الشركة القابضة
لمياه الشرب والصرف الصحى

برنامج المسار الوظيفي للعاملين بقطاع مياه الشرب والصرف الصحي

دليل المتدرب

Basic Course in Inorganic Instruments

كيميائي مياه- الدرجة الثالثة

حقوق الطبع محفوظة
الشركة القابضة لمياه الشرب والصرف الصحى
الإدارة العامة للمسار الوظيفى - إدارة المناهج

تم إعداد المادة بواسطة الشركة القابضة لمياه الشرب والصرف الصحي
قطاع تنمية الموارد البشرية - الادارة العامة لتخطيط المسار الوظيفي
الإصدار الثانى - 2019.

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مقدمة الإصدار الثانى

تهدف مجموعة البرامج التدريبية المعدة من إدارة المسار الوظيفى بالشركة القابضة لمياه الشرب والصرف الصحى إلى رفع كفاءة الكيميائين العاملين بالشركة القابضة لمياه الشرب والصرف الصحى والشركات التابعة لها وتنمية مهاراتهم ومعارفهم بالشكل الذى يضمن الوصول إلى كوب مياه نظيف وبيئة آمنة يرضى متطلبات وإحتياجات العملاء الكرام.

ويعتبر الإصدار الثانى من برامج المسار الوظيفى لوظيفة كيميائى مياه الشرب هو ثمرة جهود الكيميائيين العاملين بمعامل الشركات التابعة والمعمل المرجعى لمياه الشرب بالشركة القابضة بما تحمله من مزيج متجانس من الخبرات والكفاءات الذين لم يدخروا جهدا حتى يخرج هذا العمل بالطريقة اللائقة.

وجدير بالذكر أن هذا الإصدار يعتبر مكتبة مرجعية وافية وشاملة لجميع الجدارات المتضمنة المهارات والمعارف التي تجعل الكيميائى كفؤا لوظيفته.

ومما تجدر الإشارة إليه بأنه تم الاعتماد على منهجية للمسار التدريبى بحيث يكون المتدرب قد تلقى الدورات الحقلية بداية من التعرف على مراحل التنقية والمعالجة ثم الانتقال إلى الدورات المعملية داخل معمله طبقا للإطار الزمنى المحدد للمدد البينية لكل درجة وظيفية.

ولقد اعتمدنا على وضع معايير لكل مرحلة فى إعداد هذا الاصدار وكان من أهم هذه المعايير:

- المشاركة الفعالة للخبرات والكفاءات التدريبية بالشركات التابعة فى وضع المناهج بما يناسب عموع الكيميائيين على مستوى الجمهورية.
- عقد ورشة عمل متخصصة لكل مادة تدريبية يشارك بها جميع المدربين ذوى التخصص والخبرات سواء من المعمل المرجعى أو معامل الشركات فضلا عن أن يكون المدرب قد قام بتدريس هذه المادة مرات عديدة.
- استخدام وسيلة اتصال غير تزامنى بين جميع المدربين المعتمدين لكل مادة على حدة من خلال انشاء جروب على الفيس بوك لكل مادة على حده (مذكور فى دليل المدرب).
- وضع حقيبة تدريبية كاملة لكل برنامج معدة طبقا لأحدث النظم والمعايير العالمية تحتوى على (دليل المتدرب- شرائح العرض- ملحقات مقروءة ومرئية- دليل المدرب- بنك الأسئلة).
- بناء المحتوى لكل برنامج تدريبى طبقاً لأحدث المراجع العالمية ومن أهمها كتاب الطرق القياسية لتحليل مياه الشرب والصرف الصحى (الإصدار رقم 23) وبما يتوافق مع متطلبات آخر إصدارات الايزو(17025)، مع مراعاة التحديثات الخاصة بالتشريعات والقوانين ذات الصلة.

وجدير بالذكر أن الإصدار الثانى من البرامج التدريبية اعتمد فى تصميمه على عرض مبسط للمعلومات قدر الامكان طبقاً للأسس العلمية وطبقاً للجدارات المعتمدة على تحديد أهداف واضحة وصريحة لتدريب المتدربين، وتشتق تلك الجدارات من الفهم الواضح لدور المتدرب طبقا لبطاقة الوصف الوظيفى، وتتضمن معارف ومهارات وسلوك. مما يضمن إكساب المتدرب مهارات سلوكية بالإضافة إلى المواد التخصصية.

كما تم تصميم العديد من ورش العمل على أساس تسهيل و تسريع عمليتي التعلم و كسب المهارات بما يسمح بتعظيم الفائدة من العملية التدريبية.

كذلك تم استخدام أساليب التدريب الحديثة والاعتماد على التدريب التفاعلى والتركيز على الجوانب التطبيقية في استخدام الوسائل والأساليب المختلفة ، كما تم استخدام الطرق الحديثة للتعليم التفاعلي

والغير تزامني كمصادر مساندة للتعلم من خلال انشاء جروب على الفيس بوك للمدربين المعتمدين **(HCWW Trainers)** .

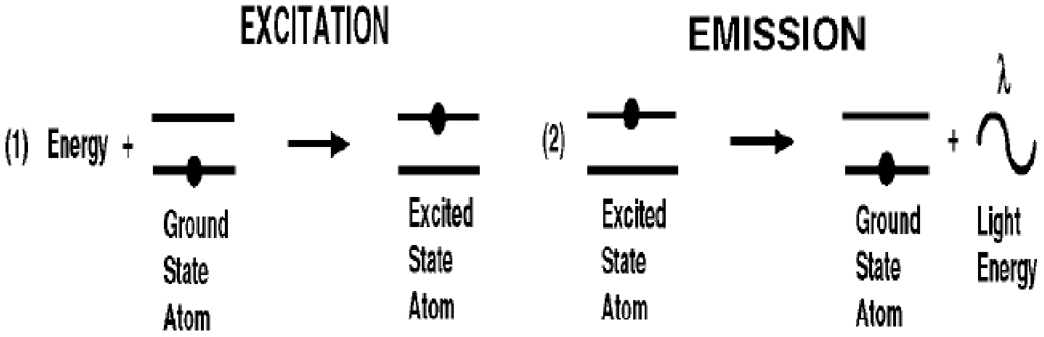
وفى الختام نرجوا من الله أن يتقبل منا هذا العمل كما نأمل أن يكون هذا العمل علما نافعا للعاملين بقطاع المعامل بالشركة القابضة والشركات التابعة لما يشمله من معلومات فنية قيمة وأن يفيد العاملين الجدد بها ليصبحوا قادرين على تنفيذ مهامهم الوظيفية بالشكل الأمثل .

والله ولي التوفيق.

1. INTRODUCTION TO ATOMIC ABSORPTION SPECTROMETRY (AAS)

1.1 Atomic absorption process

Atomic Absorption is that If light of just the right wavelength impinges on a free, ground state atom, the atom may absorb the light as it enters an excited state**.** The quantity of interest in atomic absorption measurements is the amount of light at the resonant wavelength which is absorbed as the light passes through a cloud of atoms. As the number of atoms in the light path increases, the amount of light absorbed increases in a predictable way. By measuring the amount of light absorbed, a quantitative determination of the amount of analyte element present can be made. The use of special light sources and careful selection of wavelength allow the specific quantitative determination of individual elements in the presence of others.



1.2 Principles of Quantitive Analysis by Atomic Absorption

Light at the resonance wavelength of initial intensity, Io, is focused on the flame cell containing ground state atoms. The initial light intensity is decreased by an amount determined by the atom concentration in the flame cell. The light is then directed onto the detector where the reduced intensity, (I), is measured. The amount of light absorbed is determined by comparing (I) to (Io).

Beer's Lambert law

$I_o = I_a + I_t + (I_r + I_s)$

$I_o = I_a + I_t$ $\quad$ $I_a = I_o - I_t$

- Lambert law: The rate of decreasing intensity of incident light is proportional to cell thickness (L)

$$\text{Log } I_o / I_t = K\ L \quad ..... \quad 1$$

- Beer’s law: The rate of decreasing intensity of incident light is proportional to concentration of the solution in the cell

$$\text{Log } I_o / I_t = K^{/}\ C \quad ......2$$

From 1 and 2

$$A = \log I_o / I = K\ K^{/}\ C\ L = £CL$$

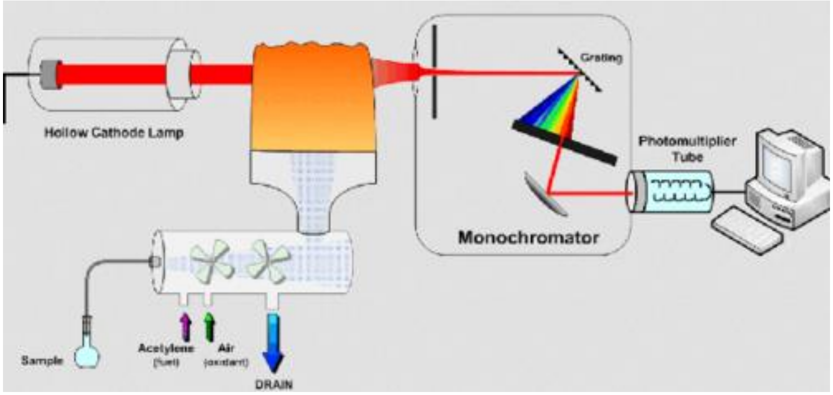
$$A = £CL$$

1.3 Atomic Absorption Spectrometry (AAS)

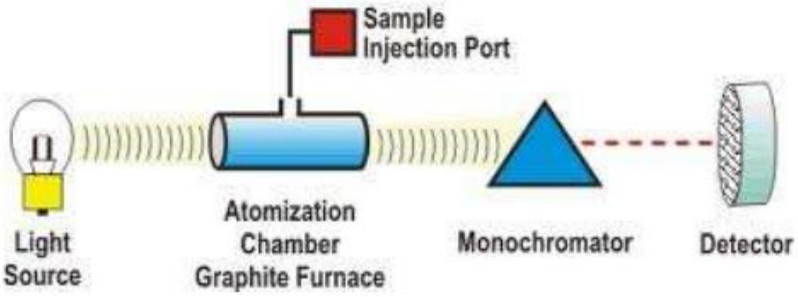
The aim of this type of devices is to determine the concentrations of the metals in different concentration levels and different matrices.

1.4 Types of Atomic Absorption Techniques

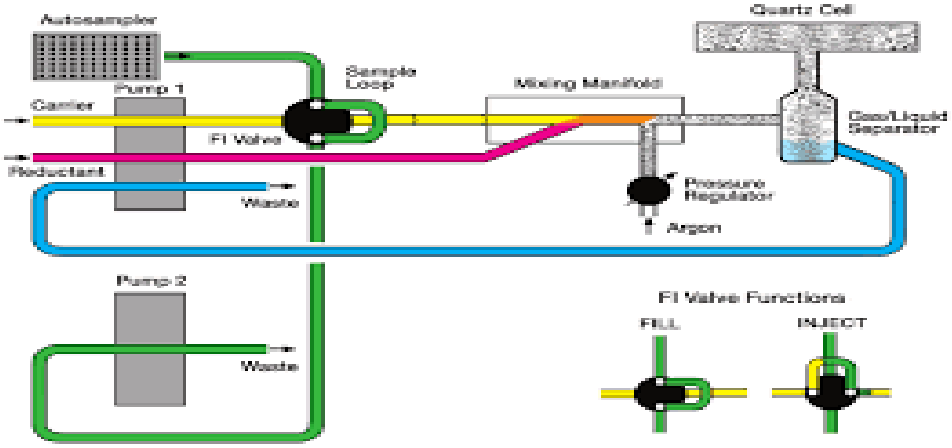
A. **Flame Atomic Absorption** (the operation instructions depends on the manufacturer manual and the analytical application is based on SM3111)



B. **Electrothermal Atomic Absorption, Graphite Furnace AA** (the operation instructions depends on the manufacturer manual and the analytical application is based on SM3113)



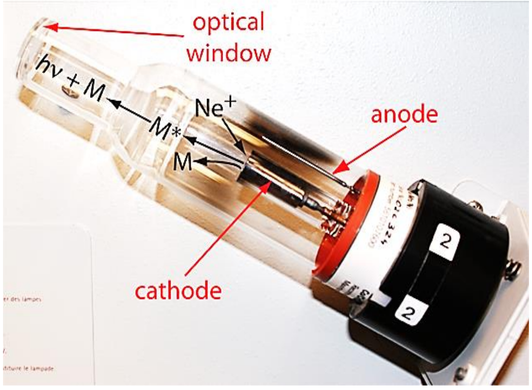
C. **Hydride Generation Atomic Absorption** (the operation instructions depends on the manufacturer manual and the analytical application is based on SM3114)



1.5 The main components of AAS

1.5.1 Light Source

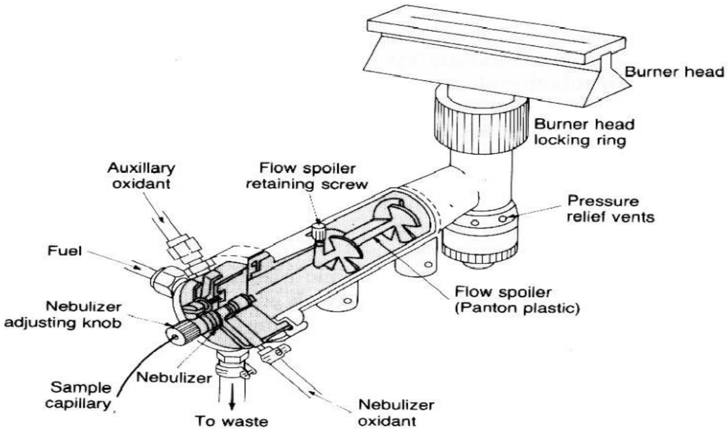
The most common light source used in atomic absorption is the ‘‘Hollow Cathode Lamp - HCL’’. It is an excellent, bright line source for most of the elements determinable by atomic absorption. It is composed of Cathode/Anode, filled with an inert gas (Ne or Ar) and the exit window composed of Pyrex or quartz depending on wavelength produced. The best alternative for HCL is Electrodeless Discharge Lamp – EDL that described as the most sensitive lamp but it is limited to some metals only.



1.5.2 The Atomic Absorption Atomizer

A. For Flame Atomic Absorption Spectroscopy Technique:

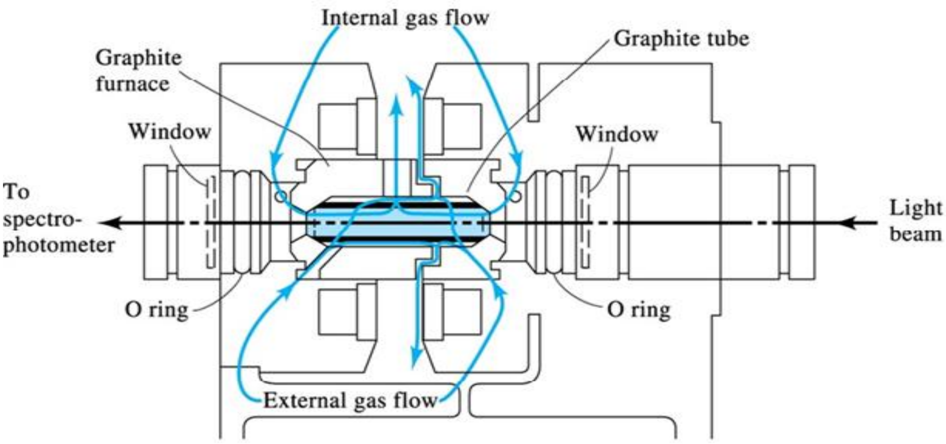
Atomization occurs in a flame created by mixing a fuel with an oxidant. Sample solution is aspirated through a nebulizer and sprayed as a fine aerosol into the mixing chamber. Here the sample aerosol is mixed with fuel and oxidant gases and carried to the burner head, where combustion and sample atomization occur.



Only a portion of the sample solution introduced into the burner chamber by the nebulizer is used for analysis. The finest droplets of sample mist, or aerosol, are carried with the combustion gases to the burner head, where atomization takes place and the excess sample is removed from the premix chamber through a drain.

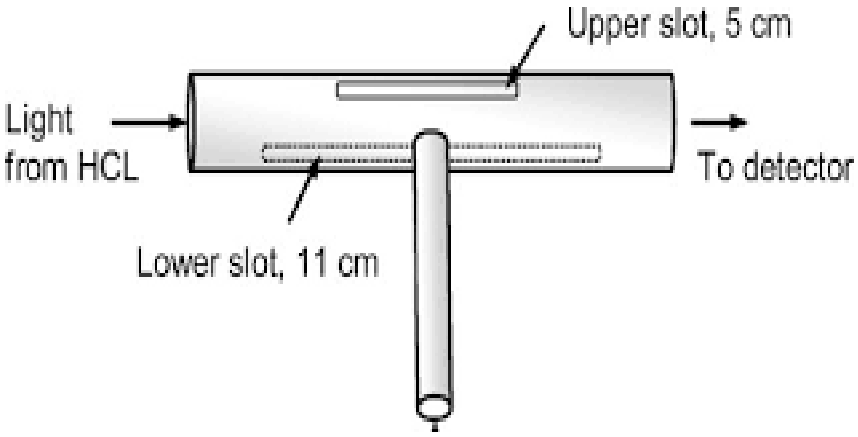
B. For Graphite Furnace Atomic Absorption Spectroscopy Technique:

the atomization in this technique is taken place by thermal energy. The sample is introduced to the Graphite Tube (the heart of the graphite furnace) surrounded by stream of Argon. The thermal program is applied to get free atoms of the analyte. The atomization process is achieved and temperature is rigorously controlled using a series of heating steps Dry (remove water), Ash or Char (destroy organics) and Atomize



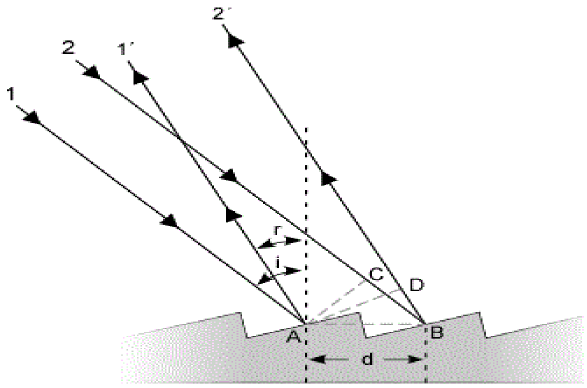
C. For Hydride Generation Atomic Absorption Spectroscopy Technique:

This technique makes use of the property that the metalloid elements (i.e.: As, Se, Sb, Bi, Se, Te, Sn and Hg) exhibit, i.e. the formation of covalent, gaseous hydrides which are not very stable at high temperatures. The hydrides of these metals are pushed by Argon gas to a quartz tube which heated by electric or flame. The heating energy gained from the heater or flame is enough to destroy the hydrides and give free atoms. In some cases this atomization is occurred in low temperature and called “ Cold Vapor Atomic Absorption”



1.5.3 Monochromator

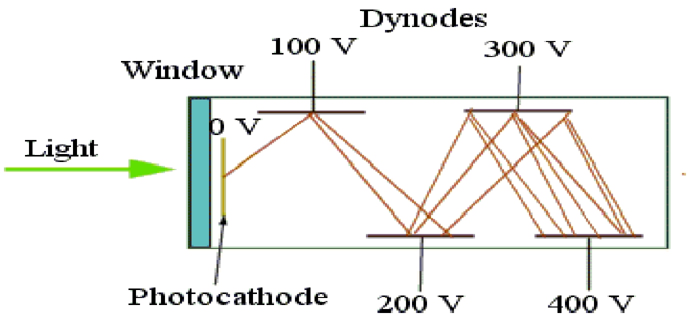
The main function of this part is to select the desired wavelength by analyzing of the light. The most common devices used for this purpose are prism and grating.



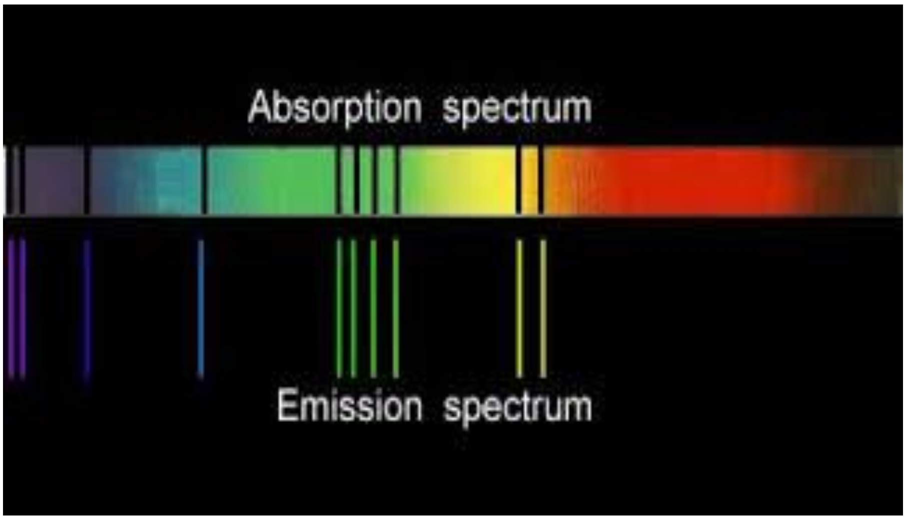
$n\lambda = d(\sin(i) + \sin(r))$

1.5.4 Detectors

The transmitted radiation is detected to calculate the absorbed part which related to the concentration of the analyte. The most common detectors are Photomultiplier Tube (PMT) and Solid State Detector (CCD).

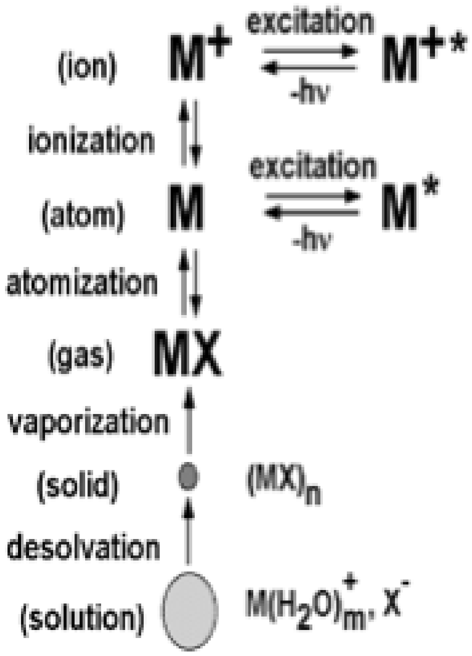


2. INTRODUCTION TO INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY (ICP-OES)



2.1. Principles of atomic emission spectrometry

It is a method of chemical analysis that uses the intensity of light emitted from a plasma at a particular wavelength to determine the quantity of an element in a sample. The wavelength of the atomic spectral line in the emission spectrum gives the identity of the element while the intensity of the emitted light is proportional to the number of atoms of the element. The high temperature of the plasma has dual functions: i) removing the solvent from the sample then atomization of the particles ii) ionize the atoms and excite both the atom and the ion. The excited atoms and ions emit specific wavelengths that used to identify the atom and quantify the amount of these atoms in the sample.

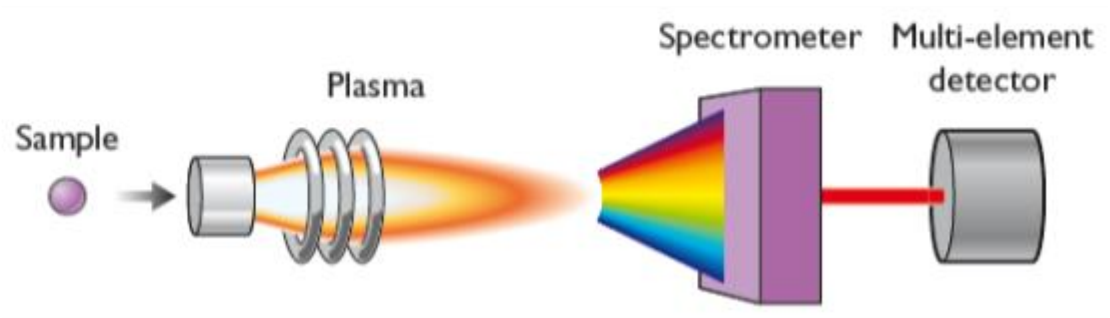


2.2. **Purpose of ICP-OES**

Inductively coupled plasma/optical emission spectrometry (ICP/OES) is a powerful tool for the determination of metals in a variety of different sample matrices. With this technique, liquid samples are injected into a radiofrequency (RF)-induced argon plasma using one of a variety of nebulizers or sample introduction techniques. The sample mist reaching the plasma is quickly dried, vaporized, and energized through collisional excitation at high temperature.

The atomic emission emanating from the plasma is viewed in either a radial or axial configuration, collected with a lens or mirror, and imaged onto the entrance slit of a wavelength selection device.

Single element measurements can be performed cost effectively with a simple monochromator / photomultiplier tube (PMT) combination, and simultaneous multi element determinations are performed for up to 70 elements with the combination of a polychromator and an array detector.

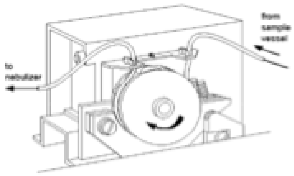


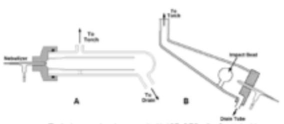
2.3. The main components of ICP-OES

2.3.1. Sample compartment:

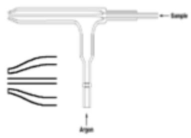
The part in which the sample is converted from liquid to mist and then to a plasma. It consists of two main parts

i) Sample introduction system in which the sample is converted from liquid to mist and it consists of pump, spray chamber and nebulizer.

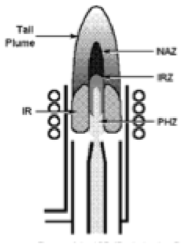




. Typical spray chambers used with ICP-OES. A - Scott double pass type, B - conical single-pass type with impact bead.

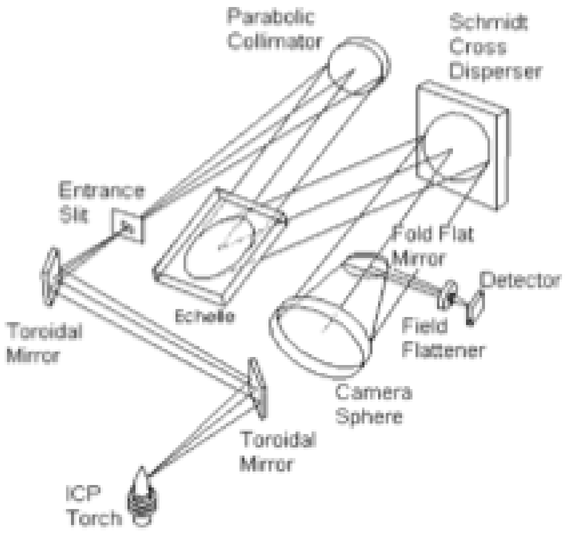


ii) the second important part of the sample compartment is the plasma torch in which the sample is atomized and excited.



3.2 Polychromator

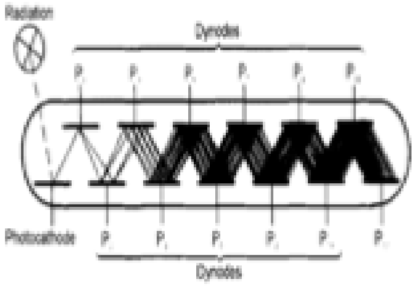
The next step in ICP-OES is the differentiation of the emission radiation from one element from the radiation emitted by other elements and molecules. The discrimination of this emission may be done in several ways. The physical dispersion of the different wavelengths by a diffraction grating is by far the most common. Other devices less commonly used include prisms, filters and interferometers.



3.3 Detectors

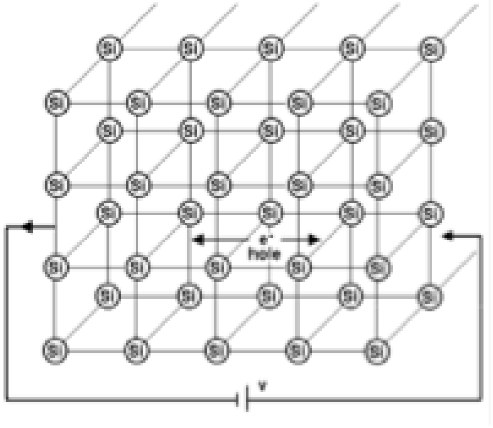
A. Photomultiplier Tubes PMT

The photomultiplier tube (PMT) is a radiation detector in which the incident radiation falling on a photocathode causes the emission of primary electrons (outer photoelectric effect) which are released into the surrounding vacuum resulting from the applied dynode voltage each primary electron is accelerated so rapidly that when it strikes a dynode two to ten secondary electrons are emitted, leading to a cascade effect.

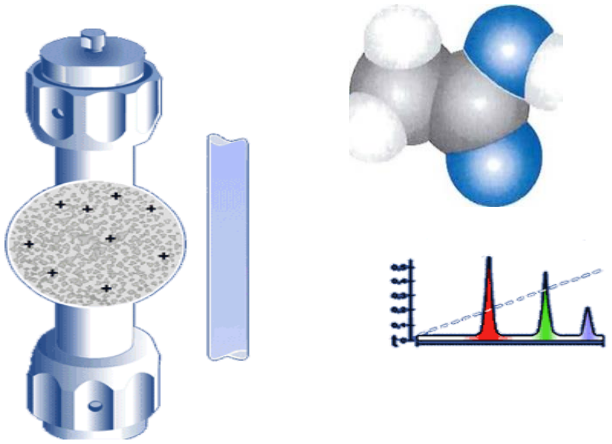


B. Solid state detector

These devices are based on the light-sensitive properties of solid-state silicon and belong to the broad class of silicon-based devices called charge transfer devices (CTD). To illustrate the principals associated with CTDs, a block of very high purity crystalline silicon is considered (Onto this silicon substrate is grown. an insulating layer of silicon dioxide (SiO_2). As shown in the pictorial, each silicon atom in the substrate is bonded to its adjacent silicon atom in a three-dimensional lattice.



3. INTRODUCTION TO ION CHROMATOGRAPHY (IC)



3.1. A General Description of Chromatography

The term chromatography is difficult to define rigorously because the name has been applied to such a variety of systems and techniques. All of these methods, however, have in common the use of a ***stationary phase and a mobile phase***. Components of a mixture are carried through the stationary phase by the flow of a gaseous or liquid mobile phase, separations being based on differences in migration rates among the sample components.

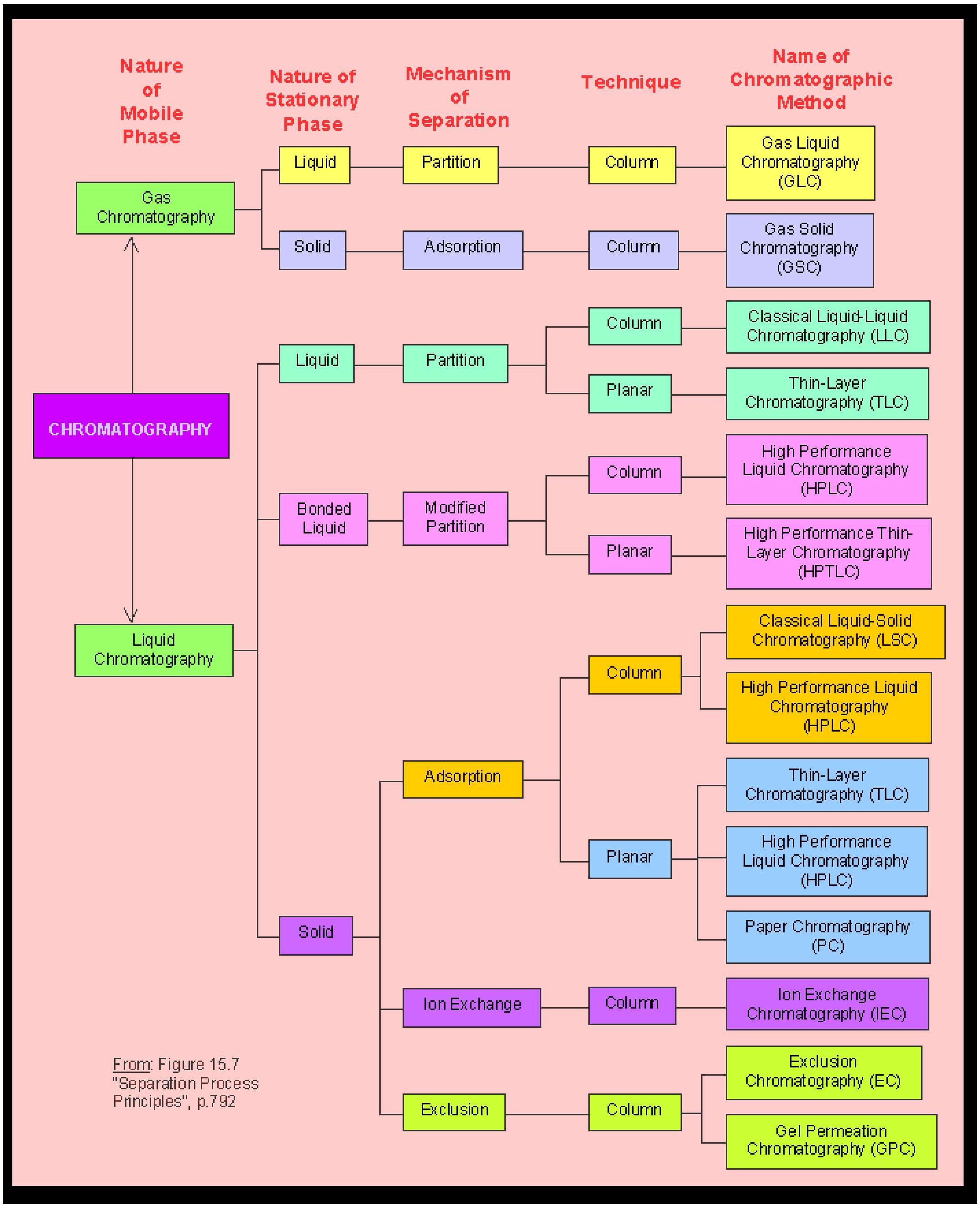


Figure A: Chromatography Classification

3.2. Ion Chromatography in Environmental Analysis

Ion chromatography (IC) is a subset of liquid chromatographyapplied to the determination of ionic solutes, such as inorganic anions, cations, transition metals, and low molecular weight organic acids and bases. Although thesesolutes can be analyzed using a number of separation anddetection modes, ion-exchange is the primary separationmode and suppressed conductivity is the primary method of detection in IC. Normally, such separations are performed on a column packed with a solid ion-exchange material. But if we define chromatography broadly as a process in which separation occurs by differences in migration, capillary electrophoresis may also be included.

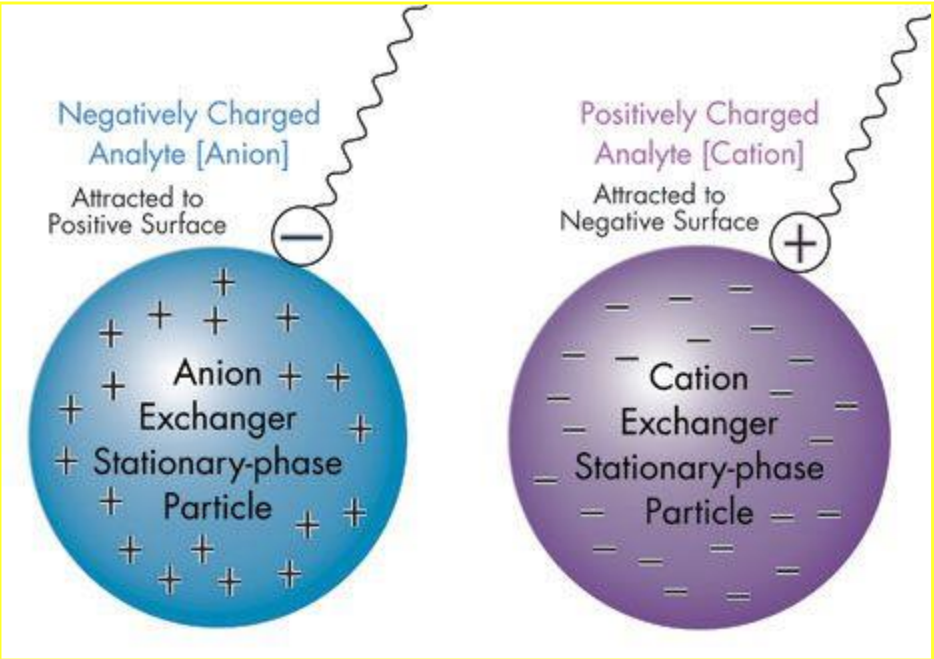


Figure B: Ion-Exchange Chromatography

IC is well established as regulatory method for the analysis of inorganic anions in environmental samples as there are few alternative methods which can determine multiple anions in a single analysis. However, there are relatively few regulatory methods for cation analysis which use IC. Methods for cation analysis tend to be based upon spectroscopic techniques, although IC offers the advantage of providing information on metal speciation.

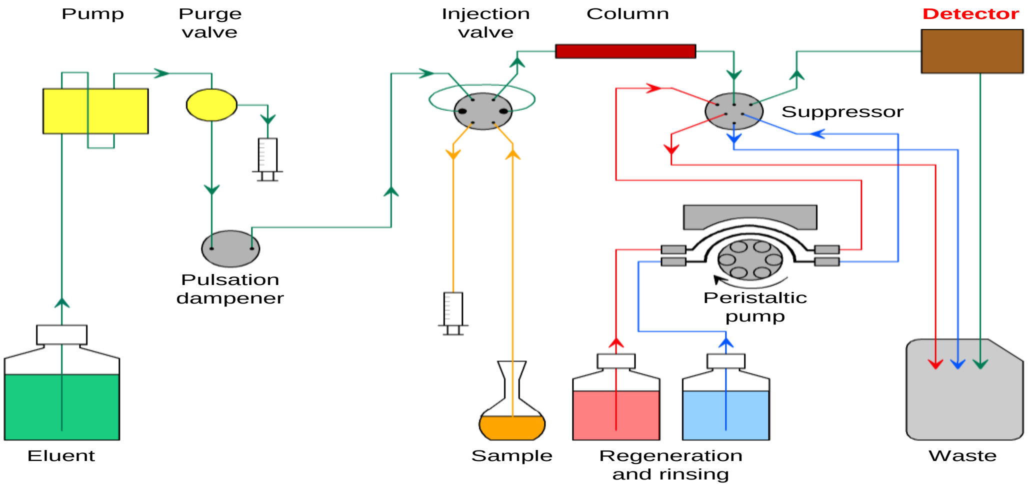
3.3. Components of an IC Instrument:

The hardware is similar to that used for high pressure liquid chromatography (HPLC) ***but does have important differences.*** Readers who are familiar with HPLC will recognize the similarity and the differences to IC hardware.

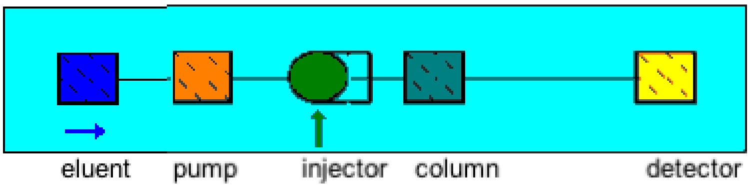
The hardware requirements for an IC include :

- *An optional Supply of eluent(s).*
- **High pressure pump (with pressure indicator) to deliver the eluent.**
- **Pulsation Dampener (to minimize base line noise).**
- *An optional Autosampler.*
- **Injector for introducing the sample into the eluent stream and onto the column.**
- **Column to separate the sample mixture into the individual components.**
- *An optional oven to contain the column.*
- *An optional Suppression Unit.*
- **Detector to measure the analyte peaks as elute from the column.**
- **Data system for collecting and organizing the chromatograms and data.**

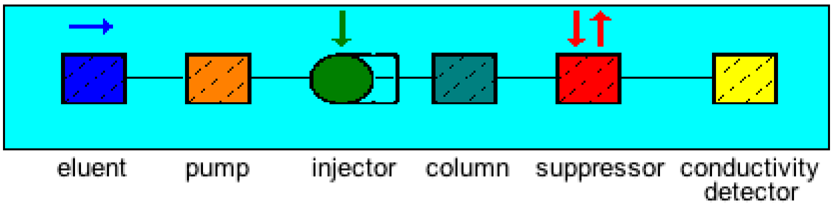
IC SET-UP



Schematic of an Ion Chromatography System

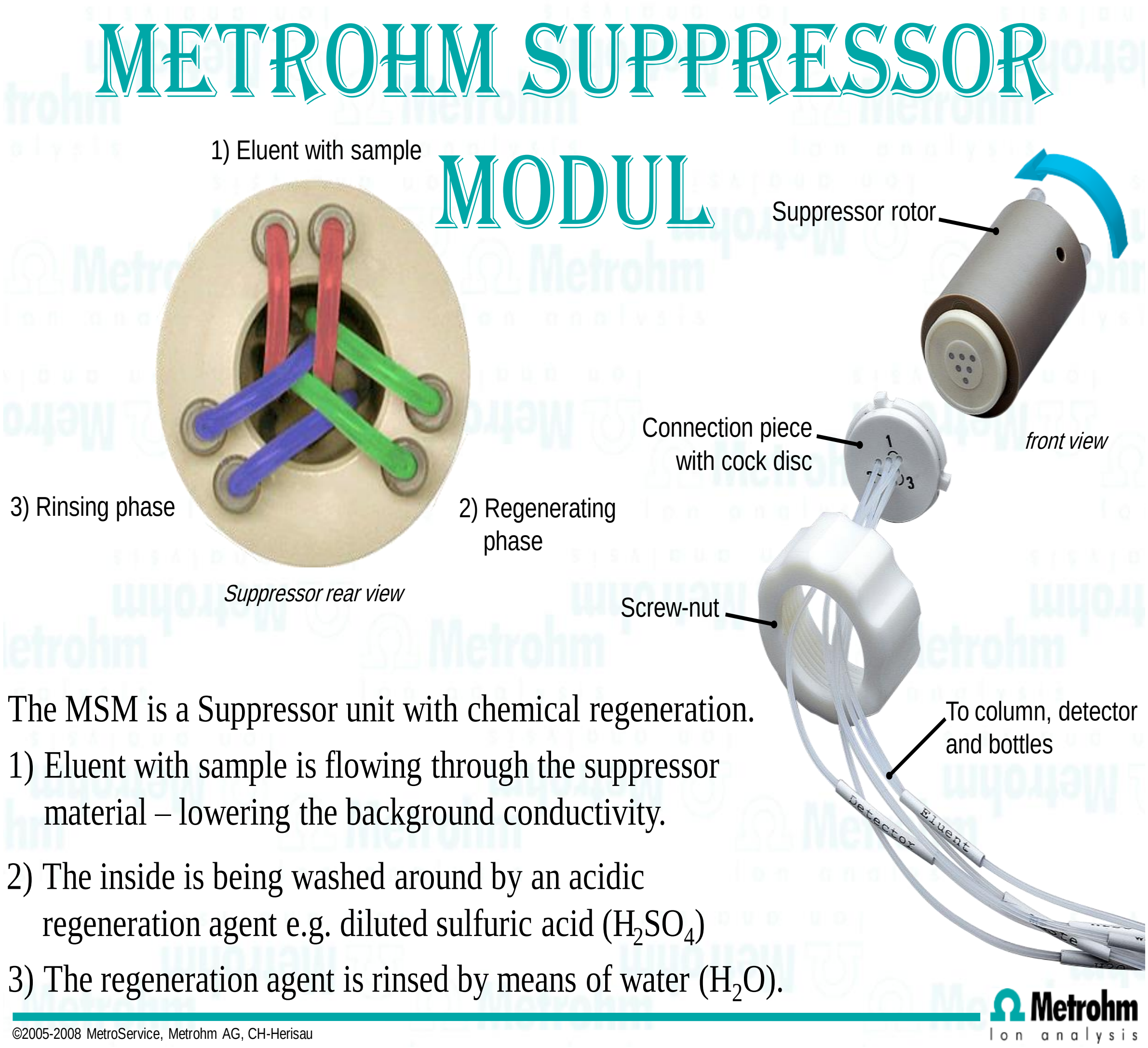


Suppression in Ion Chromatography



Metrohm Suppressor Modul(Chemical Suppression):

The suppressor used in anion chromatography is simply a cation exchanger and its job is to remove cations and replace them with an H+. So a sodium carbonate eluent (~800uS) would be converted to carbonic acid (~18uS) by the suppressor and the analyte, for example NaCl (~126uS without suppression) would become HCl (~426uS with suppression). The suppressor converts the analyte to the free acid form and the background is reduced whilst the sample signal is enhanced. The anion chemical suppression can be taken a stage further with a secondary CO_2 suppressor instrument (fitted after the MSM) which removes carbon dioxide from the suppressor reaction and carbonate from the sample, which means that the mobile phase is converted to water instead of carbonic acid so a background conductivity approaching 1uS is then achieved. This is known as Sequential Suppression and gives a greatly minimized injection peak, no system peak (from eluent), super linearity and an enhancement in the peak areas allowing lower limits of detection to be achieved.



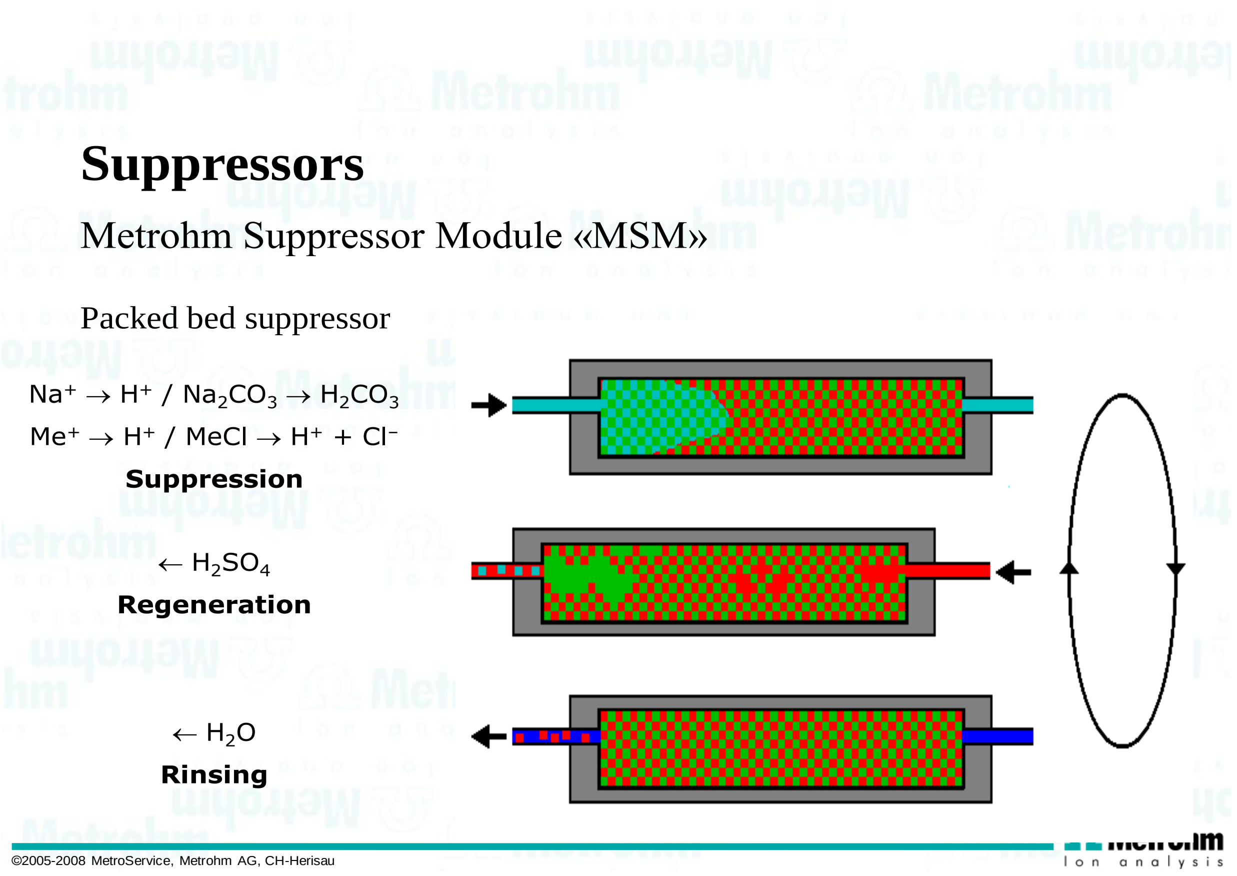
METROHM SUPPRESSOR MODUL

The MSM is a Suppressor unit with chemical regeneration.

1) Eluent with sample is flowing through the suppressor material – lowering the background conductivity.

2) The inside is being washed around by an acidic regeneration agent e.g. diluted sulfuric acid (H_2SO_4)

3) The regeneration agent is rinsed by means of water (H_2O).



Suppressors

Metrohm Suppressor Module «MSM»

Packed bed suppressor

$Na^+ \rightarrow H^+ / Na_2CO_3 \rightarrow H_2CO_3$

$Me^+ \rightarrow H^+ / MeCl \rightarrow H^+ + Cl^-$

Suppression

$\leftarrow H_2SO_4$

Regeneration

$\leftarrow H_2O$

Rinsing

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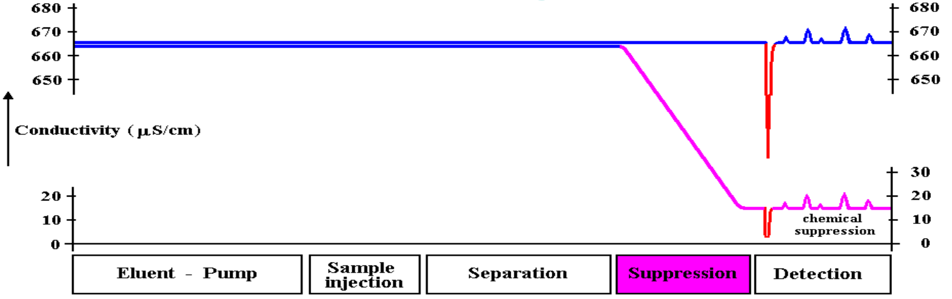
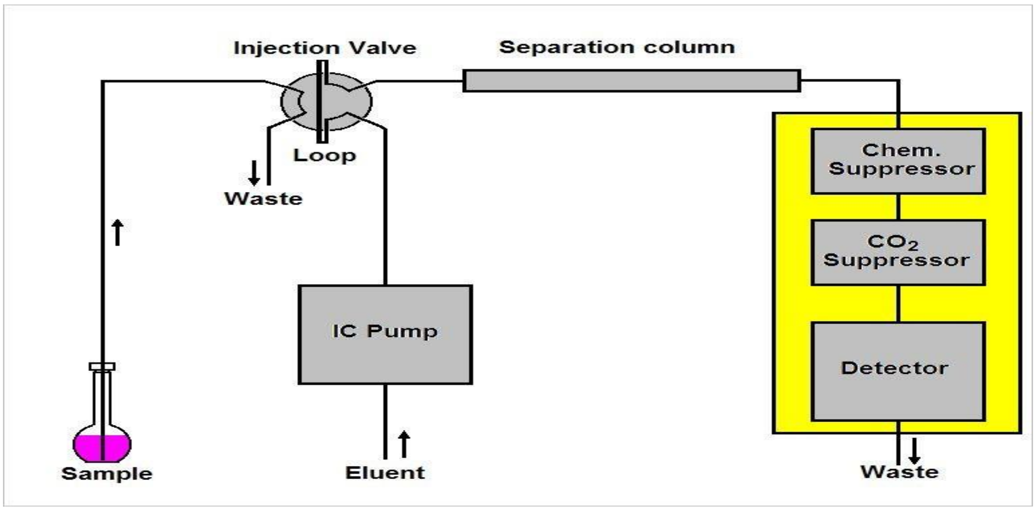
METROHM SUPPRESSOR MODUL

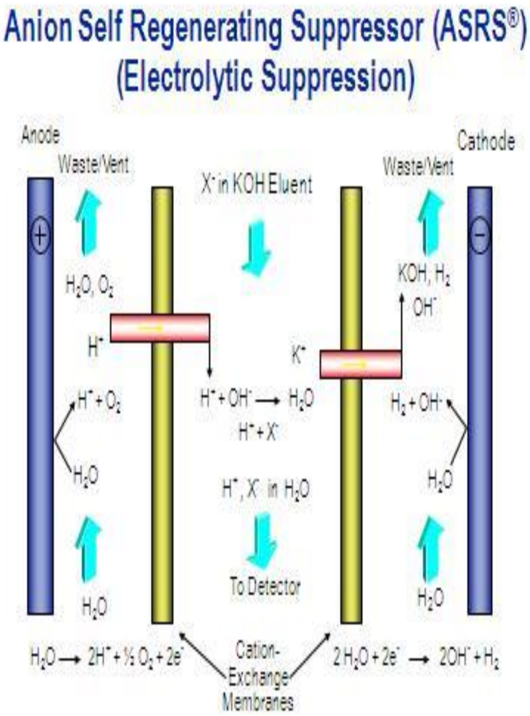


The Metrohm Suppressor Modul (MSM) is an optional component to improve the detection of the analyte.

The MSM chemically suppresses the high background conductivity of the eluent. Thus enabling a more sensitive detection of the analyte.



Dionex Suppressor Modul(Electrolytic Suppression):



The sodium hydroxide eluent flows through the channel in the suppressor between the cation exchange membranes. Under the influence of the electric field, the sodium eluent ions cross the cation exchange membranes and migrate towards the cathode. They are replaced by the hydronium ions that are continuously being generated at the anode through the electrolysis of water.

After passage through the suppressor, the eluent and the analyte anions pass through the conductivity cell. In the recycle mode the eluent stream then passes through the regen side of the suppressor, where it provides the source of hydronium ions and sweeps the sodium counterions away to waste.

Autosuppession is a form of continuous suppression, meaning fresh regenerant is continuously being supplied with no interruptions in chromatography required. The advantages of continuous suppression, as opposed to "batch" suppression in which the suppressor must be periodically regenerated off-line will be discussed in the next few slides.

Everything on the high pressure side, from the pump outlet to the end of the column, must be strong enough to withstand the pressures involved. **The wetted parts are usually made of PEEK and other types of plastics** although other materials, such as sapphire, ruby, or even ceramics are used in the pump heads, check valves, and injector of the system. ***PEEK and other high performance plastics are the materials of choice for ion chromatography.*** Stainless steel can be used provided the system is properly conditioned to remove internal corrosion and the eluents that are used do not promote further corrosion. (Almost all IC eluents are not corrosive.) Stainless steel IC components are considered to be more reliable than those made from plastics, but require higher care. The stainless steel IC is normally delivered from the manufacturer pretreated so that corrosion is not present. The reader is advised to consult the IC instrument manufacturer for care and upkeep instructions.

Dead Volume:

Dead volume is any empty space or unoccupied volume. The presence of too much dead volume can lead to extreme losses in separation efficiency due to broadening of the peaks. Eluent entering the pump should not contain any dust or other particulate matter. Particulates can interfere with pumping action and damage the seal or valves. Material can also collect on the inlet frits or on the inlet of the column, causing pressure buildup. Eluents or the water and salt solutions used to prepare the eluents neither are normally filtered with a 0.2 or 0.45 µ nylon filter.

Degassing the Eluent

Degassing the eluent is important because air can get trapped in the check valve (discussed later in this section), causing the pump to lose its prime. Loss of prime results in erratic eluent flow or no flow at all.

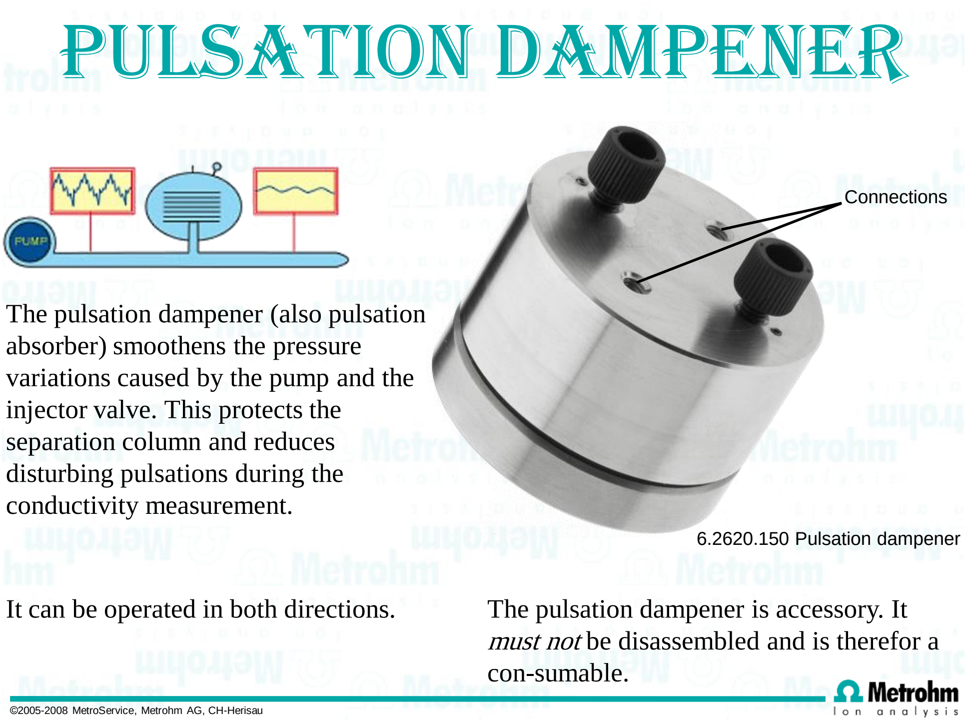
Another reason for removing dissolved air from the eluent is because air can result in changes in the effective concentration of the eluent. Carbon dioxide from air dissolved in water forms carbonic acid. Carbonic acid can change the effective concentration of a basic eluent, including solutions of sodium hydroxide, bicarbonate and carbonate. Usually, degassed water is used to prepare eluents and efforts should be made to keep exposure of eluent to air to a minimum after preparation.

Modern inline degassers are becoming quite popular. These are small devices that contain two to four channels. The eluent travels through these devices from the reser- voirs to the pump. The tubing in the device is gas permeable and surrounded by vacu- um. Helium sparging can also be used to degas eluents. Extended sparging may cause some retention shifts, so sparging should be reduced to a trickle after the initial few minutes of bubbling. Finally, it is best to change the eluents every couple of days to keep the concentration accurate.

Pumps

IC pumps are designed around an eccentric cam that is connected to a piston. The rotation of the motor is transferred into the reciprocal movement of the piston. A pair of check valves controls the direction of flow through the pump head. A pump seal surrounding the piston body keeps the eluent form leaking out of the pump head.

In single-headed reciprocating pumps, the eluent is delivered to the column for only half of the pumping cycle. A pulse dampener is used to soften the spike of pres- sure at the peak of the pumping cycle and to provide a eluent flow when the pump is refilling. Use of a dual head pump is better because heads are operated 180° out of phase with each other. One pump head pumps while the other is filling and vice versa.



PULSATION DAMPENER

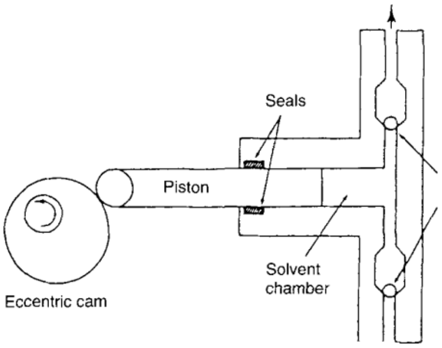
The pulsation dampener (also pulsation absorber) smoothens the pressure variations caused by the pump and the injector valve. This protects the separation column and reduces disturbing pulsations during the conductivity measurement.

6.2620.150 Pulsation dampener

It can be operated in both directions.

The pulsation dampener is accessory. It *must not* be disassembled and is therefor a con-sumable.

IC pump head, piston, and cam.



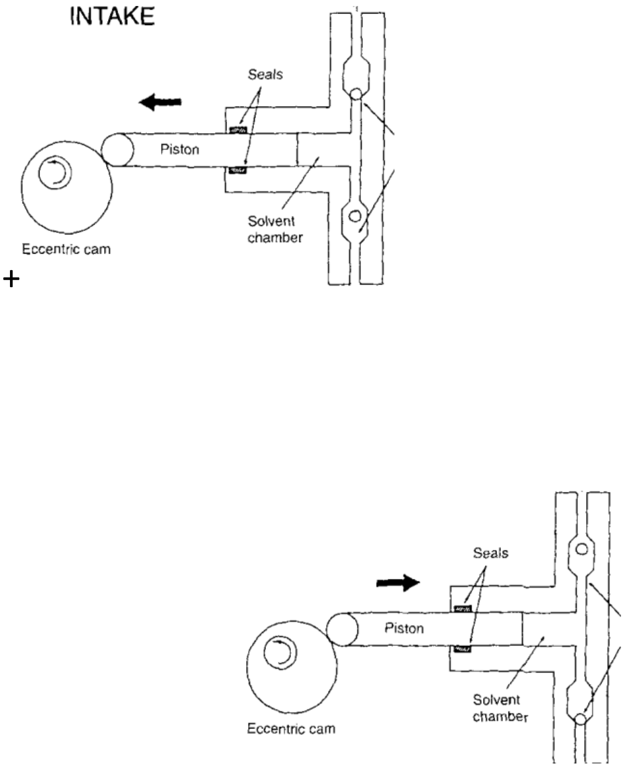


Figure 1.5. Check valve positions during intake and delivery strokes of the pump head piston.

Gradient Formation

Isocratic separations are performed with an eluent at a constant concentration of eluent buffer or salt solution. While it is desirable (simpler) to perform IC separations with single isocratic eluent, **it is sometimes necessary** to form ***a gradient of weak eluent to concentrated, strong eluent over a chromatographic run***. This allows the separa- tion of anions that may have a wide range of affinities for the column. Weakly adhering anions elute first and then, as the eluent concentration is increased, more strongly adhering anions can be eluted by the stronger eluent.

Detection and Data System:

The most common and useful detector for ion chromatography is conductivity; however, UV and other detectors can be quite useful. The results of the chromatographic separation are gen- erally displayed on a computer, although, in some older systems, recorders and integrators are used. The computer uses an AID (analog to digital) board to convert the analog signal from the detector to digital data. The digital information is stored and manipulated to report results to the user.

The type of information that is most useful are the retention times of the various peaks and the peak areas (in a few cases, peak heights are used). Retention times are used to confirm the identity of the unknown peak by comparison with a standard. Peak area is compared to standards of known concentration to calculate analyte concentrations. This calculation can be performed by the use of a

simple ratio:

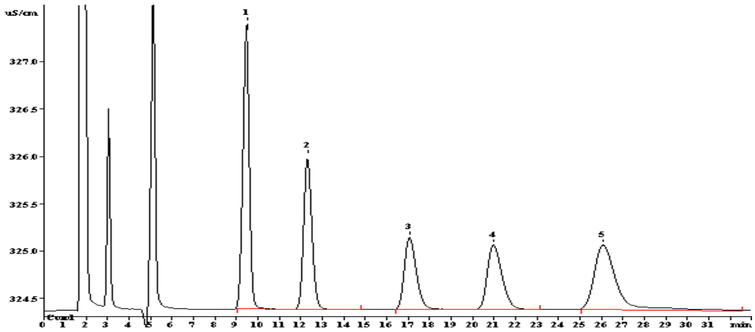
$$\frac{\text{Unknown concentration}}{\text{Known concentration}} = \frac{\text{unknown peak area}}{\text{known peak area}}$$

Therefore:

$$\text{Unknown concentration} = \frac{\text{known concentration}}{\text{Known peak area}} \text{ x unknown peak area}$$

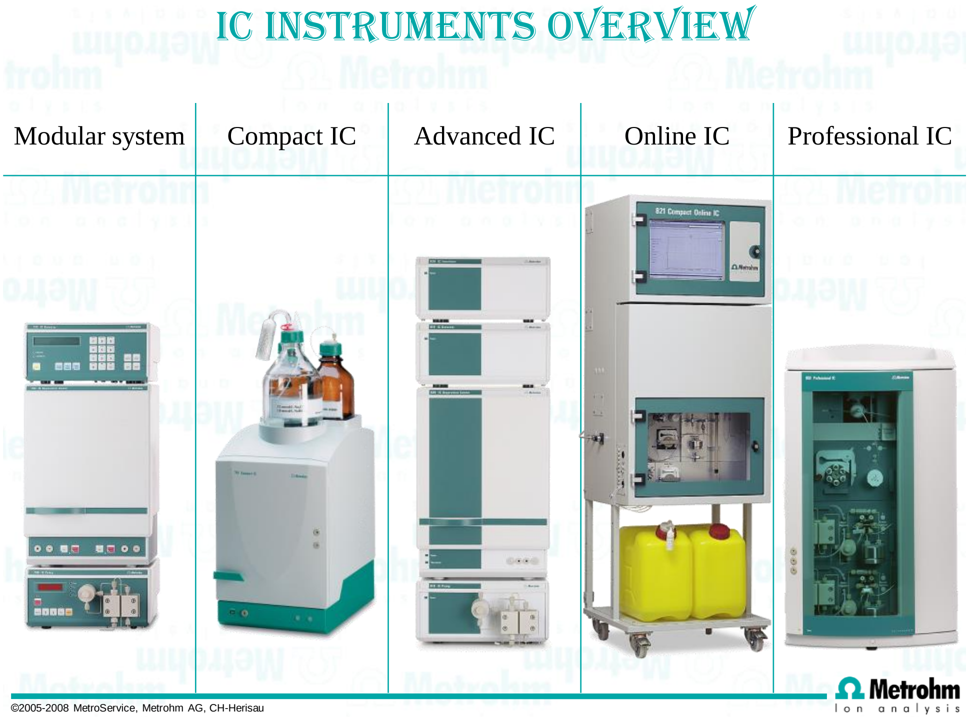
It is usually better, however, to draw a calibration curve of known peak area vs. known concentration, to find the unknown peak area on the curve and to measure the unknown concentration on the axis.

For the data system to measure peak area, the baseline of the peak must be accurately drawn. The software program will attempt on its own to draw a baseline for the peak, but frequently the user must manually mark the baseline start and finish points to accurately draw the peak baseline.



Chromatogram showing an anion exchange separation followed by direct conductivity detection (non-suppressed)

Some modern IC Instruments…



IC INSTRUMENTS OVERVIEW

Modular system | Compact IC | Advanced IC | Online IC | Professional IC

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ICS-3000 – System Accessibility

للاقتراحات والشكاوى قم بمسح الصورة (QR)

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[4] http://www.metrohm.com/

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