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EFFECT OF MICROPROCESSORS ON TRADITIONAL CHROMATOGRAPHY INSTRUMENTATION

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SUMMARY

This paper reviews the various design approaches using microprocessors for chromatography automation with emphasis on a new multi-channel data reduction system using a unique architectural design. This new architecture uses a distributed network of microprocessors organized in a hierarchical scheme. This new advance in chromatography automation is the result of recent technological achievements in microprocessor development. The availability of low-cost, powerful microcomputer elements has made it possible to assign a microprocessor to each chromatograph. The result is a major improvement in performance by providing at the chromatograph a control station and an input/output terminal. This allows the chromatographer complete access to a powerful computer even though he can still distribute the cost of this data reduction system over a large number of his chromatographs.

Possibilities of major improvements in analytical performance using microprocessors to replace analog control circuits are discussed. Advances yet to be made in this direction can lead to more reliable analytical results with less involvement of the analytical chemist.

INTRODUCTION

To a large degree, the design and performance of analytical instruments have been affected more by advances made in electronics than any other technology. Recent advances in microprocessor design have resulted in a technological revolution comparable in magnitude to that caused by the availability of transistors in the sixties. Analytical instruments incorporating microprocessors in their designs have achieved significantly improved performance at reduced costs[1–3]. With over 150,000 chromatographs in use today, gas and liquid chromatography are the two most widely used techniques in quantitative analytical chemistry[4]. Design of new chromatography equipment has been one of the most active segments of analytical instrumentation. Recent surveys[5,6] have identified over 40 major manufacturers of gas and liquid chromatography equipment, with over 150 different models available.

In recent years, automation of chromatography laboratories has become an economic necessity due to increasing labor costs and greatly increased sample throughput. Automatic sample injectors allow unattended operation and automatic data reduction systems provide quantitative results more rapidly and with much higher precision and accuracy than manual techniques. Total automation of the chromatographic analysis includes sample handling, control of analyzer operating conditions, data reduction and the generation of a final analytical report.

This paper reviews the various configurations of chromatography automation using microprocessor technology and describes a new architectural approach for the automation of many chromatographs simultaneously.

SYSTEM CONFIGURATIONS

The basic equipment in a chromatography laboratory consists of a chromatograph and a recorder. As the sample load and the demands for accuracy and precision increase, automation equipment is added. The design of most chromatographs allows automatic repetitive analyses if samples can be injected at regular intervals by an automatic injector. Data reduction for the quantitation of each sample component is a time consuming activity. This data processing task is most ideally implemented by microprocessors. Typical automatic data reduction systems provide most of the following capabilities:

Analog to digital conversion of the detector output
Automatic peak detection, area integration, and baseline correction
Calculation of component concentrations
Report of analytical results
Control of autosamplers, valves, gradient generators or recorders

The choice of automatic data reduction equipment typically depends on the number of chromatographs in the laboratory. For laboratories with one or two chromatographs, a single-channel computing integrator is preferred. But laboratories with a number of chromatographs can benefit from a multi-channel data system. The trade-offs between these two choices are summarized in Table I.

TABLE I

TRADE-OFFS BETWEEN SINGLE-CHANNEL AND MULTI-CHANNEL COMPUTING INTEGRATORS

	Advantages	*Disadvantages*
Single-channel	Low entry cost Dedicated to chromatographs Easily relocatable Flexible Highly reliable due to simplicity Optimized for one channel	Higher cost per channel in stand-alone design Limited memory capacity
Multi-channel	Low cost per channel Supported by larger, more sophisticated computer Optimized for 4+ channels Larger memory capacity	High initial investment

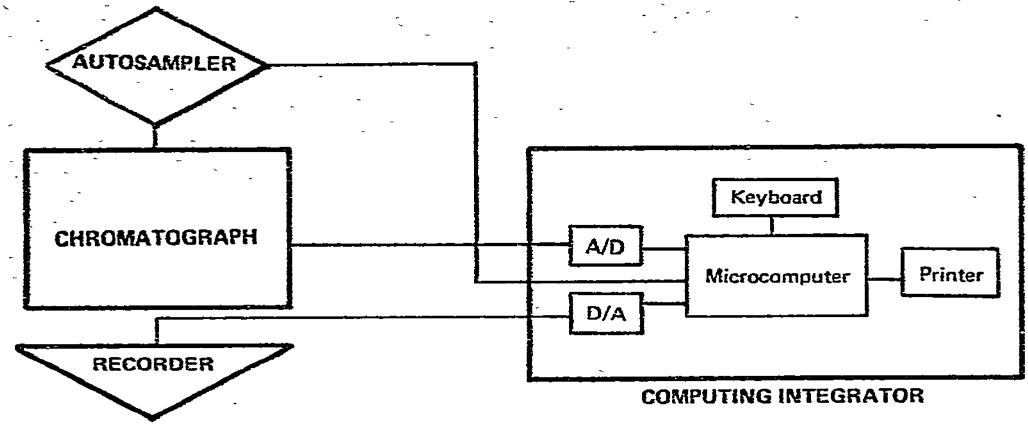

Fig. 1. Chromatography automation using a single-channel computing integrator.

The block diagram in Fig. 1 illustrates the use of a single-channel computing integrator for automation[7]. Typical designs use a single microprocessor to perform the tasks of peak detection, area integration, baseline correction, calculation of component concentrations, and the reporting of analytical results. In addition to these basic data reduction tasks, the computing integrator can interact with the chromatographic system by generating control signals at specified time intervals and by providing an attenuator controlled replicate analog record. Microprocessor-based single-channel computing integrators now provide data reduction capabilities that can automate calculation procedures used by most laboratories.

The implementation shown in Fig. 1 results in two separate records, a printed report and a chromatogram. The desire to combine these records in a single report has led to a new innovative application of microprocessors[8]. In addition to chromatographic data processing, significantly more computing power is available from current microprocessors to provide complete logic control of thermal printer/plotter type devices at very nominal costs. Thus, the technological evolution of microprocessors and thermal printing devices has led to the development of the "Printer/Plotter" used in chromatography automation for both the plotting of the analog chromatogram and the printing of the final component concentration report on a single sheet of paper.

A more recent development in the application of microprocessor technology has led to the complete automation of the chromatographic analysis function[3]. The chromatograph, with its many complex control functions (flow, temperature, detection, etc.), is an ideal candidate for microprocessor implementation. The block diagram in Fig. 2 illustrates an approach for total automation of sample handling, analyzer control, data reduction and analytical reports. By integrating the many control circuits and the data processing activities under one microprocessor, considerable economy can be achieved. For example, the front panel switches, dials, and meters necessary to select and monitor operating conditions are virtually eliminated. Operating parameters are entered by the use of the same keyboard control and digital readout shared by the data processor. For applications where a new chromatograph is a necessary investment, the integrated approach offers total automation at a cost comparable to that of a separate chromatograph, computing integrator and recorder.

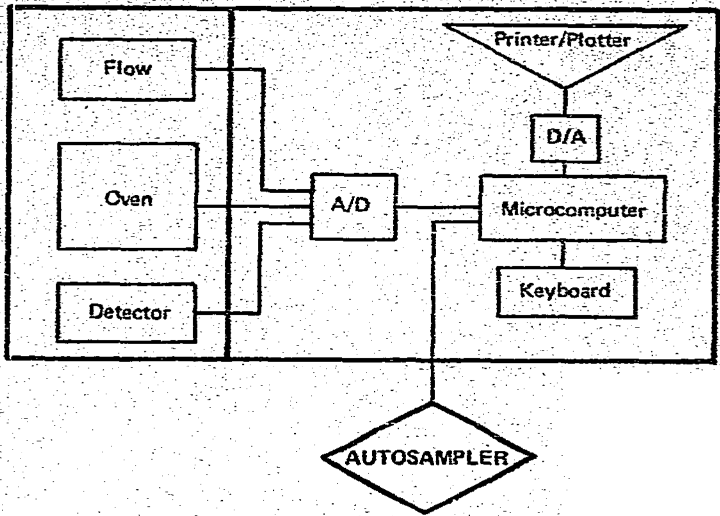

Fig. 2. Integrated chromatograph block diagram.

The combination of analyzer operation and result calculation under a single microprocessor opens up the possibilities of performance improvement. Traditional analog control techniques are being replaced by digital techniques capable of better accuracy and precision. The implementation of functional control of the chromatographic analysis by an intelligent microprocessor allows operations such as:

Automatic sequential analysis of different sample types using different analysis parameters
Repetitive analysis of selected samples
Detection of malfunctions caused by equipment failure or operator errors
Optimization of operating parameters
Evaluation of analytical results

TABLE II

COMPARISON OF SINGLE-CHANNEL AUTOMATION APPROACHES

	Advantages	Disadvantages
Single-channel integrator and chromatograph	Choice of most suitable chromatograph Easily upgradable Allows flexible equipment mix in laboratory Redundancy Reliability	Two separate records Multiple vendors May not be cost-effective if suitable integrated system is available
Integrated system	For appropriate application, most cost-effective approach Ideal for routine use with large sample throughput Single record for results Single vendor	Not easily upgradable Lacks flexibility for non-routine operation No redundancy or back-up capability Hidden controls High cost of automation for existing laboratories

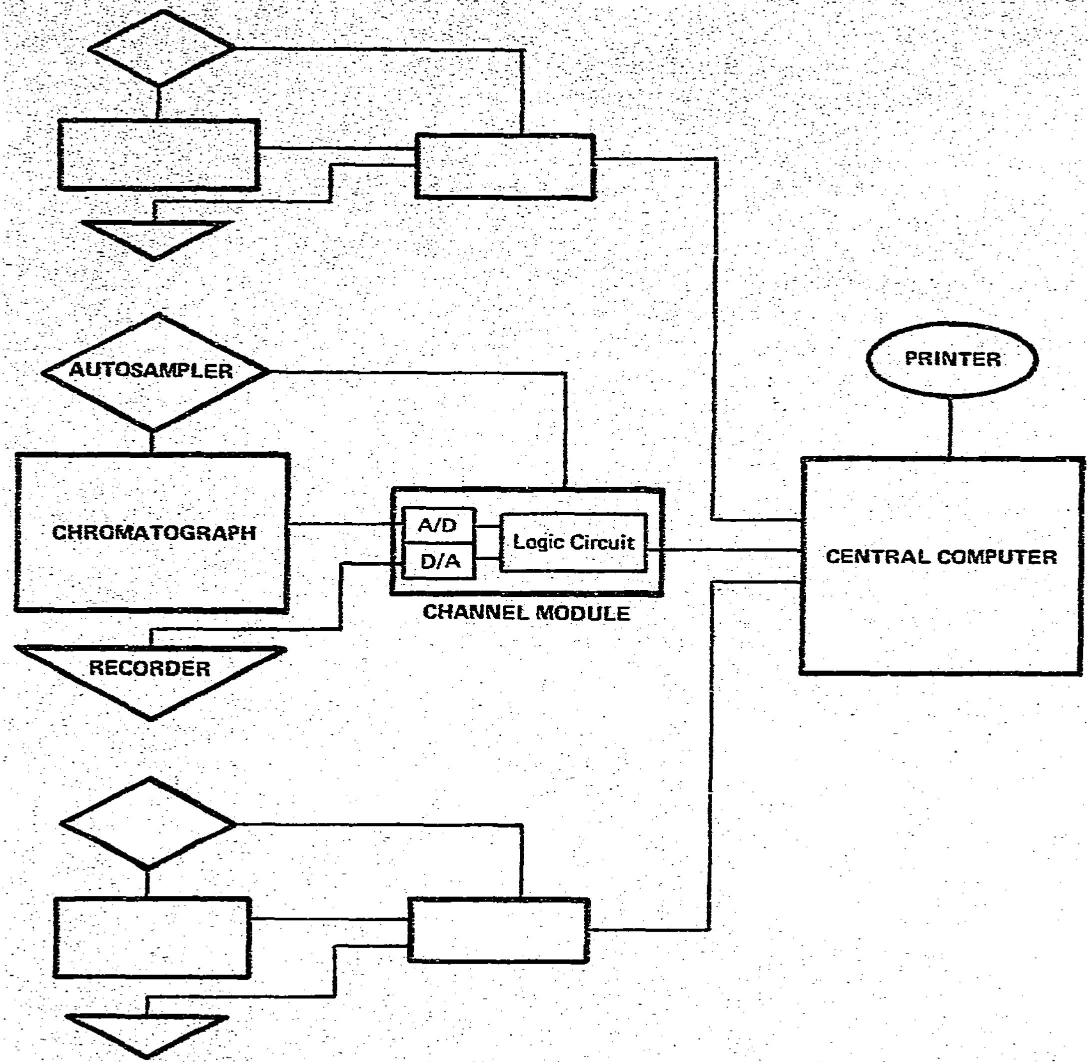

Fig. 3. Chromatography automation using a multi-channel chromatography data system.

A comparison of several approaches to single-channel automation is summarized in Table II. The alternative most appropriate to any individual chromatographer is based upon the following considerations:

The applications problem to be solved (*e.g.*, quality control, methods development, research) and the skill of the operator
Reliability
Serviceability
Cost/performance
Economics (capital investment, return on investment, obsolescence and upgradability)

Fig. 3 illustrates a configuration using a multi-channel data system suitable for laboratories with a number of chromatographs. The detector signal from each chromatograph is acquired by an interface module whose major function is to convert the analog signal into digital form. This digital signal is transmitted and then processed by a central computer, which has sufficient power to automate a number of chromatographs. The interface modules can provide control signals at selected time intervals to control autosampler, valves, gradient generators or recorders. An isolated recorder output is available to record chromatograms.

The multi-channel configuration allows the sharing of the central computer by more than one chromatograph, resulting in a lower cost per channel. The central computer may be a powerful minicomputer with sufficient memory capacity to support complete analytical reports with peaks identified by component names. The operating instructions can be written in a high-level language and may allow modifications by the chromatographer. Due to the high cost of the minicomputer and its as-

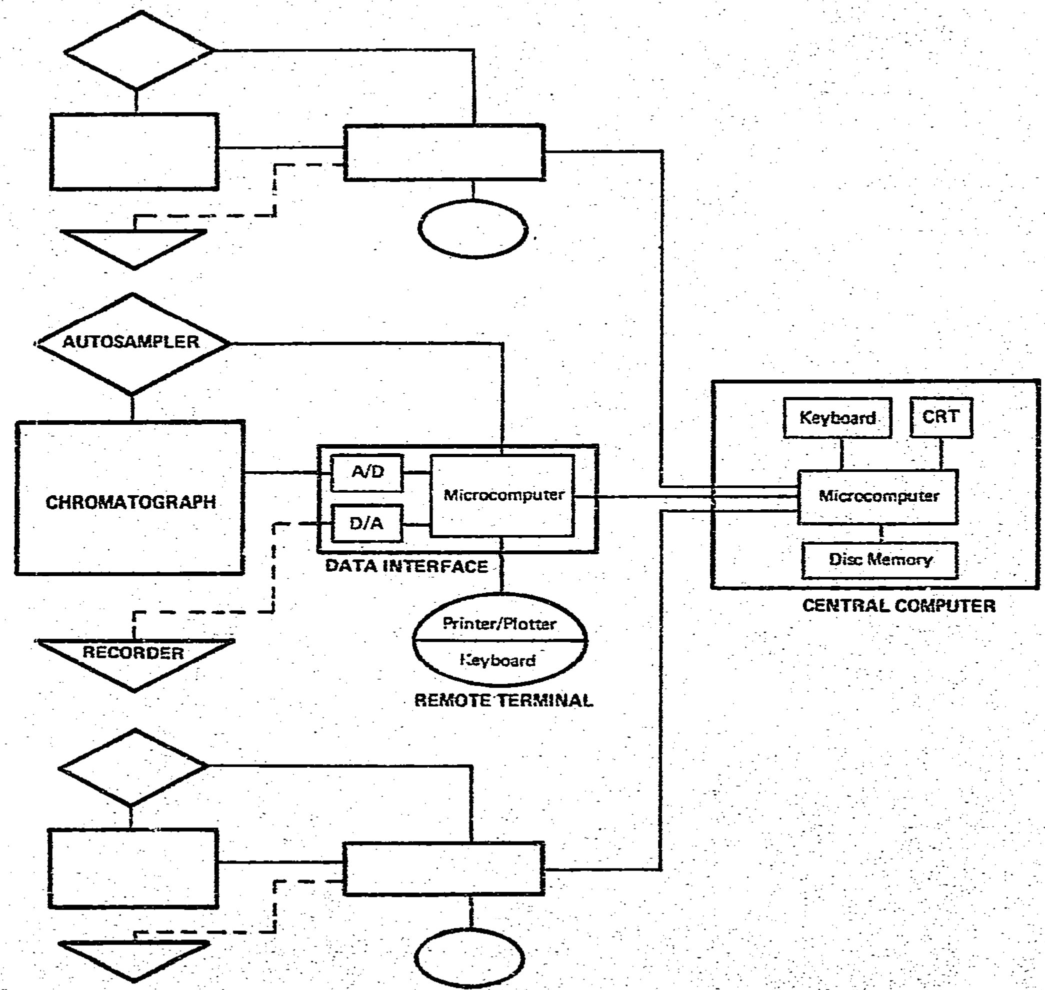

Fig. 4. Hierarchical chromatography data system.

sociated hardware, however, large data systems are easily justified only for laboratories with many chromatographs.

A NEW SYSTEM ARCHITECTURE

The approaches summarized above represent an evolution of chromatographic automation that has been influenced by available technology, market acceptance and design experience. Each has advantages and disadvantages in various applications. The design of any new system should maximize the advantages of most importance to chromatographers, which are:

- Entry cost and cost per chromatograph
- Best performance/cost for each module
- Stand-alone, dedicated operation
- Single, complete record of analytical results
- Reliability
- Flexibility

A new system architecture developed by Spectra-Physics and illustrated in Fig. 4 maximizes these advantages and yet provides the chromatographer with a wide selection of hardware configurations best suited for his individual requirements. The new architecture takes advantage of the capabilities of low-cost, high-power microprocessors for data processing and instrumentation control to construct an efficient, flexible, multi-channel chromatographic data reduction system. The new system uses a distributed network of microprocessors connected in a hierarchy of remote modules supported by a central processor. The built-in intelligence of each remote module provides an efficient but low-cost building block that can be repeated at each chromatograph. The dedicated microcomputer at each chromatograph is supported by a large amount of computation power and logic decision capabilities at a central computer. This hierarchical design provides each chromatograph with a dedicated module that is capable of data reduction, analyzer control, and input/output operations. The microcomputer at the chromatograph communicates directly with the central computer through a three-wire data transmission loop and has access to the calculation procedures, report generating capabilities, and memory capacity of the central computer. The pooling of the data reduction tasks allows a powerful central computer while still maintaining a low entry cost and low cost per chromatograph. The major benefits provided by this approach are summarized below:

- High performance/cost, expandable system supported by a sophisticated computer architecture
- Low cost per channel, additional automation provided by adding data interfaces
- Allows chromatographer to keep or buy the most appropriate chromatograph
- Back-up provided by separate recorder output
- Printer/plotter for each chromatograph to provide a single analytical record
- Ability for each channel to control an autosampler and chromatograph
- Power-fail protection using standard automobile batteries

The new system architecture uses four basic modules interconnected as shown in Fig. 4, viz. data interfaces, remote terminals, central processor, and disc memory.

As a stand-alone module at each chromatograph, the data interface with built-in microcomputer performs the following functions:

Converts analog chromatographic signals to digital format
Communicates with central processor by sending data and receiving calculated results
Generates control signals for actuating autosamplers, valves and gradient generators
Provides an isolated recorder output with attenuator controls

A remote terminal consisting of a thermal printer/plotter and alpha-numeric keyboard can be added to each data interface. The microcomputer in the data interface provides the computational power to control all the functional logic operations of the thermal printer/plotter which plots the chromatogram and generates a report. Data reduction parameters are entered on the keyboard and displayed on the printer/plotter. For output only, the data interface can be equipped with a printer/plotter without a keyboard. For data interface alone, reports can be routed to other printer/plotters resulting in the sharing of these output devices by more than one chromatograph.

Thus, at each chromatograph, the user has the choice of: (i) Installing the data interface by itself to control the autosampler/chromatograph while still using a conventional recorder for chromat grams or (ii) expanding his system with a remote terminal for combined remote parameter entry and report output.

The central processor supports the calculation requirements of the system. It also functions as a central operator control station providing convenient access to the operating parameters of every operating channel. These input/output functions are supported by an alpha-numeric keyboard and cathode ray tube (CRT) display.

The CRT feature of the central processor is particularly important because it provides a significant improvement in operator convenience. In a large multi-channel system where a large amount of chromatographic information must be input to the system, the ability of the CRT to display whole or large sections of file data allows the setup, review and editing of this information to be quickly performed and confirmed.

The central processor operates on a priority interrupt scheme, which allows each device (data interface, CRT, teletypewriters) to access the central processor in a time-shared mode. One such device of considerable value in automatic data reduction is the disc memory module, which provides bulk memory storage capacity for parameter file libraries, sample information, and analytical reports. With the disc memory module it is also possible to store the data points of an entire chromatogram for reprocessing. Controllers for specific components of the chromatograph (*e.g.*, pumps, ovens, gradient generator) can be implemented by the use of additional interface circuits.

In applications where power interruptions cannot be tolerated, an external 24-V power source (two 12-V batteries) can be connected as a back-up supply. The battery pack can continue to keep the central processor operating for several hours. When power is resumed, a trickle-charge circuit will recharge the battery. Switchover to the battery is instantaneous. A summary of the hardware capabilities of this design approach is summarized below:

1. Standard full alpha-numeric keyboard
2. CRT display of complete parameter files
3. Thermal printer/plotter to produce a single analytical report

4. Expandable memory capacity (high speed or bulk storage)
5. Multiple-channel operation
6. Simple three-wire parallel connection between remote modules and central processor
7. Accepts standard 1-V input; other input voltages optional
8. Remote input/output supported by printer/plotter
9. Optional interface to teletypewriters and other computers
10. Chromatographs can be up to 600 m away
11. Power fail protection using standard automobile batteries.

APPLICATION CAPABILITIES

Automatic data reduction requires two levels of operation. First, the area of each peak must be determined and then the results calculated such that the chromatographer can interpret the results within the context of his analyses.

In addition to providing standard calculation procedures, this new data reduction system can provide the following capabilities:

Bracketing of unstable samples with two calibration standards on either side of the sample
Multiple-level calibration to compensate for non-linear detectors
Peak grouping according to homologous series
Simplified report of only those components outside acceptable values
Statistical analysis
Combination of multiple analyses results into a single report
Reporting of Kováts' indices for component identification
Simulated distillation
Amino acid analysis using two non-linear detectors
Diagnostic programs for identification of operator errors and defective components
Reprocessing using stored data points for entire chromatograms.

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