

Power plant analysis



Quality control in power plants (process water, turbine oils, fuels, and operating materials)



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Metrohm – customized analysis for water, turbine oil, fuels, and operating materials in power plants

Energy and power plants

Energy supply has become a major issue of modern times. The importance of energy even plays a role in Greek mythology. Zeus withheld fire from humanity, thus removing all prospects of civilization. But the humans had Prometheus on their side: he stole fire from the gods and gave it back to mankind. Unfortunately, Prometheus is no longer here to help us. Humankind has had to fend for itself by developing power plants to convert kinetic (wind, water) and thermal energy (nuclear energy, chemical energy) into electrical power.

Increasing energy consumption

The rapid increase in the Earth's population, which is growing by about 80 million every year, has also led to rising energy consumption. Calculations by the International Energy Agency (IEA) predict that the global energy demand will increase by about 65% by 2035. A major fraction of the required energy will continue to be provided by fossil fuel-fired and nuclear power plants.

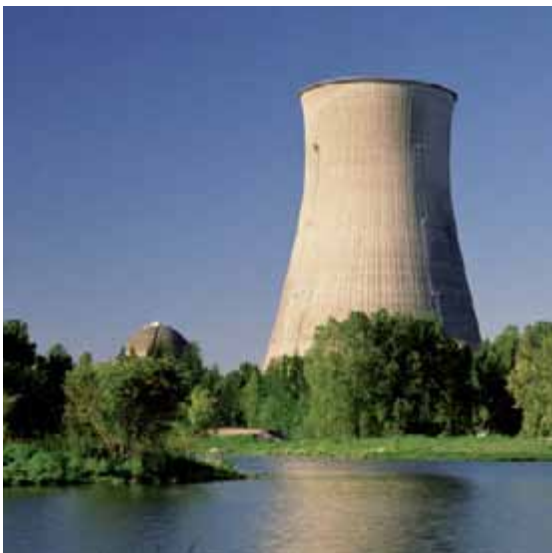
Relevance of analysis in power plants

With its high-performance laboratory and process analysis instruments, Metrohm contributes to a reliable and sustainable operation of power plants and helps to minimize costly downtimes. This includes analysis of the process water circulating in the various cooling circuits, the oils and lubricants used in turbines, as well as fuels and operating materials.

As a leading manufacturer of equipment for chemical analysis, we are well aware of the challenges. We offer state-of-the-art instruments and systems for analysis solutions in your power plant, both in the laboratory and in-process.

You can rely on our expertise

Metrohm offers not only the most modern equipment, but also complete solutions for specific analytical requirements. Your contact partners at Metrohm are specialists who will develop applications tailored to your needs and provide competent support for all analytical issues in power plants.



Power plant analysis

04

I. Process water

Water circuits in thermal power plants

Thermal power plants use the heat generated by combustion or nuclear fission to produce steam, which is fed into a turbine driving a generator that converts the mechanical energy into electrical energy. Downstream of the turbine, the steam is condensed to water in a condenser. This water is fed into a feed tank from where it is pumped back into the steam boiler. Cooling water flows through the condenser in a separate circuit and removes the heat of condensation released by the steam via a heat exchanger. Nuclear power plants with pressurized water reactors have an additional water circuit known as the primary circuit.

An optimized water chemistry is essential

All thermal power plants use water as a central (operating) medium. As a liquid, it is used for cooling and as a gas, it drives the turbines. In nuclear power plants, it also moderates the fission neutrons and thus controls nuclear fission. A well-devised water chemistry ensures safe and efficient power plant operation.

Guidelines of VGB, EPRI, EPPSA, IAPWS, and the IAEA safety standards

Nearly 50% of the unplanned downtimes in power plants are caused by contaminants or problems with the chemistry of the water-steam circuit, with corrosion being the primary factor. Various guidelines define permissible operating ranges, and they are used by power plant operators as a fundamental means of orientation. These include guidelines from the VGB (Vereinigung der Grosskesselbesitzer e.V., Association of Large Boiler Operators),

EPPSA (European Power Plant Suppliers' Association), IAPWS (International Association for the Properties of Water and Steam), and EPRI (Electric Power Research Institute). Furthermore, there are also the safety standards of the International Atomic Energy Agency (IAEA) that apply exclusively to nuclear power generation.

The water chemistry depends on the type of power plant, the cooling circuit design, and the construction materials. Every cooling circuit has a unique design and its own analytical requirements. If this brochure does not include your power plant application, please contact your Metrohm representative.

Process and laboratory parameters

With respect to instrumentation, there are two analytical objectives: determination of process parameters and laboratory parameters. The former are key criteria that are usually measured online and are used to continuously control the operating conditions. In contrast, laboratory parameters are determined offline and generally at defined intervals. They provide additional diagnostic information and supplement the online measurements; however, they are not used for primary control of the operating conditions.

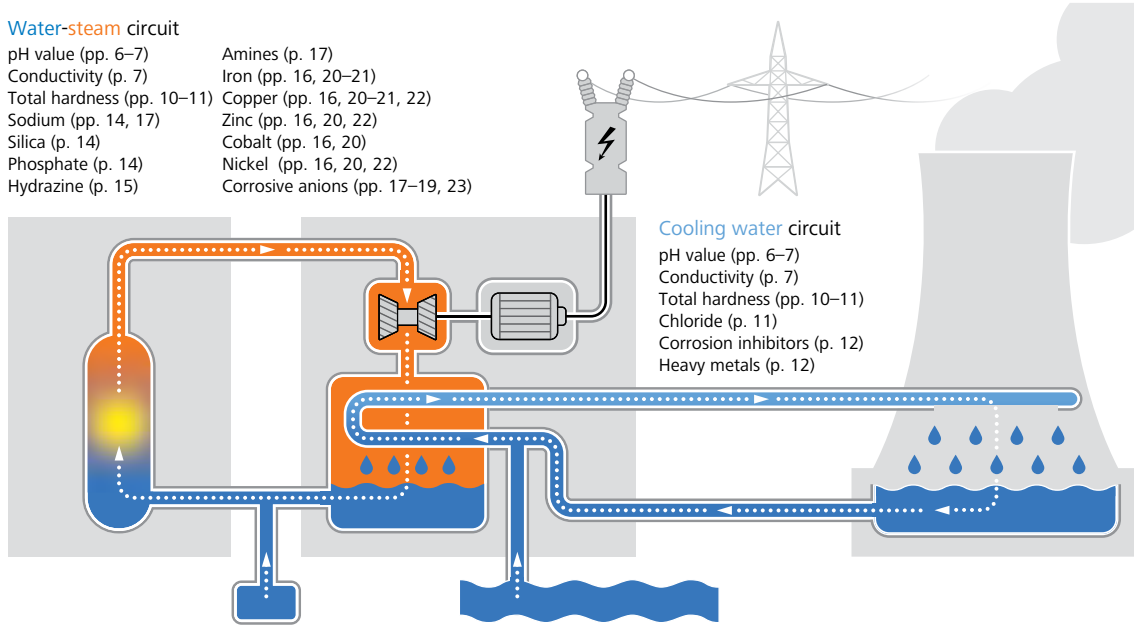
The following two flow charts show key parameters of the water chemistry that are determined in power plants with two, respectively, three circuits. The numbers in brackets refer to the page(s) on which the application is described.



Water-steam circuit

pH value (pp. 6–7)
 Conductivity (p. 7)
 Total hardness (pp. 10–11)
 Sodium (pp. 14, 17)
 Silica (p. 14)
 Phosphate (p. 14)
 Hydrazine (p. 15)

Amines (p. 17)
 Iron (pp. 16, 20–21)
 Copper (pp. 16, 20–21, 22)
 Zinc (pp. 16, 20, 22)
 Cobalt (pp. 16, 20)
 Nickel (pp. 16, 20, 22)
 Corrosive anions (pp. 17–19, 23)



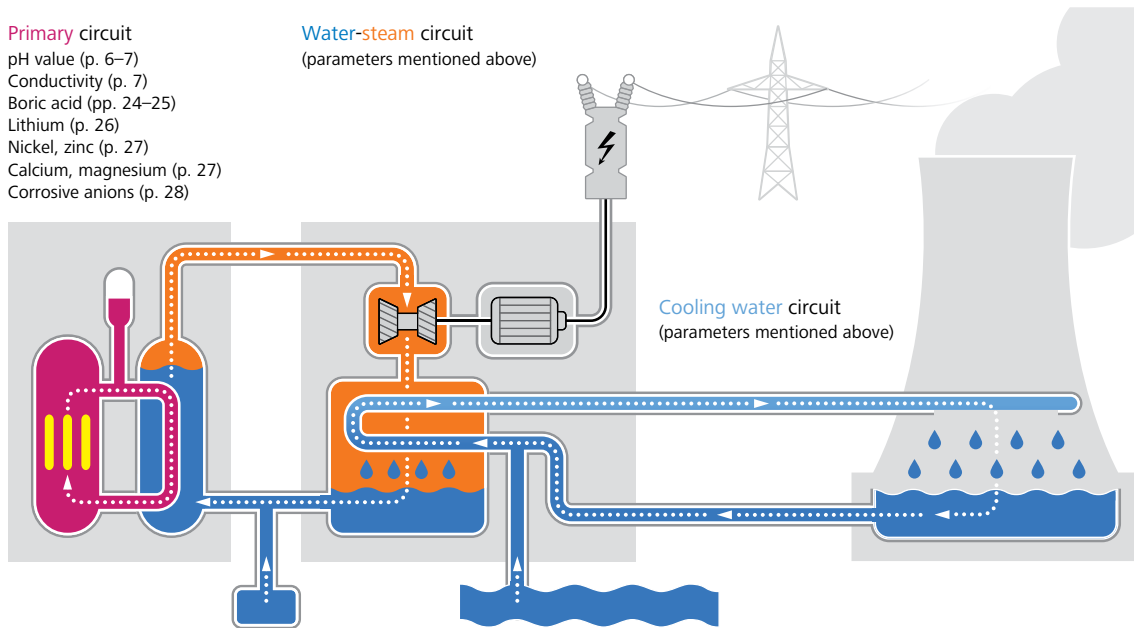
Analytical parameters monitored in a power plant with two water circuits

Primary circuit

pH value (p. 6–7)
 Conductivity (p. 7)
 Boric acid (pp. 24–25)
 Lithium (p. 26)
 Nickel, zinc (p. 27)
 Calcium, magnesium (p. 27)
 Corrosive anions (p. 28)

Water-steam circuit

(parameters mentioned above)



Analytical parameters monitored in a pressurized water reactor with three water circuits

II. Turbine and lubricating oils

Turbine and lubricating oils are exposed to extreme conditions in power plants. Numerous international standards define the requirements and test procedures for in-service maintenance of the turbines. Pages 32 to 35 of this brochure describe two test procedures defined in ASTM D 4378: potentiometric determination of the acid and base numbers and determination of the water content using Karl Fischer titration.

III. Fuels and operating materials

Pages 36 to 37 describe the use of combustion ion chromatography to determine the amounts of halogens and sulfur in all combustible samples, both solid and liquid, such as coal, refuse, in consumer goods such as latex gloves, or to investigate ion-exchange resins used to condition process water.

I. Process water in power plants

pH value and conductivity

All thermal power plants convert energy generated by combustion processes or nuclear fission into heat that is then used to vaporize water as an operating fluid. High-pressure superheated steam is fed to the blades of a high-pressure turbine causing it to rotate, thus producing electricity via a coupled generator. The steam is expanded in the low-pressure zone of the turbine and then condensed to water in a cooled condenser. This condensate is purified, preheated, and pumped as feed water to the evaporator. The primary cooling water flowing through kilometers of piping in the condenser dissipates the heat of condensation. High-purity steam is essential if the steam turbine is to operate efficiently and trouble-free.

pH value

The corrosion properties of metals are primarily determined by the oxygen content and the pH value of the water. Higher pH values (i.e., less acidic) lower the driving force of the corrosion. The strongest corrosion occurs at pH values below 8; whereas passivation reactions usually dominate at higher values. The pH value is a key parameter to control the amount of lithium hydroxide that has to be added to the primary circuit of a pressurized water reactor; in water steam circuits, the pH value controls the amount of added amines.

The pH value can be determined with the 867 pH Module – equipped with software or touch control – or with the 780 pH Meter. The recommended sensor is the Aquatrode Plus, which guarantees the highest accuracy for pH measurements in process waters with a low ionic strength.

Aquatrode Plus: the fixed ground-joint diaphragm is insensitive to contamination and guarantees a low-noise measuring signal in water matrices with a low ionic strength.



Conductivity

The conductivity indicates the amount of dissolved minerals. It is a measure of the water purity and is one of the most important parameters for any chemical control program in a power plant. It is determined at numerous sampling points in the power plant, for example, in the cooling water circuit, in feed and makeup water, at the outlet of the condensate pump, and in the primary circuit of a pressurized water reactor. In high-purity process water, it reflects the amount of added additives such as ammonium or amines. A sudden increase in the conductivity often indicates a leakage because carbon dioxide from the air has dissolved in the water.

An important parameter is the cationic or acid conductivity, which is measured at the outlet of the cation exchanger and reveals the presence of corrosive acid residues. The conductivity of the acid anions is greatly increased because the counterions – ammonium or sodium – have been replaced by the hydronium ions, which have a significantly higher conductivity. Measurement of the conductivity downstream of the cation exchanger is an important parameter for detecting leakages. The conductivity of ultrapure water in water-steam circuits is generally about $0.15 \mu\text{S}/\text{cm}$. If this value is exceeded without previous addition of additives, this is often an indication that contaminants from the cooling water circuit have infiltrated.

Determining the conductivity of ultrapure water is challenging. One solution is the 856 Conductivity Module, either software- or touch-controlled, in combination with a stainless steel conductivity measuring cell.

pH value and conductivity – process parameters

The pH value and the conductivity have to be determined quasi-continuously at numerous locations within the cooling circuit. This can be achieved with the Process Analyzer ADI 2045TI from Metrohm Applikon. It combines these direct measurements with various analytical methods and is designed for simultaneous analysis of multiple sample streams and parameters. Measurements are carried out on unpressurized sample streams in the high-temperature water circuits that have been cooled to room temperature and in which the online sensors are located.



The 856 Conductivity Module (center) with 900 Touch Control and 801 Stirrer

Corrosion

08

Corrosion of metals in power plants is a commonly occurring phenomenon due to the continuous contact of the metal with a corroding environment. According to the definition in DIN EN ISO 8044, corrosion is a physico-chemical interaction between a metal and its surroundings and which results in a measurable change in the material that can have a negative impact on the function of the metal or even the entire system. These interactions are usually of an electrochemical nature. The objective is to use suitable treatment and conditioning of the water to minimize the corrosion rate and the transport of corrosion products within the circuits.

Electrochemical analysis methods are becoming increasingly common for quantifying the corrosion rates. These are far superior to traditional methods, such as determination of the weight loss, because they provide more detailed information about corrosion phenomena with less effort and time. The capabilities of the electrochemical methods are reflected in numerous international standards.

Selection of important standards relating to corrosion measurement

Standard	Brief description
ASTM G 102, DIN 50918	Standard practice for calculation of corrosion rates and related information from electrochemical measurements Corrosion of metals; electrochemical corrosion tests
ASTM G 106, DIN EN ISO 16773	Standard practice for electrochemical impedance measurements
ASTM G 5, DIN EN ISO 17475	Standard reference test method for making potentiostatic and potentiodynamic anodic polarization measurements Corrosion of metals and alloys – electrochemical test methods – Guidelines for conducting potentiostatic and potentiodynamic polarization measurements
ASTM G 199	Standard guide for electrochemical noise measurements
ASTM G 148	Standard practice for evaluation of hydrogen uptake, permeation, and transport in metals by electrochemical technique
ASTM G 150	Standard test method for electrochemical critical pitting temperature testing of stainless steels
DIN 50919	Corrosion of metal; investigations of galvanic corrosion in electrolytic solutions

The most important electrochemical methods for corrosion testing are linear polarization (LP), electrochemical noise measurement (ECN), and electrochemical impedance spectroscopy (EIS).

The critical pitting temperature in accordance with ASTM G 150

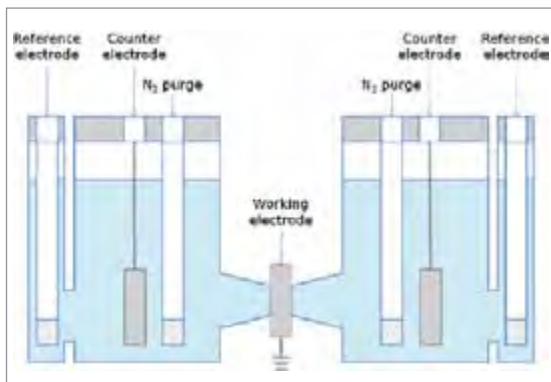
The critical pitting temperature (CPT) indicates the corrosion resistance of a material at high temperatures. It is the temperature at which a metal surface exposed to a

test solution under defined conditions shows the first signs of corrosive attack in the form of deep pits. CPT is determined at a constant polarization potential and is revealed by a sharp rise in the anodic current density. The more resistant the material is to pitting corrosion, the higher is its CPT. The CPT can be determined with the Autolab PGSTAT 302N or 128N with an optional pX1000 module. The temperature of the measuring cell is controlled by an external thermostat connected to a corrosion cell.

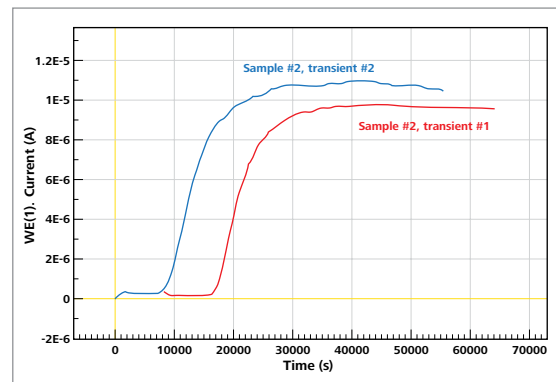
Hydrogen permeation in accordance with ASTM G 148

Electrochemically generated hydrogen, absorbed by some metallic surfaces, can permeate the material changing its mechanical properties. Determination of hydrogen permeation in metals is thus an important parameter in corrosion research. Electrochemically controlled hydrogen permeation is measured using a Devanathan-Stachurski (double) cell. It comprises two separate electrolytic cells

that are separated by a membrane made from the metal being investigated. Hydrogen is generated electrochemically at the cathode, whereas the hydrogen that has diffused through the metal membrane is oxidized at a constant potential at the anode. The oxidation current is directly proportional to the amount of hydrogen diffusing through the metal membrane.



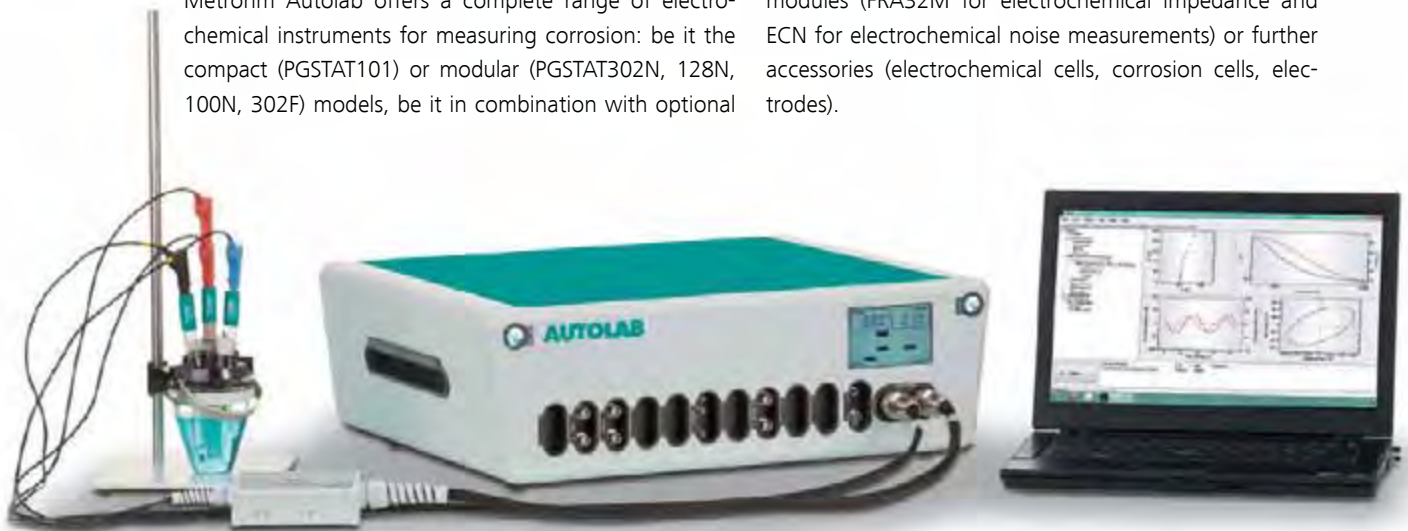
Schematic diagram of the Devanathan-Stachurski cell for electrochemically controlled hydrogen permeation measurements



Hydrogen permeation measurements on two samples of carbon steel with differing thicknesses

Metrohm Autolab offers a complete range of electrochemical instruments for measuring corrosion: be it the compact (PGSTAT101) or modular (PGSTAT302N, 128N, 100N, 302F) models, be it in combination with optional

modules (FRA32M for electrochemical impedance and ECN for electrochemical noise measurements) or further accessories (electrochemical cells, corrosion cells, electrodes).



Autolab PGSTAT128N with FRA32M module

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Cooling water

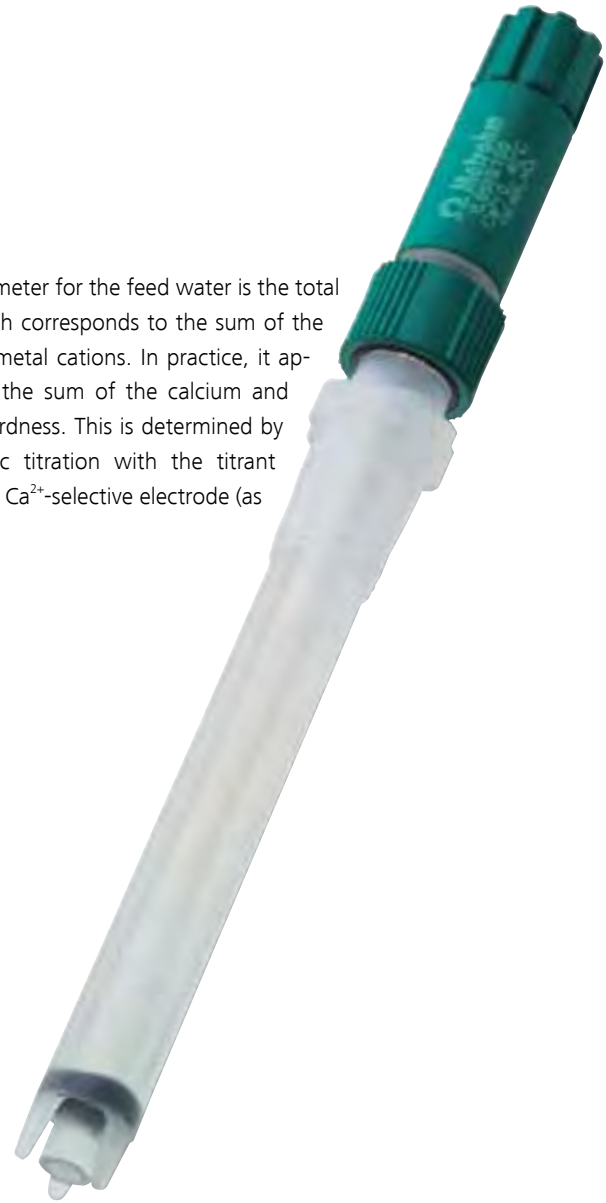
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Cooling water is used to condense the exhaust steam from the turbine to water, which is then sent back to the water-steam circuit as feed water. The heat of condensation from the steam is transferred to this cooling water as it flows through the kilometers of piping – usually made of titanium – in the condenser. The cooling water is cooled either by once-through cooling, in which the water is taken from a river and returned at a slightly higher temperature, or in a circuit in a cooling tower. In a wet cooling tower, this heat is dissipated into the atmosphere: as the warmed-up cooling water falls from a great height in the tower, the heat is transferred to the rising air stream. Continuous circulation of the cooling water increases the concentration of contaminants. This necessitates water analyses to control corrosion and deposition processes taking place in the cooling water circuit. However, the purity requirements of cooling water are much lower compared to those of the boiler feed water. Some of the parameters are discussed below.

Determination of the water hardness using an ion-selective electrode (ISE)

Alkaline earth salts dissolved in cooling water can deposit in the kilometers of piping in the condenser. These deposits form an insulating layer that hinders heat transfer and lowers the condenser's operating efficiency. The same applies to an even greater degree to the steam generator in the water-steam circuit.

A control parameter for the feed water is the total hardness, which corresponds to the sum of the alkaline earth metal cations. In practice, it approximates to the sum of the calcium and magnesium hardness. This is determined by complexometric titration with the titrant Na_2EDTA and a Ca^{2+} -selective electrode (as per ISO 6095).



Combined polymer membrane electrode for determining calcium and magnesium



The MATi1 comprises the 815 Robotic USB Sample Processor XL, several 800 Dosinos, the 856 Conductivity Module, and the 905 Titrand. It is particularly suitable for fully automated analyses of process waters in power plants.

Colorimetric determination of the water hardness – process parameters

In addition to determination with an ISE, described on page 10, the water hardness can also be determined by colorimetry. After adding the indicator hydroxynaphthol blue, a red complex forms at pH values above 7. Adding EDTA solution changes the color back to blue. The color change is proportional to the concentration of alkaline earth metal ions. Determinations in the sub- $\mu\text{g/L}$ range can be conveniently carried out in only 10 minutes using an Alert Analyzer from Metrohm Applikon. For higher calcium and magnesium concentrations, ADI Analyzers can also be used.

Chloride

Because chloride ions promote metal corrosion, their concentration in cooling water should not exceed certain limits. Chloride ions are determined by potentiometric titration with the titrant AgNO_3 after pH adjustment with nitric acid. A combined Ag-ring electrode, the Ag-Titrode, is used as the sensor. This electrode is maintenance-free because a pH glass membrane is used as reference electrode. In this way, it is no longer necessary to replenish electrolyte.



The Ag-Titrode for chloride determination



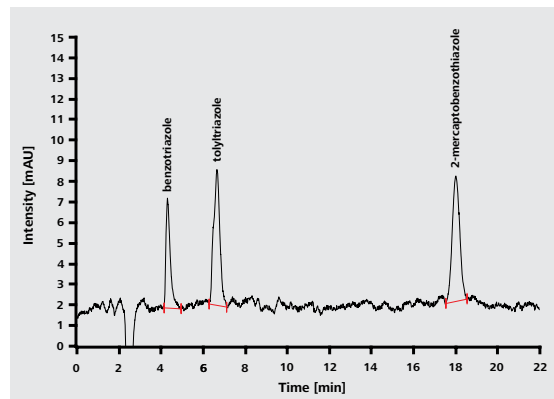


Corrosion inhibitors

Steel corrosion can be inhibited by adding zinc ions, phosphates, or phosphonates. These form thin films on the metal that protect it against corrosion. Corrosion of copper and its alloys can be inhibited by adding triazoles such as tolyltriazole, benzotriazole, and 2-mercaptobenzothiazole in the mg/L range. They form sparingly soluble compounds on the surface of the metal. Because the copper compounds of the triazoles are not resistant to oxidation and also react with added microbiocides, the triazoles have to be replenished, which necessitates regular determinations. This can be carried out by ion chromatography with spectrophotometric detection.

Heavy metals

Heavy metals such as Cu, Fe, Zn, and Pb mainly enter the cooling water via corrosion. In the branched piping system inside the condenser, they lead to incrustations with a very high resistance to thermal conduction that hamper heat transfer. They also act as catalysts for further contamination.



Sample of cooling water spiked with 1 mg/L of each corrosion inhibitor; column: Prontosil 120-3-C18-AQ 150/4.0; eluent: 0.5% phosphoric acid and 25% acetonitrile, 0.8 mL/min; column temperature 40 °C; wavelength: 214 nm (only the 2-mercaptobenzothiazole is detected at 320 nm); sample volume: 20 µL

These heavy metals can be determined with the 797 VA Computrace. This all-round measuring stand allows exact and sensitive determination of trace metals in cooling water. Sample preparation in the process water is not necessary.

Water-steam circuit – boiler feed water

Boiler feed water is the operating medium in the water-steam circuit of a thermal power plant and consists of recycled condensate from the water-steam circuit and conditioned makeup water. As it flows through the piping of the steam generator, it is heated and converted into high-purity steam that drives the turbines, thus generating electricity via the coupled generators. After the exhaust steam exits the turbine, it is condensed in a downstream condenser at the lowest possible temperatures and then recirculated back into the steam generator as feed water. The very high temperatures in the steam

generator lead to corrosion and deposits that severely reduce the efficiency of the power plant. This can be combated with an optimized feed water chemistry. On the one hand, the water must be ultrapure and on the other, the addition of conditioning agents (phosphates, oxygen scavengers) must be continuously monitored. The requirements for the water circulating in the water-steam circuit are very stringent and are defined, for example, in the standards EN 12952 (water-tube boilers and auxiliary installations) and EN 12953 (shell boilers).

EN 12953: Requirements for the feed water of steam boilers and hot-water boilers

Parameter	Feed water		Makeup water entire range
	> 0.5 to 20	> 20	
Operating pressure [bar]			
pH value at 25 °C	> 9.2	> 9.2	> 7.0
Conductivity at 25 °C [μ S/cm]	< 6000	< 3000	< 1500
Total hardness (Ca + Mg) [mmol/L]	< 0.01	< 0.01	< 0.05
Iron [mg/L]	< 0.3	< 0.1	< 0.2
Copper [mg/L]	< 0.05	< 0.03	< 0.1
Silica [mg/L]	pressure-dependent		
Oxygen [mg/L]	< 0.05	< 0.02	–
Oil/grease [mg/L]	< 1	< 1	< 1
Organic substances*		–	

* Organic substances can be degraded to products that increase acid conductivity, corrosion, and deposits. Furthermore, they can lead to foaming and/or the formation of deposits. Their concentration in boiler feed water should thus be kept as low as possible.

The methods described below apply to measuring limit values at different locations in the water-steam circuit and include the analyses of steam, condensate, boiler feed water, and makeup water. These methods are also used to control the water chemistry in the cooling system of a boiling water reactor or a pressurized water reactor and in the cooling circuit.

Steam for heating purposes and process steam

Steam is not only used to drive turbines, but also for heating purposes or as process steam in the chemical industry. Many of the applications described in this brochure also apply to these applications; however, they are not discussed here.

Sodium determination using ion-selective electrodes – process parameters

Sodium ions are nearly always present in water. The majority originates from the sodium hydroxide and trisodium phosphate additives for conditioning boiler water. Elevated sodium concentrations in the water-steam circuit also indicate leakages in the condenser where sodium-enriched cooling water infiltrates the high-purity process water. Sodium ions corrode metals and produce harmful deposits in the process water system and on turbine blades.

Sodium concentrations in power plant waters are usually less than 50 µg/L. The easiest way to measure the concentration is using ion-selective electrodes (ISE) and an ammonium or diisopropylamine buffer. In contrast, the ion-selective electrodes with polymer membranes used in the Alert Analyzers do not need a buffer. Their mode of operation is simple: the polymer membrane contains a molecule (ionophore) that binds only sodium ions. When sodium ions penetrate this membrane, they change its electrochemical properties and thus alter the potential. Detection limits are in the sub-µg/L range.

Sodium can also be determined by ion chromatography. Indeed, this is the method of choice if other cations have to be determined as well (page 17).

Silica – process parameters

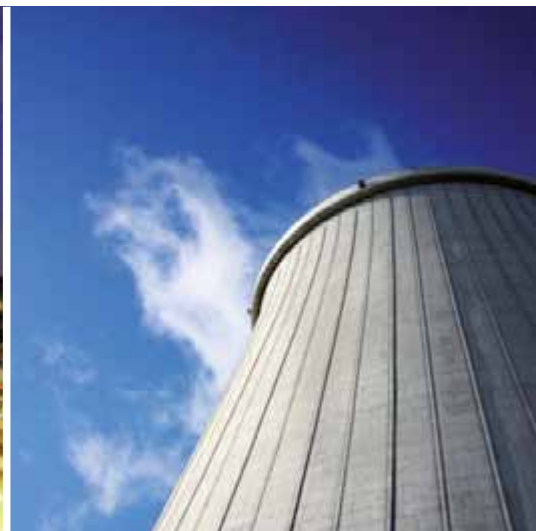
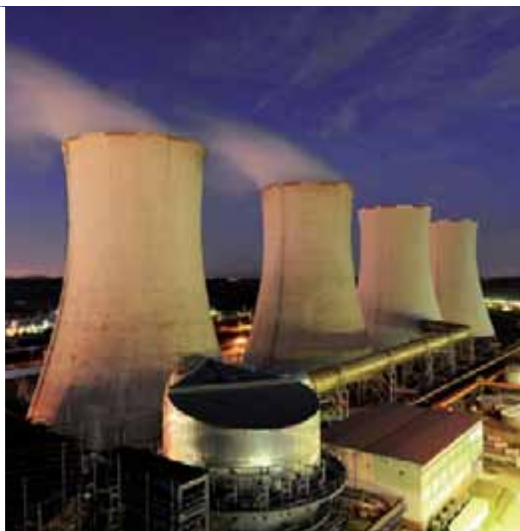
An excessive silica concentration in the boiler feed water or makeup water of power plants must be avoided. Silica (SiO₂) is a very weakly dissociated acid. During the treatment of makeup water colloidal silica is not retained by the ion exchangers and is hydrolyzed into soluble silica in the boiler. Owing to its volatility, it can enter the steam circuit at elevated pressures and then deposit on turbine blades, particularly in the presence of alkaline earth metals.

Silica is determined colorimetrically after treatment with ammonium molybdate and subsequent reduction of the resulting yellow silicomolybdic acid with ascorbic acid to blue silicomolybdous acid. Interference by phosphate, which is also determined by adding ammonium molybdate, is avoided by adding oxalic acid. Analysis is carried out with an Alert Analyzer; the detection limits are in the lower µg/L range.

Phosphate determination – process parameters

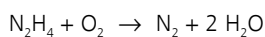
Phosphates are one of the most commonly used agents for conditioning cooling and boiler feed water. They form corrosion-resistant protective films on metal surfaces; cracks and defects are phosphatized in their presence. Boiler feed water is treated with trisodium phosphate (TNP), which reduces the residual hardness, phosphatizes, and alkalizes the water. Excessive TNP concentrations lead to undesirable foaming. Phosphate is usually added quasi-continuously.

In-process determinations are carried out colorimetrically using the phosphomolybdenum blue method at 875 nm. Ammonium molybdate reacts under acidic conditions with orthophosphate to the yellow dodecamolybdophosphoric acid (H₃[P(Mo₃O₁₀)₄]), which is reduced to phosphomolybdenum blue by strong reducing agents such as ascorbic acid. The detection limits lie in the mg/L range. Measurements are carried out with an Alert Analyzer, ADI 2019 or ADI 2045.



Oxygen scavenger (hydrazine) – process parameters

Dissolved oxygen is one of the main causes of corrosion in water circuits, which is why water is always thermally degassed before use. The residual oxygen is removed chemically – usually by adding reducing agents such as hydrazine or sulfite. Sulfite salts have the drawback that they are oxidized to corrosive sulfate and thus increase the salt content in the water-steam circuit. Although hydrazine is classified as carcinogenic, its efficiency in water circuits is hard to beat. It is an excellent oxygen scavenger, and its oxidation and decomposition products are salt free because they consist of only nitrogen, water, and ammonia.



Furthermore, hydrazine increases the pH value and is also a good corrosion inhibitor. It forms a passivating layer of magnetite in steel boilers and a protective oxide layer on copper alloys.

Hydrazine is determined colorimetrically with p-dimethylaminobenzaldehyde at 440 nm and is detected with Alert Analyzers. The analysis takes 10 minutes; the detection limit is in the lower µg/L range. Increasing use is being made of diethylhydroxylamine (DEHA) as an oxygen scavenger. It can also be detected colorimetrically with the Alert Analyzer. DEHA reduces added Fe(III) to Fe(II), which is then determined colorimetrically.

Overview of colorimetric determinations in power plant chemistry – process parameters

Analyte	Concentration range [mg/L]	Method (color reagent)	Analyzer
Water hardness (Ca, Mg)	0.005–5	Hydroxynaphthol blue (HNB)	Alert, ADI 2019, ADI 2045
Iron (Fe ²⁺)	0.005–1	Triazine	Alert, ADI 2019, ADI 2045
Copper (Cu ²⁺)	0.02–5	2,2-Bicinchoninic acid	Alert, ADI 2019, ADI 2045
Nickel (Ni ²⁺)	0.02–3	Dimethylglyoxime	Alert, ADI 2019, ADI 2045
Zinc (Zn ²⁺)	0.02–2	Zincon	Alert, ADI 2019, ADI 2045
Phosphate	0.01–7	Phosphomolybdenum blue	Alert, ADI 2019, ADI 2045
Silica	0.005–5	Molybdenum blue	Alert, ADI 2019, ADI 2045
Hydrazine	0.005–0.5	p-Dimethylaminobenzaldehyde	Alert, ADI 2019, ADI 2045
Diethylhydroxylamine (DEHA)	0.005–0.5	Fe ³⁺	Alert, ADI 2019, ADI 2045

Ion selective

- Ammonium
- Calcium
- Chloride
- Fluoride
- Nitrate
- Potassium
- Sodium



Colorimetric

- Aluminium
- Ammonium
- Chromium
- Copper
- Hydrazine
- Iron
- Nickel
- Nitrate
- Nitrite
- Phosphate
- Silica
- Zinc

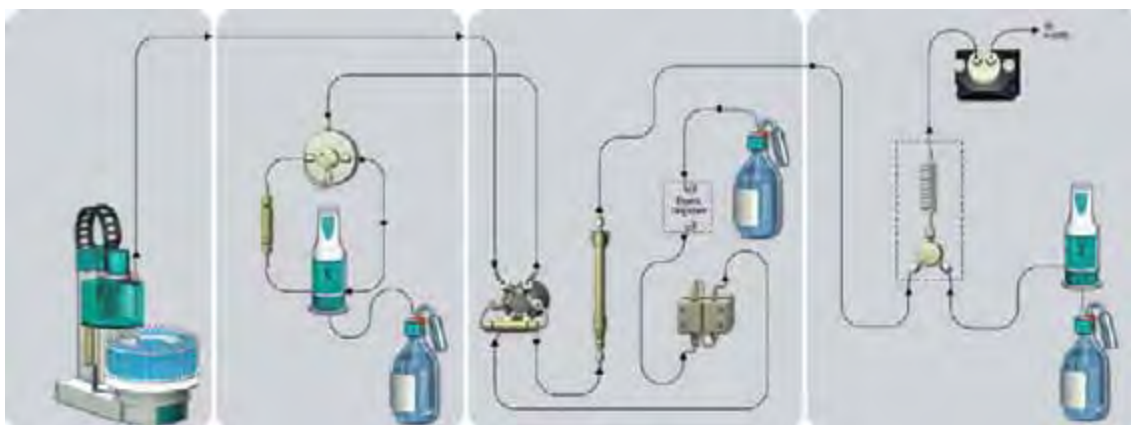
«Plug and analyze» – the two variants of the operator-friendly Alert Analyzers: the **Alert ADI 2003** for measuring with ion-selective electrodes (left) and the **Alert ADI 2004** for colorimetric measurements.

Transition metals

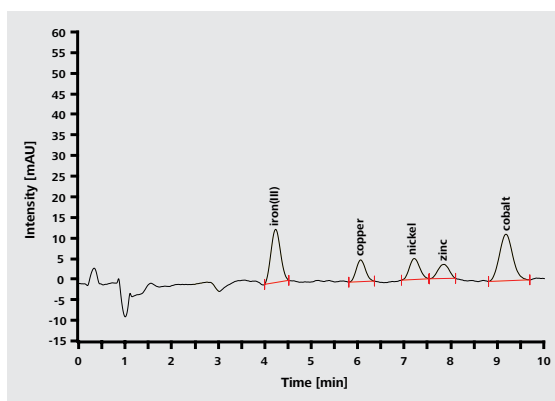
Metal surfaces in the power plant are in continuous contact with water and/or steam and corrosion occurs everywhere. Ions of the metals iron, copper, and nickel are important corrosion indicators. They are entrained by the steam and deposit on the turbine blades, thus greatly decreasing the efficiency.

Transition metals are determined by ion chromatography with UV/VIS detection. Samples are prepared using Inline Preconcentration of the metal cations. The eluent removes the metals from the preconcentration column and

flushes them in the form of their negatively charged anionic complexes onto the anion-exchange column, where they are separated. In the post-column reaction, the metals react with the chelating agent PAR to spectrochemically active complexes that are determined by UV/VIS detection. This method can differentiate between iron(II) and iron(III). As demonstrated by the cation determination in the water-steam circuit of a boiling water reactor on page 22, these can also be determined directly using conductivity detection.



Inline Preconcentration of samples in the sub- $\mu\text{g/L}$ range: The sample is withdrawn via the injection valve into a buffer volume. After the valve has been switched over, a sample volume, which depends on the preconcentration volume (0.1 to 10 mL), is applied to the preconcentration column. From here, the metals elute onto the separation column and then react with PAR in the post-column reactor to produce UV/VIS-active complexes that can be determined by the UV/VIS detector. The PAR reagent is supplied by a low maintenance, easily cleaned, and precise dosing unit.



Simulated sample from a water-steam circuit, spiked with 2 $\mu\text{g/L}$ each of iron(III), copper, nickel, zinc, and cobalt; column: Metrosep A Supp 10 - 75/4.0 (6.1020.070); eluent: 7 mmol/L dipicolinic acid, 5.6 mol/L Na_2SO_4 , 66 mmol/L NaOH, 74 mmol/L formic acid, 1 mL/min; column temperature: 45 °C; preconcentration volume: 4000 μL ; post-column reaction (PCR) with 0.11 g/L 4-(2-pyridylazo)resorcinol (PAR); flow rate: 0.2 mL/min; UV/VIS detection at 510 nm. Alternatively, microbore columns can be used; they consume less eluent and sample.

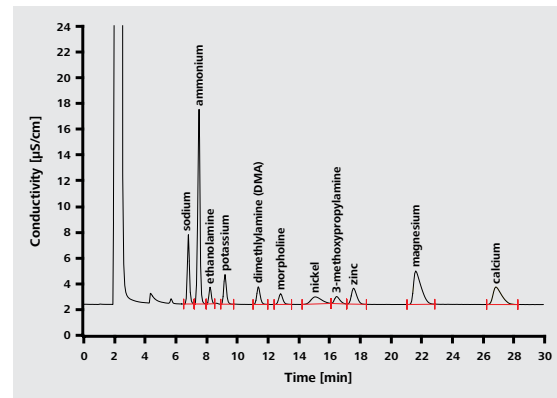
Amines and cations

Excessively low pH values increase the corrosion potential, whereas excessively high pH values destroy the protective layer on the metals. Adjustment of the pH value is challenging because the requirement for minimum corrosion and maximum protective layer leaves very little flexibility. pH values are usually adjusted with Lewis bases such as amines. Ion chromatography with conductivity detection provides an effective means to control amine addition. Alkali metals and alkaline earth metals can also be determined in the same analysis run. This enables immediate detection of leakages by infiltrating cooling water. Preconcentration volumes of 4 mL are sufficient to determine sodium concentrations down to 0.05 $\mu\text{g/L}$.

Corrosive anions at trace levels

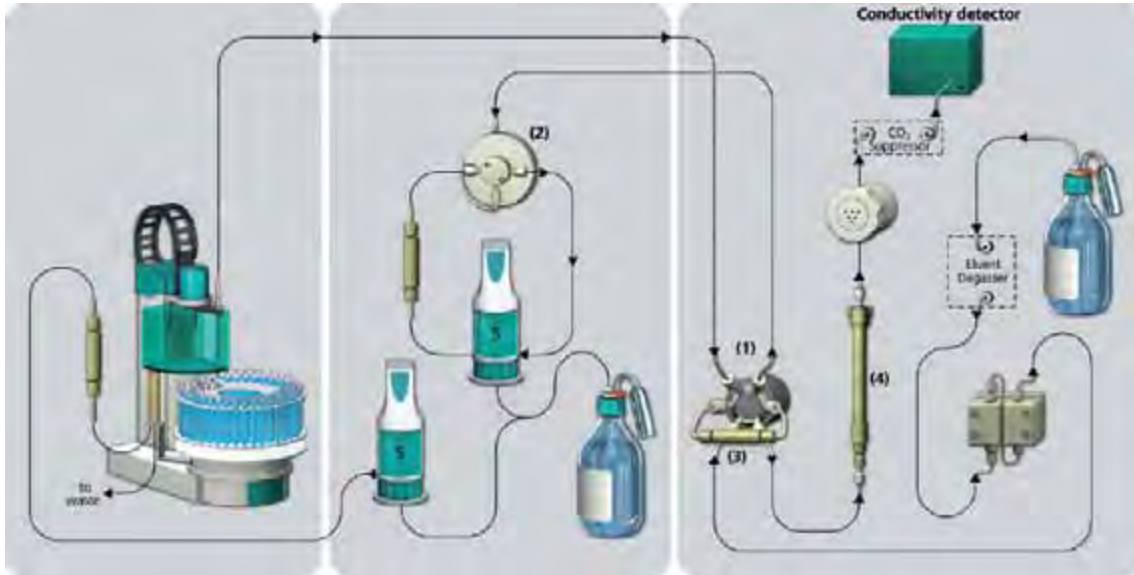
Chloride causes pitting corrosion on turbine blades and rotors. In combination with sulfate, it also leads to corrosion fatigue and stress corrosion cracking (SCC). Volatile ammonium compounds enhance these effects. Consequently, even trace levels of corrosive anions must be determined. Trace analysis of anions in the water-steam circuit is based on a combination of Inline Preconcentration and Matrix Elimination (MiPCT-ME). The target vol-

ume is transferred with microliter precision to the conditioned preconcentration column. The column is then rinsed with ultrapure water to remove the unwanted matrix. This protects the chromatography column and improves the separation efficiency. Anions can be determined in a concentration range from 0.01 to 10 000 $\mu\text{g/L}$.

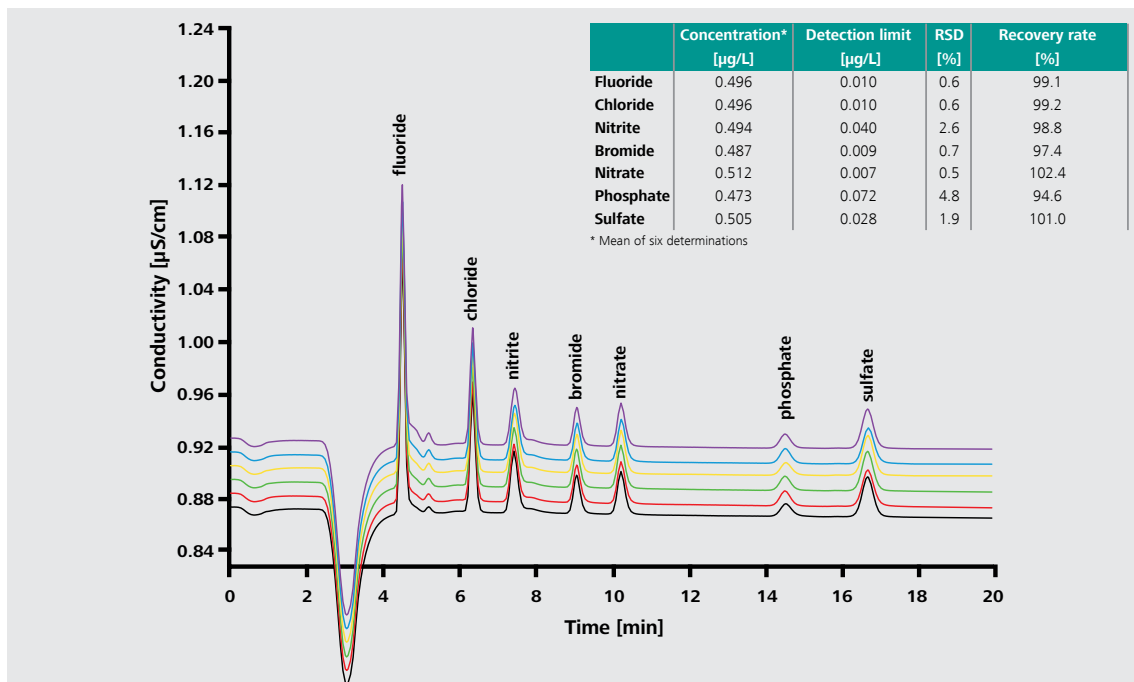


Simulated sample from a water-steam circuit treated with 1 mg/L of each amine and cation. All cations have a linear measuring range. Column: Metrosep C 4 - 250/4.0 (6.1050.430); eluent: 2.5 mmol/L HNO_3 and 0.5 mmol/L oxalic acid, 0.9 mL/min; column temperature: 32 °C; sample volume: 100 μL





Schematic diagram of combined Inline Preconcentration with Matrix Elimination (MiPCT-ME) for samples in the $\mu\text{g/L}$ range: The 800 Dosino aspirates the required sample volume through the injection valve (1) into the buffer volume (2). After the valve has been switched over, this volume is applied to the preconcentration column (3) from where the ions elute to the chromatography column (4) after the valve has been switched over again.



Simulated sample from a water-steam circuit treated with $0.5 \mu\text{g/L}$ of each anion; MiPCT-ME guarantees recovery rates and precision in the sub- $\mu\text{g/L}$ range that are otherwise only achieved in the mg/L range. Preconcentration volume: $4000 \mu\text{L}$; column: Metrosep A Supp 5 - 150/4.0 (6.1006.520); eluent: $3.2 \text{ mmol/L Na}_2\text{CO}_3$, $1.0 \text{ mmol/L NaHCO}_3$, 0.7 mL/min ; column temperature: $35 \text{ }^\circ\text{C}$

Automatic calibration

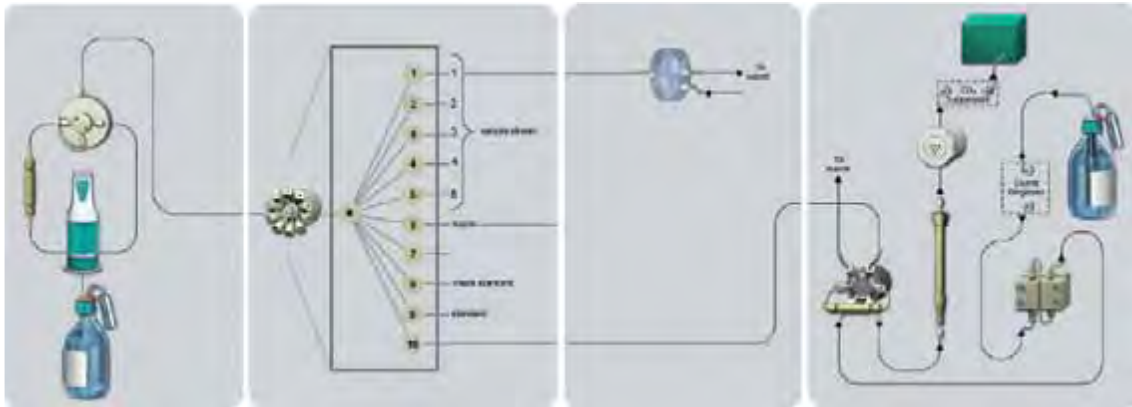
Both techniques, Metrohm intelligent Preconcentration with Matrix Elimination (MiPCT-ME) and Metrohm intelligent Partial Loop Injection Technique (MiPT) are ideally suited for routine analysis in power plants. With a single standard, a multipoint calibration is prepared and concentration ranges from the ng/L to the mg/L range are covered.



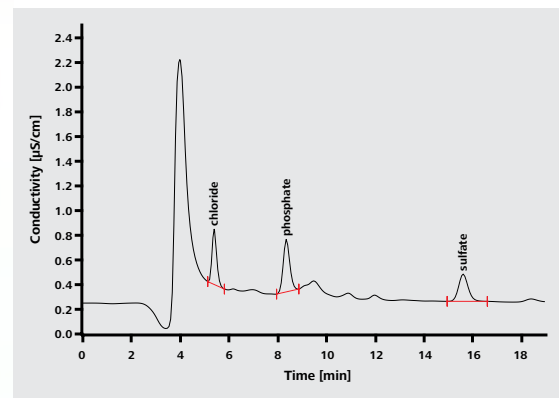
Chloride and sulfate in supercritical water-steam circuits

The higher the temperature and pressure of the steam driving the turbine blades is, the greater is the efficiency of the thermodynamic cycles. Power plants that use water above the critical point in the water-steam circuit (374 °C and 221 bar) thus produce significantly more electricity for a given amount of fuel. However, such su-

percritical boiler technologies have the highest requirements for boiler materials because these conditions promote corrosion and the formation of deposits. Metrohm's high-performance ion chromatography system provides fast, flexible, and extremely sensitive monitoring of the anion concentration in supercritical water-steam circuits.



The online monitoring system can analyze up to five freely selectable sample streams in alternation. If necessary, the samples pass through an ultrafiltration cell before being analyzed. Two further ports of the 10-port selector valve are used for the calibration and check standards.

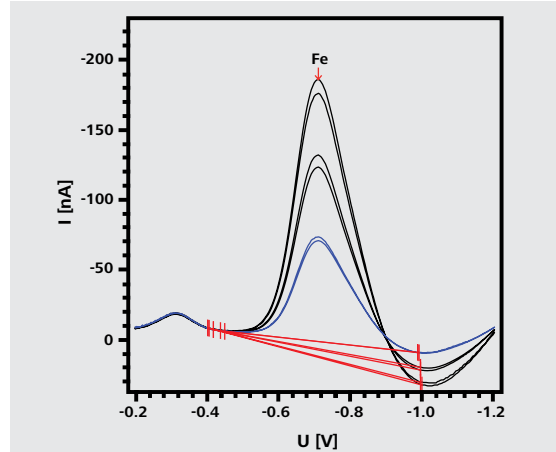


Simulated sample from the water-steam circuit of a supercritically operated reactor treated with 1 µg/L anions; column: Metrosep A Supp 10 - 100/2.0 (6.1020.210); eluent: 5 mmol/L Na₂CO₃, 5 mmol/L NaHCO₃, 0.25 mL/min; column temperature: 45 °C; preconcentration volume: 4000 µL

Iron

At high temperatures, steam reacts with the iron in the carbon steel of steam boilers. This leads to the formation of a thin layer of magnetite, an iron(II,III) oxide, which passivates the steel surface protecting it against further corrosion (Schikorr reaction). Under unfavorable conditions, the inhibiting magnetite layer can flake off, which leads to elevated iron concentrations in the water-steam circuit. A regular iron determination enables monitoring of not only corrosion processes but also the formation and destruction of the protective magnetite layer.

Adsorptive stripping voltammetry (AdSV) provides fast and sensitive detection of iron in process waters of the water-steam circuit (boiler feed water, makeup water, condensate) in power plants. This is achieved by adding suitable complexing agents to convert the iron into adsorbable complexes that are reduced on the electrode surface after a defined preconcentration time. Detection limits in the lower $\mu\text{g/L}$ range can be achieved using 2,3-dihydroxynaphthalene (DHN) as the complexing agent. Direct calibration by standard addition to the sample enables matrix-independent determination.



Voltammetric determination of iron

Copper and other heavy metals (iron, zinc, cadmium, lead, nickel, cobalt)

Copper alloys are now used almost exclusively in condensers of the water-steam circuit. The drawback is the susceptibility of copper and its alloys to corrosion by ammonia. The resulting corrosion products initiate further corrosive attack. Copper compounds already precipitate from steam in the high-pressure regions of steam turbines and deposit on the blades. They are determined voltammetrically according to DIN 38406-16. Sample preparation is not necessary.



797 VA Computrace: A user-friendly all-round measuring stand for sensitive determination of trace metals in process waters of power plants



Trace analysis of heavy metals – process parameters

The online determination of heavy metals at trace levels is performed with the ADI 2045VA Analyzer from Metrohm Applikon. The central unit of the ADI 2045VA is the 797 VA Computrace. This analyzer can be used to monitor the various water circuits in power plants using online voltammetry of up to four sample streams.

Iron and copper – process parameters

Quasi-continuous analysis of the iron concentrations in the condensate can be used for early detection of corrosion processes in turbines, pumps, or heat exchangers (water-steam circuit). Quasi-continuous analyses also guarantee that no dissolved iron reaches the condensate stream and thus the turbine blades where it would cause damage.

Adding 2,4,6-tripyridyl-s-triazine to iron(II) produces a violet complex that can be determined colorimetrically at 590 nm in the lower $\mu\text{g/L}$ range. The iron(III) content is determined after it has been reduced to iron(II).

Copper usually enters the water-steam circuit from one of its alloys. It is detected as the copper(I) ion by adding the sodium salt of 2,2-bicinchoninic acid. The resulting violet complex is detected at 550 nm down to the lower $\mu\text{g/L}$ range. The copper(II) content is determined after it has been reduced to copper(I).

Similar to the other colorimetric determinations, iron and copper are analyzed with an Alert Analyzer. Alternatively, a ADI 2019 or ADI 2045 Analyzer from Metrohm Applikon can be used.

Water-steam circuit in a boiling water reactor (BWR)

22

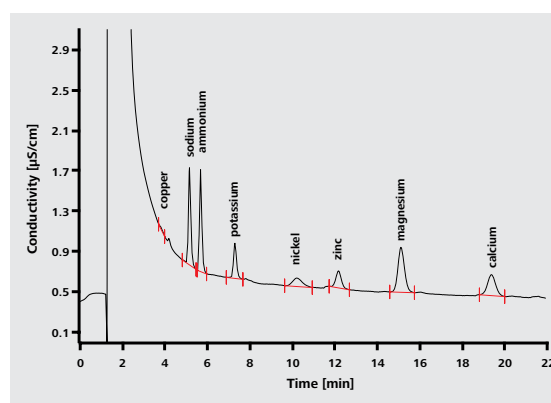
In a boiling water reactor (BWR), the energy generated by nuclear fission is used to evaporate water. The resulting steam is fed directly to the turbine driving the generators. Contaminants, such as corrosion products from the pipeline and tank materials, enter the water-steam circuit and thus reach the fuel assemblies. This also applies to leakages in the condenser that allow constituents from the less pure cooling water to enter the water-steam circuit so that they can reach the fuel assemblies where they adversely affect the operating conditions.

The water chemistry in the BWR varies according to manufacturer and plant engineering. Nobel metals are often added because they form a thin protective layer on the system surfaces.

Cations as well as copper, zinc, and nickel

Corrosion of steel and brass alloys liberates corrosive ions of the metals nickel, copper, and zinc. These are readily entrained by the steam and then deposit on turbine blades, thus considerably reducing the efficiency. In addition, the metals released by corrosion undergo nuclear reactions and thus increase radiation in the power plant. To limit corrosion of the materials and to prevent the formation of radioactive ^{60}Co (produced by neutron capture by the stable ^{59}Co isotope present in the steel alloys), the cooling water in the BWR is often treated with depleted zinc oxide (contains < 1% of the stable main isotope ^{64}Zn).

As already described on pages 10 to 11, at elevated temperatures, salts of the alkaline earth metals deposit on the heat transfer surfaces as an insulating layer of boiler scale that reduces heat transfer. Ion chromatography with conductivity detection provides sensitive determination of Cu, Zn, Ni, alkali metals, alkaline earth metals, and ammonium.



Simulated sample from a water-steam circuit, spiked with 0.5 $\mu\text{g}/\text{L}$ of the standard cations as well as copper, nickel, and zinc; column: Metrosep C 4 - 250/2.0 (6.1050.230); eluent: 2.5 mmol/L HNO_3 , 0.5 mmol/L oxalic acid, 0.4 mL/min; column temperature: 32 $^\circ\text{C}$; preconcentration volume of sample: 9800 μL

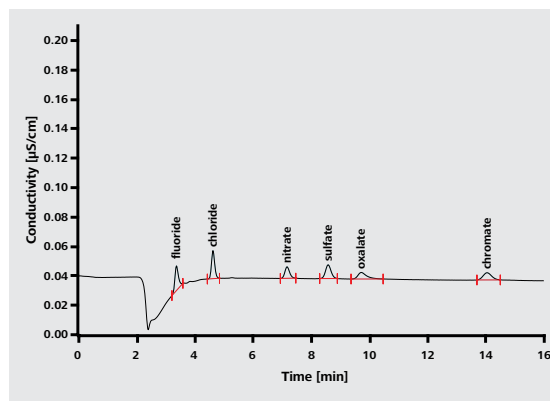
The transition metals can also be determined spectrophotometrically, as described on page 16.





Corrosive anions

The combination of Metrohm Inline Preconcentration (MiPCT) and automatic calibration enables detection of corrosive anions in the lower $\mu\text{g/L}$ range. This method is suitable for water-steam circuits in nuclear and fossil fuel-fired reactors. Chromate can also be analyzed at the same time to a detection limit of 50 ng/L. The detection limits can be lowered even further by increasing the preconcentration volume.



Simulated sample from a water-steam circuit of a boiling water reactor treated with 50 ng/L of each of the standard anions and chromate; column: Metrosep A Supp 5 - 150/4.0 (6.1006.520); eluent: 4.8 mmol/L Na_2CO_3 , 1.5 mmol/L NaHCO_3 , 0.8 mL/min; column temperature: 30 °C; preconcentration volume: 2000 μL

Primary circuit in a pressurized water reactor (PWR)

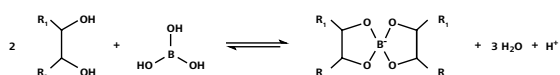
24

The most common types of nuclear reactors are the boiling water reactor (BWR) and the pressurized water reactor (PWR). The PWR has three circuits, whereas the BWR has only two. The primary circuit of the PWR circulates water under a high pressure (up to 160 bar) through the reactor core. It absorbs the heat released by nuclear fission. In the steam generator, the water – now at a temperature of approx. 325 °C – transfers its heat to the secondary circuit, which is a conventional water-steam circuit with steam generator, turbine, and cooled condenser. The additional circuit in the PWR ensures that the radioactive materials remain in the primary circuit.

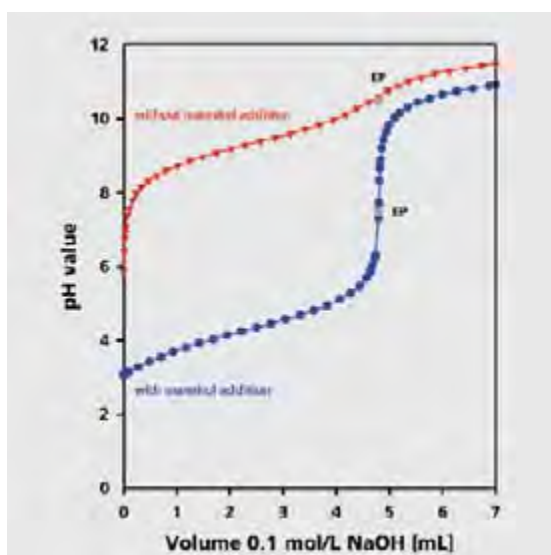
The pressurized water reactor is controlled by the control rods in the core and by the boric acid dissolved in the primary circuit. The ^{10}B isotope, in particular, acts as a moderator and captures the fission neutrons that keep the nuclear chain reaction going and which are responsible for the reactivity of the reactor. Determination of the constituents in the primary circuit is extremely important with regard to reactor safety and its efficiency.

Boric acid

A simple and quick determination of the boric acid concentration is important to control the reactivity in the PWR. Boric acid has an acidity constant K_{a1} of $5.75 \cdot 10^{-10}$ ($\text{p}K_a = 9.24$), which means it is a weak acid that is difficult to titrate. The addition of polyalcohols, for example, mannitol, leads to the formation of complexes with a greater acidic strength that behave like a monovalent acid which can be easily titrated with sodium hydroxide solution. The equilibrium of the complexation reaction lies on the right-hand side of the following equation:



In the manual determination method, the sample is pipetted into a titration cell, diluted with distilled water, and treated with a defined (excess) volume of a saturated mannitol solution. After stirring, the mixture is titrated with 0.1 molar sodium hydroxide solution to a pH value of 8.5. Accurate pipetting and determination of the blank value of mannitol are essential for precise determination of boric acid. This method can also be used to determine the boric acid content in the spent fuel pool.



Titrimetric determination of the boric acid content in a simulated sample from the primary circuit of a PWR: red line: titration without added mannitol; blue line: after addition of mannitol.



The Robotic Boric Acid Analyzer

Metrohm offers a fully automated system for the analysis of boric acid. It includes not only complete traceability, but also a high sample throughput during 365/24/7 operations.

Boric acid – process parameters

Pressurized water reactors, which use light water, do not allow the fuel assemblies to be exchanged during operation so that a fuel reserve has to be in place at the start of an operating cycle. The associated excess activity in the reactor is controlled by a higher boric acid concentration. As the fuels burn up, the boric acid concentration has to be lowered to keep the reactor running at maximum output. This is achieved by replacing water containing boric acid with ultrapure water: the boric acid concentration between two fuel reloadings varies between 2000 mg/L and almost zero.

Quasi-continuous control of the constantly changing amounts of boric acid in the primary circuit is extremely important for efficient and reliable operation. Continuous determination of the boric acid in the process is thus essential. Both the ADI 2016 and the ADI 2045TI Analyzers from Metrohm Applikon provide fast and reliable determination via potentiometric titration. As already described on page 24, the acidic boric acid esters resulting from the addition of mannitol are titrated.



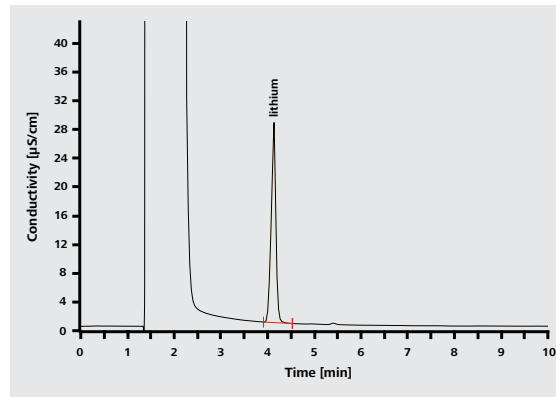
ADI 2045TI – a flexible analyzer for online applications in power plant chemistry



Lithium

An optimum pH value of the water in the primary circuit prevents attack of the metallic materials and destruction of the protective layers adhering to them. The addition of boric acid to the primary circuit of the PWR lowers the pH value and thus increases the corrosion potential. This is prevented by adding an alkalizing agent to the primary circuit. Monoisotopic lithium hydroxide ${}^7\text{Li}$ (approx. 2 mg/L) is used in most pressurized water reactors. On the one hand, ${}^7\text{Li}$ does not undergo any undesirable nuclear reactions, and on the other hand, it is already present in the reactor because it is formed by a neutron capture reaction of the boron: ${}^{10}\text{B}(n,\alpha){}^7\text{Li}$.

Lithium cations can be determined with the intelligent partial loop injection technique (MiPT). Based on the actual sample concentration, the system calculates and injects the required volume (2–200 μL) of undiluted sample. This method is fast, precise, and can be combined with ultrafiltration.

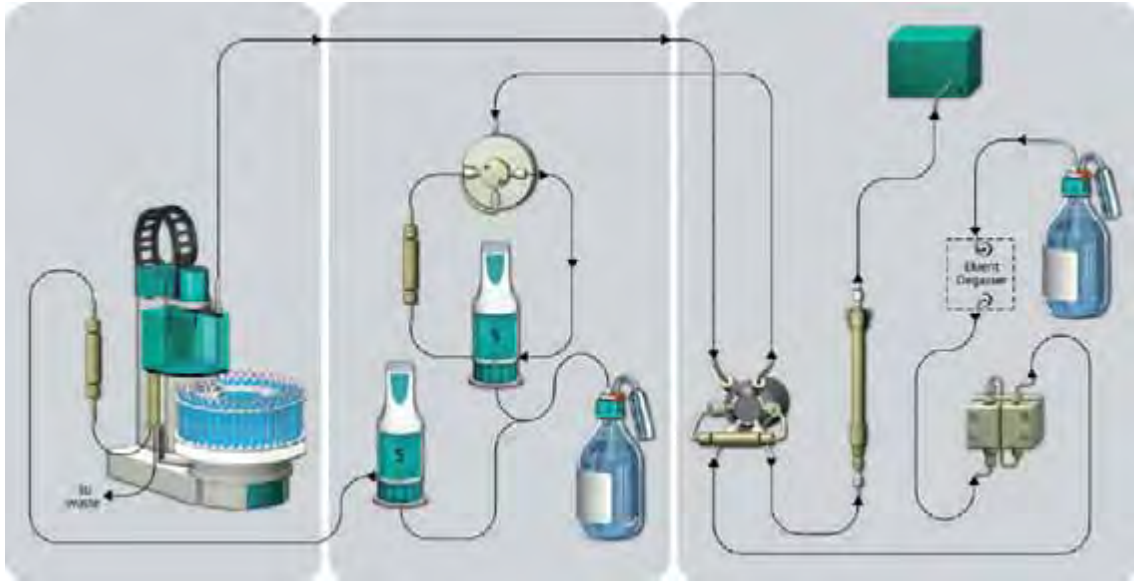


Simulated sample from the primary circuit of a PWR containing 1 g/L boric acid, treated with 1.7 mg/L lithium hydroxide; column: Metrosep C 4 - 250/2.0 (6.1050.230); eluent: 2.5 mmol/L HNO_3 , 0.5 mmol/L oxalic acid, 0.4 mL/min; column temperature: 32 °C; sample volume: 20 μL

Further cations

The same ion chromatograph as that used to analyze lithium can also be used to determine nickel, zinc, calcium, and magnesium. Even the same separating condi-

tions can be used. The only difference is the required preconcentration volumes.

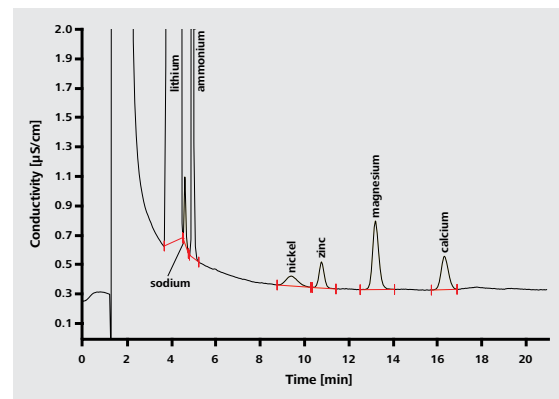


Combined Inline Preconcentration and Matrix Elimination for the determination of metals in the $\mu\text{g/L}$ range. The samples from the primary circuit are withdrawn through the injection valve into a buffer volume. After switching to the fill position, the accurately dispensed volume is transferred to the preconcentration column.

Nickel, zinc, calcium, and magnesium

Nickel is an important alloying metal that increases the corrosion resistance of steel. However, if nickel ions enter the primary circuit, they promote corrosion. Therefore, their concentration must be checked at regular intervals. Depleted zinc (containing $< 1\%$ of the stable main isotope ^{64}Zn) is often added to the primary circuit. This not only reduces the radioactivity on the component surfaces, but it also lowers corrosion of the metal surfaces in contact with the water.

In the primary circuit, which contains boric acid and lithium hydroxide, combined Inline Preconcentration and Matrix Elimination can be used to determine the metal concentrations down to the sub- $\mu\text{g/L}$ range.

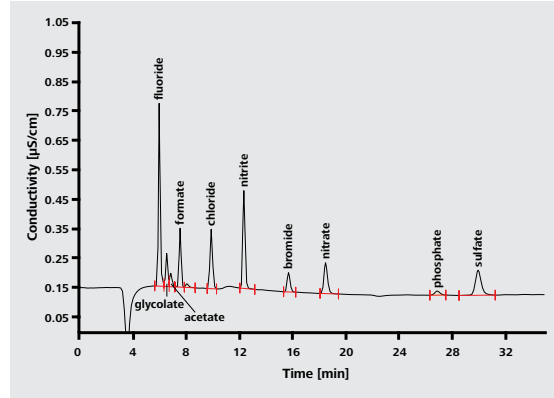


Simulated sample from the primary circuit of a pressurized water reactor containing 2 g/L boric acid and 3.3 mg/L lithium hydroxide, treated with 2 $\mu\text{g/L}$ each of nickel, zinc, calcium, and magnesium; column: Metrosep C 4 - 250/2.0 (6.1050.230); eluent: 2.5 mmol/L HNO_3 , 0.5 mmol/L oxalic acid, 0.4 mL/min; column temperature: 32 °C; preconcentration volume: 1000 μL

Corrosive anions

Anions corrode metals and their concentration must be checked at regular intervals. The analytical challenge is to detect anions in the $\mu\text{g/L}$ range alongside gram quantities of boric acid and lithium hydroxide. The analysis is carried out fully automatically using a combination of Inline Matrix Elimination (for the borate) and Inline Neutralization (for the LiOH). Successful trace analysis depends on the implemented preconcentration technique. Detection is also successful if boric acid is neutralized with ammonium instead of LiOH. A further advantage is automatic calibration. It guarantees excellent detection limits, a high reproducibility, and excellent recovery rates.

In addition to the standard anions – fluoride, chloride, nitrate, and sulfate – important organic degradation products such as glycolate, formate and acetate are determined with high precision. Their presence usually indicates defective ion exchangers that are used to condition the boiler feed water.



Water sample from the primary circuit of a pressurized water reactor containing 2 g/L boric acid and 3.3 mg/L lithium hydroxide spiked with 2 $\mu\text{g/L}$ anions; column: Metrosep A Supp 7 - 250/4.0 (6.1006.630); eluent: 3.6 mmol/L Na_2CO_3 , 0.8 mL/min; column temperature: 45 °C; preconcentration volume: 2000 μL





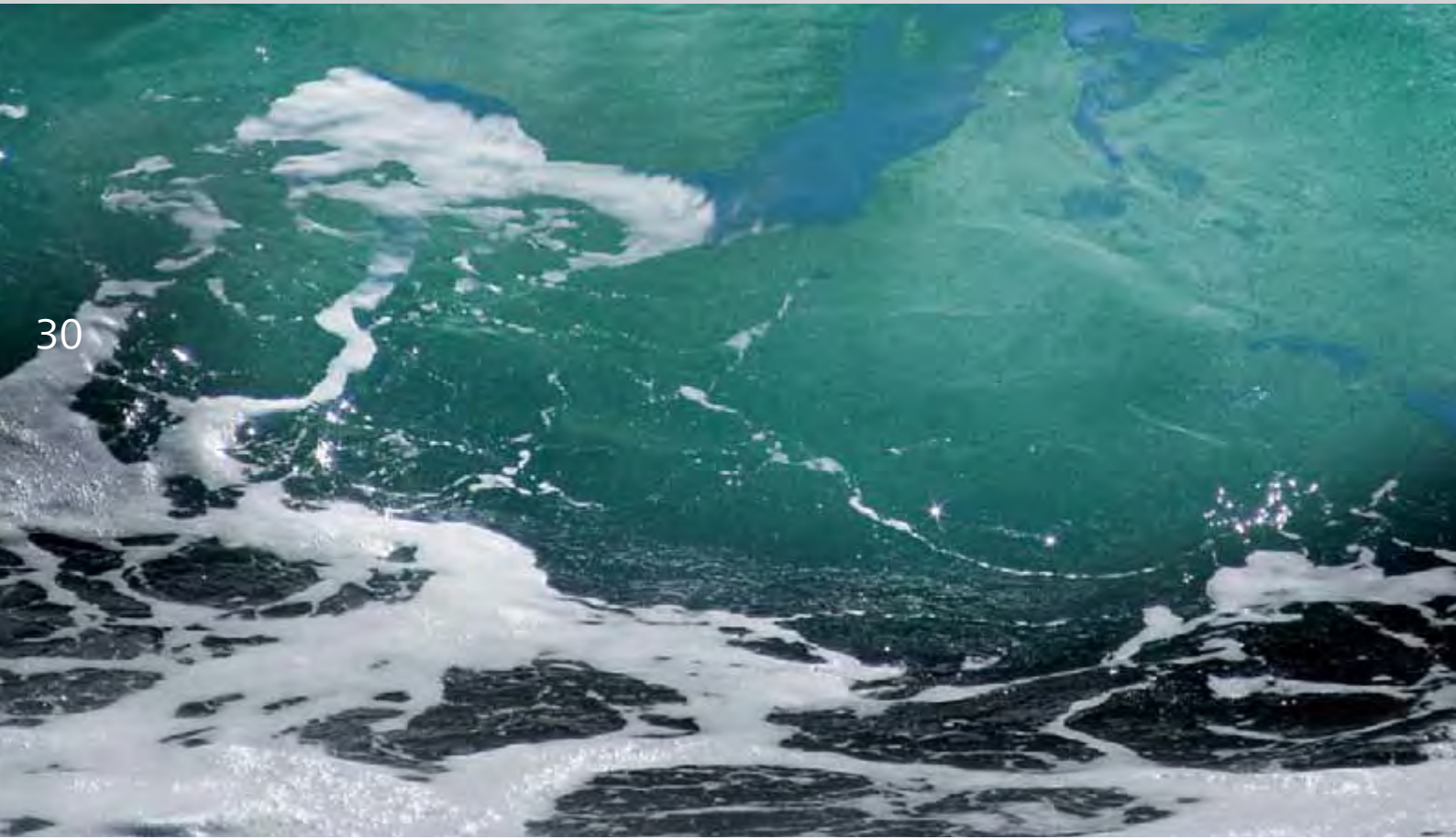
Wastewater

Recycling of process water in the cooling circuits is becoming increasingly common in modern power plants. When the water is finally discharged into the environment, it must comply with the limit values for a large number of compounds. Most constituents originate from chemicals added to the water during conditioning. These include corrosion inhibitors, oxygen scavengers, alkalis, and acids. There is also radioactive and boric acid-containing wastewater from the circuits of boiling water and pressurized water reactors. The stringent mandatory requirements entail comprehensive wastewater analysis. Parameters such as the pH value, conductivity, chemical oxygen demand (COD) as well as the concentration of heavy metals, hydrazine, chloride, and sulfate are strictly

regulated, and compliance with limit values necessitates precise analysis. Metrohm brochures on water and environmental analysis discuss numerous analytical wastewater applications. This document describes an exemplary determination of heavy metals and process analysis in flue gas cleaning plants.

Heavy metals in wastewater

In the water circuits of power plants, water is in permanent contact with metals and metal alloys. Heavy metals enter the water circuits due to corrosion and must therefore also be taken into account. Zinc, cadmium, lead, and copper can be determined with high sensitivity by voltammetry in accordance with DIN 38406 Part 16.



Wastewater from flue gas cleaning – process parameters

The combustion of fossil fuels – including incineration of domestic refuse – produces atmospheric pollutants such as carbon dioxide, nitrogen oxides, and sulfur oxides as well as dust. These all have to be removed from the flue gas. In the first step, the nitrogen oxides are catalytically reduced to nitrogen. Electric filters then remove the dust before the scrubbing towers in the flue gas desulfurization plant convert sulfur dioxide into sulfate using a limestone slurry and oxygen, turning it into gypsum.

The resulting wastewater is contaminated and must be subjected to complex chemical and physical treatments before it can be discharged into the environment. The analytical parameters that need controlling require measurement of the pH value, analysis of sulfur species, and determination of heavy metals.

This is the application field of the online or atline analyzers of Metrohm Applikon, regardless of whether it is for a single sample stream or for complex multiple sample streams. The analyzers are based on wet chemical methods such as titration, colorimetry, or measurements with ion-selective electrodes.

Flue gas cleaning

CO₂ binding capacity of amine scrubbing – process parameters

From 1930 onwards, removing CO₂ in the flue gas arising from the combustion of organic materials using amine scrubbing became increasingly widespread. This technique was used in fossil-fueled power plants for the first time in 1980. Flue gas is treated with a scrubbing solution containing amines (20–30% monoethylamine, MEA). The acidic CO₂ is reversibly chemically bound by the amines before it is released by heating, compressed, dried, and liquefied. After the amine-containing scrubbing solution has been cleaned by counterflowing steam, it is cooled and recycled back into the process.

The ADI 2045TI Process Analyzer from Metrohm Applikon determines the CO₂ binding capacity of the scrubbing solution that is required to completely remove the CO₂ in the flue gas. This means determining the CO₂ content of the flue gas. The CO₂ is captured with an NaOH solution, and excess NaOH is then determined by titrating with HNO₃.

A single analyzer can monitor several sample streams and determine the CO₂ binding capacity of several amine scrubbers in succession.



II. Turbine and lubricating oils

New power plant technologies and improvement of the efficiencies of gas and steam turbines present ever greater requirements regarding lubricant performance. ASTM D 4378 describes requirements and test procedures for in-service maintenance of steam and gas turbines. Key parameters to be determined are the acid and base numbers as well as the water content using Karl Fischer titration. The applications described below also apply to oils and lubricants used in turbines of water- and wind-driven power plants.

Acid and base numbers

Determination of acid and base numbers

With the base number, alkaline components are determined in petroleum products as a sum parameter. These include primary organic and inorganic amino compounds in particular. However, salts of weak acids, basic salts of polycarboxylic acids, a number of heavy metal salts, and detergents are also registered. The base number indicates how many basic components, expressed as mg KOH, are contained in 1 g of sample. This determination is used for the immediate detection of product changes during use.

With the acid number, acidic components are measured in petroleum products as a sum parameter. These are compounds (acids, salts) with pK_s values < 9 . The acid number indicates how many mg KOH are required to neutralize 1 g of sample. It also detects changes during the use of the product. Both parameters are determined by potentiometric titration in nonaqueous solvents or solvent mixtures. Titrimetric determinations can be completely automated – from the addition of solvents to the cleaning of the electrode. Oil samples can even be weighed fully automatically by the 864 Robotic Balance Sample Processor before titration. This guarantees complete traceability.

The Solvotrode easyClean is a combined pH glass electrode that was especially developed for this application. The detachable ground-joint diaphragm can be easily cleaned even of strong contamination. The electrostatic shielding of the electrolyte compartment also ensures a low-noise measuring signal.

Determination of acid and base numbers according to ASTM D 974 (photometric titration)

The acid and base numbers may also be determined by photometric titration with color indication of the equivalence point according to ASTM D 974. For this application, Metrohm offers the Optrode, a new sensor for photometric titration. It is 100% solvent-resistant (glass shaft) and – unlike visual endpoint recognition – enables automation of the determination.



Optrode

Solvotrode easyClean

Important standards relating to TAN/TBN determination

Standard	Parameter	Titrant	Solvent	Electrode (reference electrolyte)
ASTM D 4739	Base number	HCl in isopropanol	Chloroform, toluene, isopropanol, water	Solvotrode easyClean (LiCl in EtOH)
ASTM D 2896	Base number > 300 mg KOH/g	Perchloric acid in glacial acetic acid	Glacial acetic acid, xylene	Solvotrode easyClean (TEAB ^a in ethylene glycol)
DIN ISO 3771	Total base number	Perchloric acid in glacial acetic acid	Toluene, acetic acid, acetone	Solvotrode easyClean (TEAB ^a in ethylene glycol)
ASTM D 664	Acid number	KOH in isopropanol	Toluene, isopropanol, water (lubricants), isopropanol (biodiesel)	Solvotrode easyClean (LiCl in EtOH)
DIN EN 12634	Acid number	KOH or TMAH ^b in isopropanol	Dimethylsulfoxide, isopropanol, toluene	Solvotrode easyClean (LiCl in EtOH)
UOP565	Acid number and naphthenic acids	KOH in isopropanol	Toluene, isopropanol, water	Solvotrode easyClean (LiCl in EtOH)
ASTM D 974	Acid number	KOH in isopropanol	Toluene, isopropanol, water	Optrode
ASTM D 974	Base number	KOH in isopropanol	Toluene, isopropanol, water	Optrode

^aTetraethylammonium bromide

^bTetramethylammonium hydroxide



855 Robotic Titrosampler (with 772 Pump Unit) for TAN/TBN determination

Water determination by Karl Fischer titration

34

Water is nearly always present as a contaminant in all turbine and lubricating oils. It lowers the lubricity, promotes oxidation of the oil, reacts with water-sensitive additives, and enhances corrosion of ferrous and non-ferrous metals. At high oil temperatures, water evaporates and leads to partial degreasing. In contrast, the turbine and lubricating oils used in wind turbines are often exposed to sub-zero temperatures so that the water forms ice crystals and thus reduces lubricity.

Efficient and reliable power generation requires control of the water content in the oils used in the power plant. As a consequence of the very good reproducibility and accuracy as well as the simple handling, many international standards recommend the Karl Fischer titration method. These include ASTM D 6304 and ISO 12937. Determination can be carried out volumetrically or coulometrically.

Coulometric determination

Coulometric determination with a diaphragm cell is particularly suitable for turbine and lubricating oils that contain very little water. To ensure that the oils completely dissolve in the methanol-containing Karl Fischer reagents, solubilizing agents such as chloroform or trichloroethylene are added.

Additives in turbine and lubricating oils can undergo side reactions with Karl Fischer reagents and thus falsify the analyzed water content. Some of these side reactions can be suppressed using special KF reagents. If this is not possible, the oven method can be used instead.



851 Titrando (center) with 900 Touch Control and 801 Stirrer

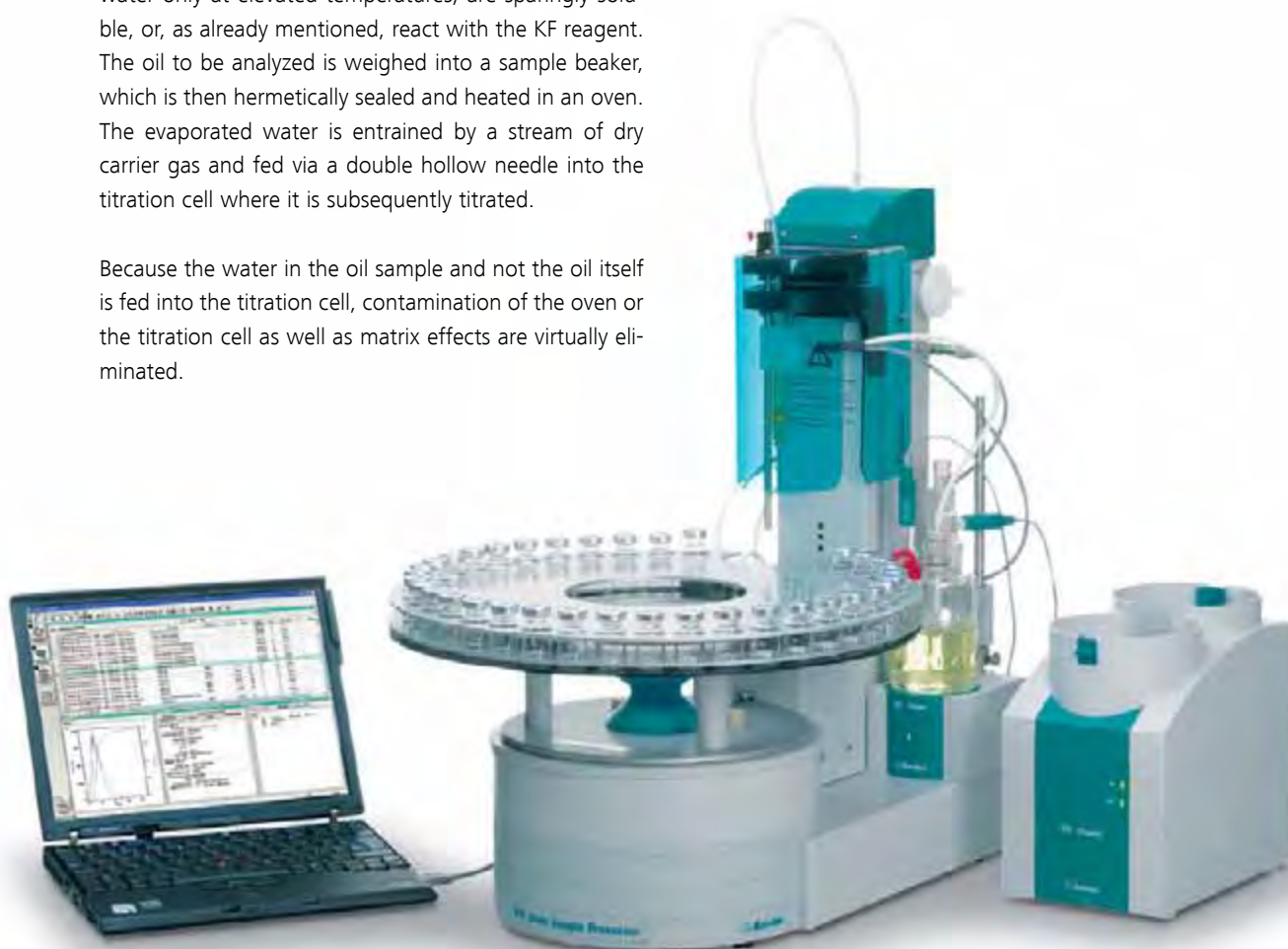


899 Coulometer (center) with 860 KF Thermoprep and USB Thermal Printer Neo's

Oven method

The oven method is suitable for samples that release their water only at elevated temperatures, are sparingly soluble, or, as already mentioned, react with the KF reagent. The oil to be analyzed is weighed into a sample beaker, which is then hermetically sealed and heated in an oven. The evaporated water is entrained by a stream of dry carrier gas and fed via a double hollow needle into the titration cell where it is subsequently titrated.

Because the water in the oil sample and not the oil itself is fed into the titration cell, contamination of the oven or the titration cell as well as matrix effects are virtually eliminated.



874 USB Oven Sample Processor with 851 Titrand

III. Fuels and operating materials

36 Halogens and sulfur using Combustion IC

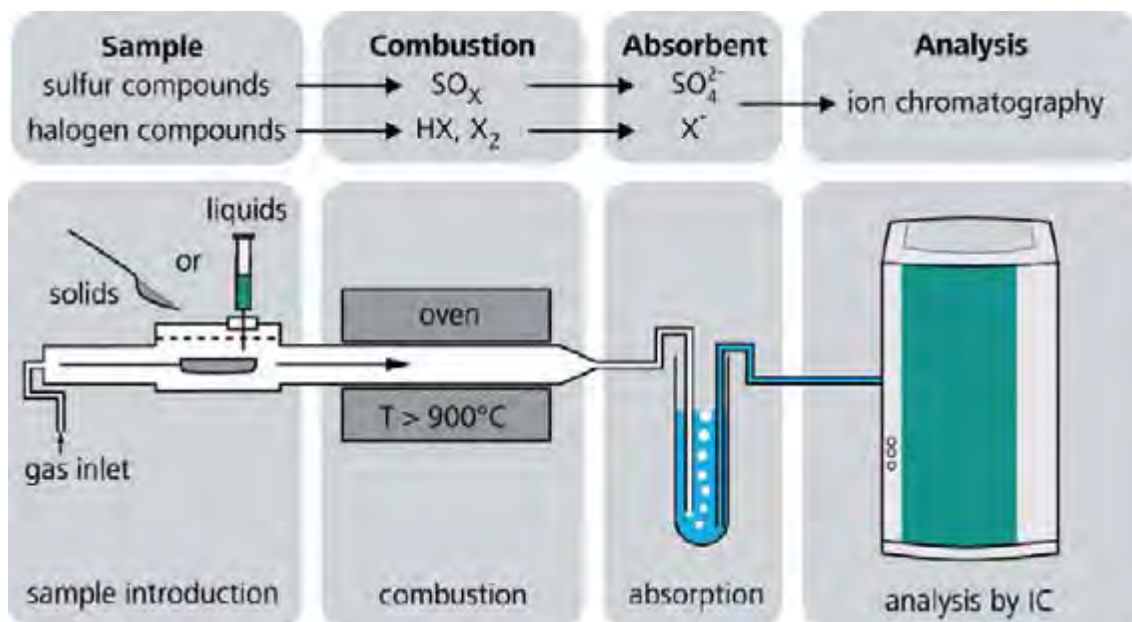
The fossil fuels combusted in power plants, such as mineral oil and coal, may contain large amounts of sulfur (e.g., mercaptans, sulfides, elemental sulfur, etc.). Combustion produces sulfur dioxide (SO_2). This reacts with water and oxygen to sulfuric acid and the resulting «acid rain» damages plants, buildings, and watercourses. In the cooling circuits of power plants, halides promote corrosion, which necessitates determining their concentrations in the operating and auxiliary materials used in power plants (e.g., seals and latex gloves).

The Combustion IC system developed by Analytik Jena and Metrohm allows simultaneous determination of halogens and sulfur in all combustible samples, regardless

of whether they are solid or liquid. The fully automated inline system is far superior to offline digestion methods and also boasts high sample throughput, precision and accuracy. A flame sensor controls the combustion process and saves on time-consuming method developments.

The principle

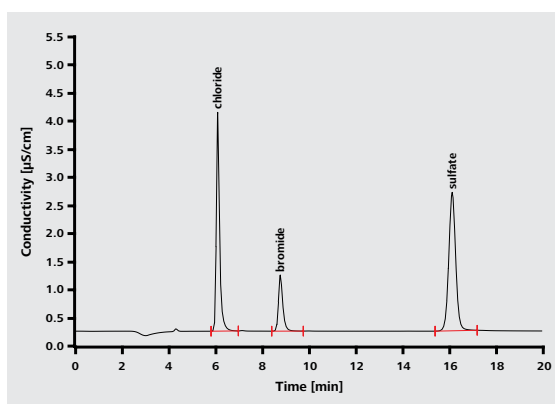
The first step of the CIC method is pyrolysis of the samples in the oven under an argon atmosphere, followed by combustion with oxygen. Tiny amounts of water are injected into the pyrolysis oven to prevent deposits or corrosion of glass by HF and to improve discharge of the combustion products.



Sulfur and halogen compounds are subjected to combustion digestion (pyrolysis) and converted into sulfur dioxide, respectively, hydrogen halides and elemental halogens. These gaseous combustion products are fed into an oxidizing absorption solution and detected as sulfate and halides by subsequent ion chromatography.

Halogen and sulfur content in plastics and combustible materials

In addition to the aforementioned determination of sulfur in fuels (p. 36), Combustion IC can also detect halogens in solid materials. Only halogen-free materials should be used in the water circuits of power plants. This prevents corrosive anions, for example, from seals, entering the process water.

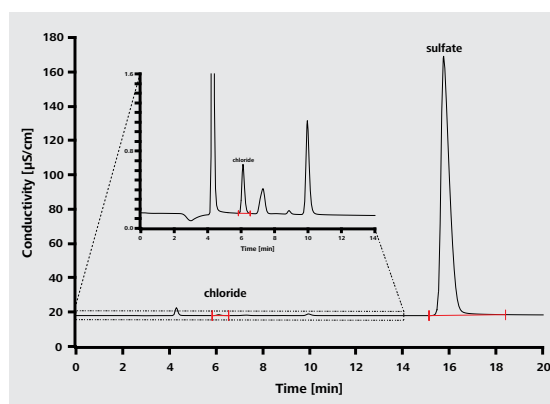


Determination of halogens and sulfur in certified PE pellets ERM-EC681k; recovery rates: chloride (102.4%), bromide (95.4%), sulfur (100.3%); column: Metrosep A Supp 5 - 150/4.0; eluent: 3.2 mmol/L Na_2CO_3 , 1.0 mmol/L NaHCO_3 , 0.7 mL/min

Halogen and sulfur contents in latex gloves

Gloves are used in cleanrooms to hold back ionic contaminants from hand sweat. In the water-steam circuit of power plants as well as the primary circuit of pressurized water reactors, only halogen- and sulfur-free materials are used to prevent contamination with corrosive halides or sulfates. The halogen and sulfur contents in latex gloves can be easily and reliably determined by Combustion Ion Chromatography.

The method is able to determine halogens and sulfur in aromatic hydrocarbons as described in ASTM D 7359 as well as the total fluorine content in coal and coke according to ASTM D 5987.



Determination of halogens and sulfur in coal reference material NIST 2682b; recovery rates: chloride (103.4%), sulfur (96.8%); column: Metrosep A Supp 5 - 150/4.0; eluent: 3.2 mmol/L Na_2CO_3 , 1.0 mmol/L NaHCO_3 , 0.7 mL/min

Halogen and sulfur contents in ion-exchange resins

Ion-exchange resins are used in power plants to remove at least 98% of the dissolved salts in the makeup water or in the condensate. This is only possible if high-purity ion-exchange resins are used. The halogen and sulfur contents in ion-exchange resins can also be conveniently determined by combustion IC.



Service you can rely on – Metrohm Quality Service

Analyses in power plants is an integral component of chemical analysis and primarily deals with investigations of the operating medium: water. If the plant is to operate reliably and efficiently, it needs an optimum water chemistry that aims to minimize corrosion and deposits. Also important is the analysis of turbine and lubricating oils as well as the fuels and operating materials used in fossil fuel-fired power plants and waste incineration plants.

Whoever is responsible in the powerplant for the accuracy of the results must not make compromises. Fortunately, systems installed and maintained by professionals on a regular basis all but eliminate the threats of instrument failure and lost profits.

Relying on the Metrohm Quality Service gives you peace of mind from the very start. From the professional installation of your instruments to regular maintenance care and – should a failure ever occur – instant quality repairs,

we do everything to make sure that you can rely 100 percent on results produced during the entire lifetime of your Metrohm instruments.

Metrohm Compliance Service

Benefit from the Metrohm Compliance Service when it comes to the professional initial qualification of your analytical instruments. Installation Qualification/Operational Qualification carried out by our experts saves you time and money, as your analytical system is configured according to your needs and put into operation fast and reliably.

Initial instructions and user trainings ensure error-free operation of your new instruments by your staff. The Metrohm Compliance Service includes comprehensive documentation and guarantees compliance with the standards of quality management systems such as GLP/ GMP and ISO.



Metrohm Quality Service

Metrohm Quality Service is available worldwide. Preventive maintenance carried out on a regular basis extends your instrument’s lifetime while providing for trouble-free operation. All maintenance work done under the label Metrohm Quality Service is carried out by our own certified service engineers. You can choose the service contract that suits you best.

With a full service contract, for example, you can rely on the optimum performance of your Metrohm instruments, incur no additional costs and benefit from complete and compliant verification documents. Thanks to our service you are perfectly prepared for audits.

An overview of Metrohm Quality Service

Our Services	Benefit for the Customer
<p>Metrohm Care Contracts that, depending on contract type, include preventive maintenance, instrument certification, on-site repair, free or discount on spare parts and consumables as well as guaranteed response times.</p>	<p>Cost certainty and cost savings, coverage of repair risk, quick response times and rapid problem solving, minimal downtimes, and ideal preparation for audits</p>
<p>Application support by our vast selection of Application Bulletins, Applications Notes, monographs, validation brochures, technical posters, and articles Personal consultation by our specialists per telephone or e-mail</p>	<p>Quick and professional solution to all arising application questions and complex analytical challenges</p>
<p>Training courses</p>	<p>Competent users contribute substantially to reliable results</p>
<p>Certified calibrations, for example, of dosing and exchange units</p>	<p>Accurate measurements Verification documentation for compliance with regulations and for efficient audits</p>
<p>Remote maintenance</p>	<p>Expeditious resolution of software questions</p>
<p>Back-up support</p>	<p>High data security</p>
<p>Emergency service, for example, express on-site repairs</p>	<p>Short response times and thus, rapid problem resolution Minimization of downtime</p>
<p>Original spare parts, made in Switzerland and available world wide Guaranteed spare parts available for at least 10 years beyond instrument discontinuation date</p>	<p>Lasting, successful repair; short delivery times Minimization of downtime Protection of your investment through long-term availability of spare parts and accessories</p>
<p>Decentralized repair workshops located around the world and a central workshop in Switzerland</p>	<p>Quality repairs done quickly, so your instruments are ready for use again</p>



Applications

40

In the previous pages of this brochure, only a small selection of available Metrohm applications from the power plant sector are described. If you do not find your specific application in the following list as well, please do not hesitate to contact your local Metrohm specialist.

Applications dealing with corrosion are available under: www.metrohm-autolab.com; all other applications can be downloaded under: www.metrohm.com/com/Applications

I. Process water

Corrosion

COR01	Corrosion Part 1 – Basic concepts
COR02	Corrosion Part 2 – Measurement of corrosion rates
COR03	Corrosion Part 3 – Measurement of polarization resistance
COR04	Corrosion Part 4 – Equivalent circuit models
COR05	Corrosion Part 5 – Corrosion inhibitors
COR06	Corrosion Part 6 – Critical pitting temperature measurements with pX1000
COR07	Corrosion Part 7 – Hydrogen permeation experiments with PGSTAT302F

Water analysis in power plants – general

AN-Q-005	Online analysis of trace anions in power plant water matrices
AN-Q-004	Online analysis of trace cations in power plant water matrices
AN-S-304	Variable Inline Preconcentration including Matrix Elimination for trace anion analysis (MiPCT-ME)
AN-S-250	Trace anions in tetramethylammonium hydroxide (TMAOH)
AN-S-032	ppt analysis (ng/L) in ion chromatography
8.000.6064EN	Microbore columns: a contribution to green chemistry

Cooling water

AN-S-218	Common anions in a closed cooling water system
AN-U-060	Corrosion inhibitors in cooling water
8.000.6065	Automated ion chromatographic determinations over six orders of magnitude
8.000.6063	Post-column chemistry for improved optical absorption detection
8.000.6005	Hyphenated techniques as modern detection systems in ion chromatography

Water treatment

AN-CIC-011	Analysis of an ion exchanger applying Metrohm Combustion IC
AN-H-012	Determination of ferrous ion content of heat exchanger wash solutions
AN-S-158	Five anions in the presence of 2 g/L nitrate in an ion exchanger eluate

Water-steam circuit

AN-C-139	Cations and amines in the water-steam cycle
AN-C-132	Traces of lithium, sodium, and ammonium in the presence of ethanolamine (Metrosep C 4 - 250/4.0)
AN-C-126	Methylamines and ethanolamines (Metrosep C 4 - 150/4.0)
AN-C-094	Methylamine, isopropylamine, diethylamine, and diethylethanolamine with preconcentration
AN-C-083	Online monitoring of trace levels of cations in boiler feed water
AN-C-078	Ethanolamines in the presence of alkali metal and alkaline earth cations
AN-C-076	Sodium and ammonium in 25% DEA (diethanolamine)
AN-C-052	Determination of cations and ethanolamines
AN-C-049	Trace cations in power plant feed water stabilized with 7 ppm monoethanolamine (MEA)

AN-N-060	Online monitoring of trace levels of silicate in boiler feed water
AN-Q-007	Online analysis of chloride and sulfate in supercritical water-steam cycles
AN-Q-003	Online monitoring of trace levels of anions in boiler feed water
AN-Q-002	Online monitoring of trace levels of cations in boiler feed water
AN-S-056	Traces of chloride in ultrapure water under clean room conditions
AN-S-157	Trace anions in boiler feed water containing 10 mg/L ammonia
AN-S-295	Anions in boiler water including sulfur speciation (sulfite and sulfate)
AN-V-179	Iron in boiler feed water for power plants (DHN method)
AN-U-059	Transition metals in feedwater using post-column reaction and subsequent UV/VIS detection
AN-U-061	Speciation of iron(II) and iron(III) applying post-column reaction and subsequent UV/VIS detection

Water-steam circuit in boiling water reactors

AN-C-137	Copper, nickel, zinc, and common cations in the water-steam cycle of a boiling water reactor (BWR)
AN-S-306	Trace anions including chromate in water-steam cycle of a boiling water reactor (BWR)

Primary circuit in pressurized water reactors

AB-044	Colorimetric determination of boron
AB-066	Potentiometric determination of boric acid
AB-266	Determination of titanium and uranium by voltammetry
AB-243	Determination of chromium at the «Ultra Trace» graphite electrode by cathodic stripping voltammetry
AB-083	Determination of sodium with the ion-selective electrode
AB-045	Colorimetric determination of silicic acid
AN-C-038	Five cations in 4% boric acid
AN-C-096	Trace analysis of monovalent cations in an ethanolamine matrix (secondary cycle of a pressurized water reactor) using Metrohm Inline Sample Preparation with an 800 Dosino for liquid handling
AN-C-140	Lithium in borated water of a pressurized water reactor (PWR)
AN-C-138	Zinc, nickel, calcium, and magnesium in borated water of a pressurized water reactor (PWR)
AN-Q-006	Online analysis of trace anions in borated water of a pressurized water reactor (PWR)
AN-S-242	Trace analysis of anions in the primary cycle of a nuclear power plant (PWR) using Metrohm Inline Sample Preparation
AN-S-066	Trace anions in boric acid after preconcentration
8.000.6071	Trace-level determination of anions in the primary circuit of a PWR-type nuclear power plant using ion chromatography after inline sample preparation
8.000.6072	Trace-level determination of cations in the secondary circuit of a PWR-type nuclear power plant using ion chromatography after inline sample preparation

Effluent

AN-N-065	Borate in borate effluent
AN-S-222	Anions in borate effluent

Flue gas

AN-T-027	Alkalinity of amine-containing gas washing solutions
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II. Turbine and lubricating oils

AB-209 Coulometric water determination according to Karl Fischer in insulating oils as well as in hydrocarbons and their derivatives

III. Fuels and operating materials

AN-K-010 Water in coal dust
AN-CIC-003 Chlorine, bromine, and sulfur in low-density polyethylene (ERM[®]-EC680k) applying Combustion IC
AN-CIC-004 Total and leachable concentration of halogens and sulfur in latex gloves using Combustion IC and a leach test
AN-CIC-005 Analysis of an ion exchanger applying Metrohm Combustion IC

Ordering information

pH value

2.826.0110	826 pH mobile with case and Primatrode
2.827.0X1X	827 pH lab with Primatrode or Unitrode
2.780.0010	780 pH Meter, including Unitrode pH electrode, rod, and electrode holder
2.140.0200	Printer Custom DP40-S4N for 780 pH Meter
2.867.0110	867 pH Module for pH and ion measuring with Touch Control including iUnitrode
2.801.0010	801 Stirrer for 780 pH Meter and 867 pH Module
6.0277.300	iAquatrode Plus with Pt 1000
6.0257.600	Aquatrode Plus with Pt 1000, plug-in head U
6.2104.600	Electrode cable, plug-in head U, plug F, 2 × B

Conductivity measurement

2.856.0120	856 Conductivity Module with Touch Control and stainless steel conductivity measuring cell $c = 0.1 \text{ cm}^{-1}$
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Titration (total hardness, chloride, boric acid)

2.905.0010	905 Titrand
2.800.0010	800 Dosino
2.801.0040	801Stirrer
6.0253.100	Aquatrode Plus
6.0257.600	Aquatrode Plus with Pt 1000, plug-in head U
6.0277.300	iAquatrode Plus with Pt 1000
6.0430.100	Ag-Titrode
6.0470.300	iAg-Titrode
6.0510.100	Combined polymer-membrane electrode for Ca^{2+}
6.2104.020	Electrode cable, Plug F
6.2104.600	Electrode cable, Plug-in head U, plug F, 2 × B

Water determination according to Karl Fischer

Coulometric KF titration

2.831.0010	831 KF Coulometer including generator electrode with diaphragm and 728 Stirrer
2.831.0110*	831 KF Coulometer including generator electrode without diaphragm
2.756.0010	756 KF Coulometer with built-in printer including generator electrode with diaphragm and 728 Stirrer
2.756.0110*	756 KF Coulometer with built-in printer including generator electrode without diaphragm
2.851.0010	851 Titrand including generator electrode with diaphragm 801 Stirrer
2.851.0110*	851 Titrand including generator electrode without diaphragm
2.852.0050	852 Titrand including generator electrode with diaphragm 801 Stirrer
2.852.0150*	852 Titrand including generator electrode without diaphragm
2.899.0010	899 Coulometer with built-in stirrer including generator electrode with diaphragm
2.899.0110	899 Coulometer with built-in stirrer including generator electrode without diaphragm

KF Oven

2.860.0010	860 KF Thermoprep
2.874.0010	874 Oven Sample Processor
2.885.0010	885 Compact Oven Sample Changer

* The stirrer has to be ordered separately.

Ion chromatography**Corrosion inhibitors**

2.850.1010	850 Professional IC Cation
2.887.0010	887 Professional UV/VIS Detector
2.858.0020	858 Professional Sample Processor – Pump
6.6059.242	MagIC Net™ 2.4 Professional

Transition metals

2.850.1010	850 Professional IC Cation
2.886.0110	886 Professional Reactor
2.887.0010	887 Professional UV/VIS Detector
2.858.0010	858 Professional Sample Processor
2.800.0010	800 Dosino
6.3032.150	Dosing Unit 5 mL
6.2841.100	Rinsing Station for IC Sample Processor
6.5330.040	IC Equipment for LH with 800 Dosino
6.1020.070	Metrosep A Supp 10 - 75/4.0
6.1020.500	Metrosep A Supp 10 Guard/4.0
6.1010.320	Metrosep C PCC 1 VHC/4.0
6.6059.242	MagIC Net™ 2.4 Professional

Amines and cations

2.850.1010	850 Professional IC Cation
2.850.9010	IC Conductivity Detector
2.858.0010	858 Professional Sample Processor
2.800.0010	800 Dosino, 2 ×
6.3032.150	Dosing Unit 5 mL, 2 ×
6.2841.100	Rinsing Station for IC Sample Processor
6.5330.040	IC Equipment for LH with 800 Dosino
6.5330.060	IC Equipment for matrix elimination with 800 Dosino
6.1014.200	Metrosep I Trap 1 - 100/4.0, 2 ×
6.1050.430	Metrosep C 4 - 250/4.0
6.1050.500	Metrosep C 4 Guard/4.0
6.1010.310	Metrosep C PCC 1 HC/4.0
6.6059.242	MagIC Net™ 2.4 Professional

Corrosive anions in the trace level

2.850.2030	850 Professional IC Anion – MCS
2.850.9010	IC Conductivity Detector
2.858.0010	858 Professional Sample Processor
2.800.0010	Dosino, 2 ×
6.3032.150	Dosing Unit 5 mL, 2 ×
6.2841.100	Rinsing Station for IC Sample Processor
6.5330.040	IC Equipment for LH with Dosino
6.5330.060	IC Equipment for matrix elimination with Dosino
6.1014.200	Metrosep I Trap 1 - 100/4.0, 2 ×
6.1006.520	Metrosep A Supp 5 - 150/4.0
6.1006.500	Metrosep A Supp 4/5 Guard/4.0
6.1006.310	Metrosep A PCC 1 HC/4.0
6.6059.242	MagIC Net™ 2.4 Professional

Chloride and sulfate in overcritical water-steam-circuits

2.850.2030	850 Professional IC Anion – MCS
2.850.9010	IC Conductivity Detector
2.872.0060	Extension Module Liquid Handling
2.800.0010	Dosino
6.3032.150	Dosing Unit 5 mL
6.5330.040	IC Equipment for LH with Dosino
6.1020.070	Metrosep A Supp 10 - 100/2.0
6.1020.500	Metrosep A Supp 10 Guard/2.0
6.1006.310	Metrosep A PCC 1 HC/4.0
6.6059.242	MagIC Net™ 2.4 Professional

Cations, copper, zinc, and nickel

2.850.1010	850 Professional IC Cation
2.850.9010	IC Conductivity Detector
2.858.0010	858 Professional Sample Processor
2.800.0010	Dosino, 2 ×
6.3032.150	Dosing Unit 5 mL, 2 ×
6.2841.100	Rinsing Station for IC Sample Processor
6.5330.040	IC Equipment for LH with 800 Dosino
6.5330.060	IC Equipment for matrix elimination with 800 Dosino
6.1014.200	Metrosep I Trap 1 - 100/4.0, 2 ×
6.1015.000	Metrosep C Trap 1 - 100/4.0
6.1050.230	Metrosep C 4 - 250/2.0
6.1050.610	Metrosep C 4 Guard/2.0
6.1010.310	Metrosep C PCC 1 HC/4.0
6.6059.242	MagIC Net™ 2.4 Professional

Lithium in the primary circuit**Nickel, zinc, calcium, and magnesium in the primary circuit**

2.850.1010	850 Professional IC Cation
2.850.9010	IC Conductivity Detector
2.858.0010	858 Professional Sample Processor
2.800.0010	Dosino, 2 ×
6.3032.150	Dosing Unit 5 mL, 2 ×
6.2841.100	Rinsing Station for IC Sample Processor
6.5330.040	IC Equipment for LH with Dosino
6.5330.060	IC Equipment for matrix elimination with Dosino
6.1014.200	Metrosep I Trap 1 - 100/4.0, 2 ×
6.1015.000	Metrosep C Trap 1 - 100/4.0
6.1050.230	Metrosep C 4 - 250/2.0
6.1050.610	Metrosep C 4 Guard/2.0
6.1010.310	Metrosep C PCC 1 HC/4.0
6.6059.242	MagIC Net™ 2.4 Professional

Corrosive anions in the primary circuit

2.850.2190	850 Professional IC Anion – MCS – Prep 3
2.850.9010	IC Conductivity Detector
2.858.0010	858 Professional Sample Processor
2.800.0010	Dosino, 2 ×
6.3032.150	Dosing Unit 5 mL, 2 ×
6.2841.100	Rinsing Station for IC Sample Processor
6.5330.040	IC Equipment for LH with Dosino
6.5330.060	IC Equipment for matrix elimination with Dosino
6.1014.200	Metrosep I Trap 1 - 100/4.0, 2 ×
6.1006.520	Metrosep A Supp 7 - 250/4.0
6.1006.500	Metrosep A Supp 4/5 Guard/4.0
6.1006.310	Metrosep A PCC 1 HC/4.0
6.6059.242	MagIC Net™ 2.4 Professional

Combustion IC

2.881.3030	Metrohm Combustion IC
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The package contains

2.881.0030	881 Compact IC pro – Anion – MCS
2.850.9010	IC Conductivity Detector
2.920.0010	Absorber Module
2.136.0700	Combustion Module
6.1006.310	Metrosep A PCC 1 HC/4.0
6.6059.241	MagIC Net™ 2.4 Compact

Optional

2.136.0800	Autosampler MMS 5000 (no sampler head/rack)
6.7302.000	MMS 5000 Kit for Solid Samples
6.7303.000	MMS 5000 Kit for Liquid Samples
6.1031.420	Metrosep A Supp 16 - 150/4.0
6.1031.500	Metrosep A Supp 16 Guard/4.0



Voltammetry

- 2.797.0010 797 VA Computrace for manual operation
- MVA-2 VA Computrace system with automatic standard addition consisting of 797 VA Computrace and two 800 Dosinos
- MVA-3 Fully automated VA Computrace system consisting of 797 VA Computrace with 863 Compact VA Autosampler and two 800 Dosinos for automatic addition of auxiliary solutions. Allows the automatic processing of up to 18 samples. This system is the optimum solution for automatic analysis of small sample series.



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