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SIMULTANEOUS OPERATION OF A POLYCHROMATOR AND MONOCHROMATOR ON A SINGLE-SOURCE INDUCTIVELY COUPLED PLASMA EMISSION SYSTEM

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ABSTRACT

Inductively coupled plasma atomic emission spectroscopy (ICP-AES) is a popular analytical technique for routine multielement analysis. An argon plasma, which is sustained by a radio-frequency field, serves as the atomization excitation source for emission spectroscopy. With commercial instrumentation the atomic emission is monitored with either a direct reader (polychromator) or a scanning monochromator. The topic of this paper is the development of software for simultaneous operation of both the polychromator and the monochrmator with a single plasma source. The hardware for this system includes an LSI-11/23 as the main computer with satellite FALCON SBC-11/21 single-board computers for hardware control and data acquisition. TSX-Plus, a multi-user operating system, is being employed for software development on the LSI-11/23.

INTRODUCTION

Inductively coupled plasma atomic emission spectroscopy (ICP-AES) has become one of the most popular techniques for routine multielement analysis of solutions in the past five years. The combination of good sensitivity, multielement capability, determination of refractory elements (e.g., P, B, W, Zr, and U), a large linear dynamic range, and reduction in chemical interferences has resulted in the recent popularity of ICP-AES.

An electrical discharge in argon gas sustained by the application of a radio-frequency electric field is known as an ICP and serves as the atomization and excitation source for emission spectroscopy. This discharge causes the flowing argon to become electrically conductive and heats the gas to a high temperature with a maximum somewhere between 8000 and 14,000°C. When a chemical sample is heated sufficiently, the element of interest is dissociated from its compounds, and some of the atoms are excited, resulting in emission of radiation at specific wavelengths. The intensity of radiation is easily measured and is proportional to the concentration of elements in the sample.

Most multielement systems are designed around a polychromator (Fig. 1). Light from the plasma enters through a single slit and is diffracted by the grating; then the dispersed radiation passes through a number of fixed exit slits, each corresponding to a different wavelength. A drawback to the polychromator (besides cost) is that if the user or manufacturer has chosen the analytical wavelengths (which define the fixed exit slit positions) and it is later decided to add wavelengths for other elements or change some wavelengths because of spectral interferences, the instrument must be modified — a process that is complex, time-consuming, and expensive.

An alternative multielement system is designed around a computer-controlled scanning monochromator. Unlike a polychromator a monochromator has only one exit slit. The different wavelengths are focused on the exit slit by rotating the grating on its axis (Fig. 1). By interfacing the grating rotation with a computer-controlled stepping motor, the system can be programmed to search for many different analytical wavelengths; when the limited set of spectral lines observable in a polychromator become restrictive, employing a scanning monochromator may be preferable.

Because our laboratory must provide analytical support for a large variety of research programs, our instrumental prerequisites included a versatile system that was conveniently adaptable to a variety of sample types and nonrestrictive in the number of elements observable. Therefore, we chose to employ both a polychromator and a monochromator with a single plasma source (Fig. 1). This instrument configuration offers the speed, accuracy, and reliability of a direct reader and also allows the necessary flexibility for unusual or special analysis requests.

Although the ICP system we purchased included both a direct reader and a computer-controlled scanning monochromator, the two spectrometers could only be used independently. This procedure resulted in double analyses for any set of samples requiring both the polychromator and monochromator. However, simultaneous operation of both spectrometers would not

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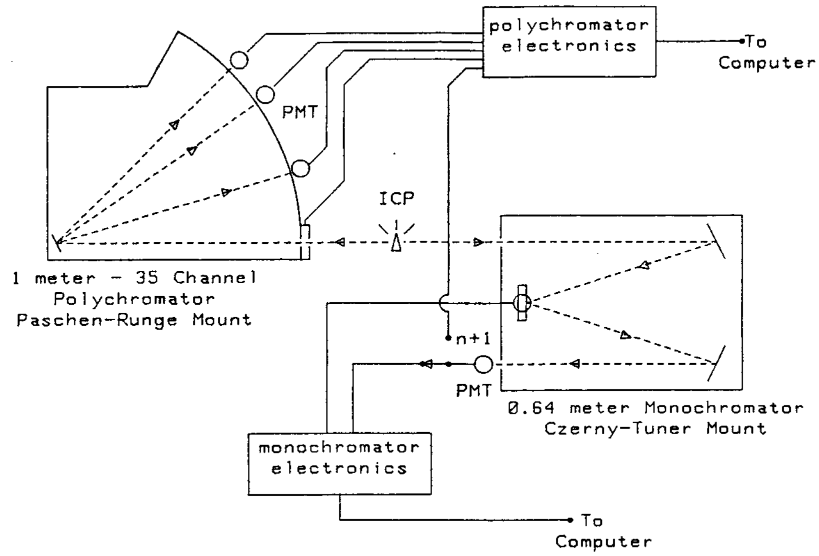

Fig. 1. ORNL ICP optical system.

only require a single analysis, which would result in timesaving, but would also offer other advantages.

The method of sample introduction to the plasma requires generation of an aerosol from the sample solution and results in less than 10% utilization of the available solution. A major portion of the sample is lost to waste. Since the sample is introduced at a constant rate, a decrease in analysis time requires less sample volume.

A major factor affecting the accuracy of ICP analysis is the spectral interference of one element upon another at a given wavelength (interelement interference), which may result in either a shift in spectral background or a direct overlap of spectral lines. A shift in background may be eliminated by doing off-peak background correction, which consists of measuring the background intensity near the spectral line of interest and subtracting the background intensity from the spectral line intensity. However, direct line overlap by an interfering species with the spectral line of interest requires calibrating the instrument for each interference. This calibration for interference involves running a series of solutions with increasing concentrations of the interferent and then determining the concentration of interferent vs apparent concentration of the interfered element correction curve. Once these curves are determined, the contribution caused by an interference may be calculated and subtracted from the apparent interfered element concentration. This method of correction implies that the concentration of the interfering element must be determinable at the time of analysis.

Certain conditions may present a problem with interference corrections when only a polychromator is employed. If the concentration of an interfering element is high enough to result in a signal that exceeds the electronic range or the interfering species is not available in the polychromator array, the required correction cannot be determined. Simultaneous operation of a monochromator with the polychromator offers a viable solution to both problems. With the monochromator, one may employ wavelengths not available with the polychromator or use weaker lines for species off scale and thus determine the required corrections.

ANALYTICAL INSTRUMENTATION

The spectrometers were obtained from Instruments SA, Inc. (Metuchen, NJ) and included a JY-48 direct reading polychromator and an HR-640 0.64-meter computer-controlled scanning monochromator. The plasma system was a Plasma-Therm ICP-2500 (Plasma-Therm, Inc.) and included the HFP-2500D rf generator with APCS-1 automatic power control. The generator operates at 27.12 MHz and is crystal controlled with a maximum output power of 2500 W.

COMPUTER HARDWARE

The ICP computer includes an LSI-11/23 with 256-Kbyte memory as the main computer. A Tektronix 4025 graphics terminal is employed as the console, and an ADM-3A (Lear Siegler, Inc.) with a Retro-graphics Option (Digital Engineering, Inc.) is available as an auxiliary terminal. Hardcopy is obtained with an LA120 DECwriter installed as a line printer. The mass storage devices include (1) a DSD880/30 (Data Systems Design, Inc.) 30-Mbyte Winchester with a single-sided double density floppy drive and (2) a DEC RX02 dual floppy drive. A Bell 212A modem is employed for communication with a DEC System 10, which maintains our data management system, and a Hewlett Packard 9835A, used for hardcopy graphics.

For hardware control and data acquisition, we plan to employ the DEC FALCON SBC-11/21 single-board computer in a configuration shown in Fig. 2. Communication between the LSI-11/23 and the FALCON will be by DLV11-J serial I/O lines and the on-board serial I/O ports on the FALCON.

An attractive feature of the computer system shown in Fig. 2 is the expandability that is gained by employing the FALCONs for hardware control and data acquisition. By employing a multi-user operating system such as TSX-Plus (S&H Computers, Inc.) on the LSI-11/23 and handling real-time applications with the FALCON computers and data processing on the LSI/11/23, subsequent addition of analytical instrumentation involves simply adding another FALCON to the system, which is depicted by the second ICP in Fig. 2.

TSX-PLUS OR RSX-11M

TSX-Plus, a multi-user operating system for PDP-11 and LSI-11 computers, offers most of the features available with the DEC RSX-11M at a lower cost and retains the functionality of the DEC RT-11 operating system for up to twenty concurrent jobs. TSX-Plus interfaces with standard RT-11 device handlers, supports RT-11 utility programs (PIP, DUP, DIR, etc.) and CCL commands (COMPILE, EXECUTE, etc.) and provides a super set of RT-11 keyboard commands. If one is already familiar with RT-11, the change to TSX-Plus requires minimum effort.

The software we received with our ICP was written for RT-11, and we plan to incorporate as much of this software as possible into the system for simultaneous operation of the two spectrometers. To employ this software with RSX-11M would have required a major revision of the programs. Modifications necessary to operate with TSX-Plus were minor; for example, only one short subroutine and a call to that subroutine in the main program were required to put the polychromator into operation.

SOFTWARE STRUCTURE FOR ORNL ICP

The general software structure for simultaneous operation of two spectrometers is presented in Fig. 3. A supervisory program running under TSX-Plus will provide the interface between the user and the instrumentation. This main program will handle the input of analytical conditions, store data, maintain calibration and correction curves, handle all calculations, and output the analytical results. The supervisory program will also initiate the instrumental operation by passing two- or three-character ASCII commands to the FALCON single-board computers.

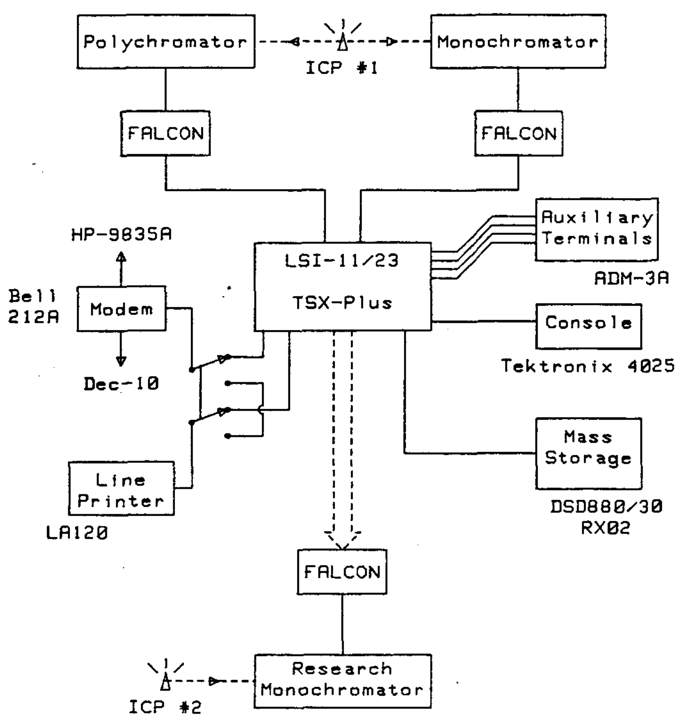

Fig. 2. Computer network for ORNL ICP laboratory.

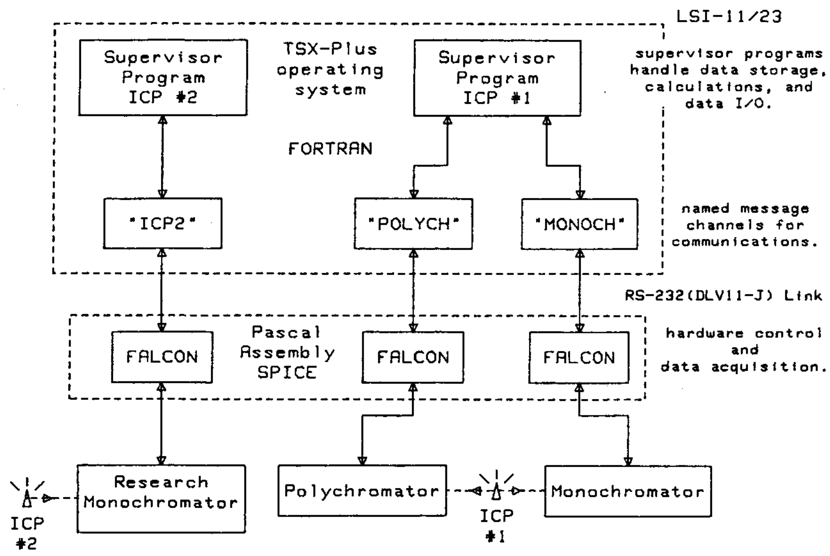

Fig. 3. Software structure for ORNL ICP laboratory.

The ASCII command will utilize the message communication facility of TSX-Plus. This job communication facility employs message channels (separate from I/O channels), having one- to six-character names that are used by both the sending and receiving programs to identify the channel. A message channel accepts an ASCII message from a sending program, stores the message in a queue associated with the channel, and delivers the message to a receiving program that requests a message from the channel.

Therefore, associated with each FALCON will be a communication program that runs as a detached job (a job that runs concurrently with other jobs under TSX-Plus but cannot communicate with a terminal) on the LSI-11/23 (Fig. 4). The detached communication program will provide several functions, including receiving queued messages from the supervisory program followed by output of the message to the FALCON via a serial I/O line, receiving data from the FALCON on the same I/O line, and queuing the data on a message channel for the supervisory program to receive.

Finally, the FALCON single-board computer will handle hardware control and data acquisition for the spectrometers. A simplified flow diagram of the required functions for the polychromator FALCON can be seen in Fig. 5 and the monochromator FALCON in Fig. 6. Routines on the FALCONs will be short and simple, and most decisions will be made by the supervisory program on the LSI-11/23.

At this time we have not decided which programming language to employ on the FALCON single-board computers. The options we are considering include the

Detached Jobs under TSX-Plus

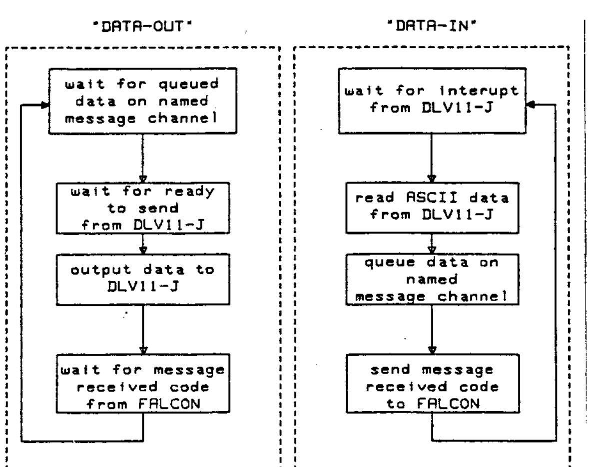

Fig. 4. Communication of LSI-11/23 with FALCON SBC-11/21.

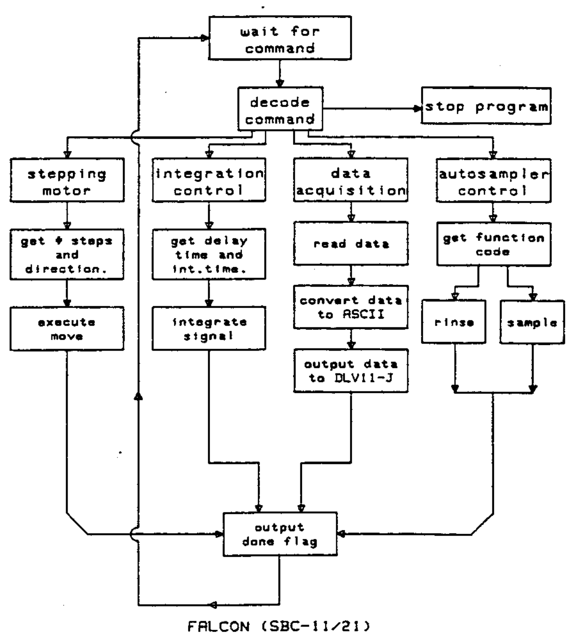

Fig. 5. Hardware control and data acquisition for polychromator.