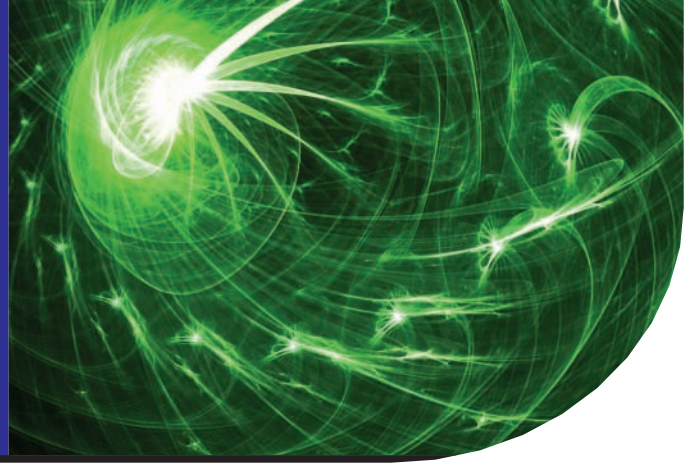

Professional Ion Chromatography used to Assay the Anion Profile of Cement

The 850 Professional Ion Chromatography (IC) range from Metrohm can be used to determine any conductive anion and cation in a variety of different everyday products. Concrete has many uses in life and IC is a technique that enables the concentration of fluoride, chloride, sulfate and phosphate of the materials being fed into the kiln (kiln feed, coal, alternative fuels etc) to be determined quickly and accurately before they are used to prevent further problems arising with the cement quality down the line.

> ***"The MagIC Net software is the next generation of chromatographic software from Metrohm"***

Author Details:

Jonathan Bruce,
Applications Manager,
Metrohm UK Ltd.
Tel: 01280 824824
Email: jbruce@metrohm.co.uk

The characteristic of cement is that after it has been mixed with water it will set hard as rock and will bind any rock or mineral fragments mixed with it. Mortar is made from a mixture of sand and cement, and bonds together bricks in a wall. Most cement is mixed with both sand and aggregate to make concrete.

The quality of the finished product cement is determined by the raw materials that are used to make cement's chemistry, and the process by which the chemistry is created in the kiln. Fluoride negatively impacts on cement quality; it affects the cement's strength and also the rate at which it sets, both of which are crucial factors.

Chlorine and sulfate are minor oxides in cement, coming from the raw materials used to make cement (limestone and shale) and the fuels that are used to power the kiln (such as, car tyres or coal). These compounds negatively affect operation of the kiln and must therefore be tightly controlled. Chloride and sulfate are volatilised in the high temperatures of the kiln, and then blown by hot gases to the entrance of the kiln, where it is cooler, before they condense back into their original material. The process occurs in cycles and leads to an accumulation of chlorine and sulfate that can cause great problems with kiln stability.

Phosphate is a minor oxide that occurs in the raw materials used in the cement manufacturing process. Phosphate has a negative effect on cement resistance if it represents greater than 0.5% total of the cement.

The 850 Professional IC and 858 Sample Processor are the result of more than 20 years of creative solutions through Research and Development to the field of IC and represent the third generation of IC instruments to come from Metrohm.

Intelligence comes as standard with the Professional IC systems and features hardware specifications not available anywhere else in the marketplace:

- **iPump** with 15% less pulsation compared to previous designs that ultimately yields lower detection limits.
- **iDetector** with a single range that no longer requires the correct selection of scale or the use of auto-ranging. The latest Digital Signal Processing and outstanding temperature stability guarantee the highest precision. Example chromatograms are stored in the detector for validation or training purposes.
- **iColumn** the only IC columns available today equipped with intelligence that automatically knows their optimum working conditions and possess full traceability with regard to number of injections performed, operating pressure and flow rates.
- **iDosing** with Dosino™ technology for very accurate liquid dispensing of volumes as low as 1µl. The iDosing technology is utilised for pre-concentration when working at trace levels or can be used for automatically inline sample dilution in combination with the intelligent MagIC Net software.
- **MagIC Net** software with smart system control and monitoring of all results. Trending of any chromatographic parameter from a method is only a mouse click away so visually all the necessary information can be shown graphically over time.

The Professional IC (ProfIC) systems comprise 11 different base modules but due to the modularity and expandability of the design any instrument can be converted into a different model, for example, a single chemistry can be converted to a dual chemistry if the analytical requirements change over time.

Size is ever increasingly an issue in most laboratories and the 850 Professional IC and 858 Sample Processor occupy a footprint of only 64cm width on the bench.

MagIC Net

The MagIC Net software is the next generation of chromatographic software from Metrohm. The software has been completely developed in-house and has a similar view on first appearance to the outstanding Tiamo software so existing titration users will have an instant empathy with MagIC Net. The prime consideration during development of MagIC Net was to develop an intuitive software platform that was easily used in the event of novice operation but for the skilled user represents a powerful data acquisition and processing package with all the information readily available.

All system parameters are monitored and optimised through the MagIC Net software with its smart control. Control cards for calibration and samples can be freely defined and should an important parameter leave a predefined tolerance than the laboratory user is informed by either email or SMS text message. Particular attention has been give to the long service interval of the Professional IC instruments and these can all be observed through the MagIC Net software along with the expiration date of all solutions, eluents and consumable hardware parts.

The results are safely stored in a database that fully complies with the analytical requirements of the FDA and the data can easily be exported into a LIMS systems or a package like Microsoft Excel®.

MagIC Net™ is a client server based data system that provides configurable user interfaces with the results stored in a single central database where they can be viewed and reprocessed by all client PC's. Network users can access the search and filter tools, control instruments, individually customise reports and execute sample tables through a fewclicks of the mouse. The system administrator decides upon the new users and assigns individual access rights to both data and methods.

The determination of the anions fluoride, chloride, sulfate and phosphate in cement is a relatively simple process. A few

hundred milligrams of dry product are added to a little ultra pure water and then a few hundred microlitres of concentrated acid (for example nitric) are added to extract the solid cement ions into solution.

Once cessation of carbon dioxide has occurred then the cement solution is diluted with ultra pure water and the pH elevated with a few microlitres of sodium hydroxide to neutralise the solution. Typical dilution would be in the region of 1:1000 and the diluted solution is injected directly into the Ion Chromatograph via the use of a sample processor. The results for all four anions are obtained in less than 25 minutes and the separation performed using the temperature selective A SUPP 15 column from Metrohm.

Depending upon the composition of the cement then the diluted cement solution may contain solid pieces of aggregate not dissolved by the acid. These particles could have a detrimental on the lifetime of the separation column (usually the main consumable item) should they get onto the column so it is recommended to remove any such particles prior to injection.

These can be removed using filtration through filter paper or the use of syringe filters prior to sample loading. Both of these are time consuming procedures that increase the sample preparation time and in the case of syringe filters they can add considerable running costs to the analysis.

Metrohm has a number of Inline Sample Preparation Modules (MISP) that can be used to automate difficult sample matrices. The use of inline ultra-filtration considerably increases the sample throughput by automatic filtration of each sample using the unique Metrohm ultra-filtration cell. Inline preparation saves the user a lengthy manual sample pre-treatment step and at the same time minimises the operational costs.

The ultra-filtration cell is fed by means of a dual channel pump system, as the sample is processed it is fed into the bottom chamber of the ultra-filtration cell and along the standard membrane to waste. The pump creates a vacuum in the upper chamber and this draws a portion of the sample typically 20% (the other 80% goes to waste) across the ultra-filtration membrane and the filtered sample is then transported to the sample loop from where it is introduced onto the separation column. The arrangement of the ultra-filtration cell and the fact that the majority of the sample goes straight to waste helps to avoid the formation of filter cake that would ordinarily block the membrane. Ultra-filtration can be added to any Metrohm IC system and represents a convenient, cost effective way of helping to extend the lifetime of the separation column.

HPLC Instrumentation Problems Troubleshooting Guide

Circle no. 614

Anachem Ltd is pleased to announce the release of the new Gilson HPLC Troubleshooting Guide that will be a valuable tool to enable users to identify and resolve possible problems. Comprehensive in its approach, this guide provides troubleshooting from a system and a component perspective. References to TRILUTION® LC software, Ethernet and GSIOC Utility software packages are made throughout the guide.

The new Gilson HPLC Troubleshooting Guide includes many reference charts for full system and component troubleshooting. The very first checklist included in the guide is a recommended form that can be used during initial system troubleshooting. General guidelines for isolating problems related to hardware, software, system (including chromatography), laboratory and computer are covered in the guide.

Also included are tables listing solutions to common problems for each component category (pumps and detectors). This guide also includes the GX products and accessories as well as an Appendix full of reference tables and conversion factors useful to any laboratory chemist.

ADVERTORIAL

Is Environmental Screening Good for the Environment?

Every day, all around the world there are thousands of gas chromatographs continuously analysing environmental samples, the majority of which will give a negative result. It is estimated that worldwide GC's running environmental samples could be using as much as 1.4 gigawatt hours of energy and producing 840 tonnes of CO_2 every single day. To put this into perspective an average 1.6 litre family car would need to drive around the circumference of the earth over 116 times to produce the same amount of CO_2. The amount of CO_2 produced could be greatly reduced by using energy efficient fast GC technology for screening samples. The 300 Series Fast GC from Cambridge Scientific Instruments directly heats the column rather than using a conventional air blow oven. As only the column itself is being heated, considerably less power is required and the heating/cool down times can be much more rapid.

A typical GC with a power consumption of 2200 VA running an application with a heating time of 20 minutes would be producing approximately 300 g of CO_2 per sample. A 300 Series Fast GC with a power consumption of only 850 VA is able to run an equivalent analysis, but thanks to its much faster heat up and has a run time of only 3 minutes. So not only is the 300 Series fast GC using less energy, but what power it does require is for a much shorter amount of time.

All together this mean the 300 Series GC produces approximately 17 g of CO_2 per sample making it nearly 20 times more energy efficient.

For further information on the 300 Series Fast GC please visit www.camsci.co.uk or if you would like to talk someone about your chromatography requirements please call 01353 669916.

Circle no. 615

Circle no. 616