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The Use of a Control Computer in a Chemistry Department

Abstract: The Chemistry Department of the Stony Brook Campus of the State University of New York is composed of a number of faculty members whose research experiments fall generally into two categories; (1) steady data acquisition and control tasks with relatively small data and demand rates, and (2) continuous scan-type measurements of various data rates. An IBM 1800 control system is employed as the nerve center of a computer-based experiment complex.

This paper discusses the multi-experiment control system used at Stony Brook Chemistry, giving some details of experiments performed by faculty members as examples of its application. It is pointed out that one of the objectives of this computerized department is creation of a course in the use of computers in chemistry. With a data-acquistition computer available in the department, the course can include actual bench practice in data acquisition and control tasks.

Introduction

The increased availability of digital computers in the recent years has made them an invaluable tool in every aspect of modern sciences. Chemistry, with its inherent nature of manipulation of data obtained from various substances under various conditions, is a major recipient of the benefits of the development of the computer technology. X-ray and neutron diffraction studies and quantum chemistry are good examples demonstrating the power of the computer for data manipulations and large-scale computations. There is little doubt that these areas will be even further influenced in the future.

The experimental part of chemistry requires that the tasks of data collection and experimental control be performed before the data manipulation stage can be initiated. Being confronted with tasks of gathering a large amount of diffraction data needed for crystal structure analyses, crystallographers have pioneered in the field of computer-based data collections. For example, at present there are a number of automated, semi-automated, or fully computer-controlled single-crystal diffractometers. Other applications of the computer in the so-called on-line mode can be found in various other fields, such as nuclear magnetic resonance, mass spectrometry and chromatography.

Out of these fragmentary computer-based instrumentation activities in the past several years, the general strategy for an optimum use of computers in the data acquisition and control fields has evolved. Although the total configurations of data-acquisition systems differ considerably from each other, it is possible to draw some common denominators for the choice of computer, hardware interfacing techniques and over-all software system to carry out the intended goals of the computer system. These functions are intimately related to each other. To obtain a successful computer-managed laboratory, it is necessary to possess good balance between these items and to understand thoroughly the nature of experiments to be controlled by the computer.

The Chemistry Department of the Stony Brook campus of the State University of New York, hereafter called Stony Brook Chemistry, has an IBM 1800 data acquisition and control system for on-line chemistry experiments and related instructional purposes. The experiments originally intended to become on-line with the computer system were x-ray single-crystal diffractometers and nuclear magnetic resonance spectrometers. Since the delivery of the computer, several other experiments in the areas of chemistry have been considered for possible connections to the computer. Some of these have already been connected to the computer and the active data collection initiated: a list as of June 1968 is given in Table 1. Glancing through this list, one notices that this combination of experiments, representing a typical university chemistry department, is an interesting example of on-line applications, especially in view of the variety of data rates, the wide range of required interfacing techniques and the

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variety of chemical results to be derived from these studies. Moreover, this data acquisition computer is in an environment similar to other general chemical, biological, or solid state research laboratories.

Although the computer is used more and more frequently for the data-acquisition and control purposes, there were no formal courses aimed at the systematic instruction in such a computer usage. One of the important aspects of the Stony Brook Chemistry system is the educational applications. The use of computers in such nonstandard applications (although these will become standard in the near future) demands more knowledge of computing techniques than simple numerical computations. One must possess extensive knowledge of input and output functions, the methodology in analysis of experimental procedures and even timing between the computer and other electronic equipment. A systematic training in this field with the help of the computer available in the department has been initiated at Stony Brook Chemistry. This subject will be dealt with in a later section of the paper.

The computer configuration

The data acquisition computer at Stony Brook Chemistry has the following configuration:

An 1802 process controller with a 16K, 4μs core memory
A number of digital input and output points
Analog input feature
Two 2310 disk drives
One 2401 magnetic tape drive
One 1442 Mod 6 card read punch
One 1627 X–Y plotter
One 1816 keyboard-typewriter.

An extensive plan of expanding the system for our future needs is now under way. The ultimate configuration would include, among others, a 32K, 2 μs core memory, a 1443 high speed printer, at least three 1053 typewriters, a sufficient number of digital input and output points, and possibly an additional magnetic tape unit. The apparent lack of graphic output capability is intended to be solved by making inexpensive cathode ray tube displays and X–Y plotters with digital output features. The programming system used with our installation has an architecture that is very similar to that of a multi-experiment control system described elsewhere in this issue by H. Cole.[1]

Table 1 contains many experiments which perform continuous-scan type measurements. These measurements generate large amounts of data, especially through the analog input feature, that are to be stored and subsequently retrieved for further analyses. The total amount of data from one single experiment or a combination of experiments can often exceed the existing free space on the disks. For example, one experiment generates about 50,000 data points per run (usually in less than 10 minutes), and the data can be analyzed only after all the repetitive manipulations are finished. It is also quite conceivable that while this experiment is going on, other equally fast data-generating experiments are being performed. This data traffic problem can be met by magnetic tapes which, in addition to the large storage capability, possess a quick, sequential access to write or read positions. Hence it might be useful to include the magnetic tape handling capability, even though this will increase the cost of the system.

Table 1

The following list includes some of the research activities pursued by present members of the department for which on-line computer application is either now in effect, under development, or under consideration.

(a) Single-crystal x-ray diffractometers
(b) Nuclear magnetic resonance
(c) Nuclear quadrupole resonance
(d) Electron paramagnetic resonance
(e) Nuclear chemistry
(f) Molecular beam kinetics
(g) Isotope exchange kinetics via mass spectrometry
(h) Solution kinetics
(i) Flash photolysis
(j) Calorimetry
(k) Ultracentrifuge
(l) High-pressure rate measurements
(m) Reaction rate constants for electrochemically generated unstable free radicals

The foregoing are research activities primarily under the direction of individual faculty members. In addition, we plan development in several general areas as listed below.

(n) Supporting data collection equipment
(o) Instrumentation for instruction purposes
(p) General instrumentation activities for efficient computer-based laboratories

It must be emphasized that the above list reflects no more than a beginning with respect to the number of applications areas envisioned. As present and future faculty members become increasingly aware of the potential benefits of on-line computer use in their programs, new applications will be devised and, hopefully, developed.

Detailed accounts of selected experiments

This section explains in some detail two of the experiments listed in Table 1 which have already been carried out under computer control. The paper by Cole[1] discusses computer-controlled diffractometers, and since our diffractometers are used in a very similar manner, no account of this application is given here. Descriptions of computer-controlled nuclear magnetic resonance studies that are similar to ours can also be found elsewhere. The applications discussed below are those with "custom made" interfacing systems designed by the electronics group of Stony Brook Chemistry. In addition to the required interfacing hardware for experimental control and data collection, each experiment is given an interrupt button and a certain number of switches for communication with the computer. The interrupt button (or switch in certain cases) is used to signal the computer so that the experiment can be placed on the list of active experiments. The routine that handles this interrupt, called ID-CALL COMPUTER, usually acknowledges receipt of the signal by causing a recognition lamp to be turned on at the equipment through the digital output feature. Ordinarily, the experimenter must wait for this computer recognition before proceeding further in his experiment. The recognition is not granted when there exist conflicts with other experiments which are being processed by the computer. The digital output used for the recognition is also used in gating some other digital outputs and most of the interrupts coming from the experiment; the main purpose of such gating functions is to assure that program or other errors do not cause unsafe operation of the equipment, to facilitate independent manual manipulations of the experiments, and to make the multi-experiment control system concise and well organized.

• *Isotope exchange kinetics via mass spectrometry, (g) in Table 1, by Professor Bonner*

The data collection from the existing (CEC 31-130) mass spectrometer, in terms of computer-controlled experimentation, is an example of measurements at constant intervals, and the general programming techniques can be applied to other such experiments except for variations in data rates. The corresponding data acquisition programs should be permitted to stay in the core memory during the course of the experiments.

The isotopic reaction study under investigation at the present time requires extensive isotopic analysis of nitric oxide from the exchange of both nitrogen and oxygen atoms in nitric oxide with an aqueous solution phase containing nitrite ion, NO_2^-. The equation for this exchange reaction can be written as

$$NO + H_2O^* + N^{**}O_2^- \leftrightarrows N^{**}O^* + H_2O + NO_2^-,$$

where asterisked atoms are partially enriched by the less abundant species, O^{18} and N^{15}. The purpose of this study is to determine the kinetics of the reaction by changing parameters such as temperature and initial concentrations.

Gas samples are manually collected into sample cells at predetermined intervals and the contents of these cells are analyzed by mass spectrometry to determine their content of N^{15} and O^{18}. For now, the computer control of the experiment is limited to data acquisition from the mass spectrometer. The hardware interfacing for the multi-sweep control has been completed and automatic data acquisition is in progress at this time. In order to obtain a better signal-to-noise ratio, at least 50 sweeps are made for

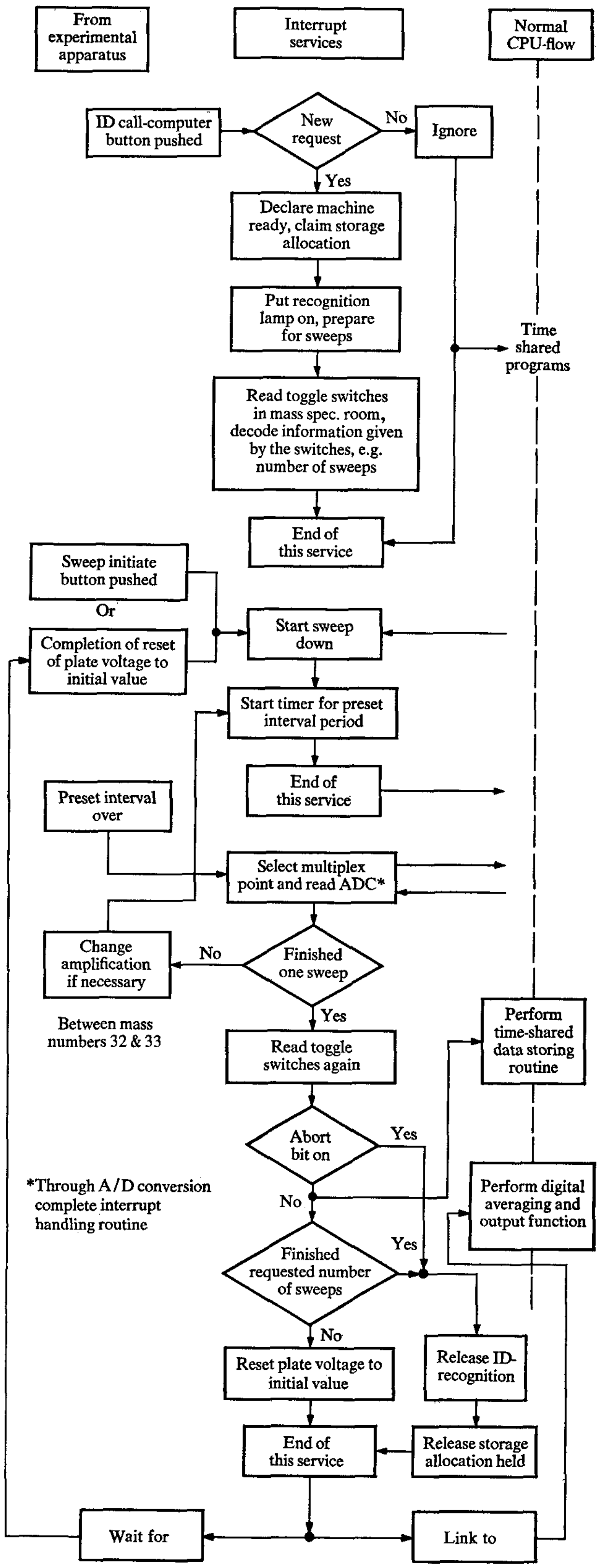

Figure 1 Program logic for isotopic reaction study.

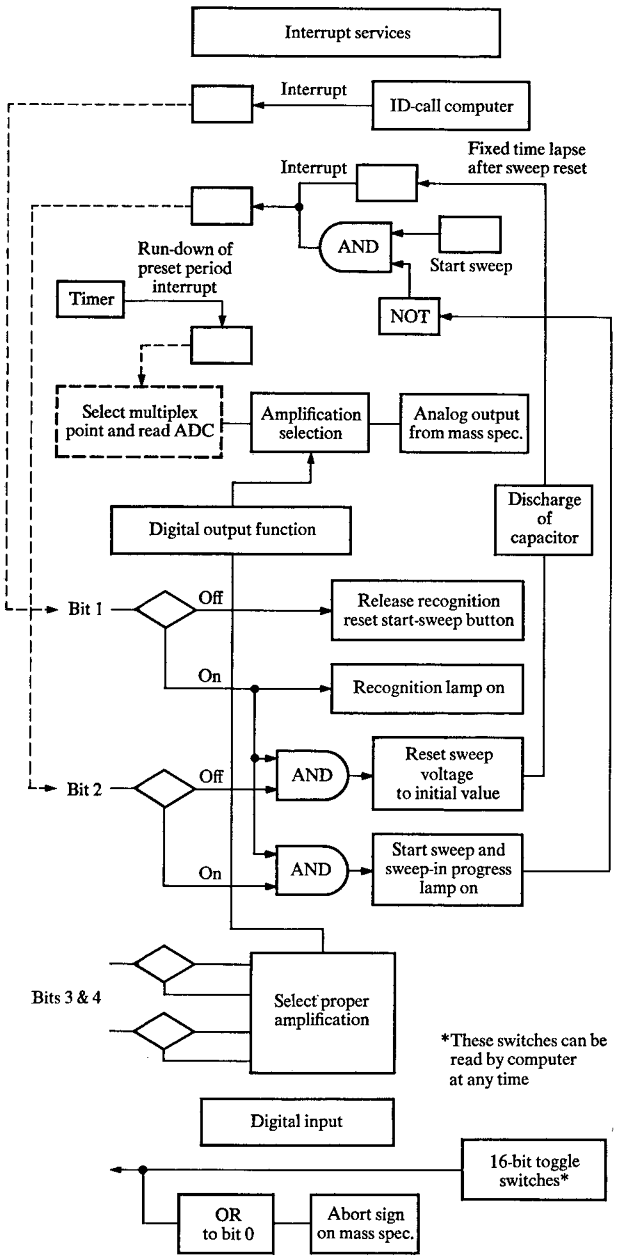

Figure 2 Logic for interface between mass spectrograph and computer, in isotope reaction study. (Dashed lines indicate possible program flows associated with the interrupts.)

each sample, each sweep consisting of about 2000 recordings of output signals at constant intervals; at the end of each sweep the computer resets the sweeping range to the starting low mass range. The number of sweeps for each run must be determined at the beginning of the measurement; this number depends on the amount of available samples and the expected amount of the least abundant molecular species. The required number can be entered into the computer via a set of toggle switches, the content of which is read into the computer at the time the ID-CALL COMPUTER interrupt is given by the experimenter. The data thus obtained are treated by a digital averaging program resulting in a noise-suppressed set of data. The characteristics of the mass distribution are such that the amplification of the output signal must also be changed by the computer in the course of each sweep.

Schematic diagrams of the program and interfacing logic are given in Figs. 1 and 2. Although the mass range used in the present study is limited to a narrow region, the hardware interfacing construction and the data collection programs are made in such a general way that they can be used in more general mass spectroscopic studies by varying only the necessary input parameters.

- *On-line data acquisition of flash photolysis experiments, (i) of Table 1, by Professor Solo.*

This reaction kinetic measurement is an example of a high data rate application. After an initial flash by a high-powered lamp, the transmission factor of light by a specimen is measured as a function of time. For example, iodine molecules at the ground state are excited and dissociate into iodine atoms by the flash and then go back to the original ground state through several paths of recombination. The typical reaction rate is such that about 200 data points are to be measured within a period of 10 to 50 msec. This fast rate requires data channel operations associated with the analog input feature of the computer. The analog input feature has been so modified that, under computer-control, the multiplexer can be by-passed and the same data point can be read continuously without advancing to the next point. After initiation of the data channel, the central processing unit is freed until an end-of-table interrupt is given. This experiment locks the analog input feature for the duration of the measurement, and simultaneous use of the same analog to digital converter by another experiment is not possible. However, this limitation imposes little problem on the total system since the initiation of the flash photolysis experiment is not time dependent. The 1800 analog to digital converter system with the multiplex by-passed permits us to sample the transmission factor every 58 μsec. This interval is found to be exact within 1%. In Figs. 3 and 4, the current data collection techniques for this application are described.

Since, for a given reaction, a number of runs must be made by changing pressure, quenching species and wavelength, on-line data collection will speed up the total experimental flow by a factor of 100 when compared to the conventional manual mode of operation which requires recording of the output on a Polaroid camera and manual handling of the data on photographs. When a computer is used, the transmission data are, within the short reaction period, already converted into useful digital form. These

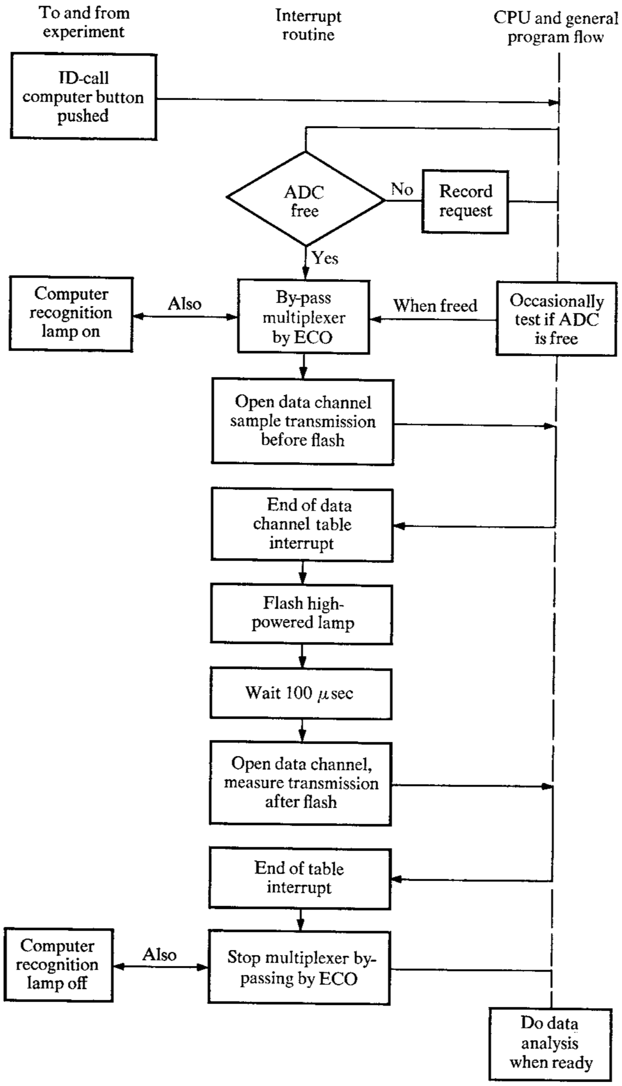

Figure 3 Current data collection scheme for flash photolysis experiments.

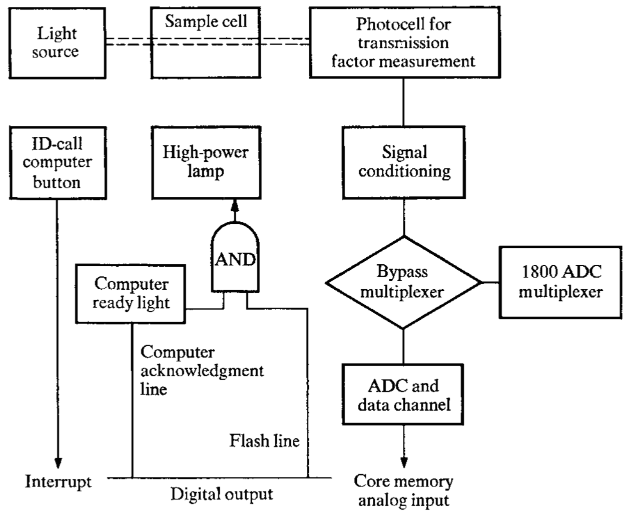

Figure 4 Experimental setup and interfacing logic for flash photolysis experiments. The AND logic at the ready light is provided to reduce chances of accidental flash by programming errors.

values can be used in subsequent treatments such as the determination of recombination kinetics.

• *Supporting data-collection equipment*, (*n*) *of Table 1.* A time-averaging computer, CAT, of the Technical Measurement Corporation, has been connected to the 1800 and data stored in the computer can be read into the memory in digital form. This connection facilitates a number of studies such as least-square analysis of nuclear magnetic resonance data stored in the computer. Other equipment items now being considered include a digitized film reader, a powder diffractometer and a specific heat measurement system.

Once a data-acquisition computer system is established in the department, the computer can also be used in smaller problems such as periodic calibration of equipment, relatively simple numerical analyses of data, graphic representations of experimental results for publication, and general clerical work. These simple tasks can be run easily under the time-shared scheme; however, such tasks by themselves would never justify the existence of even a rudimentary computer.

Instructional application of the data-acquisition computer

Although the computer is becoming increasingly important in process control operations of all kinds, systematic training programs which provide students with the necessary information and knowledge for successful participation in computer-controlled processes appear to be rare. The work involved in computer-controlled applications requires more extensive knowledge of computers than is the case for simple numerical computation. In addition to the capability of programming for numerical computation, one must be familiar with the more detailed input-output functions of the computer. It is also essential to be able to perform step-by-step analyses of processes (or experiments), including analyses of the logical decisions that would have to be made by human operators performing the experiments under manual control. It is quite important for those involved in the computer-controlled experiments to be able to perform such analyses. Otherwise, they will limit their experiments to those kinds for which programs are available; in essence, they might freeze their work—a dangerous situation. Since every individual computer has its distinct surrounding, one must also be accustomed to the generation and use of higher level problem-oriented languages. Furthermore, one must possess a certain amount of knowledge of various aspects of the "interfacing" technique. Thus, it is important to develop an integrated instructional system which covers the essential aspects of this new technology of computer usage.

Given a data-acquisition computer under the exclusive use of the Department, Stony Brook Chemistry is in the unique position of providing educational experience in this new component of chemical research methodology. A course, entitled "Computer-controlled experimentation in chemistry,"[2] has been included in the graduate study offered by the Department with the author as the principal instructor. The general objectives and broad content of the course activity may be set forth in terms of course functions:

(a) To provide understanding and actual practice of data processing techniques. The prescribed practical operations will be devised both to promote understanding of data analysis techniques and to provide experience and insight into the problem of improving data arrangements at the time of collection.

(b) To become acquainted with the basic methodology in providing the detailed analysis of data acquisition and control tasks, including those of necessary logical decisions.

(c) To participate in the computer-controlled experiments performed as research projects of members of Stony Brook Chemistry. This will involve studying a variety of data-collection programs, and will include the actual handling of some experiments. For example, consider the problem of finding the three strongest lines in the x-ray powder diffraction pattern of a specimen. Even such a simple problem teaches the student the use of shaft-angle encoders, recording of analog and digital data, and other basic techniques of data acquisition and handling. Participation in a single-crystal structure analysis or a nuclear magnetic resonance study will provide effective fundamental training of broad applicability.

(d) To define experiments and simulate them by using proper terminals. The experimental work proposed here will serve to familiarize students with the basic programming techniques which include the concept of feedback, establishment of the flow of experiments on the computer system and the selection of necessary hardware. An example of such an exercise could be to design a computer-controlled production scheme for a known organic synthesis.

(e) To define and actually design simple experiments by modularly combining actual experimental and interfacing hardware. Programs necessary for actual manipulation will be written, tested and used in running the experiments. For example, a simple hardware manipulation such as rotation of a shaft to angular settings given in a table and measure and record the values of some physical quantities at each setting.

(f) To make well-defined graphic representations of experimental results and their interpretations, including graphic techniques in monitoring experiments.

Although mainly chemical experiments are to be considered in (c) to (f), the basic technique achieved by such practice is applicable to any other data acquisition and control task. An excellent example of student participation is the computer-controlled flash photolysis experiment discussed in the previous section. In the 1968 Spring Semester, an on-line surface cardiogram system was developed by a group of three engineering students as their undergraduate research project using our 1800 system.

Acknowledgments

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References

1. H. Cole, "Computer-operated Laboratory Equipment," *IBM J. Res. Develop.* **13**, 5 (1969), this issue.
2. Chemistry course No. 626, State University of New York, Stony Brook.

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