

# Test Method

Number

# 18

## The Determination of Ions in Wipers by Ion Chromatography (IC)



# 1

## Scope

# 2

## Ion Chromatography

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The microelectronic and data storage industries are adversely affected by ionic contamination, e.g. inadvertent doping of silicon wafers and corrosion of metallization lines and disk drive components. This test method describes the ion chromatographic measurement of ionic constituents present in cleanroom wipers.

The Group IA and IIA cations of potassium, sodium, calcium and magnesium and common anions such as chloride, fluoride, nitrate, sulfate and phosphate can be determined to parts per billion levels with this method.

Additionally, procedures are provided herein for the measurement of transition metals such as iron, copper, nickel, zinc, cobalt, cadmium, and manganese. This test method\* extends and improves upon Test Method 12, published earlier by ITW Texwipe (1), which described ionic determination in wipers using Capillary Ion Electrophoresis.

*\*This test procedure does not purport to address any safety procedures associated with its use. It is the responsibility of the user of this procedure to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.*

Ion chromatography (IC) is a sensitive and rapid analytical technique for the qualitative and quantitative determination of a wide variety of ionic species in different matrices and is ideal for the determination of ionic constituents in wipers. Basically, the ions are extracted from the wipers and a sample of the extractant liquid is injected onto a separator column incorporating resins that will separate the component ionic constituents. The ions are eluted through the column and are identified and quantitated using conductivity or UV/VIS detectors and associated software (Figure 1).

This test method describes a deionized water extraction procedure for determination of anions and Group IA and IIA cations, and supplies details on eluents used for each of these categories. Since transition metals are only sparingly soluble in water, a dilute acid mixture is used to extract them from wipers. After extraction, the transition metal cations can be separated in the IC using pyridine-2, 6-dicarboxylic acid (PDCA) as a complexing eluent, and subsequently determined as the post column eluent 4-(2-pyridylazo)resorcinol (PAR) complexes using a UV/VIS absorbance detector.

A number of publications are available from standards organizations (2-5) describing the use of ion chromatography in the semiconductor and data storage industries. Additional information can be found in vendor test methods (6-9). Also, the website <http://www.dionex.com/servletw1/File-Downloader/slot114/15919/Publication-List.pdf> contains many useful references.

It is assumed that the reader is familiar with the principles and operation of ion chromatography instrumentation.

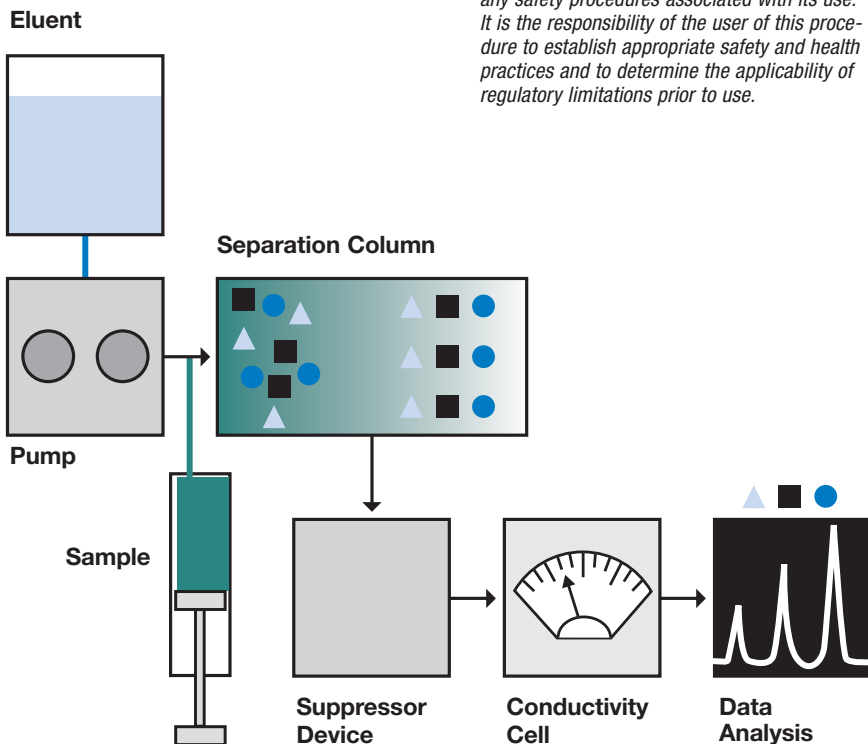


Figure 1, courtesy of Dionex Corporation

## 3 Equipment and Materials

1. IC system such as Dionex Model DX 500 (or equivalent) configured with:
  - GP50 gradient pump
  - CD20 conductivity detector
  - LC30 chromatography oven
  - EG40-KOH eluent generator (for anions only)
  - Sample loops with volumes of 25  $\mu\text{L}$  to 750  $\mu\text{L}$
  - PeakNet 5.1 chromatography workstation
  - Columns and other ancillary equipment needed for specific analysis—see Section 5.Optional: AS 50 Autosampler
2. Equipment for Group IA and IIA cations:
  - IonPac CS12A, 2 mm analytical column with IonPac CG12A 2mm guard column
  - CSRS-ULTRA 2 mm device for chemical suppression
3. Equipment for transition metal cations:
  - IonPac CS5A 2 mm analytical column with IonPac CG5A 2mm guard column
  - AD20 absorbance detector
  - PC10 pneumatic postcolumn controller and injector
  - 375  $\mu\text{L}$  knitted reaction coil
  - Sample loops with volumes of 2.5  $\mu\text{L}$  to 1 mL
  - Optional: TCC-2 Trace cation concentrator (for metals <10 ppb)
4. Equipment for anions:
  - IonPac AS15 2 mm analytical column with IonPac AG15 2mm guard column
  - ASRS-ULTRA 2 mm device for chemical suppression
5. Cast aluminum top hotplate with adjustable temperature (VWR Model Dyla-Dual or equivalent)
6. Heatable Teflon® PFA beaker, 1 L capacity (Nalgene® 1550 type HP or equivalent)
7. Teflon rods, at least 12" long
8. Cleanroom unpowdered latex gloves
9. Class 100 or cleaner laminar flow workstation
10. Polypropylene sample bottles with caps
11. Fine-point duckbill tweezers

## 4 Reagents and Standards

1. Deionized water, ASTM D1193 Type I ( $\geq 18 \text{ M}\Omega\text{-cm}$ )\*
2. 1,000 ppm solutions of ion standards, both anions and cations (High Purity Standards or equivalent)
3. Methanesulfonic acid >99% pure (Fluka, Aldrich or equivalent) for post column elution of Group IA and IIA cations
4. Pyridine-2,6-dicarboxylic acid (PDCA) (Dionex concentrate or equivalent) and 4-(2-pyridylazo)resorcinol (PAR) (Dionex concentrate or equivalent) for post column elution of transition metal cations
5. Ultra-pure HCl and HNO<sub>3</sub> (Ultrex grade, J.T. Baker or equivalent) for extraction of transition metals from wipers

\*High quality water reduces background interference and facilitates accurate determination of ppb levels of ion constituents. The water must be low in particulate matter and should not contain particles larger than 0.2  $\mu\text{m}$  to avoid plugging of chromatographic columns and other hardware.

12. High-purity compressed gas (helium or nitrogen) with regulators as required. *Helium is recommended for sparging eluents or for applying head pressure to the eluent bottles. Nitrogen is recommended for actuating valves.*
13. Vacuum pump with back-flow protection (VWR Scientific or equivalent)
14. Adjustable-volume pipettes, 0.5  $\mu\text{L}$  to 10  $\mu\text{L}$  and 10  $\mu\text{L}$  to 100  $\mu\text{L}$  (Brinkmann Instruments, Inc., Westbury, NY)
15. Top-load balance (Sartorius Model B3100S or equivalent)
16. Membrane filtration apparatus with 47 mm diameter glass base (Millipore type XX1004700, Millipore Corporation, Bedford, MA or equivalent), and 47 mm diameter membrane filters with 0.45  $\mu\text{m}$  pores (Millipore type HA or equivalent)
17. Polypropylene Erlenmeyer vacuum flask, 1L size
18. Polypropylene volumetric flasks, 25, 100, 500, 1,000 and 2,000 mL

*The cleaning of the Teflon beaker, polypropylene sample and squeezing bottles, polypropylene beakers, polypropylene flasks, vacuum flasks, and the filtration apparatus should be done in the following manner:*

- i. Clean all the polypropylene sample containers thoroughly by washing with surfactant and deionized water and rinsing the inner surface at least five times with deionized water.
- ii. Ultrasonically clean the filtration assembly and thoroughly rinse using deionized water.
- iii. Allow all containers and assemblies to drain dry in the laminar flow workstation.
- iv. Store all containers and assemblies in the laminar flow workstation to prevent environmental contamination.

## Instrument Settings

### Group IA and IIA Cations

Column	IonPac CS12A, CG12A, 2 mm
Eluent	20 mM Methanesulfonic acid
Flow Rate	0.25 mL/min
Inj. Volume	750 $\mu$ L
Temperature	35°C
Suppressor	CSRS-ULTRA 2 mm chemical suppressor
Current	50 mA
Mode	External water not recycle mode
Detection	Suppressed conductivity

### Transition Metal Cations

Column	IonPac CS5A, CG5A, 2 mm
Eluent	MetPac PDCA
Flow Rate	0.3 mL/min (for 2mm system)
PAR	0.06 g
PAR Flow Rate	0.15 mL/min
Temperature	35°C
Inj. Volume	750 $\mu$ L
Reaction Coil	375 $\mu$ L knitted reaction coil.
Detection	Visible absorbance, 520-530 nm, with 0.2 mM PAR, 10 mL cell MetPac postcolumn reagent diluent

### Anions

Column	IonPac AS15, AG15, 2 mm
Eluent	EG40 System Gradient analysis 8 mM KOH from 0–6 mins. 8–60 mM KOH from 6–16 mins. 60–8 mM KOH from 25–30 mins.
Flow Rate	0.3 mL/min
Inj. Volume	750 $\mu$ L
Temperature	35°C
Suppressor	ASRS-ULTRA 2 mm chemical suppressor
Current	100 mA
Mode	External water not recycle mode
Detection	Suppressed conductivity

## Analytical Procedure

The analytical procedure consists of 3 parts:

- I. Preparation of eluents and standards
- II. Preparation of system blank and sample solutions
- III. Analysis

All work should be performed under Class 100 or cleaner conditions.

### I-1. Preparation of Eluents and Standard Solutions for the Analysis of Group IA and Group IIA Cations

#### a. 1.0 M methanesulfonic acid\*\* (MSA) stock solution concentrate

Add 96.10 g of methanesulfonic acid to a 1 L volumetric flask containing about 500 mL of deionized water. Mix thoroughly, add additional water to bring the total volume to the 1L mark and remix.

**\*\*Caution:** Methanesulfonic acid is very corrosive and a strong irritant. Avoid breathing the vapors. Always work in a fume hood and wear appropriate gloves and goggles.

#### b. 20 mM methanesulfonic acid

**Note:** This eluent solution should not be kept for more than a month due to possible biological growth.

Prepare working eluent of 20 mM methanesulfonic acid by pipetting 20 mL of 1.0 M methanesulfonic acid into a 1 L polypropylene volumetric flask containing about 500 mL of deionized water. Add additional water to bring the total volume to the 1L mark and mix thoroughly.

#### c. Standard solutions of Group IA and IIA cations

To simplify quantitation of the ions, combined standards are used for preparation of calibration plots. Two types of combined standards are prepared—a combined intermediate standard solution prepared fresh monthly, and combined *working* standard solutions prepared fresh daily.

A combined *intermediate* standard solution is prepared by diluting combined aliquots of the stock 1000 ppm Group IA and IIA cation standards with deionized water. A combined intermediate standard containing 1 ppm each of Li<sup>+</sup>, Na<sup>+</sup>, NH<sub>4</sub><sup>+</sup>, K<sup>+</sup>, Mg<sup>2+</sup> and Ca<sup>2+</sup> has been found to be satisfactory.

Using the intermediate standard solution, prepare combined *working* standard solutions in the 5, 10, 20 and 50 ppb range, again diluting with deionized water. The combined working standards should bracket the expected Group IA and IIA cation concentrations in the wiper extractant.

## Analytical Procedure (continued)

### I-2. Preparation of Eluents and Standard Solutions for the Analysis of Transition Metal Cations

**Pyridine-2,6-dicarboxylic acid (PDCA)** allows speciation and quantification of  $\text{Fe}^{+2}$  and  $\text{Fe}^{+3}$  as well as separation of other metals such as  $\text{Cu}^{+2}$ ,  $\text{Cd}^{+2}$ ,  $\text{Co}^{+2}$ ,  $\text{Zn}^{+2}$  and  $\text{Ni}^{+2}$  (7). Oxalic acid, another transition metal complexing agent, has been used for some IC studies. However, since  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$  cannot be determined with this complexing agent, oxalic acid was not considered for this test method.

#### a. PDCA concentration

The commercially available ready-to-dilute MetPac PDCA eluent concentrates (5X) from Dionex can be used for the analysis of transition metals. PDCA acts as a strong complexing agent that separates the metal complexes by anion exchange.

The composition of the PDCA eluent mixture is as follows:

PDCA	7.0 mM
Potassium hydroxide	66.0 mM
Potassium sulfate	5.6 mM
Formic acid	74.0 mM

#### b. Working PDCA Eluent

In a 1L polypropylene volumetric flask, add 200 mL of the PDCA concentrate to approximately 700 mL deionized water and then add additional deionized water to bring the level to the 1L mark and mix thoroughly. Use this solution as the eluent.

**4-(2-pyridylazo)resorcinol or PAR** is used as a postcolumn reagent for visible absorbance detection at 520 nm-530 nm.

#### c. Postcolumn Reagent Diluents

The MetPac PAR postcolumn reagent diluent (or equivalent) is commercially available to be used with PAR to create the post-column reagent used in the absorbance detection.

1. For the detection of transition metals above 0.5 ppm, dissolve 0.12 g of PAR, 4-(2-pyridylazo)resorcinol into 1L of the diluent.
2. For detection of transition metals below 0.5 ppm, dissolve 0.06 g of PAR, 4-(2-pyridylazo)resorcinol into 1L of the diluent.
3. To enhance the dissolution of the PAR, place the solution in an ultrasonic bath for 5 minutes and mix thoroughly.

**Note:** PC10 pneumatic postcolumn controller in the Dionex system delivers the PAR to the UV/VIS detection system.

PAR Postcolumn Reagent Diluent contains the following ingredients:

2-Dimethylaminoethanol	1.0 M
Ammonium hydroxide	0.5 M
Sodium bicarbonate	0.3 M

**Caution:** To prevent oxidation of PAR, store the reagent under helium. PAR has a shelf life of only about 2 weeks. Do not use PAR that shows signs of precipitation as it might clog the postcolumn system.

#### d. Standard solutions of transition metal cations

A combined *intermediate* standard solution is prepared by diluting combined aliquots of the stock 1000 ppm transition cation standards with deionized water. The ion concentrations in the combined intermediate standard are not all the same, so as to accommodate variations in ion content in the wiper and detector response for the various ions. A combined intermediate standard containing the following concentrations of each ion has been found to be satisfactory:

$\text{Fe}^{+3}$	2 ppm	$\text{Co}^{2+}$	2 ppm
$\text{Cu}^{2+}$	2 ppm	$\text{Cd}^{2+}$	6 ppm
$\text{Ni}^{2+}$	4 ppm	$\text{Mn}^{2+}$	4 ppm
$\text{Zn}^{2+}$	2 ppm		

Using the intermediate standard solution, prepare combined *working* standard solutions in which the  $\text{Fe}^{3+}$  concentration ranges from 1.25 ppb (1600 fold dilution) to 20 ppb (100 fold dilution). Dilution factors of 1600, 800, 400, 200 and 100 have been found to be satisfactory. Note that linearity response for  $\text{Fe}^{3+}$  falls off at very low concentrations (1.25 and 2.5 ppb). The combined working standards should bracket the expected transition metal ion concentrations in the wiper extractant.

### I-3. Preparation of Eluents and Standard Solutions for the Analysis of Anions

The preferred anion eluent is a potassium hydroxide solution generated electrochemically on demand using the Dionex EG40-KOH Eluent Generator (or equivalent). The eluent concentration increases from an initial value of 8 mM KOH to a steady state value of 60 mM KOH. Refer to the manufacturer's literature for details.

Alternatively, a carbonate/bicarbonate solution can be used as the anion eluent (with different analytical and guard columns than the ones specified in this test method), but this solution causes an undesirable baseline shift early in the chromatogram which interferes with detection and quantitation of early eluting species, e.g.  $\text{F}^-$  and  $\text{Cl}^-$ .

#### Standard Solutions of Anions

A single intermediate standard solution is prepared by diluting combined aliquots of the stock 1000 ppm anion standards with deionized water. The ion concentrations in the combined intermediate standard are not all the same, so as to accommodate variations in ion content in the wiper and detector response for the various ions. A combined intermediate standard containing the following concentrations of each ion has been found to be satisfactory:

$\text{F}^-$	1 ppm	$\text{Br}^-$	3 ppm
$\text{Cl}^-$	1 ppm	$\text{NO}_3^-$	3 ppm
$\text{NO}_2^-$	2 ppm	$\text{PO}_4^{3-}$	3 ppm
$\text{SO}_4^{2-}$	2 ppm		

## Analytical Procedure (continued)

Using the intermediate standard solution, prepare combined *working* standard solutions in which the  $F^-$  concentration ranges from 1 ppb (1000 fold dilution) to 100 ppb (100 fold dilution). Dilution factors of 1000, 500, 100, 40, 20 and 10 have been found to be satisfactory. The combined working standards should bracket the expected anion concentrations in the wiper extractant.

### II. Preparation of System Blank and Sample Solutions

#### a. Deionized Water Extraction for Water-Soluble Ions

**Overview:** The procedure for extraction of Group IA and Group IIA cations and Group VIIA anions from cleanroom wipers involves placing multiple wipers in hot deionized water for a set time period, squeezing the wipers and collecting the extract solution after squeezing into a second beaker. Alternate extraction times and temperatures are described in the literature (2,5). The extract solution can also be preconcentrated by evaporation to enhance the detection limit of trace level ions (see below).

1. Clean two 1L heatable Teflon beakers (beakers 1 and 2) and a Teflon rod by washing with nonionic surfactant and deionized water and by rinsing at least five times with deionized water.
2. Fill both beakers each with 900 mL deionized water (DIW). Place the beakers on a hotplate and bring the water to a temperature of 80°C while stirring periodically using the Teflon rod. Remove the beakers from the hotplate and, after cooling, transfer 50 mL aliquots of water from each beaker into a clean polypropylene sample bottle. This water is used as the system blank solution. Empty both beakers.
3. Fill beaker 1 again with 750 mL deionized water. Place the beaker on the hotplate and bring the water to a temperature of 80°C. Remove the beaker from the hotplate.
4. Using clean tweezers to grasp the wipers, weigh sufficient wiper material to yield 50–60 grams of sample (for 9" x 9" polyester cleanroom wipers, the usual requirement is 10 wipers).
5. Document the total weight of material to the nearest milligram.
6. Place the wipers, one at a time, in the hot deionized water in the beaker, prodding and stirring periodically using the Teflon rod, for a total soak time of 15 minutes. Decant the extract solution into beaker 2.
7. Add another 150 mL of deionized water at ambient temperature to beaker 1 with the wipers. Using the bottom of a clean plastic bottle, squeeze the wipers in a piston-like motion five times. While squeezing, decant the extract solution at the same time from beaker 1 into beaker 2 by holding beaker 1 at a 45° angle over beaker 2. Allow the entire free-flowing liquid to drain into beaker 2.

8. Allow the solution in beaker 2 to cool down to room temperature. Use the aliquots of the extract solution to analyze both cations and anions using IC. Discard the wipers in beaker 2.

#### b. Acid Extraction for Transition Metal Cations

Because of the relative insolubility of transition metals in deionized water, the wipers are extracted with dilute acid if those metals must be determined. The procedure above is followed except that:

- (i) deionized water is replaced with either a 0.3 M HCl solution or a 0.15 M HCl/0.15M  $HNO_3$  solution and
- (ii) the extraction is performed for one hour at room temperature.

The following parameters for the extraction process must be noted:

1. Material ID/Lot #	Sample A / Lot # 0000
2. Material Weight	W to the nearest milligram
3. Initial DIW	750 mL
4. Additional DIW	150 mL
5. Total DIW	900 mL
6. DIW/Material	$(900/W) = R$

(as an example, if  $W = 53.430$  g, then  $R = 16.84$  mL/g)

The value of **R** is needed for the final calculation. See Section 7 and 8 for details on how to calculate **R** if a preconcentration step is used.

### III. Analysis

**Overview:** The extract solution from the wipers or other materials is analyzed for the presence of cations and anions using IC.

Instrumental sensitivity may dictate appropriate selection of sample loops and extent of sample dilutions in each case. Sensitivity of the method can be enhanced by injecting more sample. This is done by increasing the sample loop volume or by using a preconcentration technique. Methods are specific to the sizes of the sample loops. A large 750  $\mu$ L loop works well for low ppb level detection without any preconcentration, particularly for cations.

It is good practice to analyze the system blank\* first to ensure that equipment and reagents are not contaminated.

*\*If ion values in the blank exceed 15% of the corresponding ion values in the wiper extract, then the source of extraneous contamination in the blank must be eliminated or reduced before proceeding.*

The following figures show chromatograms of standards and samples for the Group IA and IIA cations, the anions and the transition metal cations.

# Analytical Procedure (continued)

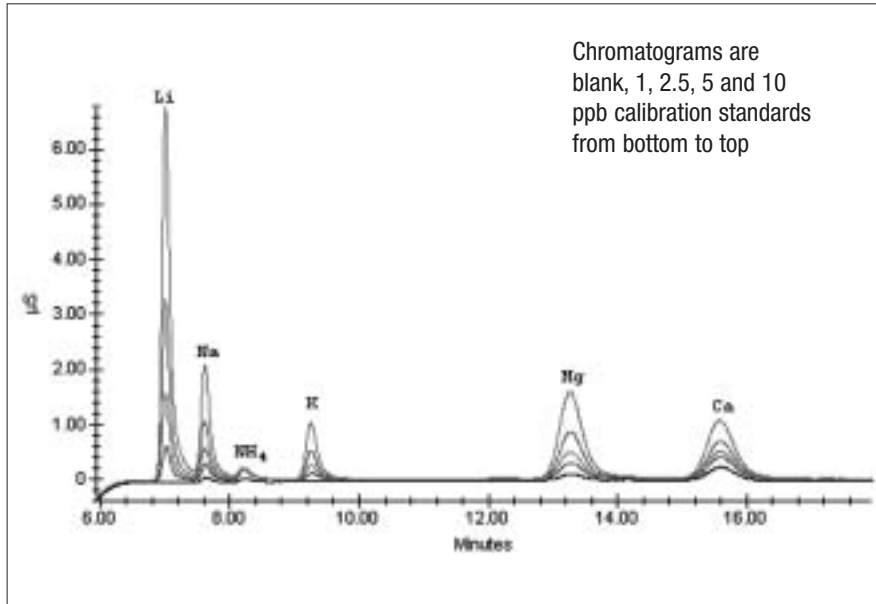


Figure 1: Cation standards by IC

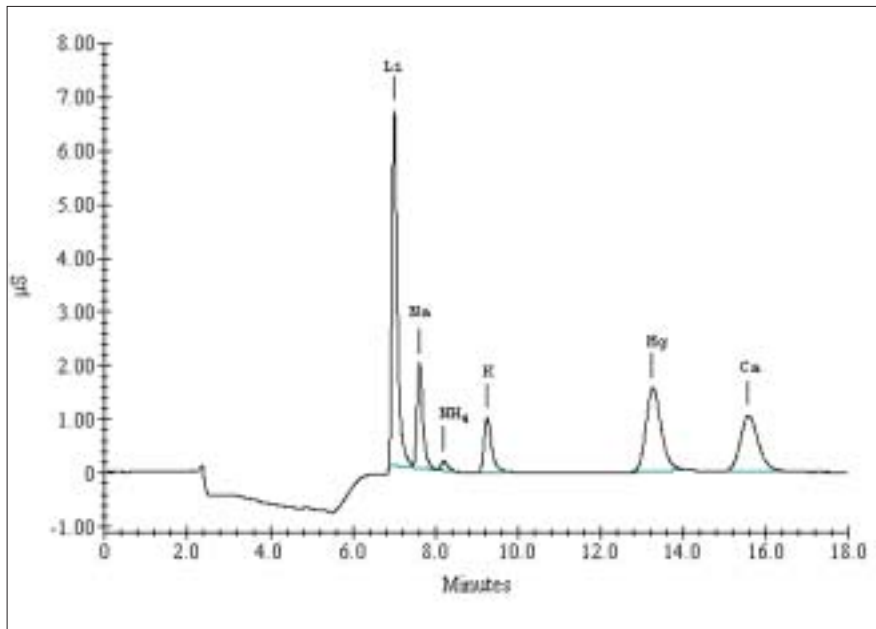


Figure 2: Typical analysis of cations in wiper extract solution by IC.

### Cation concentrations for Figure 2

Ions	Concentration (ppb)
Li <sup>+</sup>	not determined
NH <sub>4</sub> <sup>+</sup>	not determined
Na <sup>+</sup>	58.47
K <sup>+</sup>	10.60
Mg <sup>2+</sup>	28.87
Ca <sup>2+</sup>	48.41

## Analytical Procedure (continued)

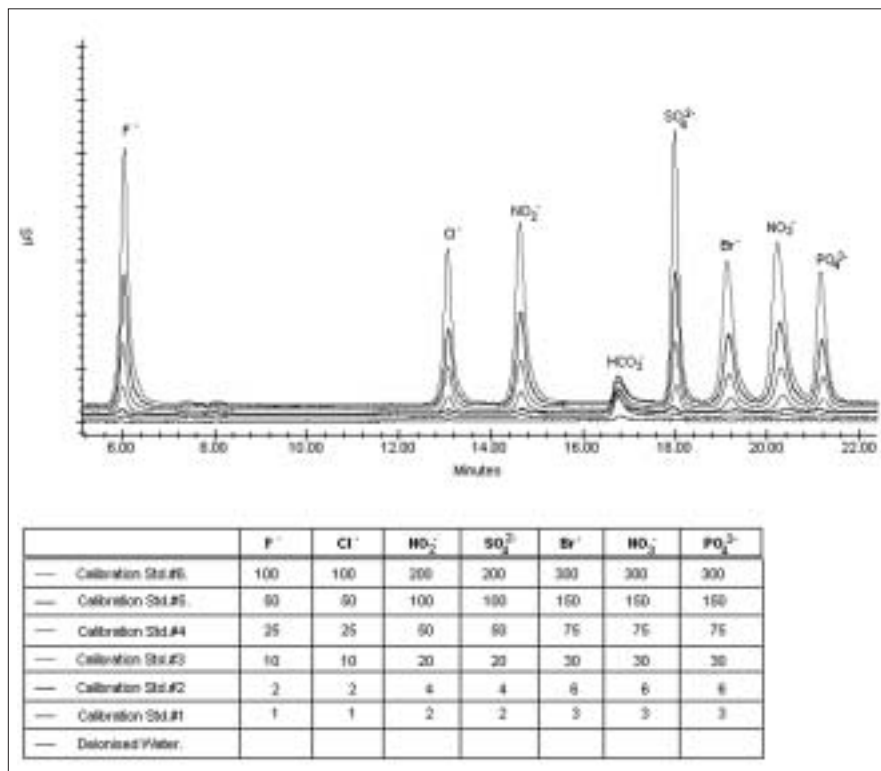


Figure 3: Anion standards by IC

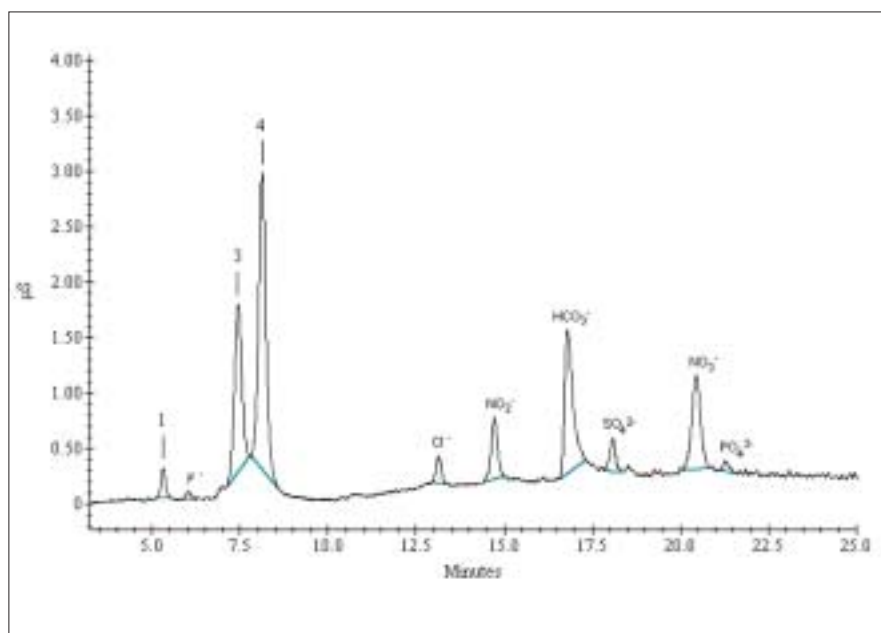


Figure 4: Typical analysis of anions in a wiper extract solution by IC.

### Anion concentrations for Figure 4

Ions	Concentration (ppb)
------	---------------------

F <sup>-</sup>	0.9
Cl <sup>-</sup>	1.8
NO <sub>2</sub> <sup>-</sup>	10.0
SO <sub>4</sub> <sup>2-</sup>	5.2
NO <sub>3</sub> <sup>-</sup>	28.0
PO <sub>4</sub> <sup>3-</sup>	5.3

Peaks 1, 3, 4 (organic species) and the peak for HCO<sub>3</sub><sup>-</sup> were not quantitated.



# Analytical Procedure (continued)

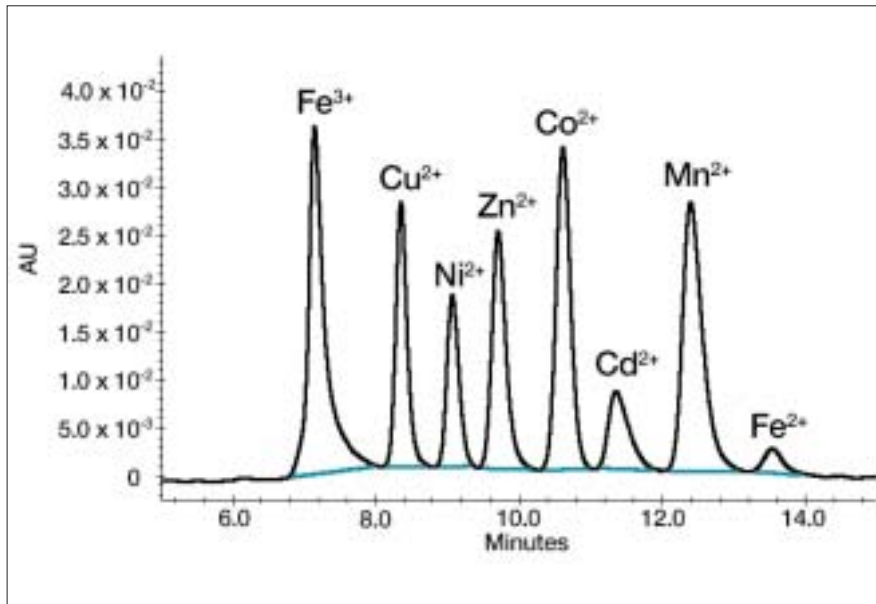


Figure 5: IC of a mixed standard solution of transition metals

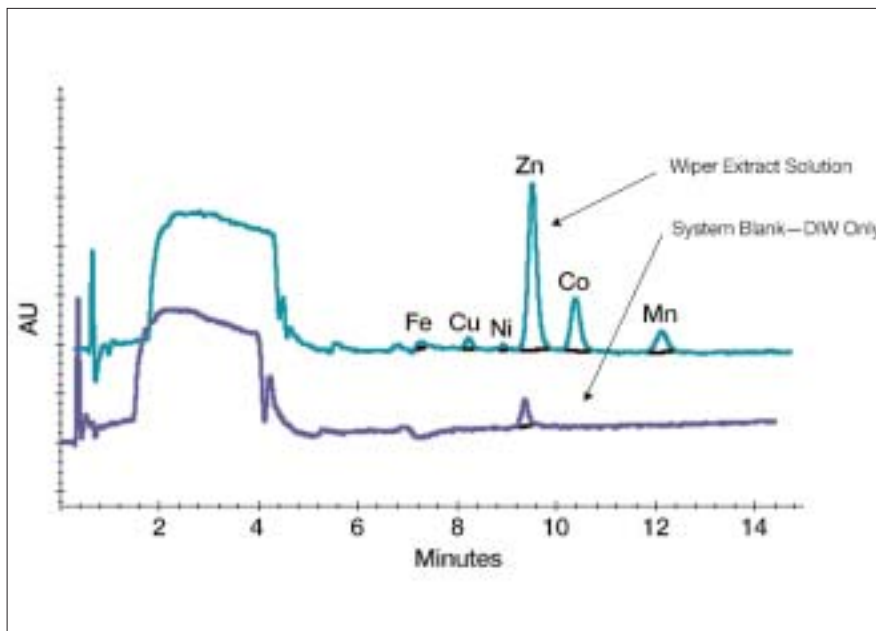


Figure 6: Typical analysis of transition metal cations in wiper extract solution by IC.

### Transition Metal Cation concentrations for Figure 6

Ions	Concentration (ppb)
Fe <sup>3+</sup>	16
Cu <sup>2+</sup>	3
Ni <sup>2+</sup>	2
Zn <sup>2+</sup>	33
Co <sup>2+</sup>	13
Mn <sup>2+</sup>	14

## Sample Calculations

To quantify each ion\*, use the computer software (PeakNet software on Dionex system) to perform regression analysis on the calibration plot for standard solutions for that ion, then have the software calculate the concentration of the unknown from the measured peak area.

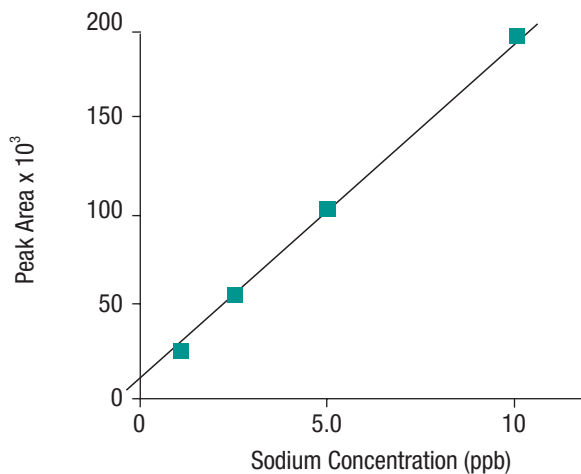
Most often, the calibration plots are linear and the software calculates the slope **m** and the intercept **b** for the best straight line for each ion\*\*. Note the calibration plots are required for each ion to be determined and each plot will have its own unique **m** and **b** values.

The concentration **x** of the ion in the wiper extract solution is calculated in software from the equation  $x = (y - b) / m$ , where **y** = the peak area for the ion in the extract. Determine the concentration of the ion in the wiper by multiplying **x** by **R**, the ratio of volume of extractant solution to the weight of the dry wipers.

As an example, consider the quantitation of sodium ions in clean-room polyester knitted, laundered wipers. The standards data might look like:

[Na <sup>+</sup> ] in (ppb)	Peak Area x 10 <sup>3</sup>
1.00	26.3
2.50	56.2
5.00	103.1
10.00	196.2

The plot for peak area vs the sodium concentration looks like:



Calibration plot of sodium standards.

From this data, the IC software produces a slope **m** of 18,656 an intercept **b** of 10,750. The sample measurement exhibits a peak area of 131,261 for sodium ion. From this  $x = (131,261 - 10,750) / 18,656 = 6.46$  ppb is determined as the concentration of sodium ion in the extractant.

Multiplying this by **R** = 16.84 mL/g, determined earlier (Section 6-II) produces a concentration of 6.46 ng/mL x 16.84 mL/g = 108.8 ppb sodium ion in the wipers.

If a preconcentration step has been employed, then adjust the value of **R** by dividing by the preconcentration factor. As an example, if the extractant volume has been reduced by a factor of 10, then in the above example **R** would be 16.84/10 = 1.684 mL/g.

\*Typically, the system blank does not exhibit any measurable peak areas. If peaks are detected in the system blank, they must be quantified and the blank contribution must be subtracted from the sample values.

\*\*The PeakNet software displays calibration plot data in the "Component Data Plot" report. In this report, concentration is displayed on the x-axis and the peak area is displayed on y-axis. However, the values of slope (labeled  $k_1$ ) and y-axis intercept (labeled  $k_0$ ) that are calculated by the software correspond to the axes reversed — i.e., concentration on the y-axis and peak area on the x-axis. The true slope value **m**, can be calculated from  $m = 1/k_1$  and the value of y-axis intercept **b** can be calculated from  $b = -k_0 k_1$ . Note that the software internally corrects the axis reversal and calculates ion concentrations for samples properly.

**Method Detection Limits**

The method detection limits (MDLs) are 1 ppb for the Group IA and IIA cations and 5 ppb for the anions. MDLs were not established for the transition metal cations. For each ion, least squares analysis was performed on replicated calibration data, and the corresponding zero concentration intercept (“b”), and slope (“m”) were determined. The MDL for that ion was determined from the equation

$$\text{MDL} = (\text{UCL}-b)/m$$

where UCL is the 3 sigma equivalent upper confidence limit for signal at zero concentration. A complete description of the calculation procedure can be found in Reference 10.

**Sample Preconcentration**

Ionic contamination in cleanroom knitted polyester, laundered wipers is typically in the sub 500 ppb range. To increase sensitivity at trace levels by IC, it is necessary to extract multiple wipers in a minimum amount of water at 80°C for 15 minutes and then use a larger injection loop, such as 750 µL, for the sample injection. It may even be necessary to preconcentrate the extract solution by evaporation prior to the injection. Preconcentration is most effectively accomplished by heating the extract solution in the Teflon PFA beaker specified in Section 3. Weigh the empty beaker, the extract before and after heating and calculate the preconcentration factor as the ratio of the extract weight before heating to that after heating. Divide “R” by the preconcentration factor in Section 7, Sample Calculations.

This can provide an improvement factor of up to 10–20. The preconcentration procedure, even though a time-consuming process, significantly enhances the ability of detecting even traces of ions present in very clean wipers.

Additionally, the Dionex DX 500 IC as well as other IC’s can be equipped with preconcentration columns. With this technique, the injection loop is replaced by a short guard or preconcentration column containing ion exchange material similar to the analytical column. This column concentrates ions from a measured volume of sample solution and thereby improves the detection limits.

1. “The Determination of Ions in Wipers and other Materials by Capillary Ion Analysis (CIA),” *Texwipe Test Method 12*, 1996.
2. “Measurement of Extractable/Leachable Cation Contamination Levels on Drive Components by Ion Chromatography (IC),” IDEMA, Document No. M12-99 (1999).
3. “Special Cations Analysis,” IDEMA, Document No. M10-98 (1998).
4. “Measurement of Extractable/Leachable Anion Contamination Levels on Drive Components by Ion Chromatography (IC),” IDEMA, Document No. M13-99 (1999).
5. “Standard Test Method for Anions in Water by Chemically Suppressed Ion Chromatography,” *ASTM Standard Test Method D4327-91*, Vol. 11.01.
6. “Determination of Transition Cations in Concentrated Acids Using AutoNeutralization™ Pretreatment/Ion Chromatography,” *Application Note 94*, Dionex Corporation.
7. “Determination of Transition Metals by Ion Chromatography,” *Technical Note 10*, Dionex Corporation.
8. “Determination of Trace Anions in High Purity Waters by High Volume/Direct Injection Ion Chromatography,” *Application Note 113*, Dionex Corporation.
9. “The Use of Concentrator Columns in Ion Chromatography,” *Technical Note 8*, Dionex Corporation.
10. “Guide to Determination of Method Detection Limits,” *SEMI C10-0698*.



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