Automated Wetchem system for fertilizer analysis

Background information about Nucomat

The Belgian company Nucomat[1], enjoys an excellent reputation in the system integration sector and as a manufacturer of customer-specific solutions for laboratory automation. The core competency of Nucomat lies in the conversion of manual laboratory methods into automated systems. Robotics and automation are frequently associated with mass production and a large number of constantly repeated steps carried out as quickly as possible. As can be seen from the text below, laboratory automation requires a different approach often involving other criteria.

Nucomat builds turnkey units using a building block system made up of autonomous work stations linked to a sophisticated control and monitoring system. Nucomat can also incorporate instruments from other manufacturers in its systems in order to solve specific customer problems.

Nucomat installed various automated systems all over the world for research applications in the fields of life sciences, biotechnology, food technology, body care and cosmetics. Other systems are used for environmental analysis, the analysis of ore samples, quality assurance/quality control and petrochemical applications.

Nucomat systems are used for applications such as:

- Automated determination of copper in ore samples from open-pit mines. The system complies with stipulated operating rules and has a built-in quality control system. It is in continuous use 24 hours per day, 7 days per week.
- Automated sample preparation for heavy metal determination in liquid samples using ICP-AES.
- Automated preparation of plutonium reference materials.
- Recording and observing bacterial growth using measurements for determining the oxygen consumption in the development of new food preservatives.
- Sample preparation of viscous liquids while monitoring pH and viscosity.
- Cryotube serum aliquotation for clinical studies.
- Automated screening with a high sample throughput for drug development.
- Automated filtration for radioligand binding assays.

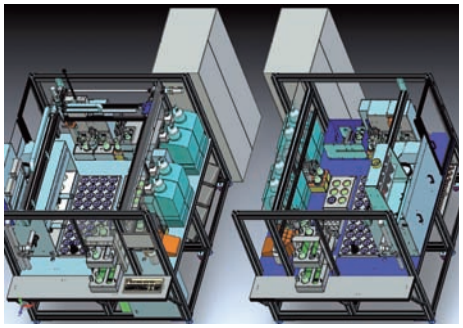

Fig. 1: One of Nucomat's product lines is the automated «Wetchem system» for the analysis of liquid chemicals. System characteristics can be described briefly as «untreated sample in, validated result out», as well as the possibility of carrying out several analysis methods.

An overview of the Nucomat system for the analysis of fertilizers

The automated Wetchem system for the analysis of fertilizer samples has recently been developed by Nucomat in close cooperation with Metrohm (see Fig. 1). The system allows multi-species determination (about 10 different species) and also carries out the complete sample preparation procedure, which is particularly worth mentioning. Sample preparation for wet-chemistry instrumental analysis methods still remains tedious and labor-intensive even in modern laboratories if it has to be carried out manually.

Outstanding features of the system are, amongst others:

- Sampling and sample preparation of liquid and solid samples
- Several reagent addition possibilities
- Classical «hotplate» digestion
- Making up the sample to a given volume by weighing (making to mass)

[1] Further information on the Belgian company Nucomat, which was founded in 1988, can be found on the internet under www.nucomat.be.

Fig. 2: The twin systems, each consisting of two 861 Advanced Compact ICs for the analysis of anions and cations, respectively.

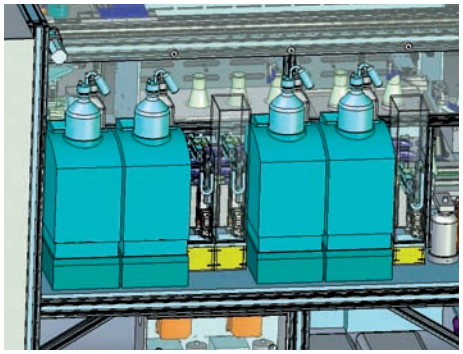

Fig. 3: Computer image of the module with the Metrohm ion chromatographs.

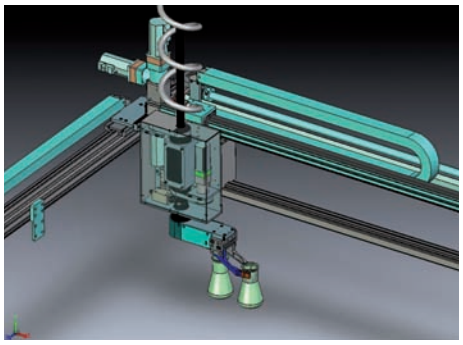

Fig. 4: The Nucomat robotic system for transporting samples and containers. The illustration shows details of the robot head with the gripper arm mounted eccentrically on the spindle axis.

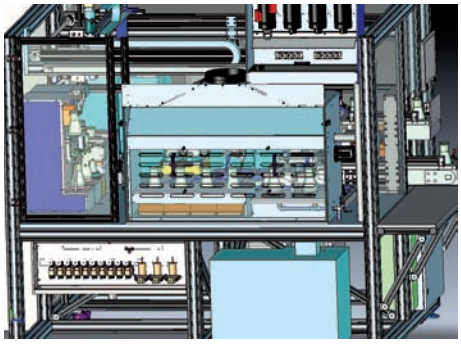

Fig. 5: As the transport robot is mounted above the working area, even Erlenmeyer flasks under the fume hood (center of illustration) can easily be accessed.

- Checking the instrument calibration by control measurements
- Integrated quality control
- Automatic system restart after power failure
- Closed-loop labware utilization (e.g. glass beakers) with built-in washing station
- Ion chromatography and titration as analysis methods
- Sample preparation for ICP-AES spectroscopy
- LIMS compatibility

Ion chromatography system

The Nucomat system described below uses 861 Advanced Compact IC ion chromatographs from Metrohm. In order to ensure as good a protection against breakdown as possible, two twin systems have been installed, each with a separate ion chromatograph for the analysis of anions and cations (see Fig. 2 and 3).

Robot with gripper arm

The main robot for sample and flask transport has four degrees of freedom. The robot head has a gripper arm (Fig. 4). Movements are based on a Cartesian coordinate system, with the belt-driven robot head moving in the X-Y plane and covering a maximum area of 2 x 2 meters. Movement along the Z axis (enabling 40 cm vertical reach) is performed by a spindle axis. The spindle can be rotated through 360 degrees for optimal use of the whole available working space. The pneumatic gripper is mounted eccentrically on the spindle axis. All the labware inside the fume hood can be accessed without any problems (Fig. 5).

The spatial position of the gripper arm is calculated on the basis of the dimensions given in the construction plans and of the index signals from the incremental sensor of the robot. No additional teaching is required for directing the robot head to any coordinates.

After installation or maintenance work (for example on the drive motor, incremental sensor/encoder or drive belt), the offset of the robot head needs to be determined.

Therefore a calibration bracket is mounted permanently on the support frame. Robot encoder index pulses are then referenced against the calibration bracket for proper alignment; no teach-in programming is necessary.

Analytical procedure

The Wetchem system analyzes liquids, suspensions, solid fertilizer granules or already ground powder samples. After scanning the barcode, the user only has to place the samples on the upper conveyor belt.

The barcode information is linked to a LIMS configuration file containing sample-specific parameters: amount of sample, acid to be added, stirring time, digestion temperature, final volume, analytical determination method and the corresponding measuring parameters.

Several working solutions can be prepared from one stock solution. In principle, each stock solution remains within the reach of the gripper arm until the preparation of the working solutions derived from it has been concluded.

By pressing a button, the user can cause the robot to reject a working solution at any time if it does not satisfy the sample preparation quality requirements. The robot then automatically prepares a new working solution from the corresponding stock solution.

Wide-neck 250 mL glass Erlenmeyer flasks are suitable for most sample preparation steps and determinations (Fig. 6). In contrast, PTFE flasks are used for the determination of boron, as digestion in glass vessels would result in contamination of the sample and therefore lead to incorrect results for this element.

After the analysis, the used sample flasks are cleaned with hot water in a washing station and are used again.

Stock solutions that are no longer required, excess working solutions, calibration solutions and samples ready for analysis by ICP-AES are transferred to the lower conveyer belt by the robot for further processing or disposal.

The ion chromatographs are controlled by the Metrodata IC Net software, which also records the measuring data. The analysis results obtained from this measuring data are stored in the internal database and simultaneously exported as ASCII-files in order to be read into the LIMS.

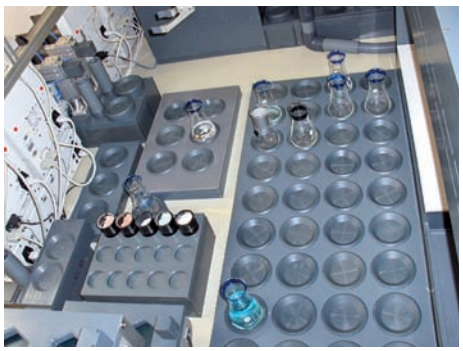

Fig. 6: View of the rear panels of the ion chromatographs and the labware of the Wetchem system (mainly wide-neck Erlenmeyer flasks) that are necessary for fully automatic sample preparation.

Calibration and verification of the ion chromatographs

Calibration and control solutions for ion chromatography are simply placed on the upper conveyor belt. The robot transfers these calibration solutions directly to the ion chromatographs and starts instrument calibration via the IC Net software. The operating personnel must ensure that all the necessary calibration solutions are available when required. A control solution is then used for the verification of the IC. The analysis result for the control solution is compared with the expected value. Depending on the result of this comparison, a control character is set to either enable the ion chromatographs for the analyses or to block them.

The operator can insert additional control solutions in order to verify the instrument performance and to evaluate the accuracy of the analysis as required by the laboratory's quality control procedures.

Instrument calibration is carried out at the same time as sample preparation.

Sampler for liquids and solids

The dosing station sampler obtains all the relevant information necessary for preparing the solutions from the Wetchem control software. The robot provides the sampler with the necessary Erlenmeyer flasks for the stock and working solutions. The sampler disposes of several hundred magnetic stirrer bars.

As soon as everything is ready, the preparation of the solutions can begin. The sequence consists of determining the tare of the Erlenmeyer flask, providing it with a magnetic stirrer bar, adding the sample, dosing in all the necessary reagents and transmitting the measured weights for each of these items.

The program is aborted as soon a serious error is detected during the preparation of the solutions. The user can terminate the preparation of a sample solution by pressing a button. The affected Erlenmeyer flask is then removed immediately and a new solution is prepared.

Integrated quality control

The accuracy of the balance is automatically checked at predefined intervals by the Wetchem system using a reference weight (Erlenmeyer flask). Each individual step in preparing the solutions, e.g. the addition of a certain volume of acid, is checked against the measured values from the balance. All measured values are permanently stored.

If the result of a control measurement differs too much from the reference value, then the availability of the particular analytical instrument is set to inactive by the system control (after confirmation by the operating personnel). In such a case, the remaining samples to be analyzed are diverted to a different, active analytical instrument or parked until the analytical instrument is activated again.

The Wetchem software

The Wetchem application program is based on Nucomat's «Robin» software architecture, an event-controlled command-line interpreter with SQL server database.

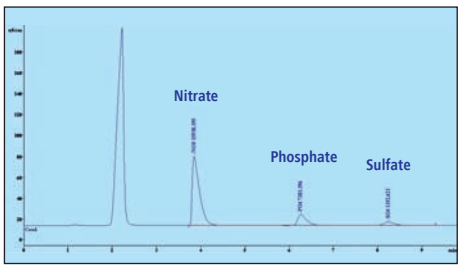

Fig. 7: This illustration shows a chromatogram obtained with a fertilizer sample by ion chromatography, in which the nitrate, phosphate and sulfate anions are determined.

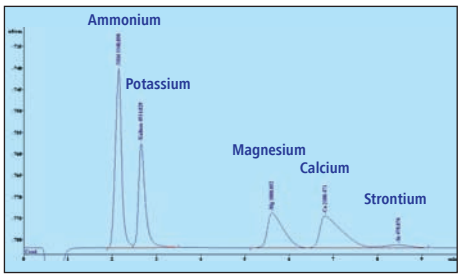

Fig. 8: Cations such as ammonium, potassium, magnesium, calcium and strontium can also be determined in the fertilizer sample by ion chromatography.

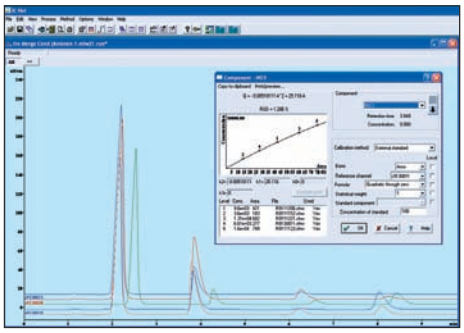

Fig. 9: This illustration shows the IC Net interface with ion chromatograms of cationic standards used for a calibration. If problems occur, the IC Net software is ideal for troubleshooting.

If a calibration point is clearly off the calibration curve (i.e. the measurement is incorrect), then the corresponding calibration solution can simply be analyzed again in order to complete the calibration. A control solution must first be measured before the ion chromatograph can be activated for the analysis.

The system allows the generation of various user profiles in order, for example, to give only the system administrator the right to alter important parameters. All alterations to the system configuration are permanently recorded and archived to ensure traceability. The same applies for each sample presented to the system after scanning, all movements of the robot, all weights and all messages from the dosing station.

If the connection to the LIMS computer should be briefly interrupted, then the Wetchem system can still continue to work; data matching takes place when the connection is reestablished.

Metrohm's compact ion chromatographs

An 861 Advanced Compact IC Metrohm ion chromatograph consists of:

- An Eluent Organizer
- A serial dual-piston pump showing extremely low pulsation with a flow range of 0.2 to 2.5 mL/min
- An injection valve allowing manual or automatic sample injection that can optionally be equipped with an «internal loop» with a volume of only 0.25 µL
- A completely metal-free flow path with all components made of PEEK or PTFE
- A separation column
- The Metrohm Suppressor Module MSM II for chemical suppression that is responsible for the extremely low noise level of only 0.2 nS/cm
- A conductivity detector with four different measuring ranges: 0...50, 250, 1000 and 5000 µS/cm
- The Metrohm data acquisition and control software IC Net that is also used for the determination of anions, cations and carboxylic acids

The sample to be injected is first pumped through an inline filter by a two-channel peristaltic pump (this prevents clogging of the separation column) and then enters the injection valve of the ion chromatograph. After the injection the valve is switched so that the filter can be rinsed with an acidified solution in reverse flow. The unit with the peristaltic pump is located directly beneath the ion chromatographs.

Applications and results

In addition to the Metrohm ion chromatographs, a titrator is also incorporated in the Wetchem system. In this way, the system described above can be used to determine the following species and parameters:

K_2O, P_2O_5, total nitrogen content, nitrogen as ammonium (NH_4^+), nitrogen as nitrate (NO_3^-), CaO, MgO, strontium, sulfate (SO_4^{2-}), chloride (all these substances by ion chromatography; Fig. 7 and 8); pH (using a pH electrode), free acid (titration with NaOH solution and a pH electrode) as well as B, Al, Fe, Cu, Zn, Cr, Ni, Cd, Mn (manual determination using an ICP-AES instrument).

The Metrohm IC Net software runs as a background process on the Wetchem computer. In normal operation, the operating personnel do not have to bother with IC Net. However, should a problem occur with the ion chromatographic analysis, the IC Net software is an extremely useful diagnostic tool that is available at any time (Fig. 9).

We would like to thank Prof. Chantal Block, Prof. Carlo Vandecasteele and Noel Van de Steene (CEO Nucomat) for writing this text and providing us with the measurement data and illustrations.