Sykam Chromatography System Ökoline

Ion Chromatography

SYKAM
CHROMATOGRAPHY

Sykam Ion Chromatograph S 135, Most Sensitive Determination of Ions Becomes Simple.

Ion Chromatography is one of the most important methods for the determination of alkaline, alkaline earth and transition metals, inorganic anions, sulfuric compounds of different oxidation levels, organic acids, and various tensides. Indeed, for aqueous solutions Ion Chromatography is one of the most sensitive methods for these substances, since in most cases the samples can be injected into the system without time consuming sample preparation. If required, e.g. for trace amounts, enrichment can be done within the chromatographic system.

The Sykam IC S135 is a compact system with modular setup. Even the basic system is designed for most sensitive anion analysis employing suppression of eluent conductivity.

The column oven integrated into the system holds measuring cell, precolumn, separation column, and suppression columns, and thus guarantees accurate retention times and quantification with superior reproducibility.

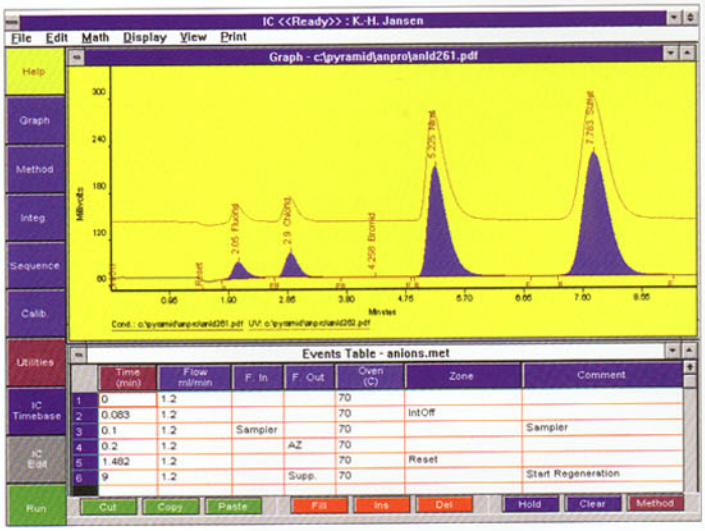
The conductivity detector's outstanding features are high background suppression, baseline stability, and signal linearity over a range of several decades. These characteristics become especially important when single column techniques are employed, e.g. for the determination of alkaline ions and alkaline earths.

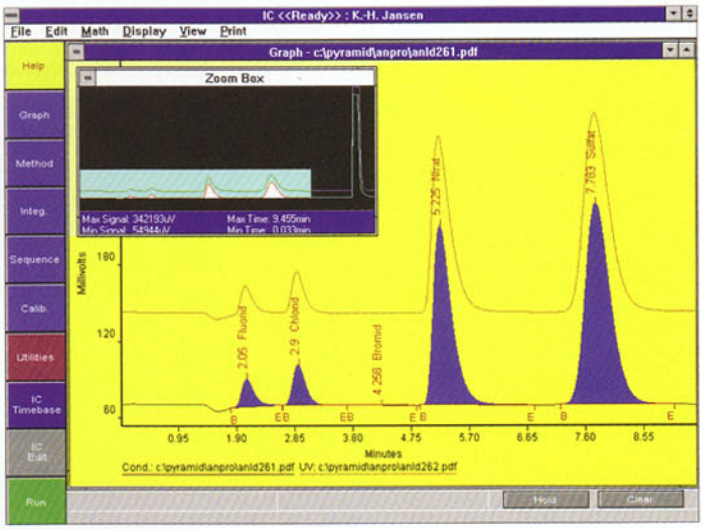
The chemically inert pump with its pump head made of PEEK meets all the requirements of Ion Chromatography. It has two serial pistons thus delivering with extremely low pulsation. This pump can even be used for HPLC, and by just adding a gradient module the system in fact can be easily upgraded for any HPLC application.

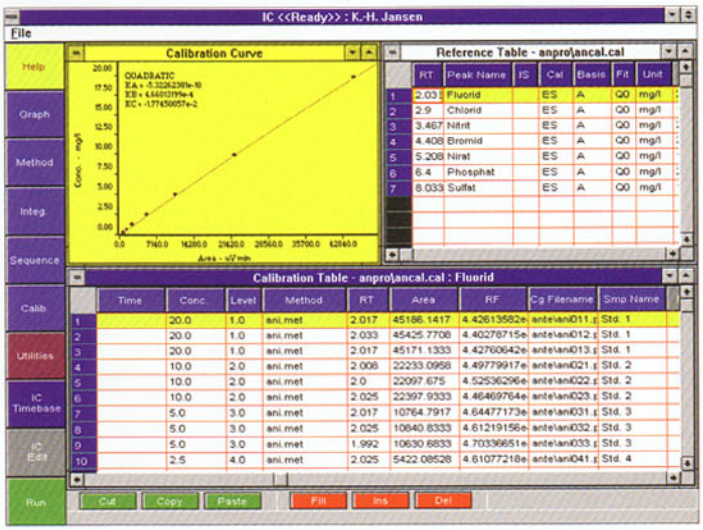
System control and data handling are performed by software running under Windows. Results can be output fully automatically with templates defined and created by the user. With automatic injection analysis of more than one hundred samples of drinking water can be done in one sequence.

Depending on applicational needs or analytical problems, the Sykam Ion Chromatograph can be easily modified by integrating additional Sykam system components. E.g. there are

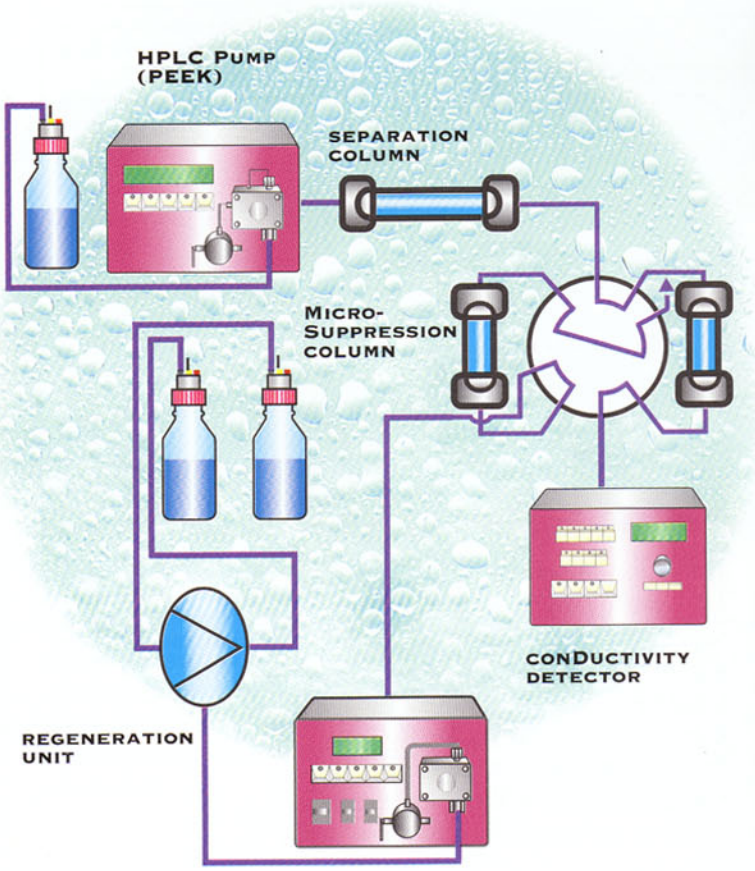
-switching valves to select columns or eluents for rapid method change.
- the UV/Vis detector with variable wavelength for high sensitivity determination of nitrite, organic acids, or transition metals after PAR post-column reaction.
- the electrochemical detector for detection of sulfide/cyanide or carbohydrates.
- automatic concentration units for trace analysis.







The Ion Chromatography module is based on the new micro column suppression unit.



The major advantages resulting from this technique are the increased sensitivity for anion determinations with improved sensitivity at low concentrations and the very short startup equilibration times needed. For each run a newly regenerated micro suppression column (resin volume 500 µl) is used, in less than one minute the background conductivity is reduced to a stable and low value (12 µS). Using two columns in an alternating mode allows automatic and continuous sequence of runs. Identical recovery rates and response times are essential for QS.

Compared to common membrane techniques the very short equilibration times and the superior mechanical stability during continuous operation are the outstanding features of the Sykam IC system.

Inorganic Anions.

Analysis of pure water or complicated matrices with high contamination: for any analytical problem the optimized method for correct results.

In most cases Ion Chromatography is used for the determination of anions in water samples. With the IC S 135 the anions are separated on an anion-exchange column, followed by a specific post-column (suppressor) reaction with subsequent conductivity detection. The suppressor reaction will replace all cations in the eluate by hydrogen ions. Selecting appropriate eluents (sodium carbonate, sodium hydroxide) leads to eluents with low conductivity and high sensitivity for the anion determination.

$$resin\text{-}H^+ + NaOH \rightarrow resin\text{-}Na^+ + \underline{HOH}\ \underline{Eluent}$$
$$resin\text{-}H^+ + NaCl \rightarrow resin\text{-}Na^+ + \underline{HCl}\ \underline{Sample}$$

The specific conductivity of the highly ionized acid formed during that reaction is about five to ten times higher than that of the respective salt. In addition, the suppressor reaction reduces the eluent conductance effectively. Sensitive determination of anions with stable baseline and without drifting effects becomes possible. In most countries this method has to be employed for drinking and waste water analysis as described in standard proceedings (DIN, ISO, EPA).

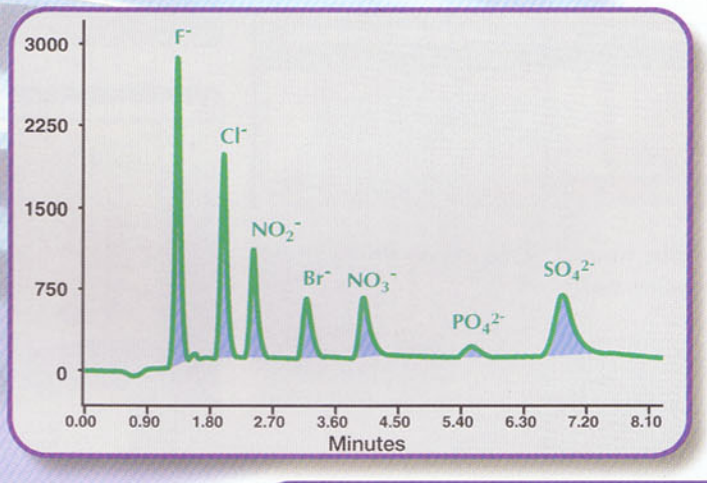
Moreover, the Sykam IC 135 allows alternative detection techniques like direct conductivity detection or indirect UV-detection. However, these methods are rarely used for anion analysis, due to lower sensitivity and prolonged equilibration times needed.

Sample Preparation.

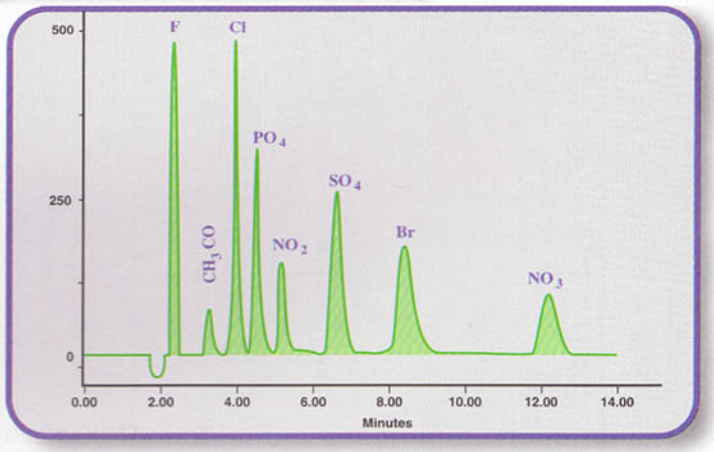
For most water samples membrane filtration is sufficient. Sample clean-up using C-18 cartridges are recommended for samples with large amounts of organic contaminations. A specific pre-column technique which is applicable for all ion chromatographic methods is used to prolong the live time of the Sykam IC columns. An unmodified macro porous polystyrene material selectively adsorbs humine substances, oils, and metal chelates, while ions pass to the separation column without interactions.

Anions in Water Samples.

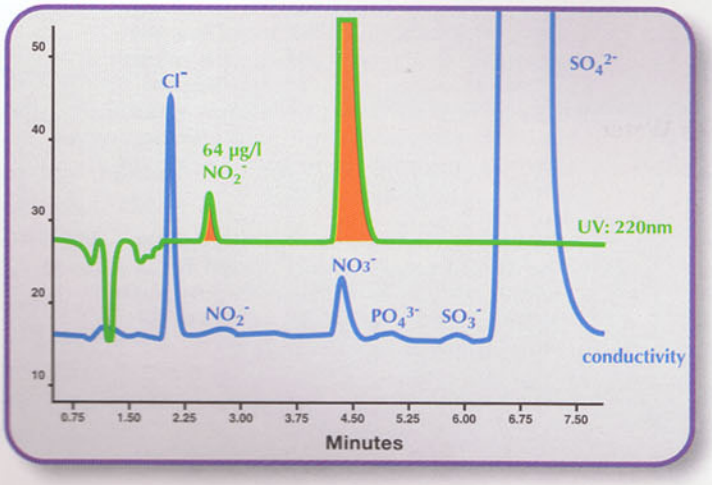
The standard procedure for anions is performed by the use of a sodium carbonate buffer solution and an anion-exchanger with additional multi-phase characteristics. This performs the elution of fluoride and acetate delayed from the water dip. The selectivity for anions can be varied via temperature and by adding organic solvents to the eluent. Due to the suppressor reaction the ions can be detected even at concentrations down to the lower µg/l range. With quadratic multi-level calibration coefficients of correlation for chloride and sulfate of 0.998 and 0.9997, respectively, could be achieved (0.2-100 mg/l).



SEPARATION OF INORGANIC ANIONS (COLUMN LCA A14) 1 MG/L EACH, WITH CONDUCTIVITY DETECTION FOLLOWING SUPPRESSOR REACTION.



SIMULTANEOUS DETECTION OF CONDUCTIVITY AND UV ABSORPTION FOR TRACE ANALYSIS OF NITRITE.



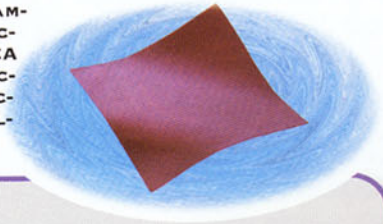
Detection of Nitrite

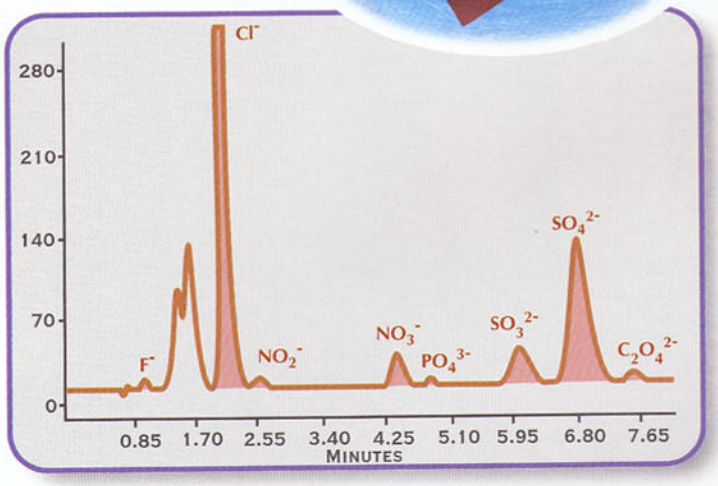
Low nitrite concentrations in the presence of a large amount of chloride, e.g. in waste water, may not be detectable by conductivity detection alone, due to overlapping peaks. By using the UV absorption at 220 mm, nitrite can be detected without interference down to concentrations of a few µg/l.

ANION ANALYSIS WITH HIGH AMOUNTS OF NITRATE. COLUMN LCA A04 WITH SPECIFIC SELECTIVITY.

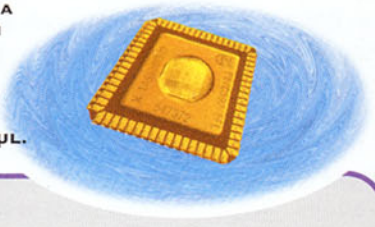
Applications for Anion Analysis: Examples.

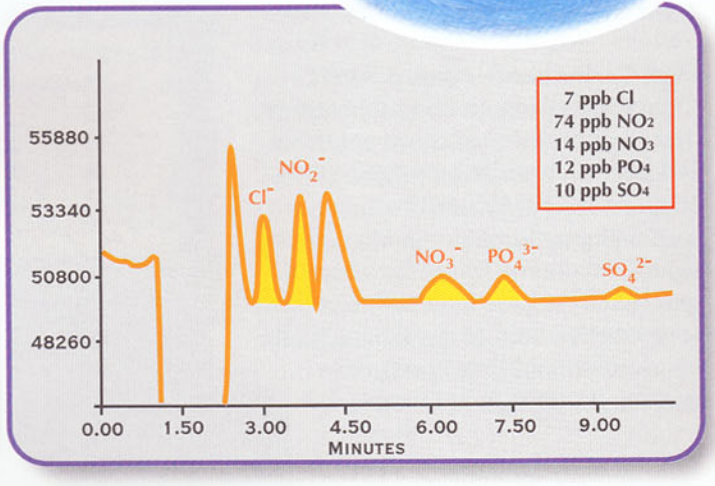
ANALYSIS OF WASTE WATER SAMPLE FROM A LEATHER MANUFACTORY. SEPARATION COLUMN LCA A14 WITH CONDUCTIVITY DETECTION AFTER SUPPRESSION REACTION. SAMPLE DILUTED AND FILTERED.



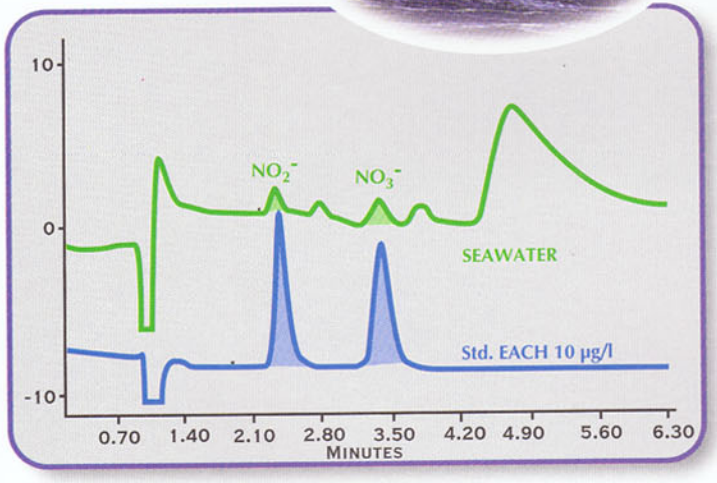


TRACE ANALYSIS OF ANIONS IN A H_2O_2 CLEAN-UP SOLUTION FROM THE ELECTRONIC INDUSTRY. SEPARATION COLUMN LCA A14 WITH CONDUCTIVITY DETECTION FOLLOWING SUPPRESSOR REACTION. DIRECT INJECTION OF 50 µL.

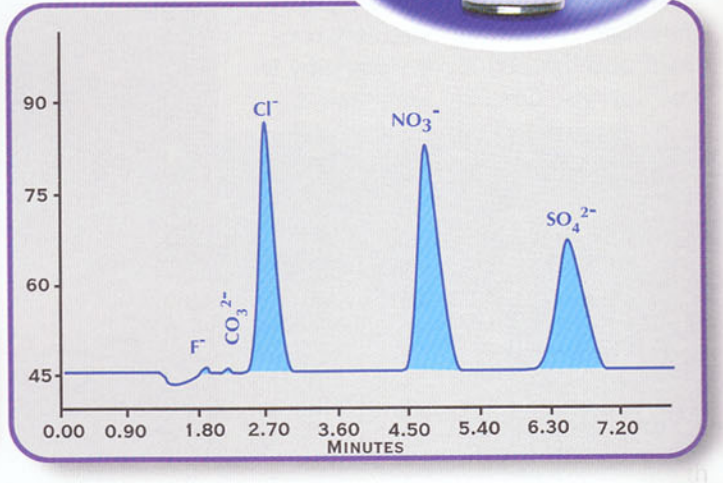




TRACE ANALYSIS (APPROX.1 µG/L) OF NITRITE AND NITRATE IN SEA WATER. SEPARATION COLUMN LCA A03, ELUENT NaCl, DIRECT UV DETECTION (220 NM).



DETERMINATION OF ANIONS IN DRINKING WATER. SEPARATION COLUMN LCA A14 WITH CONDUCTIVITY DETECTION FOLLOWING SUPPRESSOR REACTION.



Selectivity of Columns.

Several matrix interferences (e.g. high nitric acid concentration) cannot be eliminated by sample preparation or specific detection. In that case an anion-exchanger with different selectivity (e.g. LCA A04 with macro porous polystyrene material) may be employed.

Trace Elements in Sea Water.

Using non-absorbing sodium chloride as eluting solution with a stable polymeric column material (LCA A03) UV detection can be used to determine nitrite, nitrate (220 nm), bromide (212 nm) and iodine (230 nm) at levels in the low µg/l range even in the presence of about 30 g/l of chloride.

Detection Limits.

As long as concentrations of anions are all in a similar low range lower detection limits can be reached by concentrating the samples. For very dilute water samples, however, it may be appropriate, to analyze them with on-line procedures at their original location, since sample handling and transport may lead to false results by unwanted contamination.

SYKAM IC S 135, Investment for Today and Tomorrow.

The basic IC S 135 can be upgraded by a number of different Sykam Chromatography components to a system covering the entire range of ion chromatographic applications.

Optimized IC Technique.
System control and programming can be done either directly at each separate unit or via the data system. Even complex instrumentation can be operated without specific training.

Basic System IC S 135

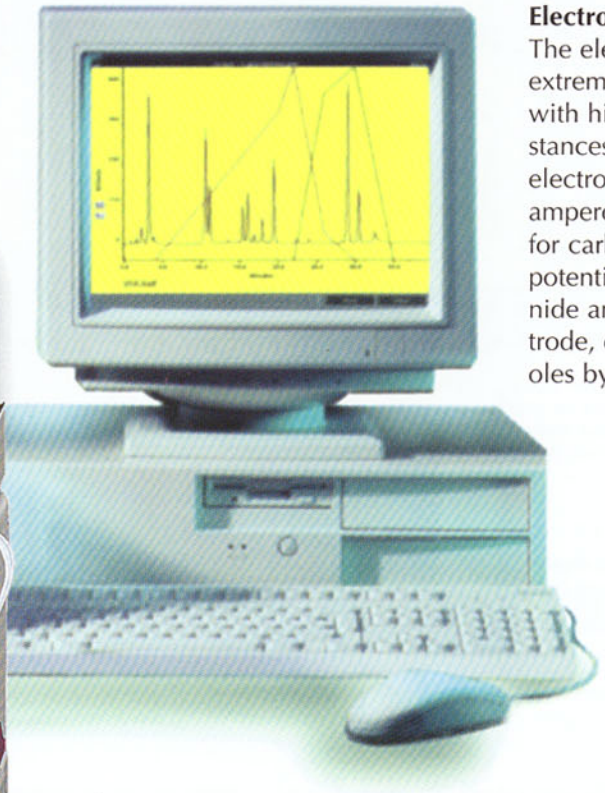
High Performance Pump S 1121.
Dual piston pump with low pulsation. The pump head is made of chemically inert Polyether-Ether-Ketone (PEEK). Programming is done via a step-by-step dialogue through the alphanumeric display. Ramp functions allow gentle start/stop of flow rate to prolong the live time of the ion-exchange resins. Optionally, the pump can be upgraded for gradient elution.

Conductivity Detector S 3111.
With its microprocessor controlled signal processing, combined with a newly developed cell design, the conductivity detector S 3111 is one of the most sensitive instruments available for the determination of ions by Ion Chromatography. The temperature of the micro cell is kept extremely constant, and this cell is convincing by its fast response time and long time stability, no matter if run in combination with suppression reaction or in direct mode with electronic suppression of the background conductivity. The dynamic ranges (0,3 µS to 10 mS) allow to determine extremely high and low concentrations in one single analytical run.

IC Module S 4260 A/B.
These two modules hold all components to perform Ion Chromatography, with the shortest flow connections possible. Inert injection valve, column oven for extremely constant control of four selectable temperatures, micro conductivity cell, micro suppressor columns with electrically actuated selection valve, inert regeneration pump, selection valve for regeneration solutions, reagent organizer, and controlling unit with panel for programming regeneration cycles. All fluid connections in the column oven are easily accessible, so column or method changes can be done without difficulty.

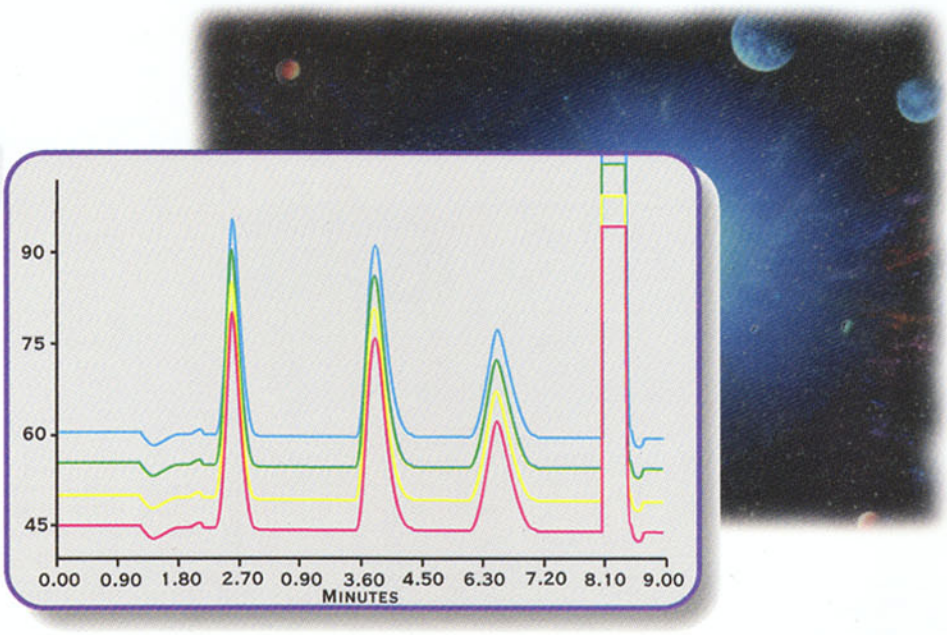
Autosampler S 5200.
Short analysis times are very advantageous for routine analysis, especially when sample throughput can be increased by automatic sample injection. The Sykam Autosampler S 5200 can handle up to 120 samples for single or multiple injections. Sample sequence is variable due to programmable x-y-z-access to the vials. A priority sample feature is implemented for maximum versatility. Automatic dilution and variable injection mode as optional features help to reduce valuable sample preparation time.

Sykam Data System.
Configurable for ion chromatographic applications, this system is capable to control the entire Ion Chromatograph in the Windows environment. Automatic integration, calibration with calibration curve analysis, freely configurable documentation of the results in reports with or without sample chromatograms and method parameters, as well as spread sheet documentation of an entire sequence's results, all these are features of this most flexible data system. Not to mention features like manual chromatogram editing, data exchange with other programs, and system security and system suitability functions.



Electrochemical Detector.
The electrochemical detector is an extremely sensitive detection system with high specifity to different substances, depending on the working electrode used. For example: pulsed amperometry using a gold electrode for carbohydrate determination, potential-free measurement of cyanide and sulfide with a silver electrode, oxidation of amines or phenoles by a glassy carbon electrode, or reduction of peroxides employing a platinum working electrode.

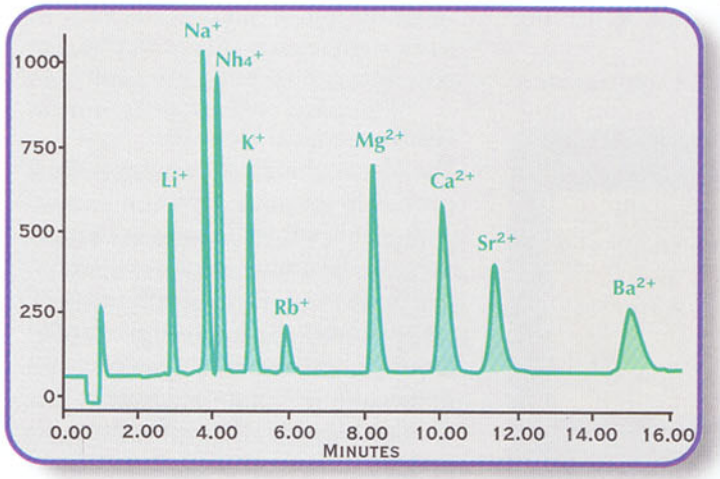
Photometer S 3205.
Due to variable wavelength programming from 195 nm to 800 nm and its single or dual wavelength capability the UV/Vis photometer S 3205 ideally matches the requirements in Ion Chromatography to detect separated compounds directly or following derivatization after separation.



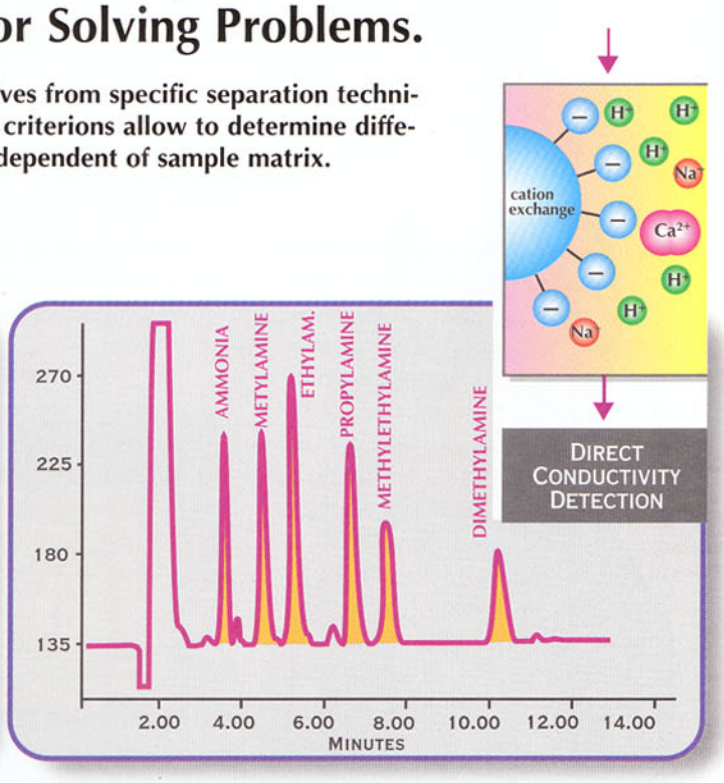
INJECTION AFTER INJECTION: STABLE BASELINE AND REPRODUCIBLE RESULTS DUE TO THE NEW SYKAM MICRO SUPPRESSION COLUMN TECHNIQUE.

Sykam IC Columns and Detection Techniques: Universal in Use, Specific Tools for Solving Problems.

The difference between Ion Chromatography and HPLC derives from specific separation techniques, i.e. columns and appropriate detection systems. These criterions allow to determine different classes of compounds with high sensitivity, relatively independent of sample matrix.



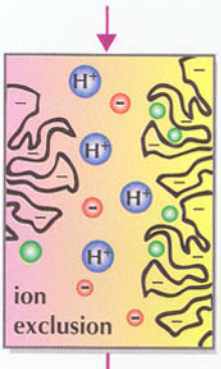
DETERMINATION OF ALKALI METALS AND ALKALINE EARTHS IN ONE ANALYTICAL RUN, USING THE CATION SEPARATION COLUMN LCA SS AND EMPLOYING DIRECT CONDUCTIVITY.

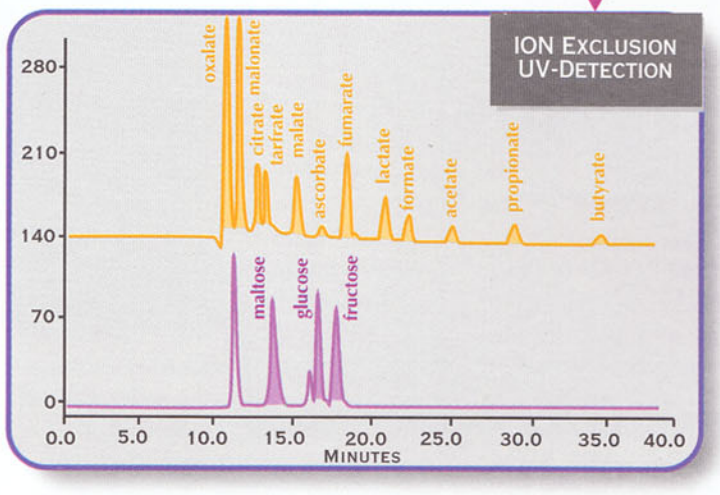


SEPARATION OF DIFFERENT AMINES (LCA SS) WITH DIRECT CONDUCTIVITY DETECTION.

Alkali Metals/Alkaline Earths.

The affinity of divalent cations to a cation-exchanger is by far higher than that of monovalent cations. Therefore retention times of alkaline and alkaline earth metals are very different. Using the Sykam cation separation column LCA SS in combination with organic acids (dipicolinic acid/citric acid) all parameters can be determined in one analytical run. For this application conductivity detection without suppression reaction is recommended, as the specific conductivity of hydrogen ions in the eluent is by far higher than the specific conductivity of the analytes, resulting in large signals when these analytes pass the flow cell. This method is ideally used for routine water analysis and is also suitable for amines.



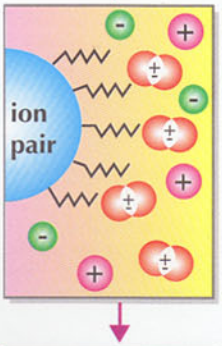


PRALLEL ANALYSIS OF ORGANIC ACIDS AND CARBOHYDRATES IN JUICES BY ION EXCLUSION CHROMATOGRAPHY (LCA E01) AND COMBINED UV AND LIGHT SCATTERING DETECTION.

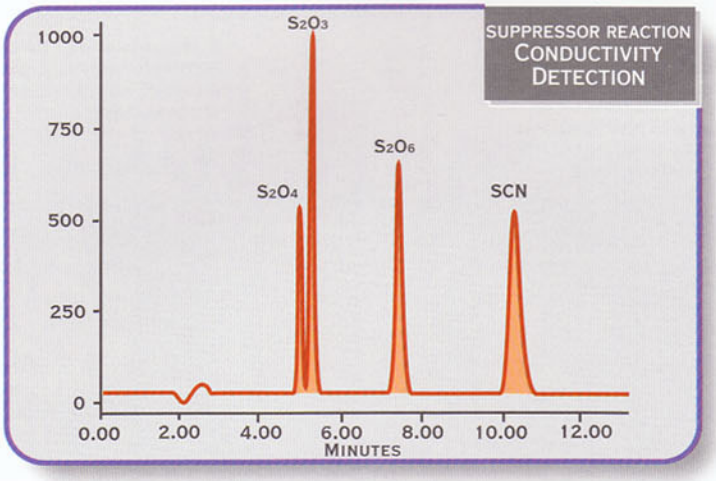
Organic Acids.

Ion exclusion chromatography with the separation column LCA E01 offers the most suitable separation technique for organic acids. The underlying principle makes use of the fact, that in the presence of an acidic eluent as sulfuric acid different organic acids will diffuse into the pores of a cation-exchange particle at different velocities. The acids will hereby elute depending on their respective pK values. Higher concentration of acidic eluent will prolong retention. Measuring UV absorption at 210 nm analyte concentrations of appr. 100 µM can be easily detected. Acetate and formic acid at lower µmolar concentrations can also be detected with direct conductivity detection. The combination of sequential UV and light scattering detection is applicable e.g. for acids and carbohydrates in fruit juices.

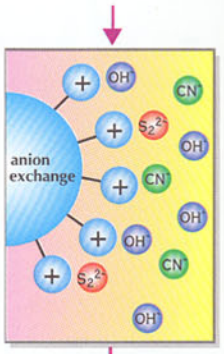
Sulfur at Different Oxidation Levels. Large and strong ions as thiosulfate, thionate, sulfonic acids, and polyphosphates are eluted from an anion-exchanger very late, if they are eluted at all. In this case ion pair chromatography is the method of choice. In the presence of tetrabutyl ammoniumhydroxide (TBAH) neutral ion pairs are formed, which can be separated on pH stable reversed phase material (Sykam LCA N01) due to hydrophobic interactions. With conductivity detection following a suppressor reaction determination is possible even at µg/l levels. When hexane sulfonic acid is used instead od TBAH quaternary amines and similar compounds can be analyzed by this method.



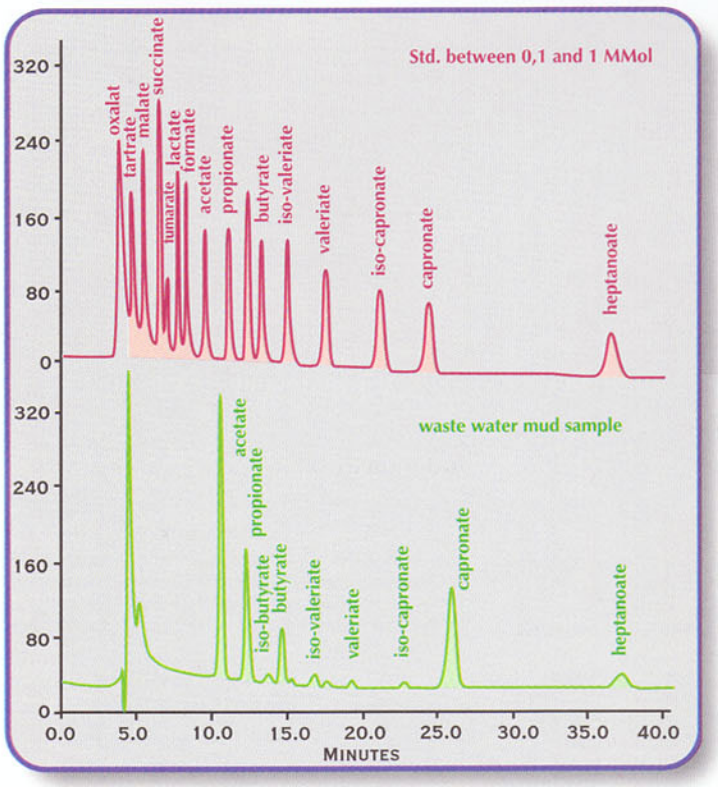
Sulfide and Cyanide. Detection of sulfide and cyanide is only possible at very high pH with electrochemical detection using a silver electrode. This specific procedure is not sensitive to other ions and thus allows to measure quantities below 10 µg/l. The Sykam separation column LCA A12 is packed with an anion-exchanger with high capacity, so that samples can be injected directly after sample preparation without loss of resolution even in the presence of 1 M NaOH.

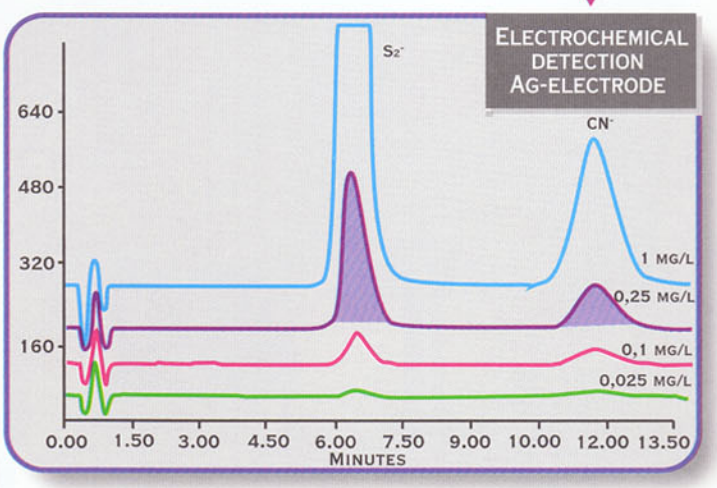


ION EXCLUSION CHROMATOGRAPHY (LCA E01) OF ORGANIC ACIDS WITH UV DETECTION AT 210 NM.



SEPARATION OF SULFUR COMPOUNDS BY ION PAIR CHROMATOGRAPHY (LCA N01), CONDUCTIVITY DETECTION FOLLOWING SUPPRESSION.

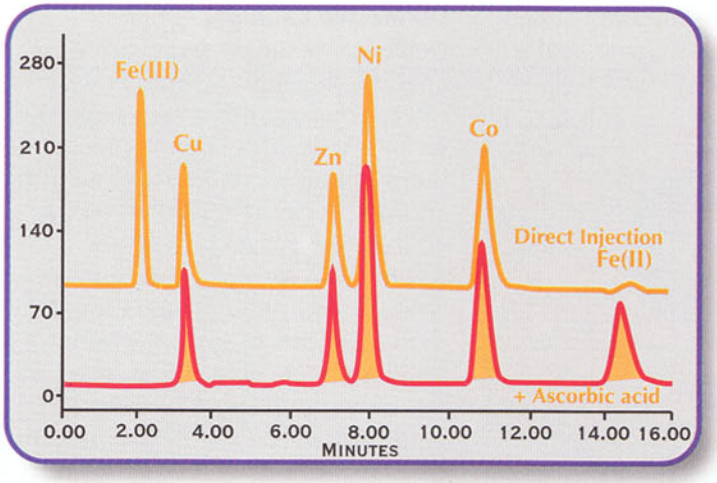




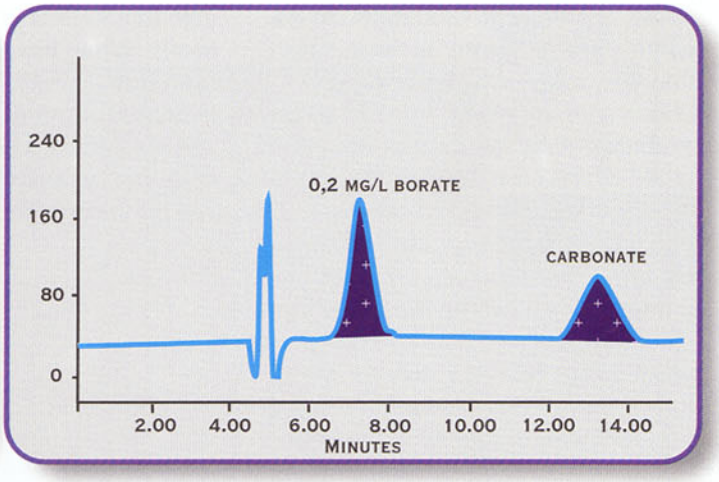
DETERMINATION OF SULFIDE AND CYANIDE USING ION-EXCHANGER (LCA A12) AND ELECTROCHEMICAL DETECTION.

Post-column Derivatization, One Further Step in the Direction of Specifity and Sensitivity.

During post-column derivatization separated compounds eluting from a chromatographic column react with a reagent to reaction products, that can be detected more sensitively. Sykam offers a wide variety of reactors, derivatization pumps and gradient modules, to upgrade the basic Ion Chromatograph for that purpose.



DETERMINATION OF TRANSITION METALS BY PAR POST-COLUMN DERIVATIZATION. BY ADDING ASCORBIC ACID, FE(III) CAN BE REDUCED TO FE(II).



DETERMINATION OF BORIC ACID. CONDUCIVITY DETECTION AFTER ION EXCLUSION CHROMATOGRAPHY AND POST-COLUMN DERIVATIZATION WITH MANNITE.

Transition Metals.

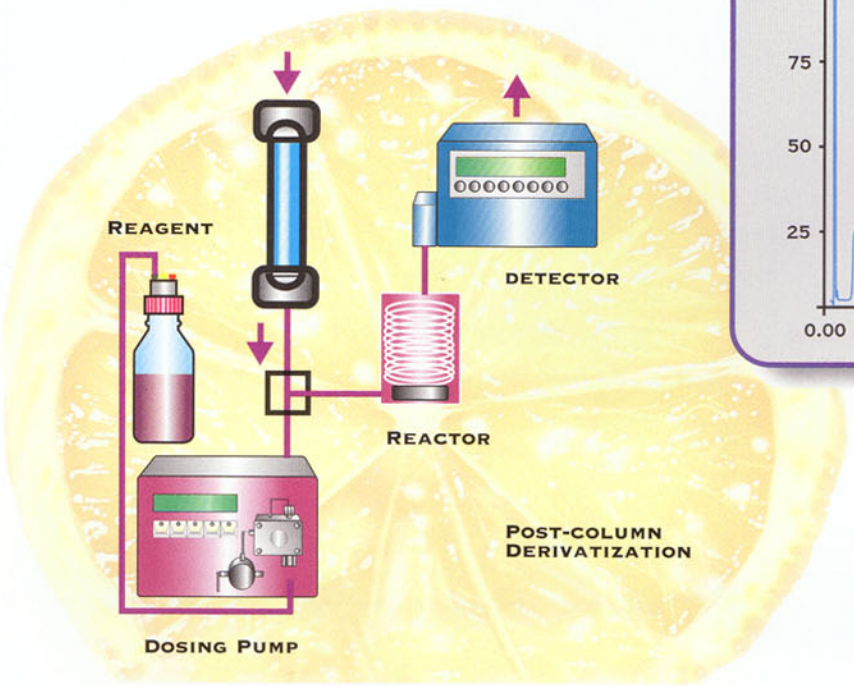
With a complexing eluent (tartaric acid) transition metals, lanthanides, and actinides can be separated on a strongly acidic cation-exchange resin, partially even according to their different valences. Photometric detection at 520 nm is performed after reaction with pyridylazoresorcinol (PAR). This method can be used not only for analytical purposes, but also for preparative clean-up of radio nuclides before radio activity monitoring.

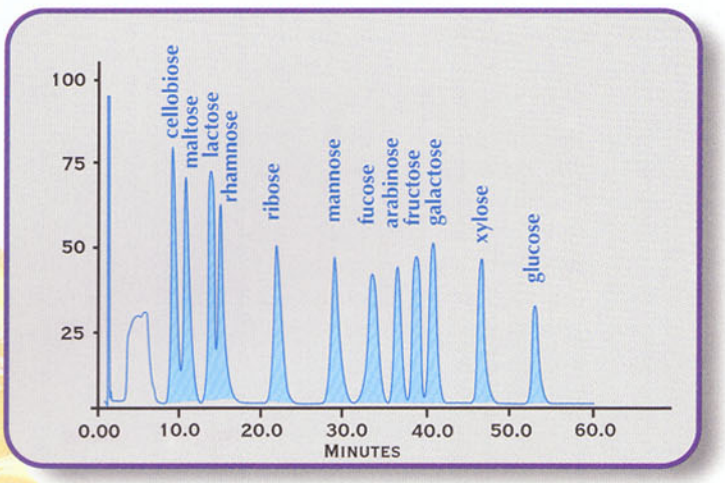
Boric Acid.

Following separation by ion exclusion (LCA E01) a mannite solution is continuously added to the eluate. Boric acid reacts with the carbohydrate to form an ionic complex detectable by conductivity detection.

Carbohydrates.

For the determination of carbohydrates as cuprous bicinchoninate complexes a high temperature reactor is needed, since reaction occurs optimally at 120 °C. The complexes are detected at 500 nm. The separation is performed by using a boric acid gradient program.

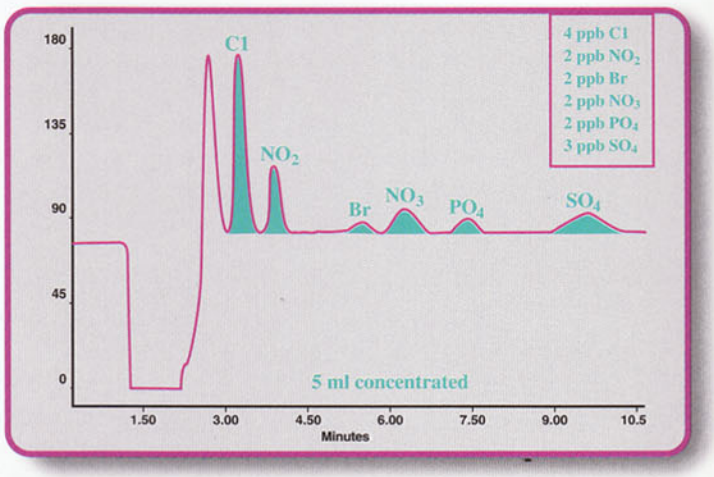




ION-EXCHANGE SEPARATION OF CARBOHYDRATES BY BORIC ACID GRADIENT. HIGH TEMPERATURE POST-COLUMN DERIVATIZATION WITH CUPROUS BICINCHONINATE, DETECTION AT 500 NM.

Valve Switching Techniques for Concentration, Fractionation, and Column Switching.

For automation of more complex analytical procedures Sykam offers various inert motor valves, ready for control by the data system.
Automatic online sample concentration for pure water samples from different sources can be performed as well as automatic eluent selection or column switching.



CONCENTRATION OF 5 ML ULTRA PURE WATER ON A CONCENTRATOR COLUMN WITH THE FOLLOWING SEPARATION (LCA A14) AND CONDUCTIVITY DETECTION AFTER SUPPRESSOR REACTION.

SAMPLE SITES.
To draw samples from varying sites for on-line detection and/or to select different standard solutions motor valves with six or ten positions are available.

Injection valve for standard addition.

ELUENT CHANGE.
Different eluents can be selected by a 6 positions valve (either manually or by motor actuation) to flush the column or to change the analytical method.

CONCENTRATION.
Using a small concentrator column connected to a 6 way/2 positions motor valve dilute samples can automatically be concentrated by a dosing pump and eluted into the analytical system.

COLUMN CHANGE.
By a 6 positions motor valve a flush channel and up to five different columns located in the column oven can be selected. The column outlets are connected to one or several detectors. Since the columns remain equilibrated with their respective eluent, fast method change is feasible.

Ordering Information

Catalog No.	Description
11 11 002	**IC Basic System S 135 for high sensitivity with chemically and electronic background compression** with HPLC pump S 1121, conductivity detector S 3111, IC module S 4260A/B, manual injection valve and reagent organizer
	IC Components:
10 10 047	**HPLC Pump S 1121, PEEK, for constant eluent dosage** (delivery rate: 0.1 - 9.95 ml/min,; pressure rate: up to 40 MPa).
10 10 053	**HPLC Pump S 1021, PEEK, for concentration** (delivery rate: 0.5 - 8.00 ml/min.; pressure rate: up to 40 MPa).
10 10 055	**HPLC Pump S 1021, PEEK, for derivatization** (delivery rate: 0.01 - 3.00 ml/min.; pressure rate: up to 30 MPa).
10 65 004	**Ion Chormatography Module S 4260A/B for reciprocal automatic selection of suppression columns with anion determination.** With integrated PEEK plunger dosage pump, column oven and reagent organizer.
10 30 003	**Conductivity Detector S 3111** Measuring range: 0.3 – 1000 µS, with temperature stabilized micro flow cell.
10 90 039	**UV/Vis Detector Model S 3205** for single and dual wavelength operation in dialog. With deuterium and tungsten lamps for a wavelength range from 190 to 800 nm.
10 90 076	**UV/Vis Detector Model S 3200** for single wavelength operation. With deuterium lamp for a wavelength range of 190 - 400 nm, optionally with a tungsten lamp (up to 800 nm).
10 90 064	**Elektrochemical Detector S 3510** for DC-Amperometry with additionally pulsed amperometric detection. Measuring range: 0.04 - 20000 nA full scale range. With glassy carbon working electrode. Optionally with gold, silver and platinum electrode.
	Upgradable to ternary gradients:
10 80 012	**Low Pressure Gradient Module S 8111** with built-in interface board for the data system
	Low Pressure Gradient Module S 8111 in combination with Controller S 2001.
10 70 008	**Reagent Organizer S 7121** with gas distributor and pressure regulator 0 - 7 bar and 3 solvent bottles, 1000 ml, with 4 in the cap integrated valves.
10 71 002	**Sykam Degasser** with digital display and analog output, for 3 solvent channels, suitable for all gradient mixing.
10 61 004	**Automatic Eluent Change** 6 positions switching valve 5012 with electric-driven valve actuator S 6361
10 61 004	**Automatic Selection of 6 sample channels** 6 positions switching valve 5012 with electric-driven valve actuator S 6361
10 61 009	**Automatic Selection of 10 sample channels** 10 positions switching valve 7610 with electric-driven valve actuator S 6361
10 61 010	**Automatic Column Change** 6 positions switching valve S 9060 with electric-driven valve actuator S 6361 for the selection of up to 5 separation columns and one flushing channel in combination with a 7 way distributor (PEEK).
10 61 011	**Automatic Switching of concentrator column** 6 way/2 positions valve 9010 with electric-driven valve actuator S 6351 for the selection/elution of concentrator columns.

Catalog No.	Description
	Data System:
900 - 100	**Pyramid Chromatography Manager P1** with 2-signal input integration software
900 - 501	**HPLC Gradient Control Module** for the control of 1 low and/or high-pressure gradient system
900 - 200	**Pyramid Chromatography Manager P2** with 4-signal input integration software
900 - 502	**HPLC Gradient Control Module** for the control of 2 low and/or high-pressures gradient systems
	Minimum Requirements for the PC with Pyramid Manager P1 and P2: MS-DOS compatible PC with at least 486 processor, min. 16 MB RAM, VGA graphics, MS DOS 5.0, Windows 3.1, MS Excel 5.0
	Separation Columns:
51 11 003	**Anion Separation Column LCA A03** for direct conductivity detection without suppression technique and direct UV measurement.
51 11 015	**Concentration Column** for the separation column LCA A03
51 11 005	**Anion Separation Column LCA A04** for anion determination with high nitrate concentrated samples.
51 11 016	**Concentration column** for the separation column LCA A04
51 11 021	**Anion Separation Column LCA A12** for the determination of sulfide and cyanide.
51 11 017	**Concentration Column** for the separation column LCA A12
51 11 014	**Anion Separation Column LCA A14** for standard anion determination with suppression technique.
51 11 018	**Concentration Column** for the separation column LCA A14
51 12 003	**Cation Separation Column LCA K01** for alkaline determination.
51 11 019	**Concentration Column** for the separation column LCA K01.
51 18 002	**Cation Separation Column LCA K02** for the determination of transition metals
51 11 020	**Concentration Column** for the separation column LCA K02.
51 18 003	**Cation Separation Column LCA SS** for the determination of alkaline and alkaline earths.
51 22 009	**Precolumn cartridge for the separation column LCA SS** in combination with pre-column holder
51 13 001	**Ion Pair Separation Column LCA N01** for the determination of sulfur at different oxidation levels
33 03 078	**Ion exclusion Separation Column LCA E01** for the determination of organic acids
33 00 546	**Pre-column for separation column LCA E01**
53 20 003	**Pre-column kit,** with 5 gr pre-column material, empty cartriges and pre-column holder
52 23 001	**Precolumn material, 5 gr;** neutral organic material
54 03 008	**Precolumn holder** for precolumn cartridges
54 02 004	**Precolumn cartridges, empty, 20 mm with** sieves and sealing rings
51 20 006	**Precolumn cartridges, packed with neutral packing material**

Sykam GmbH
Systeme + Komponenten analytischer Messtechnik
P.O. Box 1448
D- 82199 Gilching
Talhofstraße 32 a
Tel.: 0 8105/80 03
Fax: 0 8105/2 32 62