



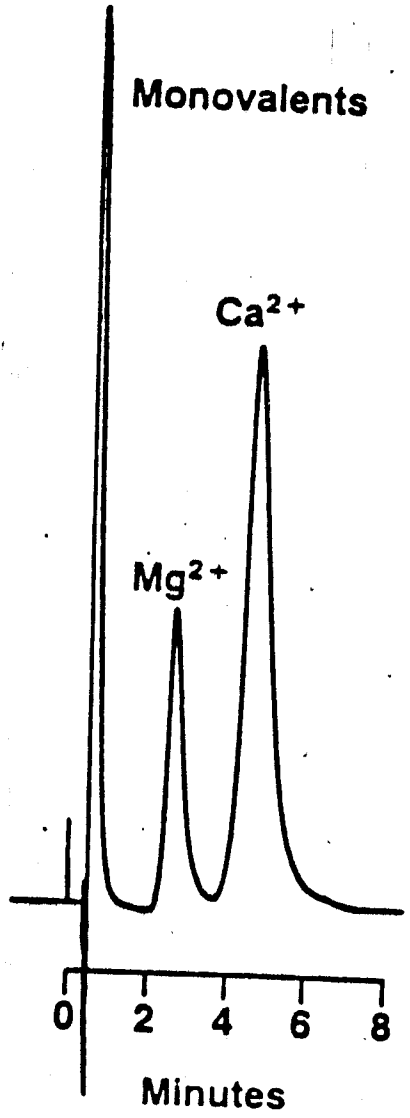


ANALYSIS OF GRANULATED FERTILIZER (ANIONS)

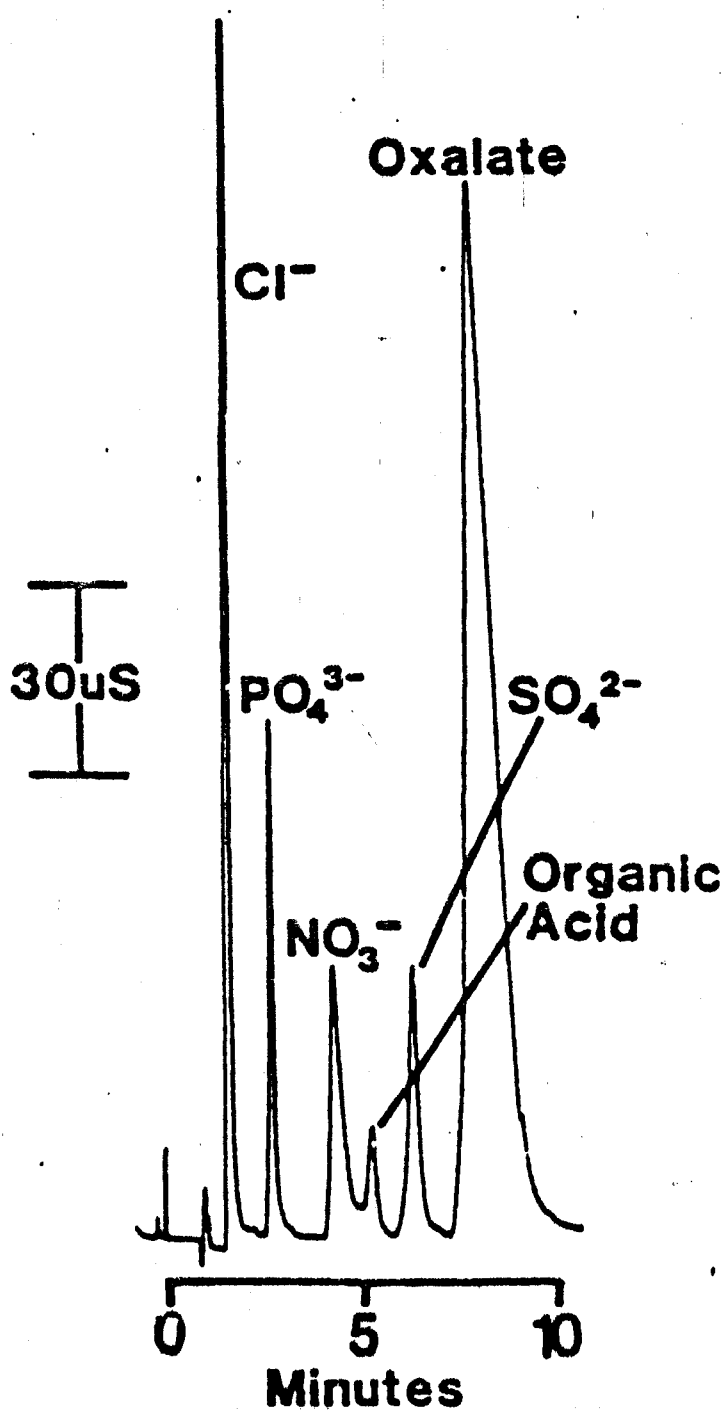


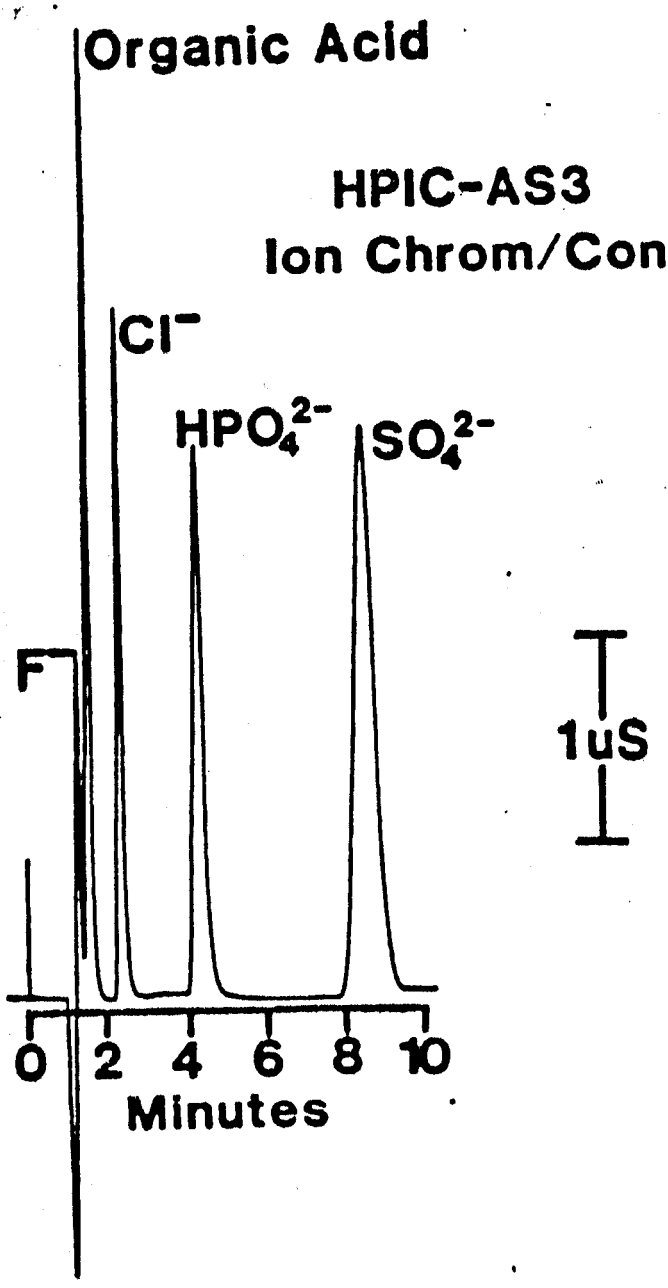
ANALYSIS OF GRANULATED FERTILIZER (MONOVALENT CATIONS)

ANALYSIS OF GRANULATED FERTILIZER
(DIVALENT CATIONS)

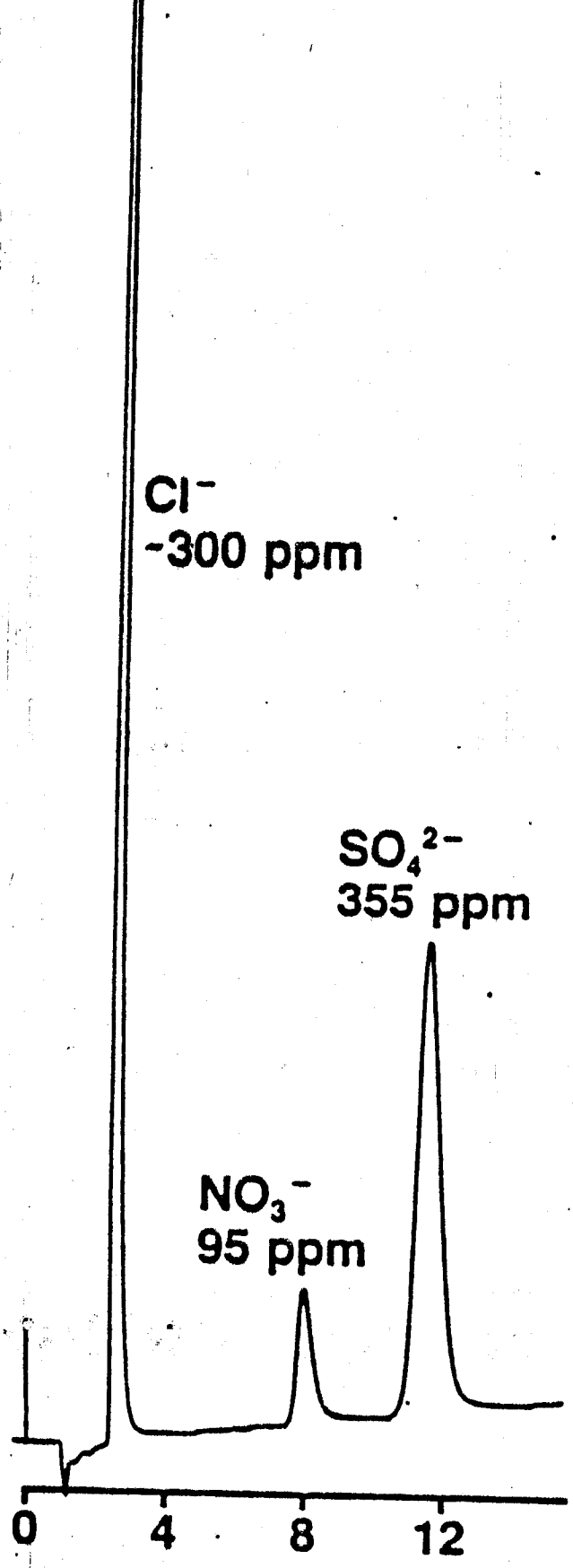


FRESH SPINACH

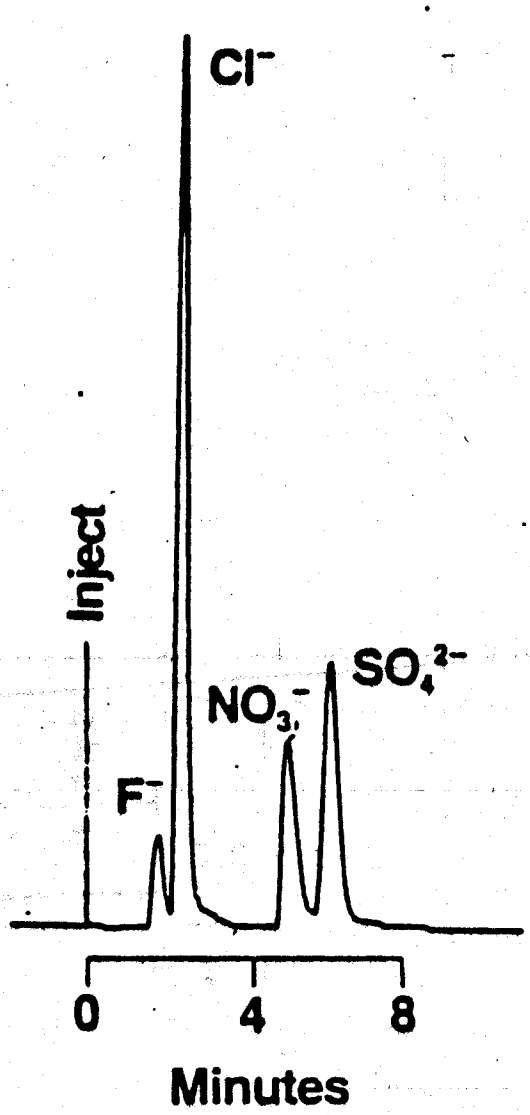




ANIONS IN FRESH ONIONS

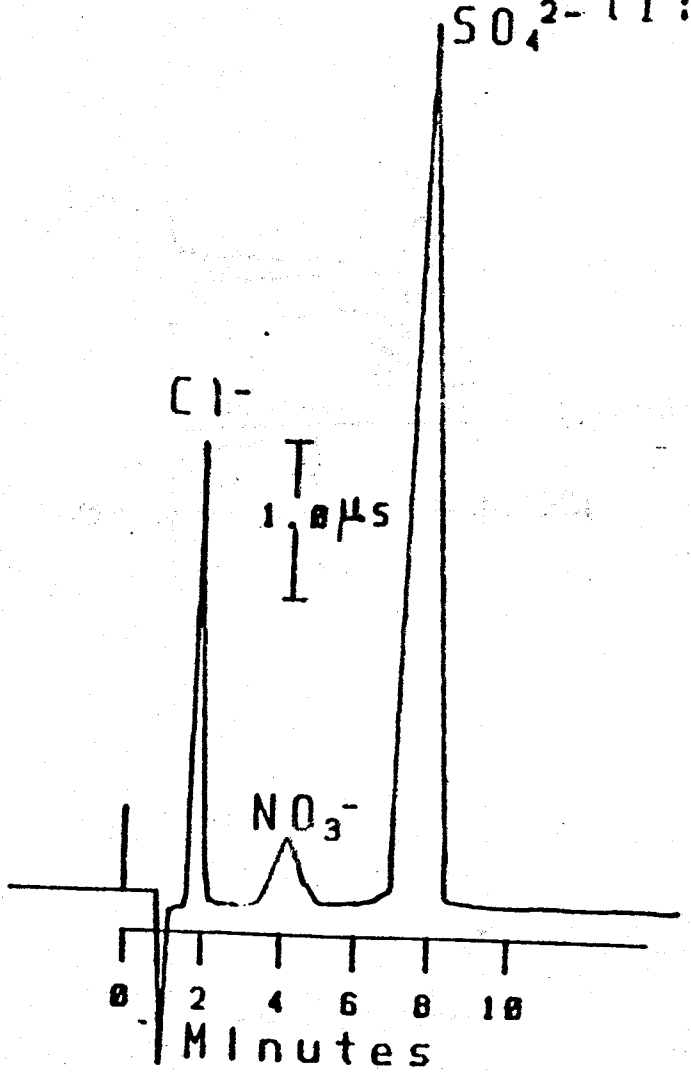


WELL WATER
TRACY, CALIFORNIA
(1:20)

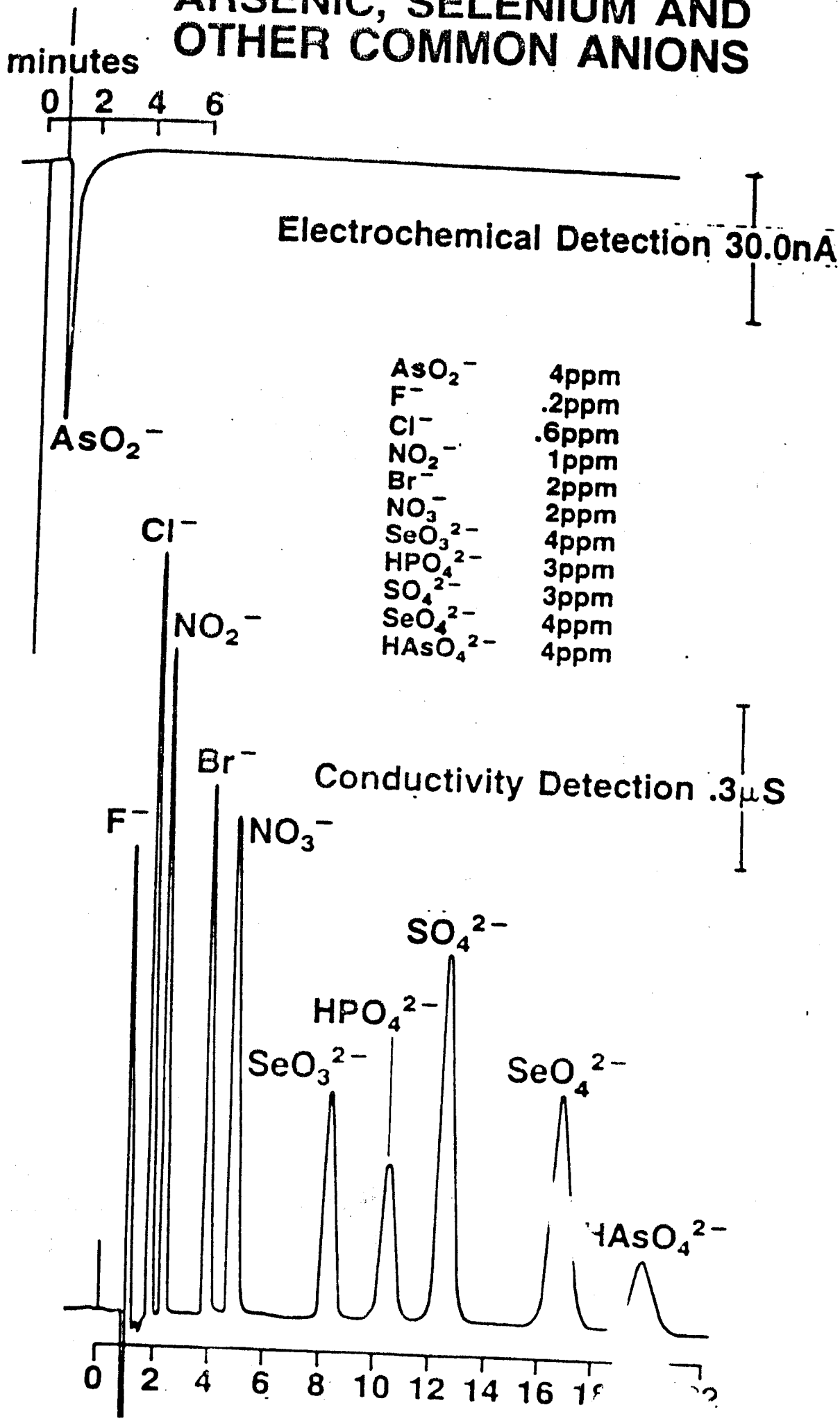


Anions in Acid Rain

COLORADO WELL WATER



ARSENIC, SELENIUM AND OTHER COMMON ANIONS



Summary

- Rapid determination of multiple ions
- Negligible interferences
- Increased sensitivity
- Small sample size
- Simplicity
- Minimized errors

1584