



**AUSTRALIAN ATOMIC ENERGY COMMISSION  
RESEARCH ESTABLISHMENT  
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**AN ON-LINE COMPUTER SYSTEM FOR A MASS SPECTROMETER**

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**October 1970**

ISBN 0 642 99385 8



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ABSTRACT

Surface ionisation mass spectrometers are commonly used to provide accurate isotopic abundance measurements of solid samples. In this report the method of mass scan control is described, together with the data logging system. Included as well are a description of the relevant parts in the PDP-8/L program, instructions for communication with the computer by the operator and the computer interface circuit diagrams.

National Library of Australia card number and ISBN 0 642 99385 8

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## 1. INTRODUCTION

Mass spectrometers are used for a variety of measurements, and their design is frequently dictated by the purpose of the measurement. This report describes an on-line computer system specifically designed for a surface ionisation instrument capable of precise isotopic abundance measurement. As such, the instrument features a high sensitivity detector incorporating an electron multiplier whose output feeds a vibrating reed electrometer, and a limited mass scan which covers the isotopes of one element only.

The output has two major features. Firstly, since the ion beams used are of very low intensity, statistical noise is large and hence lengthy reading times are necessary to attain high precision of measurement. Secondly, owing to sample depletion the ion beams decay, generally exponentially, so that with a single detector repetitive scanning is necessary in the directions of both increasing and decreasing mass to minimise systematic errors. This involves a substantial amount of data logging and subsequent processing.

The on-line computer system created for the mass spectrometer provides facilities for automatic control of the magnetic field, data logging and data processing, all under the supervision of an operator who instructs the computer using a conversational language through a teletype console. The system (shown in block form in Figure 1) comprises a small computer, an interface which provides a data input terminal and allows direct adjustment of the magnetic field, and a teletype console for two-way communication.

## 2. THE COMPUTER INTERFACE

The mass spectrometer computer interface has two major functions: it controls the magnetic field and it collects output data from the ion flux detector. It comprises two 19-inch rack-mounted modules, one containing the logic circuitry and the other, called the motor unit, containing the mechanical transducers. The wiring and digital logic of the motor unit and computer interface are shown in Figures 2-7.

### 2.1 Magnetic Field Control

A ten-turn Helipot potentiometer driven by a Slosyn stepping motor is used in the fine field adjustment circuitry of the mass spectrometer to give the computer control over small selected ranges of the magnetic field. There is a gear reduction of 5 to 1 between the motor and the potentiometer and as the motor requires 200 steps per revolution, the ten-turn potentiometer requires 10,000 motor steps to cover its full range from 0 to 100 kilohms. This gives a minimum incrementing capability of 0.01 per cent.

Over-running of the potentiometer range is prevented by pointers attached

to the shaft which intersect beams of light to a set of three photodiode detectors. Whenever the zero end of the range is reached a signal is sent to the computer so that it can check the stored potentiometer position with the actual position.

Direct manual adjustment of the magnetic field is provided by two pushbuttons marked INCREASE and DECREASE respectively. A dial gives a visual indication of the potentiometer setting so that the relative field setting can be obtained. The absolute field depends on the coarse settings in the mass spectrometer console.

## 2.2 Data Collection

The data consists of a d.c. voltage level from a Cary vibrating reed electrometer which is connected to the ion flux detector within the mass spectrometer. The voltage level is converted to digital form by means of a voltage-to-frequency converter (VIFC) and a 23-bit scaler. The VIFC gives a pulse frequency proportional to the input voltage up to a maximum of 10 kHz for a 1 volt input. This output is fed into a scaler which is gated for a predetermined time. The count in the scaler is then proportional to the voltage level for any given gate time and is read directly into the computer.

## 2.3 Sensitivity Switching

There is a facility for switching the sensitivity of the electrometer-VIFC combination by changing from the 1 volt output-line to the 30 volt output-line of the electrometer. A relay across the lines from the electrometer switches them under computer control, and allows readings of small signals to be taken with 30 times the sensitivity of large signals.

## 2.4 Timing

The gate interval is controlled by the computer, which synchronises it to timed pulses from a tuning fork oscillator of 1 kHz. These pulses are also used to give correctly timed pulses to the stepping motor, avoiding the possibility of losing steps by moving it too quickly or suddenly.

## 3. THE PROGRAM

The program can be classified into three sections: input/output routines, mass spectrometer control routines and floating point arithmetic routines. Descriptions are given of the parts of the program relevant to the operation of the mass spectrometer. Further information on the more general aspects of input/output and arithmetic routines, together with a complete program listing, may be obtained from the A.A.E.C. program library.

### 3.1 Program Format

The program consists of three separate punched paper tapes: a long tape referred to as the MS5 8K version, and two short tapes referred to respectively

as the 4K overlay and the 8K overlay. These tapes give two versions of the program. The first version (corresponding to the main tape) can be changed to the second version by loading the 4K overlay after the main tape. Using the 4K overlay the whole program can be executed in 4096 words of memory with some restrictions on the maximum number of peaks and scans that can be accommodated. The first version may be recalled by loading the 8K overlay. The 8K overlay causes the program to operate in 8192 words of memory with an increase in the maximum number of peaks and scans. The memory allocations (octal) and maximum parameters are as follows:

#### 4K version

```

0000 - 5577   program
5600 - 6427   peak accumulation data area
6430 - 7777   scan accumulation data area

maximum number of peaks = 4
maximum window           = 200
maximum number of scans = 30

```

#### 8K version

```

0000 - 5577   program
5600 - 7777   peak accumulation data area
10000 -17777  scan accumulation data area

maximum number of peaks = 7
maximum window           = 500
maximum number of scans = 85

```

### 3.2 Interrupt Procedures

Four devices can interrupt the mass spectrometer mainline program and each device is serviced by its own interrupt program. The term, mainline program, refers to that part of the program which can be interrupted at any time by an external device requiring immediate attention. The part of the program which services external devices is referred to as the interrupt program and cannot be interrupted by another device. A description of the general method of handling interrupts is contained in the PDP-8/L software handbook.

The clock interrupt routine CLOCK is used to operate the motor at 500 steps per second and also to time accurately the operation of the scaler gate. When a motor adjustment is required the mainline program sets the flag MBLCK1, which is reset when the clock routine has moved the motor to the desired position. If the scaler is to be used, the mainline program deposits the gate time in milliseconds into the location CFLAG; the clock routine then opens the gate for the required

time, closes it, and resets CFLAG to zero. The CLOCK routine also checks the push-button status and steps the motor up or down accordingly at a rate of 50 steps per second.

The three other interrupt routines service the motor zero and the keyboard and teleprinter of the teletype console. The latter interrupts are part of the input/output procedures and are not dealt with here. The motor zero interrupt is considered in the next section.

### 3.3 Motor Drive Procedures

The actual position of the motor is stored as a double precision number in the locations ACTHI and ACTLO. To move the motor to a particular position between 0 and 10,000 motor steps, the desired position is deposited as a double precision number in locations HI and LO, and is followed by the instruction JMS I LMOTOR.

Three photosensitive diodes illuminated by light beams and intersected by pointers give the range and zero position of the potentiometer to the computer. Each time the zero position is reached the mainline program is interrupted and the routine MOZERO is entered. This routine checks that the actual position register contains zero; if not, it adjusts the register and causes ZERO ERROR to be typed out by the teleprinter. To allow for inertia the motor is started with a slow start procedure. It is stepped slowly at first and then progressively faster until it attains full speed. SS1 and SS2 are the slow start counters.

### 3.4 Data Collection Procedures

After the initial manual location of peaks the automatic peak-scanning facility can be used. The facility incorporates the following subroutines.

ACCUM - Accumulate. Readings from the VTFC are taken and stored for each motor step unit across a peak. The scan starts from  $A - W/2$  (where  $A$  is the peak address and  $W$  is the window, both in motor step units), moves upwards to  $A + W/2$  and then downwards to  $A - W/2$ . The successive values are stored as double precision numbers in the table TABST until the whole peak is scanned.

SUMPK - Sum the peaks. The values in TABST are scanned and the range between the two half-maximum values is selected. This range is reduced by one-tenth at each end and the average of all the readings between these two limits is taken. A background is obtained by taking the average of ten readings about the minimum reading. The difference between these two averages is the peak value and is stored as a floating number in the table CALPK.

CALC - Calculate. This subroutine uses the method of Whitten (1961, see Appendix 1) to calculate isotopic abundances from the peak data collected. A subroutine of CALC types out the isotopic abundances and their standard deviations.

#### 4. OPERATING INSTRUCTIONS

Communication between the operator and the computer is conducted through the teletype send-receive set. The operator types instructions at the keyboard and the computer requests further information and types out results on the teleprinter. All instructions consist of a slash followed by a two-letter mnemonic. Section 4.1 gives a complete list of the two-letter instructions available, together with an explanation of their usage. If a mistake is made by omitting the slash or by typing in an invalid two-letter combination (one not contained in the instruction repertoire), the instruction is not accepted by the computer and there is no print-out of the incorrect instruction. Following the acceptance of an instruction, the computer may request further information by typing out questions. The parameters requested must be provided by the operator before the next step in the sequence can take place. If an error is made in typing in the parameters then the instruction must be typed in again and all parameters must again be supplied. The computer monitors parameters as they are entered and will type out ERROR if the value is outside the limits. These limits are stated in Section 3.1 where relevant. On receiving an ERROR indication the operator must re-type the instruction and the parameters.

Appendix 2 demonstrates the use of these instructions during an actual scan.

##### 4.1 Table of Valid Instructions

/SF	Set field	}	Manual scan instructions
/ZF	Zero field		
/RF	Read field		
/IF	Increment field		
/ST	Set scaler gate time		
/TC	Take count		
/CR	Change range		
/TF	Teletype off	}	Computer operation instructions
/TN	Teletype on		
/OF	Set program end flag		
/CP	Cancel program		
/PH	Punch heading		
/PL	Punch leader		
/??	Re-initialise	}	
/SE	Search for peaks		
/DP	Define peaks		
/SC	Scan peaks	}	

/PO	Print out isotopic abundances	} Automatic scan instructions
/MX	Print out matrix of peaks	
/DO	Do calculation of isotopic abundances	

#### 4.2 Instruction Usage

The operator uses the various instructions as follows:

- /SF Set field. The operator types in the field required in motor step units (range 0 to 10,000) followed by a colon. When the required setting is reached the computer acknowledges this by typing out a carriage-return line-feed statement.
- /ZF Zero field. The motor unit is set to zero. If ZERO ERROR is typed out there is a discrepancy between the stored field value and the actual field value, which is automatically corrected when this error message occurs.
- /RF Read field. The current field setting in motor step units is typed out.
- /IF Increment field. The operator types in the required movement from the current field, in motor step units, followed by a colon. If a decrement is required a minus sign precedes the number.
- /ST Set scaler time. The gate time of the scaler, in milliseconds, is typed in and followed by a colon. Note that the /SE instruction automatically sets the gate time to 100 ms.
- /TC Take count. The computer types out  
Current scaler gate time setting in milliseconds  
Current field setting in motor step units  
Reading from the VTFC.
- /CR Change range. The current setting of the electrometer range is changed to the alternative setting. If it is 1 volt it is changed to 30 volts and vice versa.
- /TF Teletype off. This suppresses all print-out of alphameric messages other than error messages. The operator types in the required parameters as though the print-out was present.
- /TN Teletype on. This makes available all print-out of alphameric messages.
- /OF Set program end flag. This terminates a scan at the end of a sweep instead of leaving an incompleted sweep.
- /CP Cancel program. This instruction terminates any operation during its execution and restores the computer program to the waiting state.

/PH Punch heading. After this instruction the operator can type an alphameric heading for identification purposes.

/PL Punch leader-trailer. This is used to punch out a length of blank tape.

/?? Re-initialise. This resets all peripherals and restores the computer program to the waiting state.

/SE Search for peaks. The computer types out

ADDRESSES OF PEAKS = WINDOW = PEAK VALUE =

With the current setting of the motor unit used as the starting point, the field is incremented by intervals of ten motor step units, and at each step a reading is taken from the VTFC. Peaks are detected by monitoring these readings until one above 0.1 volts is obtained. This is taken as indicating the beginning of a peak. The field is set back ten motor step units and then incremented in steps of two, with readings taken at each step over the peak until the VTFC output again falls below 0.1 volts. This is taken as the end of the peak. The computer then types out the peak address, the window and the peak value.

Peak address : the field setting for the maximum reading

Window : the range of field between the two minimum readings of 0.1 volts

Peak value : the maximum reading

After the peak parameters are typed out, the computer reverts to incrementing the field in intervals of ten motor step units, monitoring the VTFC readings at each step until another reading above 0.1 volts is obtained. The process for obtaining the peak address, window and peak value is then repeated for this new peak. The operation of the /SE instruction continues until the whole range of the motor unit is covered. The instruction /CP can terminate the operation at any time.

/DP Define peaks. The computer types out

NO. OF PEAKS =

WINDOW =

ADDRESSES OF PEAKS =

RANGE (1 OR 30)

PK1 = PK2 = PK3 = PK4 =

After each equals sign the operator types in the parameter required and follows it by a colon. These parameters should be typed in as follows:

NO. OF PEAKS =. Type in the number of separate peaks to be scanned. Note that with the 4K version of the program the maximum number of peaks is 4, and with the 8K version the maximum number of peaks is 7.

WINDOW =. Type in the width of the field, in motor step units, required to contain the whole peak. The same window is used for all peaks so the maximum window for the set of peaks should be entered. Note that with the 4K version the maximum window is 200 and with the 8K version the maximum window is 500.

ADDRESSES OF PEAKS =. Type in the field settings, in motor step units, which give the approximate maximum readings for each peak. During the scan the computer takes one reading per step between the values  $A - W/2$  and  $A + W/2$ , where A is the peak address and W is the window. As each peak address is typed it is followed by a colon.

PK1 = PK2 = etc. Type in 1 or 30 to select sensitivity for each peak. For example PK1 = 1 PK2 = 30 would increase the sensitivity of the VIFC thirty times for the second peak and this range would be used whenever readings are taken for the second peak during the scan.

/SC Scan. The computer types out

NO. OF SCANS =

SCAN TIME IN MINS. =

After the first equals sign the operator types in the parameter followed by a colon. The parameter is arrived at as follows:

NO. OF SCANS =. A complete scan consists of one upward sweep over the peaks nominated in the /DP instruction followed by one downward sweep over those peaks. For the purposes of reducing systematic error in the abundance calculation the scan always ends with an upward sweep, so the number entered represents the number of upward-downward sweeps plus one further upward sweep. Note that with the 4K version the maximum number of scans is 30 and with the 8K version the maximum number of scans is 85. Before each sweep across each peak is commenced there is a ten-second delay to allow the mass spectrometer field to settle down to a steady value.

After the second equals sign the computer types out the time for a complete scan, informing the operator when to expect the results. This calculation is only approximate, to within two or three minutes.

During a scan the computer may type out the word

## DRIFT

This occurs if the peak is no longer in the window of field nominated in the /DP instruction. The results of any scans done before the drift occurred may be obtained by typing the /PO instruction.

During a long scan the teletype console should be switched off to reduce wear. The operator can then obtain the results of the scan by turning on the teletype and typing in /PO.

/PO Print out isotopic abundances. The computer types out

COMPLETED SCANS =  
Isotopic abundances  
Standard deviations

for the isotopes nominated in the /DP instruction. The results are typed out along the line in the order in which the peaks were typed in. Along the next line are typed the standard deviations of the accumulated data for each peak. If the number of completed scans is fewer than requested then a drift has occurred.

/MX Print out matrix of peaks. The computer types out

NO. OF PEAKS =  
NO. OF SCANS =  
Matrix of the peaks

After each equals sign the operator types in the relevant parameter followed by a colon. This instruction at the completion of a scan prints out the result of each scan over each peak. It is used to sort out any noise reading which is far in excess of the other values for the same peak and hence is causing a large standard deviation for that peak.

/DO Do calculation of isotopic abundances. The computer types out

FIRST ROW =  
LAST ROW =  
Isotopic abundances  
Standard deviations

After each equals sign the operator types in the relevant parameter followed by a colon. This instruction is used in conjunction with the /MX instruction. The rows of the matrix of values printed by /MX are numbered so that the row containing the error due to noise can be eliminated from the calculation of isotopic abundances. The computer calculates the results from the measurements contained in the

nominated first row up to and including those contained in the nominated last row.

5. REFERENCE

Whittem, R. N. (1961). - Methods of calculation of isotopic abundances from MS5 mass spectrometer scans. AAEC/TM72.

## APPENDIX 1

### ISOTOPIC ABUNDANCE MEASUREMENT

To reduce the systematic error due to source decay in isotopic abundance measurement a method employing the average of peak values from adjacent scans is used to obtain isotopic abundances (Whittem 1961). The method is illustrated by arranging raw data from a scan in a matrix P whose rows are peaks of all the isotopes from a single sweep and whose columns are peaks of the same isotope from successive sweeps. From the elements of P a new matrix P' is calculated where

$$P'_{m,n} = \frac{P_{m,n} + P_{m+1,n}}{\sum_i (P_{m,i} + P_{m+1,i})}$$

where  $i = 1$  to  $N$  and  $N$  is the number of peaks.  $P_{m,n}$  is the reading of the  $n^{\text{th}}$  peak during the  $m^{\text{th}}$  sweep. The means of the columns of P' are the isotopic abundances of the source.

APPENDIX 2

SAMPLE SCAN ON RUBIDIUM

The computer output on the following page is the result of a scan on a sample of rubidium. Initial investigation has shown that two peaks occur around about the positions 2068 and 2919 motor step units and this information is entered using the /DP instruction. /ST allows the scaler gate to be set to 166 milliseconds and finally /SC starts the scan. Ten scans are requested and the computer indicates that this will take 17.92 minutes.

At the completion of the scans the computer types out that 10 scans have been done. Hence there has been no drift of the peaks out the scan windows and the isotopic abundances and standard deviations are typed out. These are read as follows:

	<u>Peak 1</u>	<u>Peak 2</u>
Abundance	0.7244	0.2755
Standard deviation	0.0012	0.0012

The operator can request the individual peak values using /MX which types them in columns for each peak. Then using the /DO instructor, abundances and standard deviations are worked out for two groups of 5 scans. This method is used for eliminating a sweep which is obviously in error because of noise.

Finally in the example the operator has used the /PH instruction to type in the result that  $^{85}\text{Rb}$  constitutes 72.46 per cent of the sample and  $^{87}\text{Rb}$  constitutes 27.54 per cent.

APPENDIX 2 (continued)

/DP  
NO. OF PEAKS= 2:  
WINDOW= 150:  
ADDRESSES OF PEAKS= 2068:2919:  
RANGE (1: OR 30:)  
PK1= 1: PK2= 1:

/ST  
166:

/SC  
NO. OF SCANS= 10:  
SCAN TIME IN MINS. = 17.92:  
COMPLETED SCANS= 10:  
0.7244: 0.2755:  
0.0012: 0.0012:

/MX  
NO. OF PEAKS= 2:  
NO. OF SCANS= 10:  
1: 637.6: 243.2:  
2: 646.0: 242.2:  
3: 647.3: 246.2:  
4: 651.9: 247.1:  
5: 654.6: 248.5:  
6: 658.9: 248.6:  
7: 656.9: 251.7:  
8: 666.7: 252.8:  
9: 668.4: 254.5:  
10: 676.7: 254.7:  
11: 679.8: 268.8:  
12: 709.4: 269.1:  
13: 713.0: 271.8:  
14: 720.2: 272.0:  
15: 719.0: 274.1:  
16: 727.0: 273.3:  
17: 720.2: 277.7:  
18: 730.3: 277.8:  
19: 732.0: 279.0:  
20: 743.6: 279.0:  
21: 737.6: 282.4:

/DO  
FIRST ROW=1:  
LAST ROW=11:  
0.7246: 0.2753:  
0.0011: 0.0011:

/DO  
FIRST ROW=11:  
LAST ROW=21:  
0.7242: 0.2757:  
0.0013: 0.0013:

/PH

ABUNDANCE      RR 85 72.46%  
                  RR 87 27.54%



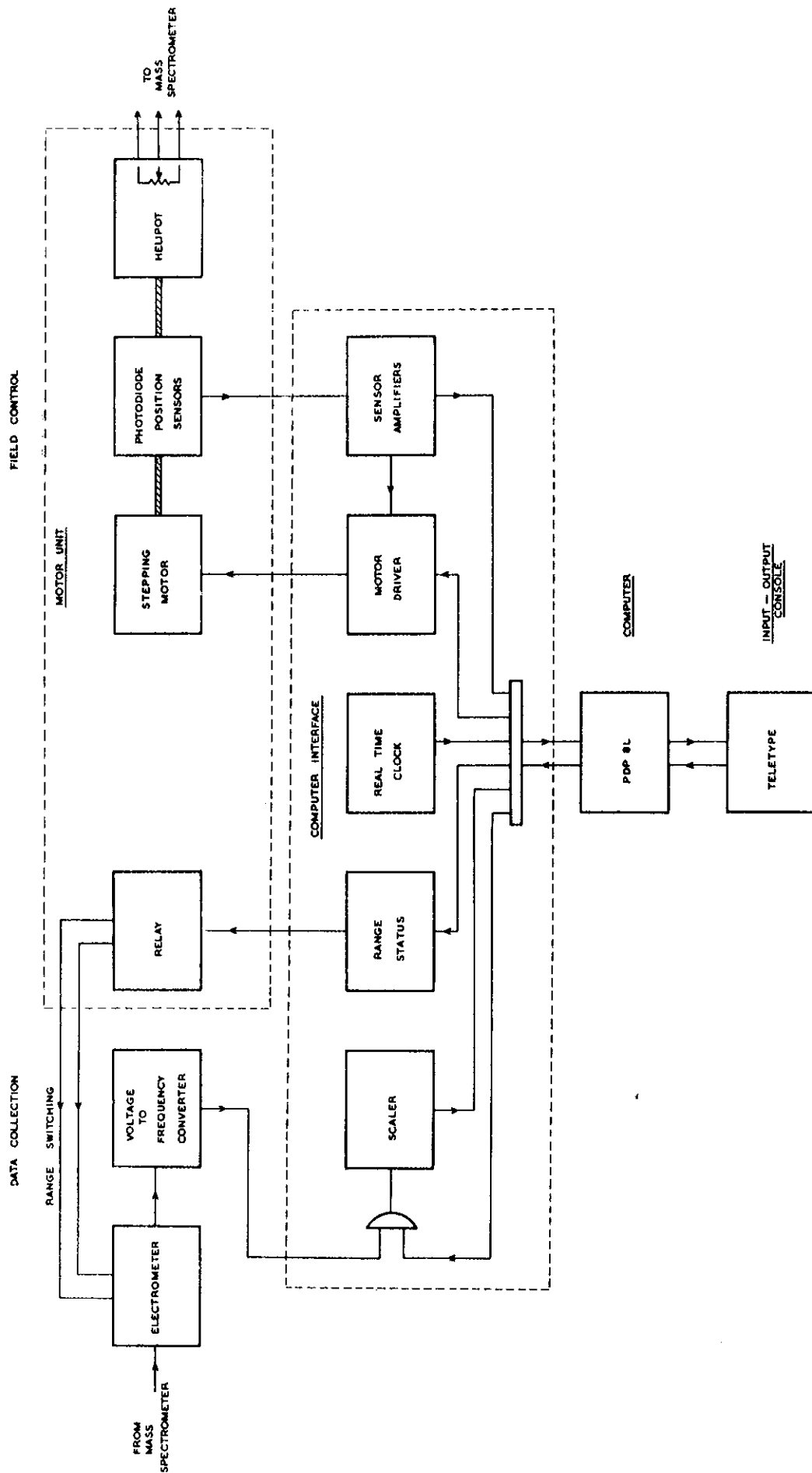


FIGURE 1. MASS SPECTROMETER INTERFACE BLOCK DIAGRAM

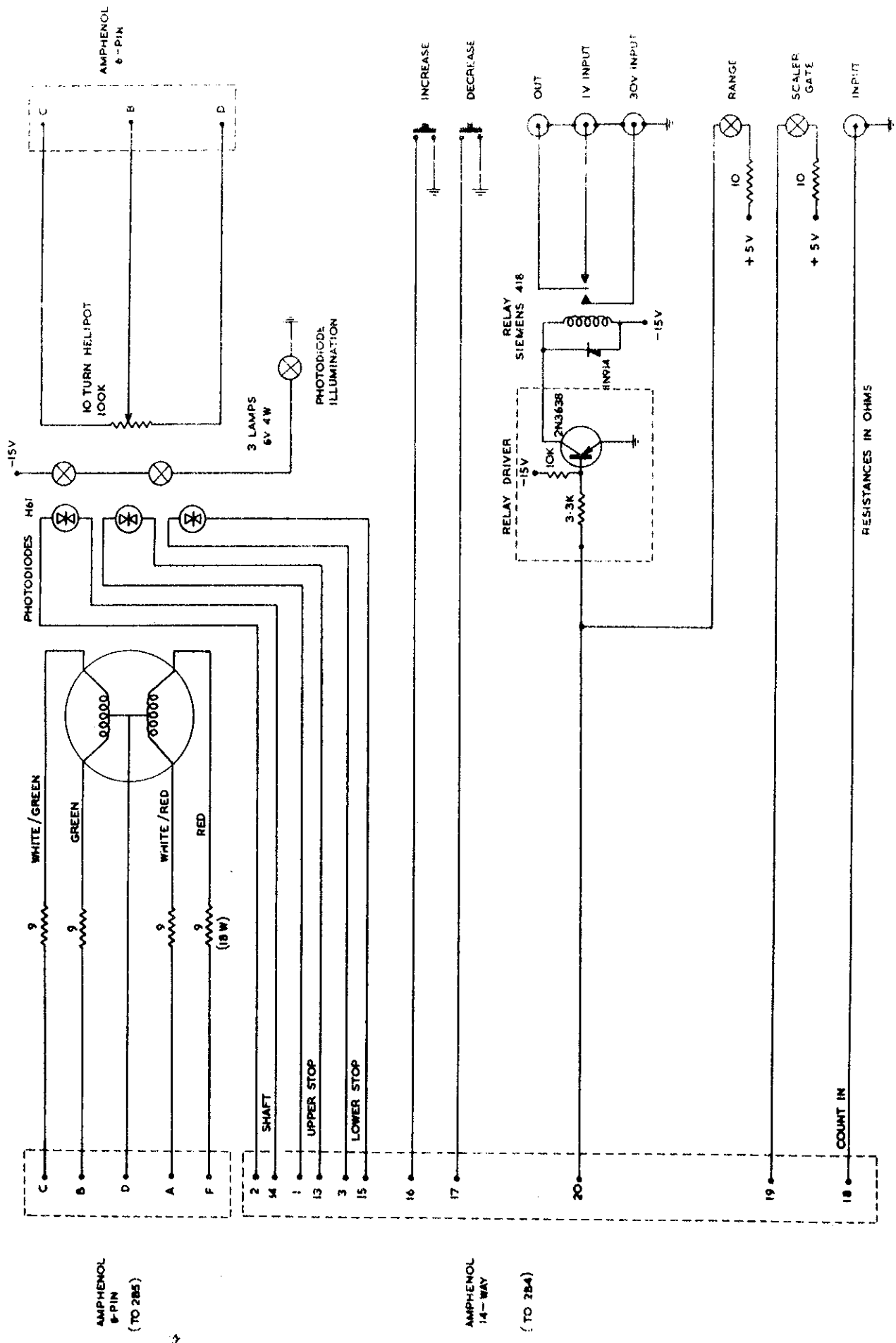


FIGURE 2. MOTOR UNIT CIRCUIT



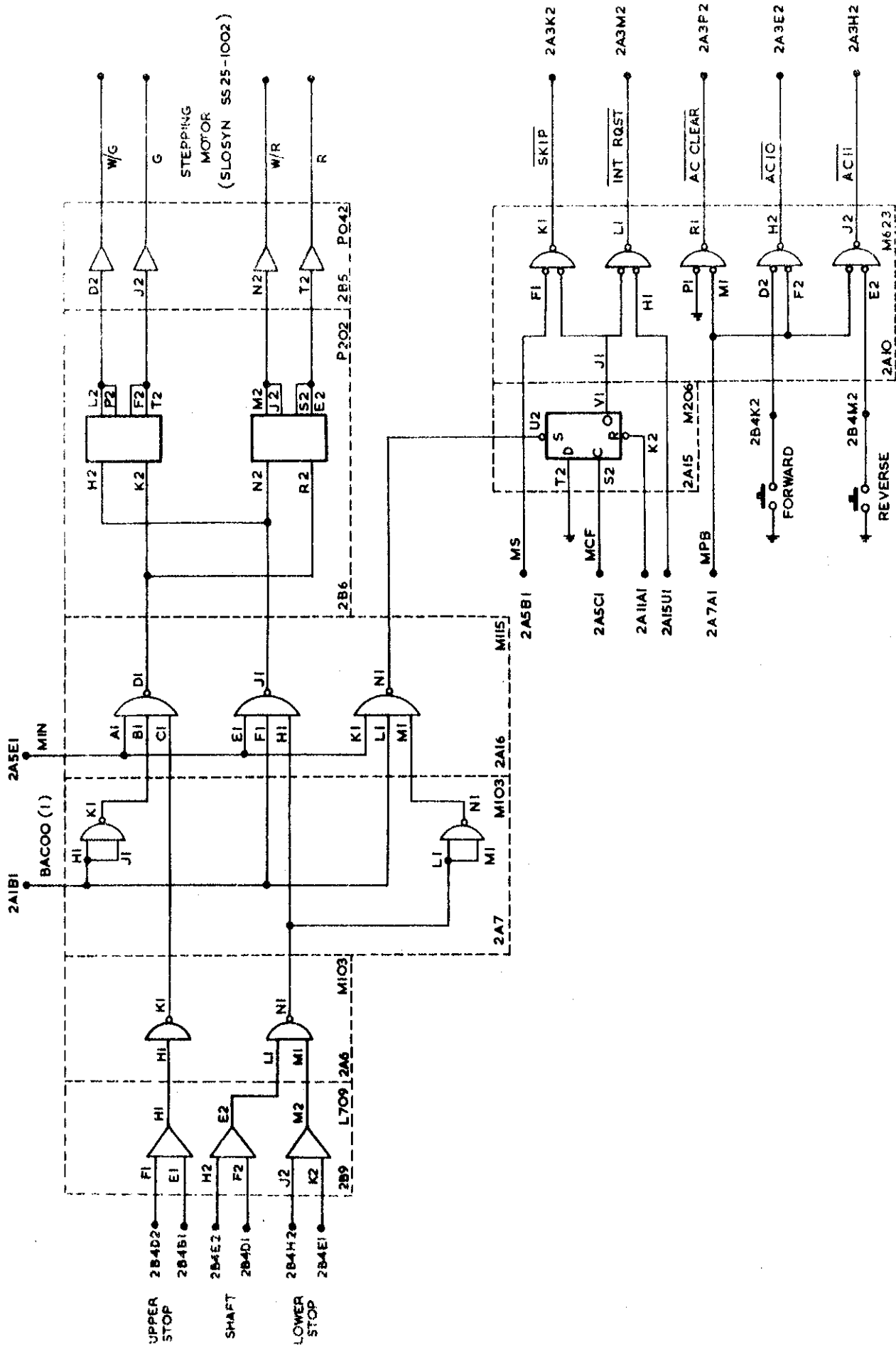


FIGURE 4. MOTOR DRIVER CIRCUIT

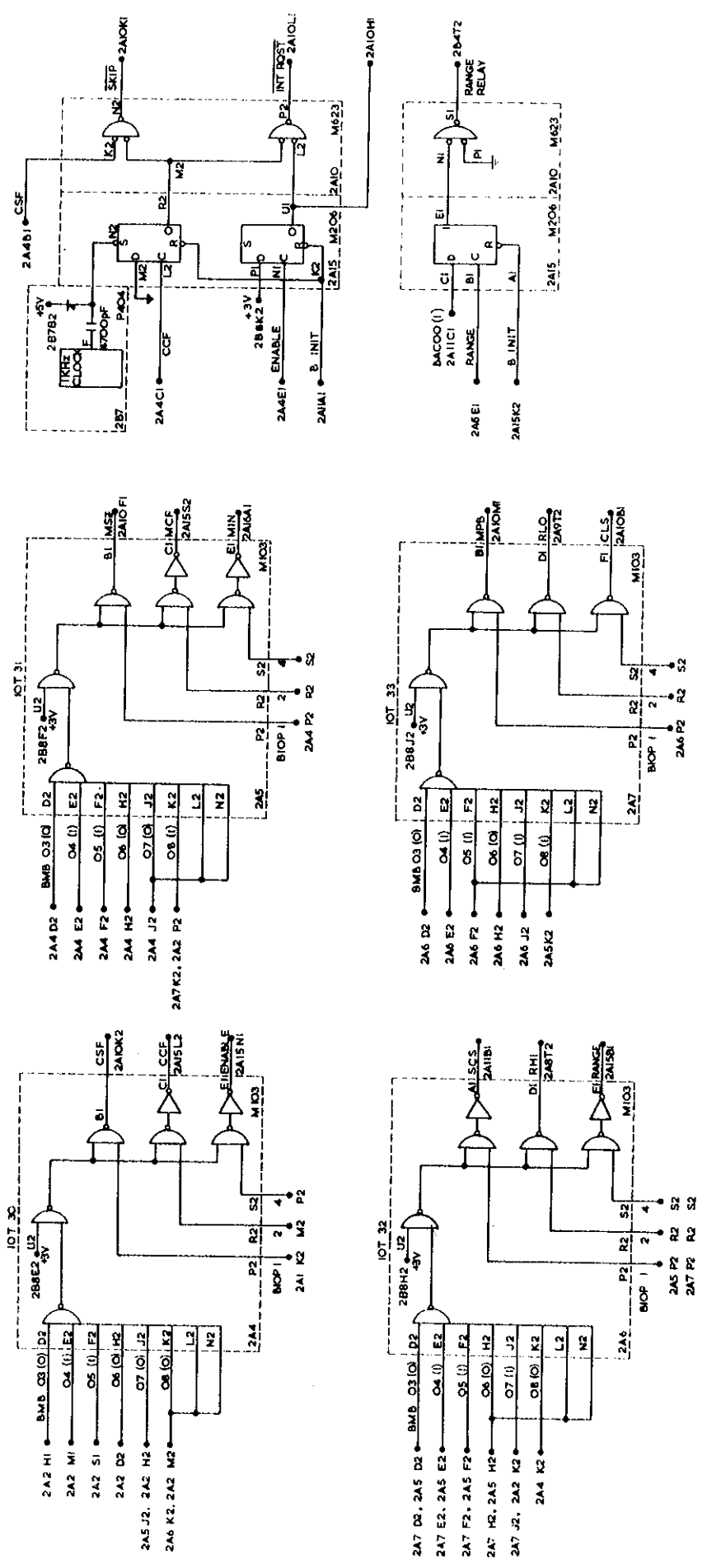


FIGURE 5. DEVICE SELECTOR AND REAL TIME CLOCK CIRCUITS

2A1	2A2	2A3	2A4	2A5	2A6	2A7	2A8	2A9	2A10	2A11	2A12	2A13	2A14	2A15	2A16
M903	M903	M903	M103	M103	M103	M103	M623	M623	M623	M206	M206	M206	M206	M206	M115
BAC OO TO II  BIOP 1,2,4  B INIT	BMB OO TO II  AC OO TO II SKIP INT RQST AC CLEAR	AC OO TO II SKIP INT RQST AC CLEAR	M103	M103	M103	M103	M623	M623	M623	M206	M206	M206	M206	M206	M115
2B1	2B2	2B3	2B4	2B5	2B6	2B7	2B8	2B9	2B10	2B11	2B12	2B13	2B14	2B15	2B16
M903	M903	M903	M903	PO42	P202	P404	POO3	L709							
BAC OO TO II  BIOP 1,2,4  B INIT	BMB OO TO II  AC OO TO II SKIP INT RQST AC CLEAR	AC OO TO II SKIP INT RQST AC CLEAR	M903	PO42	P202	P404	POO3	L709							

FIGURE 6. CARD ALLOCATION SCHEDULE

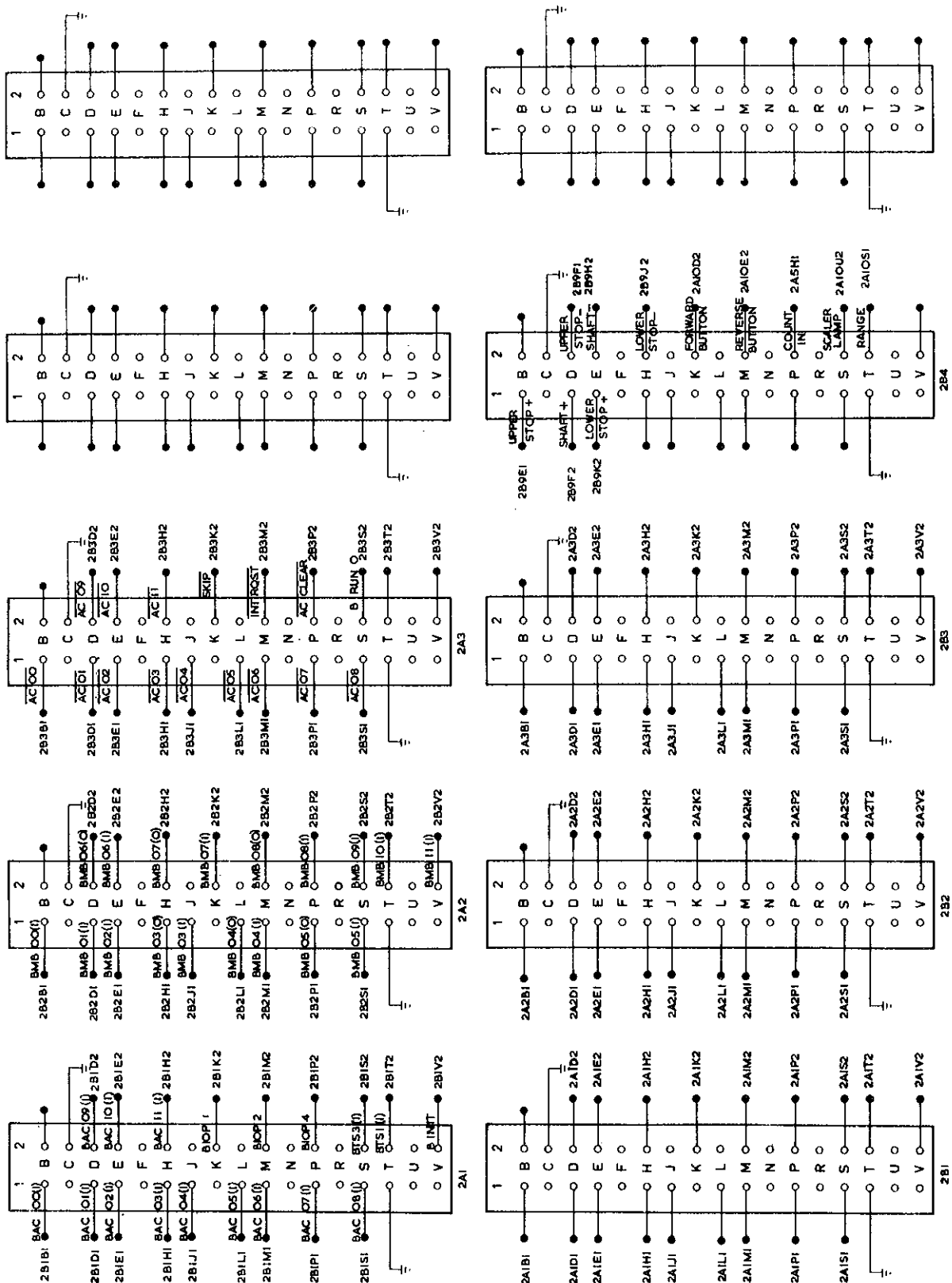


FIGURE 7. INPUT/OUTPUT WIRING SCHEDULE

