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Project Summary

The Standard Calibration Instrument Automation System for the Atomic Absorption Spectrophotometer

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The Environmental Monitoring and Support Laboratory-Cincinnati (EMSL-Cincinnati) has, as part of its mission, the development of enhanced data acquisition and reduction systems which provide high quality analytical data from environmental samples. The standard calibration instrument automation system for the flameless atomic absorption instrument supports this mission in the area of metals analysis. This paper summarizes the capabilities of the system and directs the reader to other documents which fully explain the related hardware, software, and user environments.

This Project Summary was developed by EPA's Environmental Monitoring and Support Laboratory, Cincinnati, OH, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

The standard calibration instrument automation system for the flameless atomic absorption instrument is part of the EPA laboratory automation system.[1] This system utilizes a Data General NOVA 840 minicomputer with an extended BASIC Language which is modified to allow for real time data acquisition in a multiuser, time sharing environment.[2] The instruments involved are commercially available and may vary considerably in relation to their output.

The instrument standard calibration (ISC) system can be adapted to collect and process data from a variety of instruments including single or double beam systems. with or without background correction, and sequenced through manual injections or in concert with an automatic sample device.[2] Interfacing with microprocessor based "intelligent" instruments has also been implemented with this system.[3] The system provides for the following functions:

- calibration using regression or interpolation,
- plotting of calibration curves,
- determination of concentrations,
- quality control assessments in real time for spiked samples, duplicate samples, laboratory control standards, laboratory reagent blanks, and instrument check standards,
- compensation for dilution,
- reagent blank subtraction,
- editing of suspect results,
- remeasurement of questionable samples,
- printing of progress reports and final reports,
- bidirectional communication with the EPA national Sample File Control data base,

- checking the instrument and interface hardware, and
- plotting of raw data when applicable.

The system can also operate in an unattended mode when an automatic sampling device is available.

The system has been successfully implemented on a number of direct asperation instruments, and may be adapted to TOC and UVVIS applications.

The following paragraphs describe the major features of the system and reference the three volumes of a complete report which can be obtained from the EPA.[4,5,6]

Nature of the System

The ISC system is a command structured single analyte standard calibration system. In concert with an appropriately interfaced Flameless AA instrument, the ISC system may be used for measuring metal concentrations in environmental samples. It has discrete measure/edit capabilities or it can be used to analyze a set of samples automatically. It has applications in both research and production environments and can be used in conjunction with the EPA Sample File Control System[7] or as a stand-alone automation system.

The ISC system may be adapted to other instrument systems which produce a response which varies linearly with the presence of an analyte (as in total organic carbon analyses).

Data Acquisition

The ISC system may easily be adapted to acquire instrument response data of the following types: 1) fast (60 Hz) analog signals from single or double beam instruments, 2) serial ASCII character data transmitted through EIA-RS232C compatible interfaces, or 3) manually entered data via a terminal keyboard.

When the analog-interfaced instruments are used, the system utilizes analog to digital (A/D) converters and related system software[2] to translate the incoming signal into a series of digitized numbers. This data array is then processed through a software module which quantifies the peak absorbance value of the measurement. Figure 1 shows an example plot of the peak absorbance data array.

Many newer instruments are capable of accurately producing a fully reduced absorbance value and outputting this result via a string of ASCII characters.

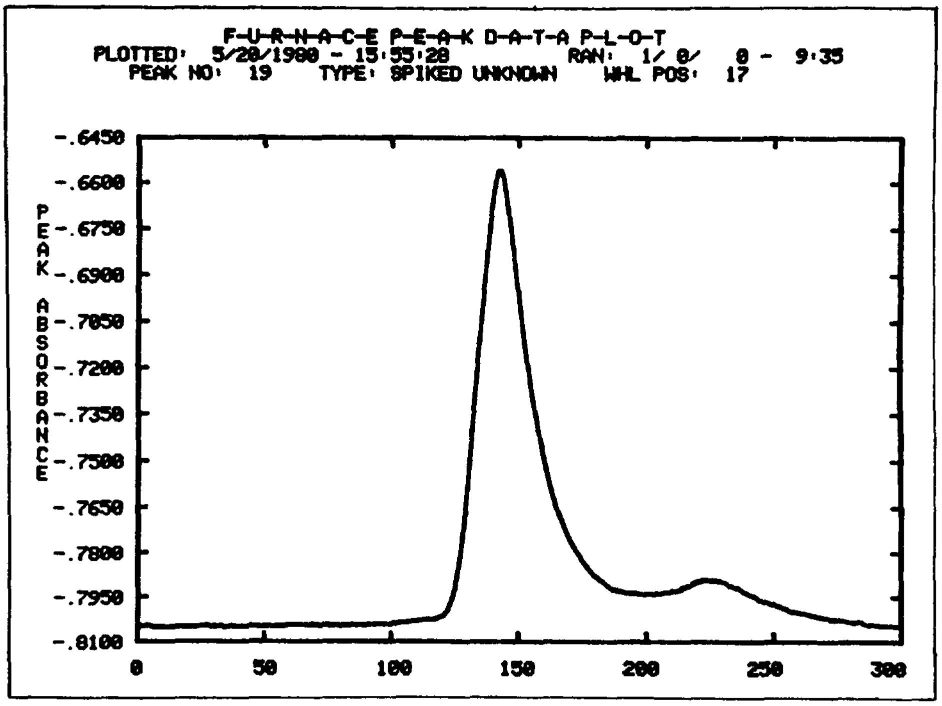

Figure 1. Example peak plot.

These instruments require interface hardware which can receive and store the ASCII characters. Ths ISC system utilizes an ASCII Character Buffer interface[3] for this purpose. Instrument data may be buffered in this device for subsequent use by the data acquisition module. The software "reads in" a string of data and extracts the absorbance value contained within.

In situations where the interface hardware and software are not easily available, the ISC system may be used in the manual data entry mode. After keyboard data entry, the analyst is still able to use the computational, quality assurance, and reporting capabilities of the system.

Calibration and Plotting

The system is capable of accumulating absorbance measurements on instrument calibration standards and generating regression equations relating absorbance to known concentration. Any number of standards may be identified in the sample wheel pattern and their order is unrestricted. As many as six replicate measurements may be made on a given calibration standard and all replicates will be used in the subsequent regression. The system will automatically perform the regression on the standards in an automatic sequence and it will produce one or more equations during the process. Forced-zero linear, regular linear, quadratic, and cubic equations and their corresponding fitting errors will be formed for a given set of standards unless the number of standards prohibits higher order equations.

After a successful calibration is formed, all subsequent absorbance measurements will result in the display of sample concentration using the current computational mode.

The user may change the computational mode at any time without necessitating a recalibration. The user will also have the opportunity to edit poor standard measurements and recalibrate the remaining standards during post-run processing.

The ISC system is configured in such a way as to allow the user to view the calibrations curves after regression has been performed. Figure 2 illustrates an example calibration curve. These curves are generated to an appropriate graphics terminal at the request of the analyst. An automatic run will continue during the curve generation process. At the completion of the run, the analyst may use the data gained from the plots to selectively edit the standard data and recalibrate, or he may select another computational mode to calculate concentrations and generate an updated run status report.

Quality Control

Quality control assessments are provided seconds after the related samples are measured. Spiked samples, duplicate samples, laboratory control standards, and reagent blanks are all automatically evaluated against limits provided by the Sample File Control System (as described below) or by the analyst. Instrument check standards are automatically assessed against limits provided by the analyst.

In addition, all non-standard samples receiving multiple replicate measurements will cause the ISC system to supply a mean and standard deviation of the computed concentrations. This will assist greatly in those cases where precision is a problem.

Compensation for Dilution

Dilution information is taken into account so that the system reports true concentrations. Samples which exhibit instrument responses above the highest calibration standard are flagged as off scale and the analyst is given the opportunity to dilute the sample and remeasure it. Sample information may be easily modified to purge old absorbance data and install new dilution volumes prior to the remeasurement of the sample.

Spike volumes and concentrations are considered along with dilution volumes in order to compute concentrations and recoveries with a high degree of integrity. A special feature is available for spiked samples so that negative spikes may be made. (This is sometimes called spiking by successive dilution and should not be confused with the Method of Standard Additions.[8])

Reports

The ISC system produces a variety of reports at various stages of the run. Before the run begins, a wheel pattern report may be generated. During the run, an on line report is generated to the user terminal after each sample is measured. After regression is performed, a calibration report is generated to the selected output device(s). After a run has been completed a status report is generated to the selected output devices. A "final report" is also available to the analyst after all post processing is completed as shown in Figure 3.

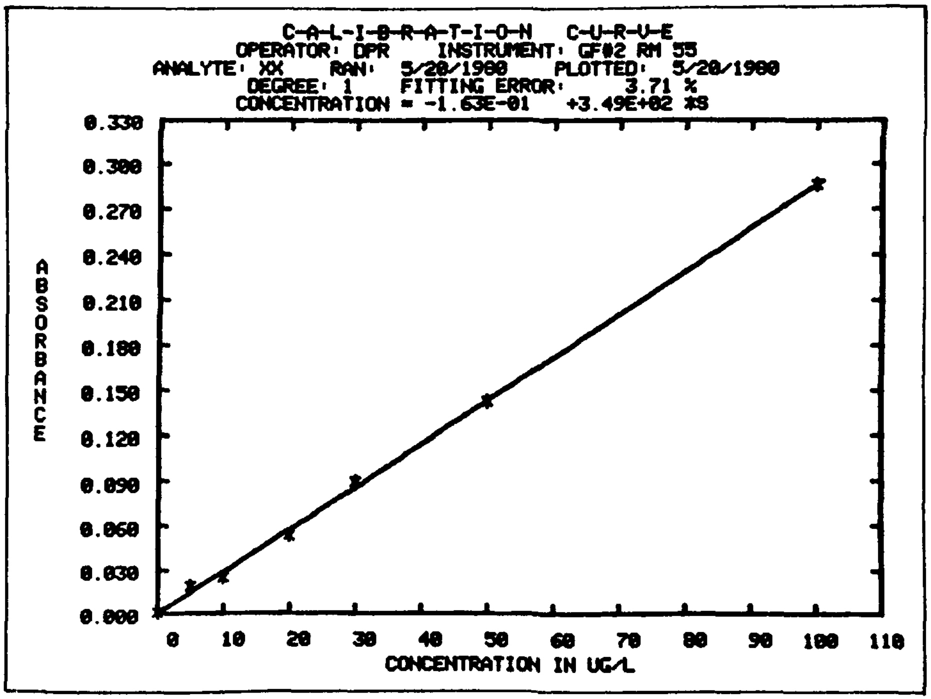

Figure 2. *Example calibration curve.*

FLAMELESS AA STANDARD CALIBRATION FINAL REPORT

INSTRUMENT: PE5000 RM464 DATE: 5/ 7/1981 14:28
ANALYST: TLR ANALYTE: CD
UNITS: MG/L RUN FILE NAME: 1CD75.RF
PARMETH CODE: 2134001 RF CREATED: 05/07/81 13:34

WHEEL POS	SAMPLE IDENTIFIER	SAMPLE CONCENTRATION	KNOWN SPIKE OR CONTROL	VIOLATION LIMIT	QC
9	LRB	+3.612E-03			OK
10	81-06542 LD1	+.83281			
11	81-06543	+.424872			
12	81-06544	+.101642			
13	81-06545	+1.171E-02			
14	81-06546	+3.612E-03			
15	81-06547	+3.612E-03			
16	81-06548	+3.612E-03			
17	81-06549	+1.171E-02			
18	81-06550	+4.422E-02			
19	81-06551 LS0	+8.517E-02			
20	81-06551 LSF	+.539918	+.472135		OK
21	81-06542 LD2	+.860977			OK

Figure 3. *Example final report.*

Sample File Control Interaction

This automation system can communicate bidirectionally with the EPA's laboratory management information system, Sample File Control. It can automatically accept a "run file" from the Sample File Control system containing a list of the samples which need to be measured, together with the quality control limits currently in effect for each unique combination of parameter, method, preparation procedure, and environmental source.

The system can also send back to the Sample File Control computer a "run results file" containing the measured concentration of the samples and related data.

Documentation

The ISC system is currently operating reliably in a number of EPA Laboratories but the conceptual framework of the system is available to any laboratory through the system specifications document.[4] This document contains project definition, functional requirements, and system design information. A second document is available as a guide to system usage.[5] This manual is packed with figures and examples to aid the user in learning about or operating the system. Finally, a detailed program description document is available.[6] This document contains a flowchart, a variable description table, a program listing, and a summary description for each of the twelve major BASIC programs in the system. These documents do not fully describe the instrument interface aspects of the system.[2,3]

Conclusion

The ISC system is a flexible approach to environmental analysis. It facilitates increased production while performing on-line quality assurance tests. It is user oriented in that is minimizes superfluous or redundant keyboard entry. And it is laboratory conscious in that it develops "clean" final reports and supports the interface with the SFC data base management system. This system, along with others developed at the EMSL-Cincinnati, could extend laboratory budgets through increased production, and improve laboratory evaluations through increased production and increased data quality.

References

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The complete reports entitled:
"The Standard Calibration Instrument Automation System for the Atomic Absorption Spectrophotometer,"
Part I - Functional Specifications (Order No. PB 82-187 832; Cost: $9.00)
Part II - User's Guide (Order No. PB 82-187 840; Cost: $10.50)
Part III - Program Documentation (Order No. PB 82-187 857; Cost: $19.50)
The above reports will be available only from: (prices are subject to change)
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