MASTER

Technical Progress Report

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Molecular Structure Studies:

A Gas Phase Electron Diffraction Unit Employing Counting Techniques

June 1 through December 18, 1969

INSTITUTION: The University of Toledo, Toledo, Ohio

PRINCIPAL INVESTIGATOR: H. Bradford Thompson

Professor of Chemistry

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This report covers progress during the first 6 1/2 months of a program to develop an apparatus of unique design for gas phase electron diffraction. The apparatus and program are described in considerable detail in the original proposal [referred to below as OP]. In brief, the unit is to employ particle counting techniques rather than the conventional combination of rotating sector and photographic plates. The design is to be competitive with the photographic method in terms of sample required, and superior in versatility and freedom from a number of sources of uncertainty. The automated character of the unit will make possible a precision greater than presently attainable, and thus permit studies of larger molecules and of isotopic effects on bonding. The techniques to be tested should apply to gas phase x-ray and neutron diffraction as well.

PERSONNEL:

Principal Investigator: As per OP, Dr. H. Bradford Thompson has directed this work, and has devoted at least 50% of his time to the program. In actuality, during the summer the project was Dr. Thompson's principal activity, and the time contribution probably approached 80%. A period of illness during the fall quarter (a period covered by the University contribution to the program) somewhat slowed progress. However, during this time the University continued its full support of the program and the principal investigator.

Other Personnel: A graduate student, Mr. Gregory Fehn, has undertaken software development for data acquisition as his M.S. thesis project. Mr. Fehn's contribution is very valuable (see below). As he is a part-time student holding a local industrial position, neither the AEC nor the University are supporting him financially on this project.

Several undergraduate students have been employed in technical and secretarial tasks, including Mr. Charles Ritz (now a graduate student at the University of Michigan), Miss Christine McLaughlin, Mr. William Kint, and Mr. Clinton Spevak.

Fortuitous Consultation: During the summer, Dr. Kozo Kuchitsu, Professor of Physical Chemistry at the University of Tokyo and an internationally-recognized worker in electron diffraction, made a three-day visit to our department, and went over plans for this apparatus in considerable detail. He had a number of valuable comments, suggestions, and cautions, particularly regarding possible effects of extraneous local ac and dc fields, vacuum oils, sources of incompletely-understood background scattering in the photographic process, and choice of molecules for original checkout of the unit.

PROGRESS TO DATE (Through December 18, 1969)

Data Acquisition System: The basic hardware and software for data acquisition, from the discriminator through the prescalers to the computer memory, has been tested, and is ready to use. The computer is a PDP-8/I, and the interface has been constructed using Digital Equipment Corporation modules and accessories. While the logic design follows established practice, its application to the present system has required testing, minor alteration, and statistical analysis. The extensive logic wiring reflects the patient efforts of Miss McLaughlin. Figures 1, 2, and 3 show the prescalers, buffer, deadtimeless transfer into the computer accumulator, and output relay-control buffer. The circuitry shown has been tested on all control channels, and on two input channels using a clock module to simulate incoming counts.

The basic software is ready. The data acquisition requires about 60% of the computer's time (worst-case), leaving time for checking, display, etc. Mr. Fehn is at present developing programs to check incoming data for significant drift or other symptoms of malfunction.

Analysis of the statistical problems involved from prescaling roundoff and other sources has been completed. In the present design, counts can be prescaled by 64 or 128. Within very wide limits of total count and of dwell-time per position, errors from this source will not be significant. Detailed analysis will be included in Mr. Fehn's thesis, and will be submitted as a later technical report.

Electron Optics: The 40-kv supply with rectified 4-volt, 5-ampere filament supply has been completed and tested. The supply is enclosed in a steel mesh cage with a power-cord disconnect attached to the door. The high voltage is measured using a Beckman Instruments 100-kv voltage divider. The electron gun has been constructed and tested. Electron-beam optics, including two sets of beam deflectors and an electrostatic lens, have been constructed and await testing. The optics resemble quite closely those in the electron diffraction unit of Dr. L. S. Bartell at the University of Michigan, except for the use of an electrostatic lens. The divided beam trap, designed to detect and allow correction for beam drift, has also been constructed. Control circuitry for all phases of the optics is complete, largely through the efforts of Mr. Ritz.

Mechanical Design: The vacuum shell has been assembled, complete with the required stands and supports. The axis of the unit is aligned along the local direction of the earth's magnetic field; adjustment has been built into the stands for minor correction of this orientation as required.

The larger of two oil diffusion pumps (a 6" NRC pump) can maintain a vacuum in the closed system below 10^{-7} torr without outgassing or cooling, and below 10^{-8} torr using liquid nitrogen in the trap. Since the pressure is much higher during sample injection, this is deemed an adequate vacuum.

The sample-injection system has been tested using CCl_4 , a 0.28 mm square-end hypodermic needle as a nozzle, and a pressure behind the nozzle of about one torr. This approximates a typical flow in the photographic process. Under these conditions, the pump and liquid-nitrogen trap could maintain a pressure of 2×10^{-5} torr, which compares favorably with pressures in photographic units. Prior degassing of the sample is found to be very important if pressures this low are to be achieved during steady flow. Some back-diffusion of pump oil onto the trap was experienced in the sample-injection test. Since there is no baffle at all between pump and trap, this is not unexpected. Installation of a simple baffle may be indicated.

The mechanical-motion system, including bellows, synchronous motor, and ball-bearing worm drive, have been assembled and tested. The bellows and associated system have been vacuum tested, and the drive tested separately and found adequate, but the counterbalance system required to use the drive on the system is incomplete, so the two have not been tested together. [Atmospheric pressure exerts a force requiring 400 pounds of counterweight.]

Detector System: Progress in this area has been slowest, since any testing required operational assemblies in all other areas. Two detection systems are under consideration: (1) scintillator-photomultiplier combinations, and (2) direct electron multiplication, using a device such as the Bendix Spiraltron. Both are to be tested. An assembly for testing the simplest scintillator design is complete. The preamplifier for a Bendix multiplier is likewise ready; a small amount of auxiliary circuitry is required for first tests.

ANTICIPATED PROGRESS (To March 31, 1970):

Effort is to be concentrated on the items below, with priority in the order listed. As a best estimate, items through number 4 will be completed during the next 3 1/2 months.

1. Detector System: The two possible detector systems will be tested, and a choice reached, using the fixed monitor detector system. The Bendix devices will be tested for changes in sensitivity with position and angle of electron incidence, by using the motion controls normally employed for sample injection to move the multiplier while under test. A hybrid system, using these compact and simple devices at small angles and scintillators at large angles, is an appealing possibility. On the basis of these tests, detection devices will be purchased and final construction of the "front end" of the detection system begun.

2. The Electron Lens: The crucial tests of the practicality of an electrostatic lens in this system will be made, so that if necessary, work can be started on the alternative magnetic lens. Design of an appropriate magnetic lens is available from the Michigan apparatus. [Reasons for preliminary choice of an electrostatic lens are given in OP.]

3. Mechanical Motion System: The mechanical motion system is to be installed and tested in conjunction with the vacuum, and one detector channel tested through the cyclic mechanical motion, using the entire data acquisition chain including the PDP-8/I computer and software.

4. Vacuum system: Two important questions regarding the vacuum system require attention--the possible need for a baffle above the 6" diffusion pump (see above), and the adequacy of the smaller 2" pump which evacuates the gun chamber. Tests on this pump when used to evacuate the entire system were unsatisfactory--the pressure could not be reduced below 10^{-5} torr. Tests are required with this pump evacuating the gun chamber alone.

5. Software: Mr. Fehn will continue work on the software. In addition to data checks (see above) we are working on oscilloscope display and periodic printout of certain experimental parameters, and automatic system shutdown when something goes awry. In connection with this, more time for such computation may be obtained by a change in the hardware (following section).

6. Data-Acquisition by Direct Memory Increment: Mr. Fehn and I have discussed use of the Memory Increment feature of the PDP-8/I to greatly decrease the data-acquisition time. The time fraction required for input of scaled data pulses might be reduced to 15% (worst case), thus greatly increasing the data-checking possibilities. This system would also simplify the programming and remove certain annoying sources of possible slight timing error. The required hardware does not seem overly complex, but design has yet to be completed.

7. Voltage-Divider Comparison: The voltage divider is to be checked against that in use at the University of Michigan. Plans for this cooperative effort have been made with Professor Bartell. Our divider (which is more portable than his) is to be transported to Michigan and tested within his high voltage cage (which is much larger than ours). If a severe discrepancy results, plans may be made to have our divider calibrated at the National Bureau of Standards.

Prescaler logic. Located near e.d. unit.

Cable to Diffraction Unit. For test purposes module with jumper from clock can be substituted for cable.

Output cable takes pulses from pins V2 when in normal position, pulses from S1 when inverted. H2 input and -15v supply for M502 module returned via same cable.

FIGURE 1

Positive logic pulses from counters via cable at B27

Codes:

- 6651 EDRP: read ones into AC if prescaled pulses have reached Hold Flags.
- 6652 EDCC: clear counters and Hold Flags.
- 6654 EDCF: reset Hold Flags corresponding to ones in AC.
- 6655 EDRS microprogrammed pulse read sequence.

"Clear" to counters via pin v, cable at B27

FIGURE 2

Codes:

6641:	clear channel switching	EDBC
	buffer	
6642:	read ones from AC to	EDBO
	switching buffer	
6643:	microprogrammed buffer	EDBL
	load sequence	
[6644:	unused	EDxx]

Four R203 modules comprise channel switching buffer, which controls relays directing count-to-prescaler routing.

Figure 3