Spotlight

Sample Preparation & Storage

REPRESENTATIVE SAMPLE PREPARATION FOR XRF ANALYSIS

The successful use of spectrometers in the analytical laboratory requires an understanding of the method and profound practical experience. Everyday routine often does not leave the users enough time to develop and optimise the methods. Some analysis technologies require elaborate external trainings of the laboratory workers which delays the use of the instruments and reduces their acceptance among the users.

X-Ray Fluorescent Analysis is an exception as the sample is analysed in solid form and the measurements are easily carried out. Therefore, this method is well established in areas where quick results are essential, e.g. for quality checks during production.

As XRF measurements are so simple to carry out, the importance of reliable sample preparation is often neglected. This can lead to insufficient reproducibility and even to wrong analysis results.

For XRF analysis, the laboratory sample consisting of a few grams often has to represent a total amount of several tons. Beside the quality of the spectrometer, the quality of the sample preparation has a decisive influence on the precision and reproducibility of the analysis results.

Figure 1. From laboratory sample to pellet

"The 'art of milling' consists in turning a laboratory sample into a representative part sample with homogeneous analytical fineness"

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PREPARING HOMOGENEOUS SAMPLES BY MILLING

The 'art of milling' consists in turning a laboratory sample into a representative part sample with homogeneous analytical fineness (*Figure 1*). When selecting a suitable mill and grinding tools it should be taken into account that the material properties to be determined (e.g. heavy metal traces by XRF analysis) are not altered in any way during the sample preparation process. This not only requires a thorough knowledge of the instruments but also some experience in the preparation of different materials. Finally, care should be taken that possible abrasion from the grinding tools does not interfere with the analysis results.

XRF ANALYSIS OF DOLOMITE

The deeper the x-ray enters the sample, the more it interacts with atoms. This means that the part which is absorbed by the sample is increasing so that from a defined thickness onwards, the x-ray light can no longer penetrate the sample. In turn this also applies to the fluorescent light which needs to leave the sample in order to be detected.

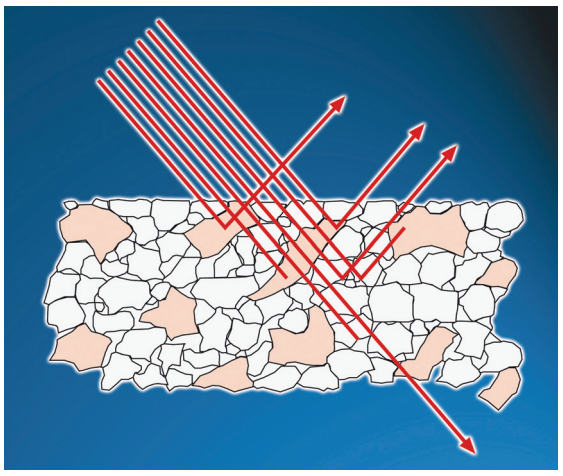


Figure 2. Saturation depth. Only a part of the fluorescent light leaves the sample

The lowest detectable sample layer is called saturation depth (*Figure 2*). It depends on the intensity of the x-rays, the wave length (i.e. the type of detected atom) and the density of the sample's surroundings (the matrix). If different elements are analysed in the same surroundings, the saturation depth increases with increasing atomic number of the element in question. Table 1 shows this correlation for dolomite.

Table 1. x-ray saturation depth of different elements in a dolomite sample.

Element	Atomic number	Saturation depth
Fe	26	170 µm
Mn	25	140 µm
Ca	20	100 µm
K	19	80 µm
S	16	30 µm
Al	13	10 µm

The general rule says that the saturation depth decreases with the atomic number which means that the element becomes more difficult to detect and with less reproducibility. That is the reason why elements such as carbon and boron emit very weak fluorescent signals.

PREPARING SAMPLES FOR XRF ANALYSIS

When preparing samples for XRF analysis, care should be taken that the size of the particles to be examined lies within the saturation depth of the x-rays to obtain a representative analysis result. This means for the dolomite sample that a fineness of 80 microns is only necessary if elements lighter than potassium have to be analysed.

Otherwise, a grind size of 100 microns, which can be obtained with any suitable laboratory mill quickly and easily, is sufficient.

Frequently, sample materials come in large amounts and great feed sizes which makes a preliminary size reduction necessary. After preliminary size reduction a part of the sample is subjected to fine grinding. This part sample must be representative, i.e. have the same properties as the total amount in order to obtain reliable information about the composition of the complete sample. Dry, pourable bulk samples can be fed to rotating dividers via vibratory feeders whereas sample splitters are suitable for heavily flowing materials.

The part sample thus obtained is then subjected to pulverisation. The most frequently used mill for the size reduction of hard and brittle sample materials for subsequent XRF analysis is the Vibratory Disc Mill (*Figure 3*). Inside the grinding jar the grinding tools, usually a puck and a ring, are moved in such a way that the sample is crushed by impact and friction effects. With this size reduction principle the required reproducible analytical fineness is achieved after very short grinding times. This is a decisive advantage when the analysis results are needed quickly, e.g. for a product approval.

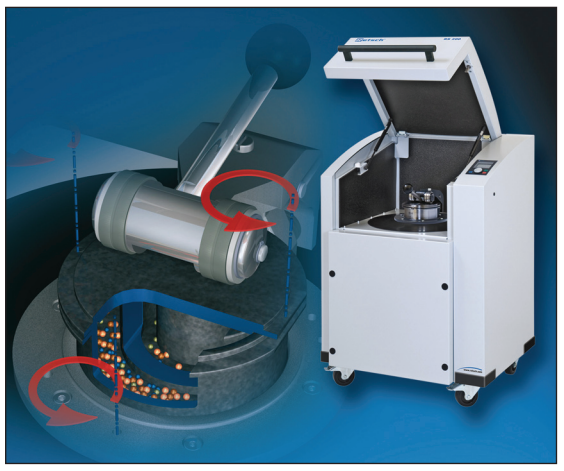


Figure 3. Retsch Vibratory Disc Mill RS 200

Small sample volumes can also be processed in a Mixer Mill (*Figure 4*). Here, the grinding jars perform radial oscillations in a horizontal position. The inertia of the grinding balls causes them to impact with high energy on the sample material at the rounded ends of the grinding jars.

Figure 4. Retsch Mixer Mill MM 400

PRODUCTION OF PELLETS

For most XRF applications pellets with a plane surface are used. In contrast to loose powder, a pellet has the advantage that the element concentration detected by the x-ray is higher because the material is more compact. In addition, a smooth surface is preferable to a rough one from an optical point of view. Usually, pellets are either produced by fusion of the sample with salt or by pressing the sample.

Fusion of the sample with lithiumtetraborate is a very effective method of producing a bead.

The sample is weighed together with the flux in a platinum crucible, then the crucible is heated up in a fusion machine to more than 1000 °C. This process destroys the original matrix and creates a homogeneous borate glass. This method yields highly reproducible results, regardless of the original material.

However, this method also has a few disadvantages. Volatile elements such as thallium or cadmium tend to escape during the fusion process and therefore cannot be detected. Moreover, the sample is heavily diluted with lithium salt (factor 10 - 50) which impairs the detection limit compared to pellets. Certain elements (e.g. boron, iron, carbides) could even damage the very expensive platinum crucible. Finally, it takes much more time to produce a bead than a pellet (15 minutes compared to approx. 2 minutes).

Therefore, pressing a pellet is the most common procedure for many applications – even though calibration of the spectrometer is more elaborate due to the sample matrix. A pressed pellet should basically fulfil the following quality criteria:

1. it must be homogeneous
2. the pellet must be absolutely solid as loose particles pollute the x-ray tube
3. the pellet should be stable (and storable)

Pressing the sample can be carried out with or without auxiliary materials. The most frequently used materials are cellulose-based or paraffin-based. Cellulose has the advantage of acting as grinding aid at the same time thus avoiding caking of the sample inside the grinding jar. Cellulose can be used in vibratory disc mills as well as mixer mills.

Wax is added after the sample has been ground, either manually or by mixing it with the help of polyamide balls in a plastic jar in the mixer mill. The addition of wax makes the pellet's surface indelible. Moreover, wax is more inexpensive than cellulose and not hygroscopic which is important if the pellets are to be stored. To stabilise the pellets either steel rings or aluminium cups are used (*Figure 5*). The cups can be labelled on the reverse side and are useful for storing the pellets.

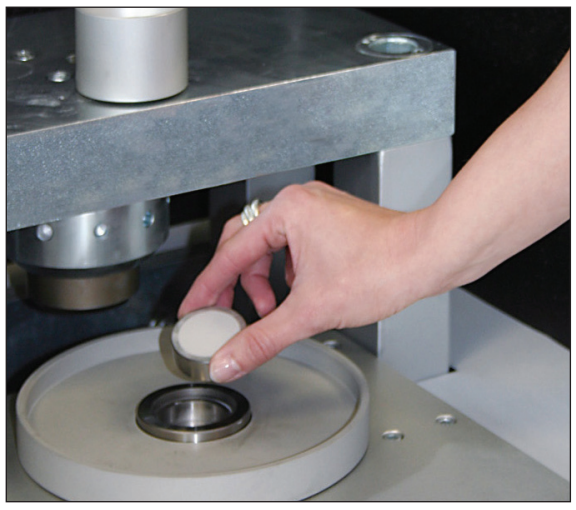


Figure 5. Retsch Pellet Press PP 40

New EVOLUTE® CX for Improved Sample Preparation

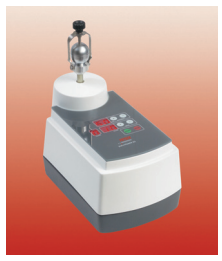
Biotage announced the introduction of the new EVOLUTE® CX sample preparation range. EVOLUTE CX is a mixed-mode resin-based SPE sorbent for the extraction of basic drugs from biological fluids using a generic procedure, minimising method development time for bioanalysts. EVOLUTE CX removes protein, phospholipids, and salt interferences, delivering cleaner extracts with high reproducibility for accurate quantitation and trouble-free analyses through pre-clinical and clinical trials.

Biotage's EVOLUTE CX uses a combination of robust polymer chemistry, uniform coverage of sulfonic acid (SO3-) groups and optimum exchange capacity to provide greater than 85% recovery on a broad range of sample matrices. The surface characteristics of every batch are tested to ensure consistency. A combination of optimised pore size, sorbent chemistry and associated generic method results in extremely clean extracts for LC–MS/MS.

The Biotage EVOLUTE family of products now includes EVOLUTE ABN (30µm and 50µm) and EVOLUTE CX. EVOLUTE products provide highly effective solutions to the problems of ion suppression and matrix effects from dirty extracts. These high performance products allow scientists to use a generic approach for a wide range of compounds, reducing method development time while improving extract cleanliness.

Circle no. 111

Drug Testing via Hair Analysis



In order to proof the consumption of drugs (cocaine, cannabis products, amphetamines) even after long periods of time, hair is analysed frequently. It is dissolved in certain acids and then analysed via gas chromatography or the mass spectrometer. The dissolving in the acid is accelerated and improved through the enhancement of the specific surface area of the sample. Here the ball mill Fritsch PULVERISETTE 23 is used. In a 15ml steel cup, 300-500mg hair is weighed in. As a grinding body a 15mm steel ball is used. After a grinding duration of 5 minutes a fine powder is attained (< 100µm), which can be used for the following analysis. For larger amounts or for the grinding of two samples simultaneously, the Fritsch Micro Mill PULVERISETTE 7 can be used. In a 45 ml grinding cup made of zirconium oxide, sintered corundum, silicon nitride approximately 0,1-1g of hair is weighed in and 7x15mm grinding balls are added. The grinding time is 5 - 20 minutes. Afterwards a fine powder is obtained (< 100 µm), which can be used for additional analysis.

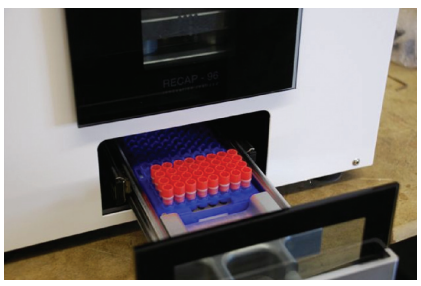
Circle no. 110

Innovative Kits for Large-ScalePlasmid DNA Isolation

Two new products for large-scale plasmid DNA isolation are being introduced by QIAGEN. The CompactPrep Plasmid Mega and Giga kits offer greater convenience and faster extraction than other methods. The purification capacity is up to 2.5 mg of plasmid DNA for the Mega Kit and up to 10 mg for the Giga Kit. With commonly used techniques, isolation of large quantities of plasmid DNA was often very time consuming, requiring as much as several hours to complete. This posed significant challenges for researchers facing increasing time pressures. In contrast, the new kits allow isolation of plasmid DNA in less than 50 minutes due to a vacuum-driven protocol. In addition, by using the company's QIAvac 24 Plus vacuum device in combination with the new QIAvac Holder, it is possible to process up to 12 preparations in parallel. Virtually no hands-on work is required during the entire procedure. Another advantage of the CompactPrep Plasmid Mega and Giga kits is the highly concentrated, low-endotoxin plasmid DNA produced. The new kits use small columns that enable elution of DNA in a very low buffer volume, resulting in DNA concentrations of up to 2.5 µg/µl. In addition, by using a supplemental wash buffer, endotoxin levels are usually lower than 1 EU/µg DNA. The resulting purified plasmid DNA is suitable for all routine downstream applications, including cloning, enzymatic restriction, in vitro transcription and translation. Due to the kits' proprietary wash buffer, the extracted plasmid DNA is ideal for transfection experiments, even with cell lines as sensitive as Huh-7.

Circle no. 112

Automated Handling of Screw Top Storage Tubes



Micronic BV has introduced a new automated instrument that accelerates the time consuming and tedious process of applying or removing screw top caps from sample storage tubes.Easy to install and operate the Automatic Screw Cap Handler automatically applies or removes the screw caps from a full 96-tube rack in less than 15 seconds. In addition to delivering significant productivity gains the Automatic Screw Cap Handler lowers the risk of sample contamination and eliminates the chance of repetitive strain injury inherent with manually applying / removing large numbers of screw top caps. To ensure optimal seal quality on samples the Automatic Screw Cap Handler simultaneously processes all 96 tubes in a rack with each cap receiving the same torque.

Designed for stand-alone use the user friendly Automatic Screw Cap Handler requires just a mains power source for operation and fits neatly on a laboratory bench or inside an environmental control cabinet. Built for versatility the instrument is compatible with the complete Micronic range of screw cap tubes (0.5ml, 0.75ml, 1.1ml and 1.4ml) and tube racks.

Circle no. 113

New, More Compact High-Temperature Digestion System

Responding to the needs of smaller labs, SCP Science has added a new product to its line of high-temperature digestion systems. Previously only available in 20- and 40-position models, the new 10-position DigiPREP HT offers the well-known high quality of SCP SCIENCE digestion systems in a more compact and affordable package.

Offering the same level of versatility and quality as its larger counterparts, the new 10-position DigiPREP HT also presents these new features include: a Teflon®-coated two-tier racks, offering corrosion resistance to acid attack; 33% more depth in each graphite well, providing increased contact for faster digestion; A silicon carbide-coated graphite block, preventing the warping that occurs with aluminium blocks; and an integrated dual-glass window, making it easier to view samples when running a method

Circle no. 114

Long Term Stored Sample Integrity Enhanced by New Formulation Tube Caps

Micronic has announced the availability of a new generation of TPE coloured caps that set a new benchmark for ease of use and long term sample storage integrity.

Extensive research by Micronic has resulted in a USP Class VI certificated Thermo Plastic Elastomer (TPE) that provides a tube seal that is easily pierced, highly hydrophobic, better resealing properties after repeated piercing and no discolouration even after long term exposure to oxygen.

Designed to increase productivity and eliminate expensive tube capping systems, the new generation Micronic TPE coloured caps provide single action capping of a single tube, a row of tubes, or a complete 96-tube rack. With a choice of eight different colours, the new TPE caps provide a simple, yet highly effective means of visually differentiating stored samples by person, department, project or even by the day of the week they were submitted.

Each TPE coloured cap cluster is made of two components: the cap, and a retaining film. This film is easily removed, leaving a high integrity seal on individual tubes. The novel cap cluster design of the TPE coloured caps virtually eliminates cross contamination problems associated with some traditional capping systems.

Manufactured from chemically resistant and hydrophobic polymer, each TPE coloured cap product is multi-piercable ensuring long-term sample integrity. Combining operational flexibility with absolute sample integrity, TPE coloured caps are compatible with all Micronic standard or coded tube products. Micronic TPE coloured caps may be seamlessly integrated into automated drug discovery and high-throughput screening systems.

Circle no. 115

New HyperSep Retain SPE Range for Faster Sample Preparation Launched

Thermo Fisher Scientific, Inc announces the launch of the Thermo Scientific HyperSep™ Retain™ Polymeric Solid Phase Extraction (SPE) range. Featuring unique functionalised polymer chemistry, HyperSep Retain products deliver high recovery levels and high reproducibility. HyperSep Retain SPE products provide sample preparation options for a wide range of applications and provide enhanced retention for a wide range of analyte types.

The Thermo Scientific HyperSep Retain range offers benefits over silica-based SPE offerings. Fast method development can be achieved and problems associated with material drying out after conditioning are eliminated. With the addition of the HyperSep Retain polymeric-based range, Thermo Fisher now offers scientists an array of products in varying formats and phases to meet the demands of their specific chromatography analysis by providing faster throughput in chromatography analysis, easier method development and enhanced selectivity in analysis.

HyperSep Retain is available in three chemistries: a polar-enhanced polymer material for balanced retention of polar and non-polar compounds; cation exchange for enhanced selectivity of basic compounds and anion exchange for enhanced selectivity of acidic compounds.

Circle no. 116