A LARGE CAPACITY SAMPLE CHANGER FOR FULLY AUTOMATED GAMMA RAY SPECTROSCOPY

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ABSTRACT

An automatic sample changer has been developed for use in Ge(Li) gamma ray spectroscopy. The sample changer features remote storage to prevent cross-talk and to reduce background. It has a capacity for 200 samples. It also features automatic setting of the sample to detector distance to optimise the count rate performance. An additional feature is the rotation and vibration of samples during counting to assure that powdered samples are compacted and improve the precision and reproducibility of the counting geometry. Three systems have been equipped with the changer and these are connected to an in-house computer on a first-come-first-served basis via a high speed data interface (153,6Kb).

INTRODUCTION

Many investigations in which the technique of neutron activation analysis is used involve the processing of large numbers of samples. For example, in our laboratory at the University of the Witwatersrand, several geological and biological projects are in progress simultaneously each of which involves the multi-element analysis of hundreds of samples. Typically, each sample must be measured at several decay times. An automatic sample changer is necessary if efficient use is to be made of the counting systems, of which there are three.

The demands made on a sample changer for use in gamma ray spectroscopy following neutron activation analysis are severe. First of all such a sample changer must be able to accommodate a large number of samples, both solid and liquid. These samples can differ in the level of their activity by several orders of magnitude. The sample library or store must therefore be very efficiently isolated or shielded from the detector and from the other systems, and it must be possible to change the sample to detector distance in order to keep the count rate in the dynamic range of the detector system while not sacrificing efficiency unnecessarily.

These considerations, together with the desirability of restricting most of the handling of radioactive material to the area of the radiochemical laboratory, and away from the vicinity of the gamma ray spectroscopy system, have led to the design of an automatic sample changer that has a sample store or library at a remote location. Transfer of the samples is effected pneumatically, and the system can be controlled from either the store or the counter. The sample - to-detector distance is set automatically so that the count-rate is equal to or less than a pre-set value.

In order for both solid and liquid samples to be accommodated by the apparatus, a liquid scintillation counter vial was used. This vial can contain 22ml of liquid and it has a liquid-tight cap.

A commercially available sample table that can hold 200 samples was used. In our laboratory batches of about 30 samples are usually processed, and each sample is measured at several decay times. The large capacity of the sample store makes it possible for the samples to be left in the store until all the measurements on the batch have been completed.

One of the most important considerations in order to obtain accurate results in the reproducibility of the sample to detector geometry during the measurement. This requirement has been given careful consideration in the design of the counting head which will be described below.

The system developed to meet these requirements incorporates a movable lead castle that provides a shield for the detector during the measurement and a biological shield around the sample store. It also incorporates a digital read-out of the sample to detector distance, a time-of-the-year clock, and an interface system to transfer the relevant data direct to the in-house computer.

DESCRIPTION OF THE SYSTEM

The sample changer consists of two distinct sections, the sample store and the counting station. Transport between these positions is effected through pneumatic tubes. A diagram of the system is shown in Figure 1. In our implementation the store and the counting station are separated by some 180 metres. The following sections give details of the main features of the system.

Sample Store

The store of 200 samples is based on a commercially available sample changer for a scintillation counter. It consists of an endless ringed belt on an aluminium platform. It has been modified by the addition of a sample-transfer tube and a pneumatic lift for introducing the sample into the tube (Figure 2).

Sample Transport

When the sample is transferred from the store to the counting station the pneumatic cylinder is activated and lifts the sample into the transfer tube. As it reaches its limit of travel a rubber ball is compressed and closes the bottom end of the transfer tube. A photocell (No. 1 in Figure 1) senses the sample and pneumatic valves A and F are activated. Valve A blows the sample over to the counting station, and valve F releases the air during transfer. The vial accelerates to approximately 60 km/h. Just before it arrives at the counting station the sample is detected by photocell 3. Valves A and F are deactivated while E is opened for approximately 300 ms. This produces a back-
pressure and decelerates the vial to a gentle landing at the counting station. Photocell 4 detects the vial and commands the analyser to start counting, or, alternatively the distance is determined and set automatically before the command is given to the analyser.

When the sample is returned to the sample store the reverse sequence takes place. Valves D and C open and, when the vial passes photocell 2, valves D and C close and B supplies the back pressure. When photocell number 1 detects the sample the pneumatic cylinder lowers the sample back into its original position in the chain. The chain advances and the next sample is despatched.

The Counting Head

Many samples for neutron activation analysis consist of powder encapsulated in quartz vials, and when these powdered samples are counted the powder is unevenly distributed inside the quartz vial because of the acceleration and deceleration during transfer. This can produce errors in the counting geometry.

The sample is therefore vibrated at a frequency of 2 to 90 Hz ramping up and down at a rate of 0.1 Hz for a duration of approximately 60 s. This ensures that all powder particles of different sizes will have been compacted. In addition the sample is rotated at 10 r/min during the entire count. This reduces the effects due to possible inhomogeneities in the sample and eliminates distance setting errors even if the sample is not exactly in the centre of the vial. This method greatly improves the reproducibility of the

Figure 2. A view of the sample store with lead shield removed.
counting geometry. The counting head incorporating these features is shown in Figure 3.

**Shielding**

A 50 mm thick lead shield is built around the sample store (Figure 4). The shield is constructed in such a way that the store is completely enclosed except at the bottom, where a space of 120 mm is left open so that the basket can be placed under the platform of the changer. Samples are removed from the system by allowing them to drop into the basket through a remotely controlled port in the platform.

Loading the changer with the shielding in position is accomplished through a port in the transfer pipe (Figure 1). A radiation monitor is mounted on top of the lead shield. If the activity exceeds 25 μSv/hr, an alarm is sounded, indicating that the maximum permissible total sample activity has been reached.

The lead shielding is easily removed by using two sets of aero castors which are located on the bottom sides of the lead shield. Compressed air at 80 kPa is fed to the aero castors and lifts the lead shield from the floor. The shield floats on a thin film of air. The shield can then be pushed away from the changer with very little effort.

The counting station is equipped with a movable lead shield that surrounds the snout of the Ge(Li) detector and the counting head (Figure 5). This shield reduces the background and prevents interference from samples being counted on adjacent counting systems.

**Control Units**

Master and remote control units are made available. The master control is situated at the sample store and is built into an instrument-type cabinet. The remote-control unit is built into a double-width NIM module and situated at the counting station (Figure 6). At both control units the following facilities are provided.

- A three digit sample position number indicated by LED display.
- Pushbuttons to provide the following functions. Forward and reverse movement of the chain that holds the samples.
- Transfer and return of the sample.
- Unloading of the sample.
The front panels are engraved with a mimic diagram and fitted with corresponding indicator lights that indicate whether the sample is present in the sample store, in transit, or at the counting station. The remote control unit at the counting station is electrically isolated from the master control to prevent groundloop noise interfering with the sensitive counting equipment.

### Automatic Distance Setting

When a sample arrives at the counting station its activity is measured by means of a logarithmic ratemeter which is connected to the main linear amplifier. The output of the ratemeter is fed into an analogue comparator that compares the output of the ratemeter against a level set by the operator. The resulting difference in output from the comparator is fed into a voltage-to-frequency converter, and the pulse output of the converter drives a stepping motor. The stepping motor (Figure 1) drives a lead screw, and the counting head will move away from the detector, depending on the activity of the sample.

The counting head will move at a constant rate of 30 mm/s if the measured count-rate level is not within ± 10 percent of the set level. When the measured count rate level falls within this range the movement of the counting head is progressively slowed to a base speed of 1 mm/s. When the measured count rate is within 1.5 percent of the set level, and this rate is maintained for 100 ms, the stepping motor is disabled and the analyser is enabled.

The distance between the counting head and the detector is determined by counting the number of pulses supplied to the stepping motor, and feeding the pulses into an up-and-down counter.

The stepping motor steps at angles of 1.8° per step, and the lead screw that moves the counting head has a pitch of 2 mm, which means that one step will displace the counting head by 0.01 mm. The maximum sample to detector distance can be set to 950 mm.

The system is calibrated by driving the counting head towards the detector where a limit switch will disable the stepping motor and set the counter to a fixed number (in our apparatus this distance is 17.00 mm, the distance of the sample holder from the face of the detector).

### Interface

The interface between the computer and the analyser consists of a receiver unit at the computer and the transmitter module at the analyser. The transmitter module can be up to 500 m away from the computer using Belden cable 9184, at a transmission rate of 153,600 bps. Multiple transmission channels are input available so that multiple counting systems can make use of the same interface in a first-come-first-served mode. The transmitter module situated at the counting station is built into a standard single width NIM module. The data to be transmitted is converted from 8 bit parallel to serial by means of a Universal Asynchronous Receiver Transmitter (UART) at a continuous rate of 153,600 bps and the clock driving the UART also generates the strobe signal required to retrieve the data from the analyser memory. When data is to be transferred a LOOK AT ME (LAM) command is transmitted every two seconds until this is acknowledged by a READ (RD) command. When the RD command is received the ancillary data from the printing NIM modules is transmitted, followed by the data from the analyser. After the last channel has been transmitted an END OF FILE (EOF) signal is generated.

If an error is detected by the computer a NOT ACKNOWLEDGED (NACK) signal is transmitted, and the sample is counted again. If no errors are detected an ACKNOWLEDGE (ACK) is sent, the sample is changed, and a new cycle is started.

The ancillary information transmitted at the beginning of the data transfer from the printing NIM modules is as follows -

1. Counting system identification code.
2. Day of the year. (When the counting of the sample began).
3. Time of day. (When the counting of the sample began).
4. Sample number.
5. Sample-to-detector distance.

The ancillary information relates to the sample that has been counted and is required for the processing of the spectral data on the computer.

If required, more modules can easily be included, as the printing module operates in a daisy-chain mode. Provision has been made for a maximum of 15 units.

The receiving unit converts the data from serial to parallel format and the data is presented in half-word mode via a universal logic interface to the SELECTED CHANNEL BUS (ESCHLCH) of the computer with associated control bits EXAMIN (EK), ERROR (ERR), END OF FILE (EOF), LOOK AT ME (LAM), BUSY (BDY), and DEVICE UNAVAILABLE (DU). Multiple input channels are made available so that more than one system can make use of the same interface on a first-come-first-served basis. All available input channels are initially inactive. When a transmit request LAM is received on any of the available channels, all remaining channels are disabled for one second. During this time the processor must acknowledge the LAM with an RD; if it does not, the sequence will be repeated one second later. All data files are transferred onto a disc, and the analysis program retrieves the data to complete the processing.
DISCUSSION AND CONCLUSION

The effective use of neutron activation analysis depends on the availability of a suitable automatic sample changer. The sample changer described here has been specifically designed to take account of the demands of the neutron activation technique. The remote location of the sample store allows the handling of samples to be restricted to a radiochemical laboratory remote from the detectors.

This greatly reduces the possibilities of contamination and cross-talk, which occur when large numbers of active samples are stored close to the detector systems.

The ability to count liquid samples has proved to be an important feature, and the maximum volume of 22 ml or mass of 30 g has proved to be sufficient. Another useful feature of the system is that, as a result of the large capacity of the sample store, the samples can remain in the store until the series of counts is completed. This greatly reduces the handling of the samples and the exposure of the operators to radioactivity.

The accuracy of the neutron-activation method depends on the accuracy and reproducibility of the counting geometry, and on the effective utilisation of the count-rate capabilities of the Ge(Li) detector system. In view of this the features incorporated in the counting head, such as the facility for rotating the sample reduce errors due to off-centre positioning of the sample in the transfer bottle or to possible inhomogeneities in the sample. Subjecting the sample to vibration ensures that powdered materials will have a reproducible position. This is particularly important at small sample-to-detector distances.

The automatic setting of the sample-to-detector distance allows samples of widely differing activities to be included in one run, and the digital read-out has eliminated errors arising in the manual measurement of the sample-to-detector distance. The on-line connection in our in-house computer has reduced the error rate attributable to human handling, and has greatly increased the amount of data that can be processed.

The fast data-transmission rate (153,6Kb) gives a transmission time per 4K spectrum of less than one second. This means that it is possible to process data from several systems connected to the central interface on a first-come-first-served basis. Since no magnetic tape unit/interface is required the cost of transferring the data is considerably reduced.

The apparatus has so far proved very reliable. Three counting systems have been implemented (Figure 7) and several thousand samples have been processed.

Figure 7. Overall view of counting room showing three counting systems.

ACKNOWLEDGEMENTS

The authors thank the National Institute for Metallurgy for permission to publish this work, and Professor J P F Sellschop of the Nuclear Physics Research Unit of the University of the Witwatersrand, for his interest in, and support of, this work.

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