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Principles and Applications of Robotics in Analytical Chemistry

The following are summaries of two of the papers presented at a Meeting of the North East Region held on November 24th, 1991 in the ICI Wilton Research Centre.

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Robotic System for Microwave Dissolution of Titanium(IV) Oxide

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In this laboratory a large number of trace-element determinations are carried out on titanium^{IV} oxide powders. These are done by a variety of spectroscopic techniques, including flame atomic absorption, electrothermal atomic absorption, inductively coupled plasma atomic emission and cold vapour mercury atomic fluorescence.

Some time ago it became apparent that the number of such samples coming into the laboratory exceeded the ability to cope with them. The instrumental capability was available, but despite a large number of staff, the samples could not be prepared quickly enough, and a backlog of work built up. Obviously something had to be done about this situation, and as automation was present in the form of autosamplers on all of the instruments, automation of the sample preparation process seemed to be the best option.

The first step was to consider the sample preparation procedures currently in use. These differed depending on the elements to be determined and the sample type. Titanium^{IV} oxide powders range in composition from 99.9% TiO_2 for high purity material, through pigmentary grades, down to about 75% for sunscreen material, the balance being largely Al_2O_3 and SiO_2. The sample preparation procedures in use were: slurry, involving grinding the sample and preparing a slurry with Viscalex as a dispersing agent, rather like making an emulsion paint (although this technique had been widely used for many years several limitations had recently become apparent); HF–HCl digestion, which has become increasingly used particularly since the use of closed digestion vessels in a microwave oven has replaced use of digestion in open vessels on a hotplate; fusion, which was only used for a small number of applications and is not really appropriate for trace-element determinations.

It was clear that HF–HCl dissolution in a close vessel in a microwave oven would be the preferred technique in the future, and that this should be automated. Consideration was given then to the requirements of an automated system. This formed the basis of the specification. The main requirements were as follows. (*i*) Carry out complete task: this would involve weighing out the sample, adding acids, capping the digestion vessel, microwaving, uncapping the digestion vessel, diluting to volume and transferring into a beaker. In summary the solid sample would be presented in a bottle and the final solution collected in a beaker, ready for analysis. (*ii*) Clean and reuse digestion vessels: there seemed no point in carrying out the digestion automatically to be confronted with a large number of digestion vessels and caps to be cleaned at the end of a run. (*iii*) Accurate weighing: ± 0.0005 g for a sample mass of 0.5 + 0.1 g. (*iv*) Accurate liquid volume dispensing: +0.5 ml for both acids, 7 ml HF and 3 ml HCl and the final volume of 50 ml. (*v*) Acids not to be under pressure: considering that HF was to be used it was decided that the acids would not be under any pressure and that gravity feeding should be used. (*vi*) Spillages to be contained: any potential spillages were to be confined to the minimum area. (*vii*) No sample contamination: there was to be no possibility of cross-sample contamination. This was relevant to the selection of the sample handling system and the digestion vessel washing technique. (*viii*) Facility to alter operating conditions: it was considered desirable to have some flexibility in selecting certain operating parameters, particularly the digestion programme, sample weight, acid types and volume, and final solution volume. (*ix*) Throughput: it was required to be able to process 60 samples in an overnight run.

It soon became apparent that it was not possible to purchase an 'off-the-shelf' robotic system capable of meeting these requirements, although many parts of the proposed process had already been automated. Whilst a robotic system carrying out most of the process could easily be envisaged, there were some areas of uncertainty, particularly the sample handling of TiO_2. It was therefore arranged for a feasibility study of these areas to be carried out by Peerless Systems Ltd.

The study was carried out satisfactorily and an order was placed with Peerless for the complete system. The robotic microwave digestion system for dissolution of titanium^{IV} oxide and its operation and performance have been described elsewhere.[1]

The system has now been installed for nearly 18 months and during that time it has generally worked well without any problems. Performance tests have shown that the reproducibility and accuracy of the robotic system are equal to those which can be achieved by a competent analyst. Meanwhile, the competent analyst is released to do more demanding tasks compatible with his or her qualifications.

References

1 Norris, J. D., Preston, B., and Ross, L. M., *Analyst*, 1992, **117**, 3.

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Robotics: Past, Present and Future

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Over the last decade, laboratory automation has progressed from being a little studied curiosity to providing a major benefit to analysts. The increase in robotic and non-robotic based systems offered by vendors has simplified the change from manual to automatic methods for the analytical chemistry community. Automation of some laboratory techniques offers significant advantages when compared with the equivalent manual operation. Often the question is not 'is it advantageous to automate a procedure?', but 'When and how should the procedure be automated?'.

Our experience with robot-based systems at BP has been mixed, but the knowledge gained allows us to select the most suitable procedures to automate in the future. Advantages of each robot geometry will be discussed.

Why Automate?

Originally, when automation was suggested for laboratory operations, the main advantages were expected to be: cost saving for automatic methods over the comparable manual methods; improved quality of the final result; increased productivity of automated laboratories; improved safety of laboratory operations.

As experience has increased, the prime goal of automation has become the replacement of manual activities aiding staff to add value to the activities of their organization. Improvements to the following can be achieved: accuracy or repeatability of results; length of working day; throughput of samples; better sample tracking using a comprehensive audit trail.

Reduction of costs and improvements of safety are often the criteria used to justify the purchase of an automated system. It is the job of laboratory managers to identify applications that can benefit from automation, and recognize that it is not always easy to make the transition from manual to automated procedure. It is the job of the scientist to ensure that workable and practical systems are selected, developed and implemented. When both jobs are done well the results can be dramatic. In addition, these sytems enable new regulatory requirements, which demand higher levels of sample tracking and method audit trails, to be undertaken in a cost-effective manner.

In an analysis scheme, there are three major stages (as shown in Fig. 1). The sample to be analysed usually requires some form of pre-treatment. After the analysis, the data is collected, analysed and then some form of deduction/result is returned to the sample author. Usually, it is the sample preparation stage that is labour intensive. Most high sample volume analytical equipment is available with auto samplers and computers to perform the latter two stages. Most robot-based laboratory systems are installed for sample preparation.

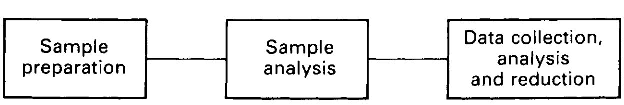

Fig. 1 The three stages of the analyses of a sample

Choosing a System and Robot Geometry

Types of Automation

The choice between dedicated automation (*e.g.*, auto samplers) and flexible automation (*e.g*, robotics) is application dependent, and the main criteria are difficulty of analysis and volume of sample (see Fig. 2). Automation of a particular analysis where only low volumes of samples pass through a laboratory rarely can be justified except on the grounds of safety (*e.g.*, toxic or radioactive analytes). Very high volumes of samples, such as blood analysis in a hospital laboratory, are best handled by a dedicated piece of equipment. If the method requires a degree of flexibility, then consider automation using a robot-based system.

There are three major geometries of robots available commercially: anthropomorphic (multi-jointed, human-like configuration); cylindrical (parallel action arm pivoted from a central point); Cartesian (three mutually perpendicular axes). Each geometry is used in a variety of industries, and each has advantages. The key to the problem is determining which application to automate and which system to use. Once the application is selected, choose commercially available hardware (robot and peripherals) to reduce the amount of integration that you have to do. Underestimating the amount of effort required to integrate, install and test a new automated system is a commonly made mistake. In our experience, an installation will take at least a year between the time the equipment is delivered to the time the system is performing useful tasks.

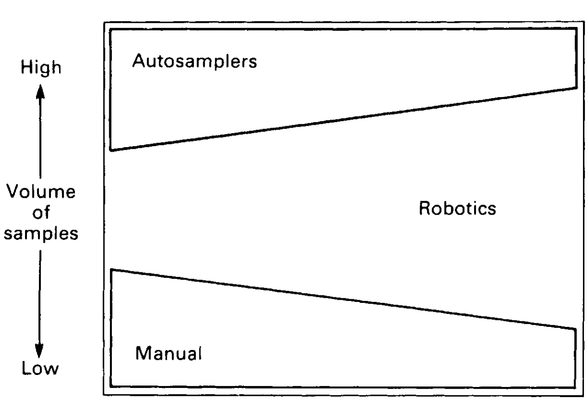

Fig. 2 Sample volume and its effect on sample preparation

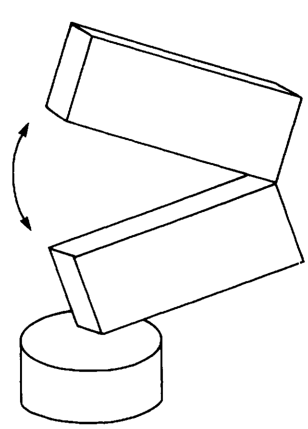

Fig. 3 Anthropomorphic geometry

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Table 1 Advantages and disadvantages of each robot geometry

Robot geometry	Applications	Advantages	Disadvantages
Anthropomorphic	(a) Five joints, light assembly applications, *e.g.*, Mitsubishi RM501 (b) Six or more joints, industrial applications, *e.g.*, Puma series (c) Laboratory Systems, *e.g.*, Hewlett-Packard ORCA (six axes) is an anthropomorphic arm mounted on a precision track	(a) D.c. motor versions are very powerful for size; circular 'waist' motion gives good access to equipment for laboratory type operations (b) Usually d.c. motor versions; very powerful; very dextrous (c) Laboratory system commercially available	1 Robot path difficult to predict when the robot is in motion 2 Require complex control algorithms 3 Limited work area unless mounted on a track
Cartesian	(a) Most commercially available systems are aimed at light/medium engineering applications (b) Laboratory Systems, *e.g.*, UMI Labman 1200	(a) Fit in well with general bench style laboratory designs; easy to set-up laboratory type handling procedures (b) Commercially available	1 Linear motion gives limited access to equipment for laboratory type operations 2 Limited applications available
Cylindrical	(a) Mixture of radial and linear motions based around a circular waist motion (b) Laboratory systems, *e.g.*, Zymark, Peerless Systems	(a) Circular 'waist' motion gives good access to equipment for laboratory type operations; easy to set-up laboratory type headling procedures (b) Commercially available; multitude of applications already completed	1 Tend to take up a large expanse of laboratory space

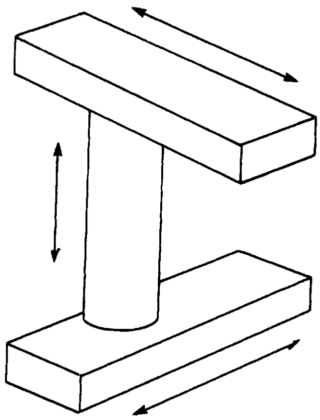

Fig. 4 Cartesian geometry

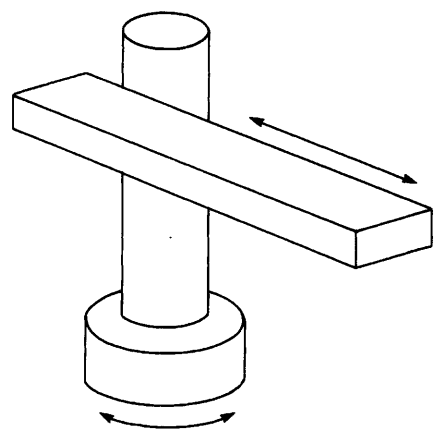

Fig. 5 Cylindrical geometry

Anthropomorphic

Originally, multi-jointed (or anthropomorphic) arms were considered for laboratory use (Fig. 3). These were implemented successfully in the automobile manufacturing and nuclear industries and small-scale (light industrial fabrication) versions were commercially available. However, studies undertaken at a number of companies proved the general unsuitability for laboratory use. The main reasons are summarized in Table 1. BP's initial experience was the use of a Mitsubishi RM501 robotic arm for an experimental automation test-cell. The robot was used to load and unload the sample probe of a small quadrupole mass spectrometer. The design and construction of the robot was of a very high quality, but the geometry made the control and operation of this robot unsuitable for laboratory use for the reasons summarized in Table 1.

Cartesian

The attraction of cartesian robots (see Fig. 4) is their compatibility with standard laboratory benching. Only very expensive, heavy-duty systems were available at the time of our first interest in laboratory robotics. BP defined a specification, and commissioned the design and construction of a cartesian robotic system. The operating computer software was written 'in-house' to give a fully flexible laboratory automation system suited to a wide range of applications. A series of designs was built. The Mark IV version is in use in a number of laboratories in Great Britain.

Applications include: sample preparation of used lubricating oils prior to inductively coupled plasma emission spectrometry (ICP-ES) analysis; separation and analysis of additives in polyethylene; manufacture of environmental monitor tubes and subsequent disassembly and analysis; novel network research, using local operating network (LON) technology to control the robot motors; cloth handling in a much larger manufacturing production line.

Cylindrical

This geometry (Fig. 5) was adopted by the most successful of the robot manufacturers in the laboratory automation market. The Zymate robotic system has proved to be very amenable to most laboratory applications and is installed in thousands of laboratories world-wide. BP has used the Zymark robot in its laboratories in both the USA and Britain. An example is the integration of a Zymark robot in a gas chromatography (GC) laboratory with the chromatographic data system, to allow the analysis of petroleum fractions.

Future

As the world-wide requirements for automation increases, driven particularly by tightening regulatory requirements and

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safety, the number of commercial systems available will increase to fulfil the market. More systems addressing particular analytical problems (*i.e.*, dedicated automation) will become available, and user will begin to develop automatic methods, rather than trying to replace manual methods using automation. Where flexible automation is required, new robotic systems will offer improved reliability and easier operator control.

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THE ROYAL SOCIETY OF CHEMISTRY: ANALYTICAL DIVISION
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INTERNATIONAL SYMPOSIUM ON ELECTROANALYSIS IN BIOMEDICAL, ENVIRONMENTAL AND INDUSTRIAL SCIENCES

April 20–23, 1993, Loughborough

The programme of this conference, which will be held in Loughborough University of Technology, embraces all aspects of electroanalysis related to the theme areas, especially the development, mechanistic studies and applications of sensors. A major aspect of the meeting will be recent significant developments in the fields of process and waste management. The sessions will be introduced by eminent invited lecturers, and supplemented by contributed papers.

Invited lectures will be presented by M. R. Smyth (Dublin), P. N. Bartlett (Southampton), J. D. R. Thomas (Cardiff), P. R. Fielden (UMIST), A. K. Covington (Newcastle upon Tyne), R. Briggs (City University), C. M. G. van den Berg (Liverpool), A. R. Hillman (Leicester), S. Bruckenstein (New York) and a speaker from British Nuclear Fuels Ltd.

The cost of registration will be £150.00 for members and £200.00 for non-members, with a single-day fee of £60.00 (members) or £75.00 (non-members). Accommodation for the full symposium (inclusive of meals) will cost £170.00 and the single night cost (including lunch, dinner and breakfast) will be £60.00, except for Monday 19 and Friday 23, when bed and breakfast will cost £20.00 per night.

For further information, or to register, please contact Dr. A. G. Fogg, Department of Chemistry, Loughborough University of Technology, Loughborough, Leicestershire LE11 3TU.