This paper discusses the use of a computer to control data-taking in the laboratory—including the case of "closed-loop" control.

Illustration is provided by describing a particular system involving computer control of an x-ray diffractometer.[1]

A computer-operated laboratory data-taking system

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laboratory data-taking

A large amount of time is spent in laboratory work in the two important operations of data-taking and data-analysis. Further experiments often accept as a base the accuracy of the present data and the reliability of the analyses. Thus, wherever it is possible, one attempts to use the best instruments available for the data-collection and the best in computing techniques for the data-analysis. In addition, in any new work, a procedure is established as quickly as possible so that reproducibility can be obtained. Then, as in any industry, whenever either a data-taking or a data-analysis procedure becomes fairly standardized, and achieves the status of a "tool", efforts are made to mechanize it, to make it faster, to make it more reproducible, and to make it less susceptible to human error. Many accepted laboratory techniques, or more broadly speaking, production techniques, are presently passing through this mechanization stage.

The next stage through which these techniques are predicted to pass, in laboratories, as in production, is a stage of "automation". By this we mean that an input/output (I/O) device and a control box may be attached to the mechanized instrument so that enough control information, prepared elsewhere, may be given to the instrument to keep it running, say, all day, and the data is accumulated in an output form that can also be analyzed either on the spot, or later, but in a device essentially separate from the instrument. The justification for this stage is the same as in any industry, plus the added feature that in laboratories a highly trained individual may be relieved from performing a tedious task and can spend more time working at a creative professional level. Instrumentation directed toward this type of "automation" is currently being developed and is appearing on the market.

The "automation" stage, as described above, does not involve any "feed-back" in the sense that the data-analysis influences the data-taking in any direct manner. For many instruments, and techniques, feed-back is not needed, and so this development may be the ultimate one. The ultimate form, of course, may involve a computer, operating under a stored program, which generates the control instructions and feeds them directly to the instrument, and receives the data back directly into its memory and performs the analysis, using another stored program. Whether the computer is a small full-time one or a large time-shared one there is still no necessity to close the loop, except for occasional readjustment of the instrument, and the two programs will probably remain separate.

There are, however, quite a few data-taking tasks which are, in a sense, today, even though mechanized, too complicated for "automation", or open-loop systems. Substituting for the human operator in these situations just an "automated", open-loop system is a step backwards, because a great deal depends on the decision-making ability of the operator. Judgments are required at certain points, and a choice can be made to follow one of a number of alternative paths. That is, the making of "local" decisions during the data-taking tends to avoid waiting for the completion of the data-analysis to determine whether the data-taking has been done correctly, or has to be repeated. The branching abilities of computers, however, permit this decision-making ability to be incorporated in a system if a computer is directly in the system and if the decisions are programmable. As soon as the cost of an open-loop system plus reruns (or rejects) is greater than the cost of a computer-controlled closed-loop decision-making system, then the computer is no longer an ultimate form but a present necessity.

The task of taking x-ray, or neutron, diffraction data from single crystals in order to determine such things as the structure (the arrangement of the atoms in the crystal) or the lattice vibrational spectrum (how the atoms are moving) is an example of a task in a laboratory that is well-defined, tedious and necessary. Moreover, we believe it is a task requiring a closed-loop computer-run instrument, if any type of relief is to be given to the operator. The rest of this paper is a description of a system which has been built, largely from commercially available items, to provide a computer-run instrument to carry out the type of closed-loop data-taking task described above. Although built with x-ray diffraction study in mind, its features are broad enough to include many other possible operations.

Example of a computer-controlled x-ray diffractometer[1]

approach

The general problem facing anyone who wishes to automate an x-ray diffractometer "Geiger counter" data-taking system is roughly the same as that faced in constructing a numerically controlled machine tool. A number of mechanical shafts need to be set to prescribed angular positions; a number of counting (or scaling) devices need to be activated; and certain data needs to be accumulated and presented in a usable form. Except for the special requirement of small, lightweight shaft angle encoders, the "hardware" part of the automation is currently available commercially in many forms.

The unique task facing the designer of the x-ray equipment is the problem of getting the proper control information to the automation. For example, the question of what is the proper manipulation of sample and detector that produces the most significant data is still currently a debatable one among crystallographers.[2] Therefore, any attempt to build prescribed search patterns within the automated hardware may lead to abandonment later. Also, crystallographic data-taking is not a repetitive task. The crystallographer never repeats himself; he reruns only to change. Thus, a scheme which produces control information in a fixed form that has to be reassembled and changed for each run may lead actually to decreased efficiency. As one attempts to simplify the tasks performed by the hardware, and at the same time permit more flexible input of control information, one inevitably arrives at the concept of computer control of the automation. This, of course, is not a new concept; however, a computer-controlled instrument represents a significant departure from the purely automated systems which have been constructed previously, if one permits current data to flow back into the computer where it can be used to select the next control instruction. With this feed-back feature, computer control permits the closest approximation to human operation of a diffractometer.

From the computer point of view, the diffraction data-producing system is merely another I/O device into which it "writes" control information and from which it "reads" data. Adequate programming of a computer of sufficient capacity then permits the decision as to "what to do next" to be left to the computer, and can be based on the current results. With the computer such an integral part of the total system, the user may easily change the manner of taking data by changing the program under which the computer operates. Thus the automation hardware may be simple, yet the equipment may be utilized easily on many different levels of sophistication, depending upon the judgment of the user, and the programs or programming skill available. Long, complex data-taking tasks may then be undertaken, as the system is in many aspects essentially self-correcting.

The combination of capacity and speed, in the IBM 1620 computer which was used here, is such that many crystallographic calculations can be done on the computer while it is also running the diffractometer. These computations are interleaved with the program which controls the diffractometer so that the computer is time-shared.

the configuration

The computer-controlled x-ray diffractometer system was assembled as much as possible from standard commercially available items.

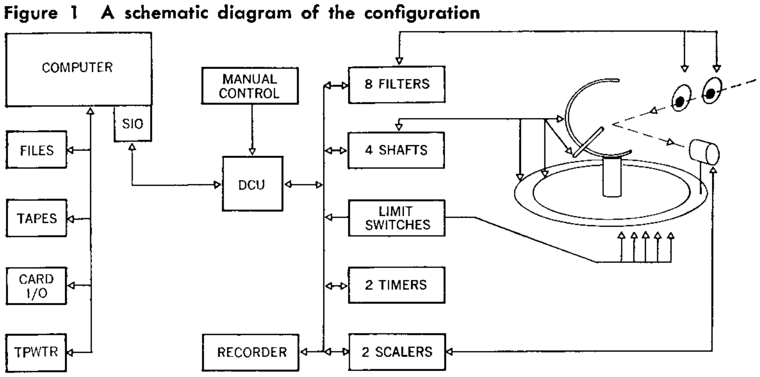
An over-all block diagram of the system is shown in Figure 1. Beginning at the left, the IBM 1620 configuration is considered first. Programs, input of basic parameters and output of information is presently handled through punched cards in this system, and thus the card read-punch is the primary I/O device. The 1620 has a magnetic core memory with the capacity to store 20 K decimal digits. Additional core memory is available and this system has 20 K additional. One 20 K block of memory appears to be more than sufficient for the diffractometer control program, so the additional block of 20 K is available for normal computing. The 1620 console also contains a typewriter so that parameters could be entered and a printout of data obtained by this means, if so desired.

Attached to the 1620 is a serial I/O cable (SIO) which permits the connection of special I/O devices to the 1620. The diffractometer, as a special I/O device, begins at the end of this cable; it begins in a "black box", the diffractometer controller unit (DCU), which is the nerve center for the automation.

The DCU, as shown schematically in Figure 1, is connected to the various motors, encoders, scalers, timers, filter wheels, limit switches, etc. which constitute the normal x-ray diffraction single-crystal electronic-detector configuration.

The computer-controlled diffractomer is digital throughout; there is no specific analog-to-digital converter. The 1620 computer "writes" binary-coded decimal digits into the DCU just as if it were producing a punched card. The DCU stores the numbers in a relay memory, and then interprets them as either desired shaft positions, control information for starting or stopping motors, or

Figure 1 **A schematic diagram of the configuration**



scaler-timers, etc., depending upon the format of the transmitted numbers. The DCU also contains within its memory the current readings of all encoders in the system and the current status of all switches, which information can be read from the DCU into the 1620 whenever the particular "read" command is given by the 1620. The DCU was built using standard logic and components; because the circuitry within the DCU is straightforward and, in addition, depends somewhat upon the characteristics of the particular computer used, circuit details will not be presented here, but instead the general features on the total system will be described.

The x-ray diffraction items include an x-ray spectrometer with a single-crystal orienter mounted on it. Together, these two assemblies provide *four* rotary axes which intersect at a common point in space, at which point the small crystal sample is placed and through which point the x-ray beam passes (see Figure 1). Three of the axes are used to manipulate the crystal and one is used to swing the detector around the crystal. By changing the orientation of the crystal and the position of the detector, the diffracted beams, generally called reflections, which can be visualized as directional beams in space, can be brought one by one into the detector. Four axes are not needed, theoretically, to reach a desired reflection; however, in practice, certain reflections which would be blocked mechanically by one approach can be reached by another approach. Also, in taking integrated intensity measurements, i.e., determining the strength of the reflections, one needs to scan through the reflections in certain ways, as well as reach the reflections. The four axes give this desired flexibility. The two axes on the spectrometer base, 2θ and ω, were provided with slewing motors and shaft angle encoders, in addition to the scan motors provided normally within the base. The two axes of the crystal orienter had encoder assemblies attached to them, in which a motor, shaft angle encoders, and the necessary gears and clutches for providing both slewing and scanning speeds were contained. The four shafts are controlled to five significant figures (0.01°) by the DCU as instructed by the 1620. Limit switches are set on these instruments so that mechanical motion can be limited to safe regions. If a limit switch is hit, the motors stop and an error signal is sent to the 1620, which can be used to cause it to branch to a subroutine to determine the cause of the error.

Eight switches can be set or their present position sensed by the 1620. At the moment these eight switches are embodied in two four-position filter wheels, shown schematically in Figure 1. Thus filter, attenuators, or apertures in the beam may be changed by computer command; other operations, such as turning on furnaces, etc., may also be performed.

The soild-state scaler and timers associated with the electronic x-ray detector, which may be thought of as a pulse generator, can be preset by the computer to desired upper limits of count and time, reset, and started, and they will run until either the preset time or count is reached, and then both will stop. The contents of the scaler and timer, six digits each, already in digital form, can be read by the 1620, through the DCU, and stored in its own memory. Provision has been made for two sets of scalers and timers to be operated through the DCU to permit monitoring of the x-ray intensity.

A manual control panel was included so that the automated equipment may be operated through the DCU without the presence of the 1620. This is intended primarily for preliminary alignment of each new crystal on the goniometers. The present position of any axis, selected by a knob, is displayed on a small "TV screen" in the middle. This positional information is always available, and gives a visual readout of all encoders at any time. The desired positions of the four axes can be set into appropriate number wheels on the manual panel; the appropriate speed, slew or scan, and the "go" command is given when the switch beside each set of number wheels is thrown.

The automation is a parallel system in the sense that all four shafts may be turning to their desired positions at the same time. This affords the possibility of trying new modes of data-taking, not currently being employed, as well as saving time during a run. For each encoder there is a register in the DCU which contains the present reading of the encoder; there is also a register for each shaft containing the desired position of each shaft. A comparator circuit, gated by a pulse from the anti-ambiguity track on the encoder, compares the desired position with the present position, determines the necessary sense of the required shaft motion and permits the motor to be activated. When all shafts are at their desired positions, a signal is sent from the DCU to the 1620, setting a sense switch in the 1620 (a branch indicator) which indicates to the program running in the 1620 that the diffractometer is ready for further attention. Some logic of this type in the interface frees the computer from continuous monitoring of the automation.

Since new angular positions differing from the previous position by 0.01° will activate the system, step scanning on all four axes in increments of 0.01° is possible. There is no special hardware for step scanning; the program in the computer may ask for axis changes of as little as 0.01° at a time.

The scaler and timer may be activated while shafts are turning, thus permitting the taking of data from a moving crystal. Since the halting of the shafts is noted by the DCU, the scaler and timer may then be stopped when the motion ceases, in addition to responding to computer preset-count-and-time.

If the x-ray detector counting rate, as noted by calculation within the 1620, is too high, a calibrated attenuator or filter may be turned into the x-ray beam by command to one of the filter wheels.

Provision has been made for the DCU to turn on a strip chart recorder, under command from the 1620, if a tracing of the output

Table 1 List of DCU commands available to programmer

Control commands (*K* 34)	*Write numeric mode* (*WN* 38)	*Write alphameric mode* (*WA* 39) *Read numeric mode* (*RN* 36)
Stop all motors	Preset scaler #1 to a count of 10^x; $x = 1$ to 6	Read present value of 2 θ axis encoder (5 digits)
Start all motors	Same as above for timer #1, scaler #2, timer #2	Same as above for ω, χ, ϕ axes encoders
Reset to zero scalers and timers	Set filter wheel #1 to position x; $x = 1$ to 4	Read contents of scaler #1 (6 digits)
Start scaler and timer #1	Set filter wheel #2 to position x; $x = 1$ to 4	Same as above for timer #1, scaler #2, timer #2
Start scaler and timer #2	Select 2 θ axis (one digit), select shaft speed (previous digit flagged or not), store 2 θ desired position (5 digits)	Read present position of filter wheel #1 (1 to 4)
Stop scaler and timer #1		Read present position of filter wheel #2
Stop scaler and timer #2	Same as above for ω axis, χ axis, ϕ axis	Read contents of status register
Start strip recorder		
Stop strip recorder		

of a rate-meter is desired while the diffractometer is moving through a particular diffraction peak.

data-taking

The system is operated by programming the 1620. The programming is standard, that is, no changes have been made internally in the computer. Within the language of the 1620, the DCU has an address, and whenever this address appears as part of an I/O instruction the DCU becomes involved. The normal "write numeric" and "write alphameric" commands of the 1620 are used to put information into the DCU, and the normal "read numeric" instruction is used to transfer information from the DCU into the 1620 memory. The normal "control" instruction is used to turn on, or off, motors, scalers, timers, move filter wheels, etc. A list of the "DCU commands" available to the programmer is given in Table 1.

The 1620, operating under the stored program, calculates the setting for the first reflection and through the DCU, moves the spectrometer to this position, selecting either slew or scan speed as the program indicates. While the spectrometer is moving, the 1620 is released and branches to another program; the first few peaks will undoubtedly be step-scanned, to determine the actual crystal setting and thus refine the knowledge of the orientation and possible misalignment of the crystal on the instrument, so the calculations for this job can be carried out at this time. When the DCU signals the 1620 that all motors have stopped, the 1620 reads the encoder positions, checks that these are the desired positions and, if so, presets and starts scaler and timer.

While the scaler and timer are running, the 1620 is again released. The scaler-timer signals the 1620 when it stops, and when ready to branch back to the automation control routine, the 1620 reads the scaler and timer. It computes the counting rate and, if too high, inserts an attenuator, and repeats the count. If the counting rate is reasonable, it proceeds to carry out the step-scanning as programmed. If no peak is found, the program may ask that the next peak be looked for, or a general search routine is instituted.

After the first, or first few, peaks are located, their integrated intensities are measured, or computed from the stepping information, using any technique chosen by the programmer.

In this manner the 1620 carries out the data-taking. Perhaps after every fifth measurement, certain previous measurements are repeated. If an inconsistency is found, that is, if the new number differs by a certain percentage from the old one, the peak position may be redetermined to see if the crystal has moved slightly; if it has, the new orientation or lattice parameter is stored and used thereafter. If other sources of inconsistencies are discovered, checking for these may be included in later control programs.

The sequence of reflections may be selected by the program in such a way that travel time is minimized. The integrated intensities, stored in the 1620, may be corrected for absorption, polarization, Lorentz factor, etc. If desired, and it usually is, a punched card can be produced by the 1620 containing all the pertinent information about each reflection. However, as data accumulates, calculation may be carried out depending on computer capacity, leading to certain mathematical results. These results are usually in the form of contour maps of electron charge density. With an x-y plotter, available with the 1620, the output may be the actual density maps.

Subroutines which carry out many of the above manipulations have been written and tested, and are being assembled into a master control program.

The significant feature of the computer-controlled system, of course, is its ability to work its way through a complicated task, unattended, under control of a good master program.

ACKNOWLEDGMENT

The DCU and manual control panel were built by an IBM group headed by F. Stern–Montagny with G. O. Boehm and R. Love being largely responsible for the final form of the unit.

CITED REFERENCES AND FOOTNOTES

1. This part of the paper is an abridgment of an article, "Computer-Controlled Diffractometer," published by the authors in *Rev. Sci. Instr.* **34**, 872 (1963). Permission of the American Institute of Physics to reprint from this paper is appreciated. Crystallographers interested in greater detail are referred to the latter paper.
2. L. E. Alexander and G. S. Smith, *Acta Cryst.* **15,** 983 (1962).