

CHARACTERIZATION OF COAL-DERIVED LIQUIDS AND OTHER FOSSIL FUEL
RELATED MATERIALS EMPLOYING MASS SPECTROMETRY

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CHARACTERIZATION OF COAL-DERIVED LIQUIDS AND OTHER FOSSIL FUELS
RELATED MATERIALS EMPLOYING MASS SPECTROMETRY
(FE 2537-9)

ABSTRACT

During the past quarter the standard electron multiplier of the CEC 21-110B mass spectrometer was replaced with a Hamamatsu electron multiplier. The change allowed the electron multiplier to be operated at a higher gain with about the same noise level on the output signal as with the standard electron multiplier operating at lower gains. The dynamic resolution of the mass spectrometer was determined when the static resolution was set for a value of 13,000.

Field-ionization mass spectra, FI/MS, were obtained for the following saturate fractions: Arco Shale Concentrates, 200-325°C, 325-425°C, and 425°C+; South Swan Hills Concentrate 370-535°C; and Utah Crude Oil #72054. The molecular ion intensities in the FI/MS of each sample were converted into carbon number distributions as a function of Z(H). The weight percents across each Z(H) series were summed to obtain total weight percents as a function of saturate type.

Steps in completing the interfacing of the Nova 3/12 data acquisition system to the comparator-microdensitometer have been taken during this quarter. An outline of the steps which have been completed and those which will be completed to implement the interfacing is given. Also preliminary designs are given in this report for the interface circuitry and the interface software.

High- and low-resolution field-ionization mass spectral data were acquired for 19 monoaromatic, diaromatic, and polyaromatic polar concentrations. These samples were analyzed for the Separations and Characterization Group at the Bartlesville Energy Technology Center. Also acquisition of high-resolution field-ionization mass spectral data for the remaining asphaltene fractions from the COED coal liquid was completed.

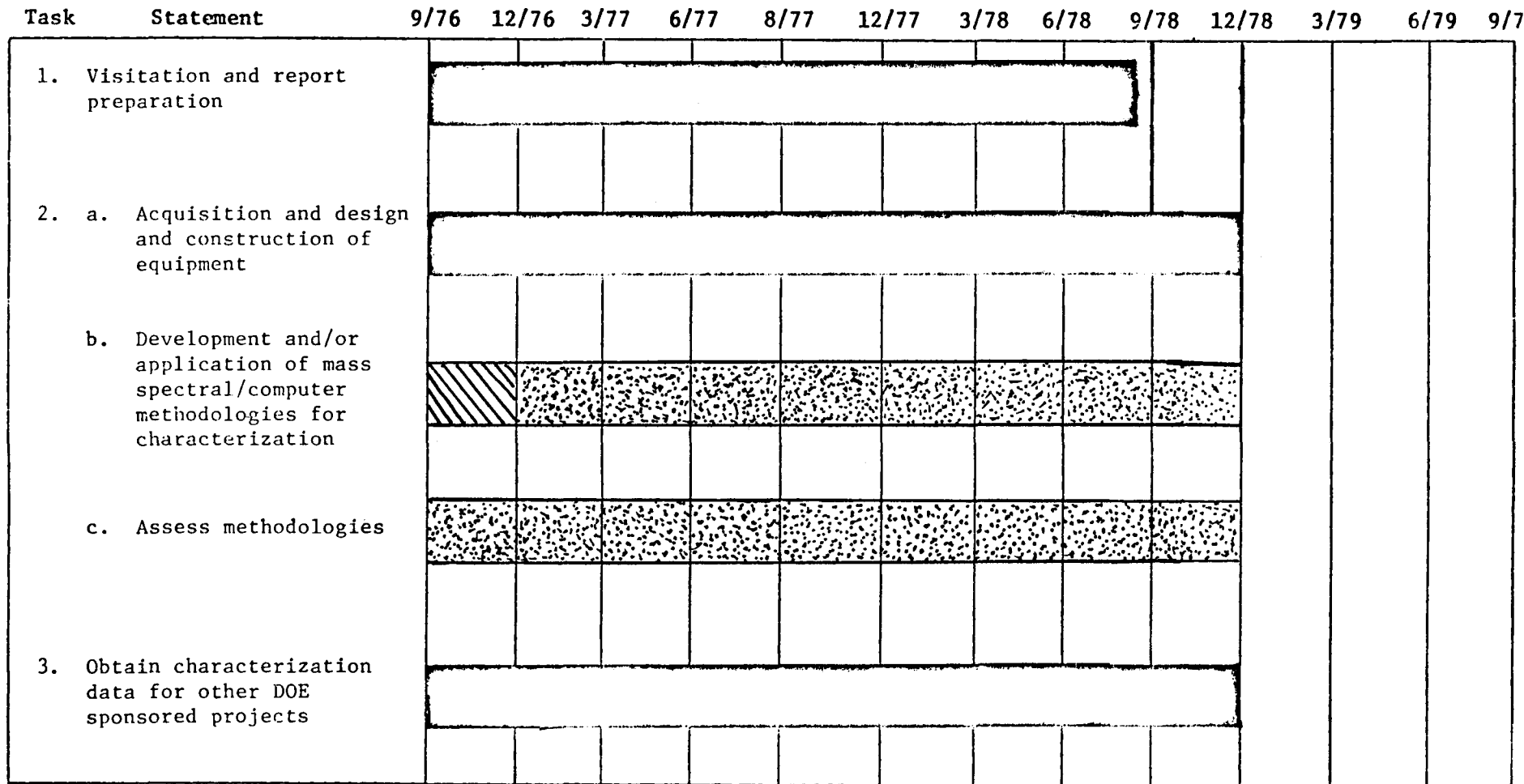
OBJECTIVE AND SCOPE OF WORK

The objectives of this program are as follows. First, to develop new and refine existing mass spectrometric techniques for obtaining routine and detailed characterization data for coal-derived liquids and other fossil fuel related materials. The existing mass spectrometric facilities are being augmented by addition of a dedicated data acquisition system, a comparator/microdensitometer, a temperature control display module for the solid introduction probe inlet, modification of the FI/EI source to operate in the field-desorption mode, and ancillary gas chromatographic equipment. Hardware and software to permit computer acquisition and processing of data from the mass spectrometer and peripheral instrumentation will be developed in detail. Using this augmented system, the following techniques will be routinely applied to the characterization of fossil fuels: a) high- and low-resolution electron-impact, field-ionization, and field-desorption mass spectrometry and b) micromolecular probe distillation and simulated distillation in conjunction with mass analysis. The utility of the newer techniques for conducting such analyses will also be evaluated. Second, the existing mass spectrometer facility and the equipment being acquired and the associated analytical methodologies available and under development will be used to obtain analytical data for other DOE sponsored projects. Third, preparation of a document for DOE which assesses both the current state-of-the-art in mass spectral analysis of alternate fossil fuels and the needs of technical personnel for characterization data. Fourth, to provide interdisciplinarily trained analysts capable of meaningful participation in the overall effort to achieve greater national energy independence and in other contemporary scientific problems of national significance.

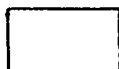
SUMMARY OF PROGRESS TO DATE


The following chart summarizes the progress to date. Task 1 has been completed. Task 2 activities include: 1) installation of the DS-50S data acquisition system, 2) installation and distribution of well regulated 120V AC and an excellent electrical ground to MS laboratory instrumentation, 3) optimization of CEC 21-110B mass spectrometer performance via major electronic modifications and maintenance, 4) optimization of system parameters for the acquisition of both low- and high-resolution mass spectral data from the CEC 21-110B in real time via the DS-50S data system, 5) implementation of communications between the NOVA 3/12 mini - computer and the IBM 370/158, 6) development of software and acquisition of hardware for implementing micromolecular probe distillation in combination with field-ionization mass spectrometry, 7) modification of the FI/EI source to operate in the field-desorption mode and construction of an apparatus for preparation of FD emitters, 8) installation of the gas chromatographic digital integrater, and 9) preparation of an initial design for an interface between the NOVA 3/12 and the comparator/microdensitometer. Task 3 activities include support of our characterization research in collaboration with J. E. Dooley's and B. L. Crynes' DOE-sponsored projects at BETC and OSU, respectively.


As of December 29, 1978, 71.0% of the total budget has been spent. This rate of expenditure is consistent with our schedule.




4

 Scheduled

 Early Start

 Schedule Extension

 Progress

 Unscheduled

DETAILED DESCRIPTION OF TECHNICAL PROGRESS

Task 1 - Preparation of a Document Which Assesses Both the Current State-of-the-Art in Mass Spectral Analysis of Alternate Fossil Fuels and the Needs of Technical Personnel for Characterization Data

This task was completed during the quarter March 30, 1978 - June 29, 1978. The document has been published by the Technical Information Center as DOE report FE 2537-7, Dist. Category UC-90d.

Task 2 - Modification and Augmentation of Existing Mass Spectrometer Facilities and Their Application to the Routine Characterization of Fossil Fuels

Task 2 research is under the direction of S. E. Scheppele and G. E. Hedrick in the Departments of Chemistry and Computer Science, respectively. Other individuals participating in these activities are Drs. Q. G. Grindstaff, and C. S. Hwang, Ms. Michele Derrick and Messrs. P. A. Benson, N. B. Perreira, and K. N. Detwiler. Dr. Scheppele is currently on a leave of absence and is at the Bartlesville Energy Technology Center. During this period of time, Dr. Grindstaff is supervising activities of the group in the Department of Chemistry with Dr. Scheppele consulting on a regular basis.

The electron multiplier of the CEC 21-110B mass spectrometer was replaced with a Hamamatsu electron multiplier (1). The change was made because the Hamamatsu multiplier can be operated at higher gains with less noise being produced on the signal from the multiplier than with the standard CEC electron multiplier. The change allows us to acquire data at a lower source pressure which in turn results in the ion source of the mass spectrometer remaining clean for a longer period of time. Also the lower noise factor improves the mass data taken by the computer (Nova 3/12 - DS-50S) since excessive noise can cause peak splitting which results in poor mass assignment.

The high-resolution electron-impact spectral data given in the previous quarterly report (2) was taken at a static resolution of 13,000. In that report it was stated that a dynamic resolution of at least 8,400 was achieved at a static resolution of 13,000. By the use of a benzene-pyridine mixture it was found that a dynamic resolution of approximately 10,000 is achieved at a static resolution of 13,000. The dynamic resolution measurements were made at magnetic scan speeds of 88 and 156 seconds per decade, and both were found to give the same dynamic resolution. Thus, the dynamic resolution at a static resolution of 13,000 is considerably higher than first estimated.

Field-ionization mass spectra, FI/MS, were acquired in order to obtain a qualitative and quantitative analysis of the following saturate fractions: Arco Shale Concentrates 200-325°C, 325-425°C, and 425°C+; South Swan Hills Concentrate 370-535°C; and Utah Crude Oil #72054. These saturated hydrocarbon fractions were produced by separation of the original samples with high pressure liquid chromatography on an alumina-silica column equipped with a sensitive UV detector. The separation was performed by the Separation and Characterization Group at the Bartlesville Energy Technology Center.

Field-ionization mass spectral data allow for a molecular-ion group-type characterization of these saturate fractions by providing carbon number distributions as a function of hydrocarbon type utilizing available sensitivity coefficient data. At high ion source temperature (300°C), the relative gram sensitivities for FI of the n-alkanes are essentially independent of the number of carbon atoms in the n-alkanes. Thus only the relative gram sensitivity for the first member of a saturated-hydrocarbon-type series has to be determined in order to calculate compositional data. Prediction of the relative gram sensitivities of the first member of a saturated-hydrocarbon-type series can be made by using the following equation (3):

$$\frac{s(g)_i}{s(g)_R} = \frac{(MW_R) \{(\Delta/C_i) [N(C)_i - N(C)_R] + 1\}}{14 N(C)_i + Z}$$

where $s(g)_i$ and $s(g)_R$ are the gram sensitivity of the i^{th} and reference compounds, respectively, MW_R is the molecular weight of the reference compound, Δ/C_i is the relative mole sensitivity per carbon, and $N(C)_i$ and $N(C)_R$ are the number of carbons in the i^{th} and reference compounds, respectively. The values of the relative gram sensitivities used in the present analysis of the saturate-hydrocarbon fractions are given in Table I. The weight percents for each saturate group type in each of the saturate fractions are given in Tables II-VI. Each sample was introduced into the batch inlet system at a temperature of 320°C. The ion source was also maintained at a temperature of 320°C during the runs. The mass spectra were acquired at a magnetic scan speed of 45 seconds per decade by the Nova 3/12 - DS-50S data acquisition system. These results are currently being compared with the corresponding analyses obtained using conventional 70-eV fragment ion group type methods. In addition the carbon number distribution in the +2 series and distribution of weight percents across Z series for Utah Crude (see Table VI) are being compared with results obtained from gas chromatographic analysis of the sample.

The comparator-microdensitometer is being interfaced with the Data General Nova 3/12 computer in order to allow the electro-mechanical acquisition of data from photographic plates. This interface is proceeding as described below.

An additional hardware logic board is required for the Nova 3/12 CPU (central processing unit) in order to allow a VDU (visual display unit) to be dedicated to the program which is operating in the foreground mode. (The Nova 3/12 allows separate and independent programs to operate at the same time. The first of these is said to operate in the background [mode], the second is said to operate in the foreground [mode].) This logic board includes an EIA RS-232-C interface (Electronic Industries Association Standard serial data transfer circuitry) which will be used to transfer information directly to the VDU and indirectly to the comparator-microdensitometer. Thus this additional hardware logic board will allow the computer to do two jobs at the same time. That is, it can take data from the mass spectrometer and the comparator at the same time.

The complete AEI data acquisition system uses the Data General RDOS operating system (real-time disk operating system). This system has been modified by AEI for use in the data acquisition system. These modifications

TABLE I
RELATIVE GRAM SENSITIVITIES VALUES
FOR SATURATE HYDROCARBONS

Z Series	First Member of Series	$s(g)_i/s(g)_R$
+2	C ₁₀ H ₂₂	1.0
0	C ₆ H ₁₂	2.2
-2	C ₁₀ H ₁₈	4.1
-4	C ₁₃ H ₂₂	5.2
-6	C ₁₆ H ₂₆	5.5
-8	C ₁₉ H ₃₀	5.8
-10	C ₂₂ H ₃₄	6.1

TABLE II
Z-SERIES DISTRIBUTION FOR SATURATE
HYDROCARBON FRACTION ARCO SHALE OIL 200-325°C

Z Series	Mass Range	Carbon Number Range	Weight Percent
2	170-254	12-18	23.8
0	168-280	12-20	37.6
-2	152-278	11-20	28.6
-4	164-276	12-20	8.7
-6	218-274	16-20	1.0
-8	244-300	18-22	0.3
-10	---	---	---

TABLE III

Z-SERIES DISTRIBUTION FOR SATURATE
HYDROCARBON FRACTION ARCO SHALE OIL 325-425°C

Z Series	Mass Range	Carbon Number Range	Weight Percent
2	268-380	19-27	18.3
0	266-392	19-28	22.7
-2	250-404	18-29	19.1
-4	248-416	18-30	21.1
-6	246-400	18-29	13.6
-8	272-412	20-30	4.2
-10	312-396	23-29	1.0

TABLE IV

Z-SERIES DISTRIBUTION FOR SATURATE
HYDROCARBON FRACTION ARCO SHALE OIL 425+°C

Z Series	Mass Range	Carbon Number Range	Weight Percent
2	380-548	27-39	13.9
0	378-560	27-40	12.5
-2	376-572	27-41	9.8
-4	374-556	27-40	9.3
-6	358-554	26-40	18.3
-8	356-552	26-40	32.9
-10	368-550	27-40	3.3

TABLE V

Z-SERIES DISTRIBUTION FOR SATURATE
HYDROCARBON FRACTION SOUTH SWAN HILLS (HPLC)

Z Series	Mass Range	Carbon Number Range	Weight Percent
2	338-548	24-39	13.5
0	308-560	22-40	15.4
-2	292-544	21-39	13.1
-4	304-556	22-40	14.2
-6	288-540	21-39	23.7
-8	328-538	24-39	15.3
-10	326-536	24-39	4.8

TABLE VI

Z-SERIES DISTRIBUTION FOR SATURATE
HYDROCARBON FRACTION 72054-UTAH CRUDE (HPLC)

Z Series	Mass Range	Carbon Number Range	Weight Percent
2	170-464	12-33	65.3
0	168-462	12-33	22.4
-2	180-432	13-31	7.5
-4	192-430	14-31	3.4
-6	260-414	19-30	1.0
-8	370-412	27-30	0.4
-10	---	---	---

are not available from any source other than AEI. A different form of RDOS is required for using the data system with two VDU's instead of with a single VDU. Consequently, such a form of RDOS must be ordered from AEI in order to use the second VDU.

After the VDU has been tested with the new AEI system, the implementation of the I/O (input/output) software logic drivers may begin. The final implementation and testing of the software logic drivers will not be completed until all new hardware is attached to the system. These software drivers are in the design stage at this time and will be discussed later in this report.

At the same time as the programmed logic drivers are being implemented, two stepper motors (HDM-170-2000-8) and two hardware logic drivers (VSD-8-3) will be ordered. These stepper motors and logic drivers will move the table of the comparator in the x-y direction upon command from the computer. These items should be attached to the comparator by a technician qualified to install finely tuned mechanisms.

At this time, the interfacing circuits should also be fabricated. These circuits must be the interface between the appropriate parts on the DG NOVA 3/12 and the comparator. Circuits will be required to interface with the AEI ADC (analog to digital converter) port and the RS-232-C port on the NOVA 3/12. Circuits will be required to interface with each hardware logic driver and with the %-intensity output of the comparator. The required circuits are shown in Figure 1.

The control signals for the vertical and horizontal stepper motors are derived from signals programmatically generated by the RS-232-C interface board of the Data General Computer. The circuit operation appears to the user, logically, as a serial signal arriving from the foreground VDU. The signal from the RS-232-C interface is translated using a 741 operational amplifier to map -12V to 5V and 12V to 0V. The translated signal is indicated as SI in Figure 1. This signal is applied to the serial input pin (pin 20) of the UART (universal asynchronous receiver/transmitter; a parallel to serial transmitter and a serial to parallel receiver). The UART converts the serial signal into seven signals (d_0 , d_1 , d_2 , d_3 , d_4 , d_5 and d_6) in parallel. The parallel signals are decoded to determine the control signal to be sent to the stepper motor hardware logic drivers. The decoding is done by the 74318 multiplexer shown in Figure 1. The control signals to the stepper motor control interface are:

- 1) FWD(H) - step the horizontal stepper motor one increment forward. This is generated when an ASCII 00000001 is received from the RS-232-C board. ASCII stands for American Standard Code for Information Interchange.
- 2) BK(H) - step the horizontal stepper motor one step backward. This is generated when an ASCII 00000010 is received.
- 3) FWD(V) - step the vertical stepper motor one step forward. This is generated when an ASCII 00000011 is received.
- 4) BK(V) - step the vertical stepper motor one step backward. This signal is generated when an ASCII 00000100 is received.
- 5) HALT1 - stop the process and lock out any further signals [BK(V), BK(H), FWD(V), and FWD(H)] until the reset button is pushed.

The clock for the UART is generated by the 555 timer. As shown in Figure 1 the clock speed is selected using the 7493₁ counter (4-bit binary counter). The control select signals for parity, stop bits, number of bits, and type of

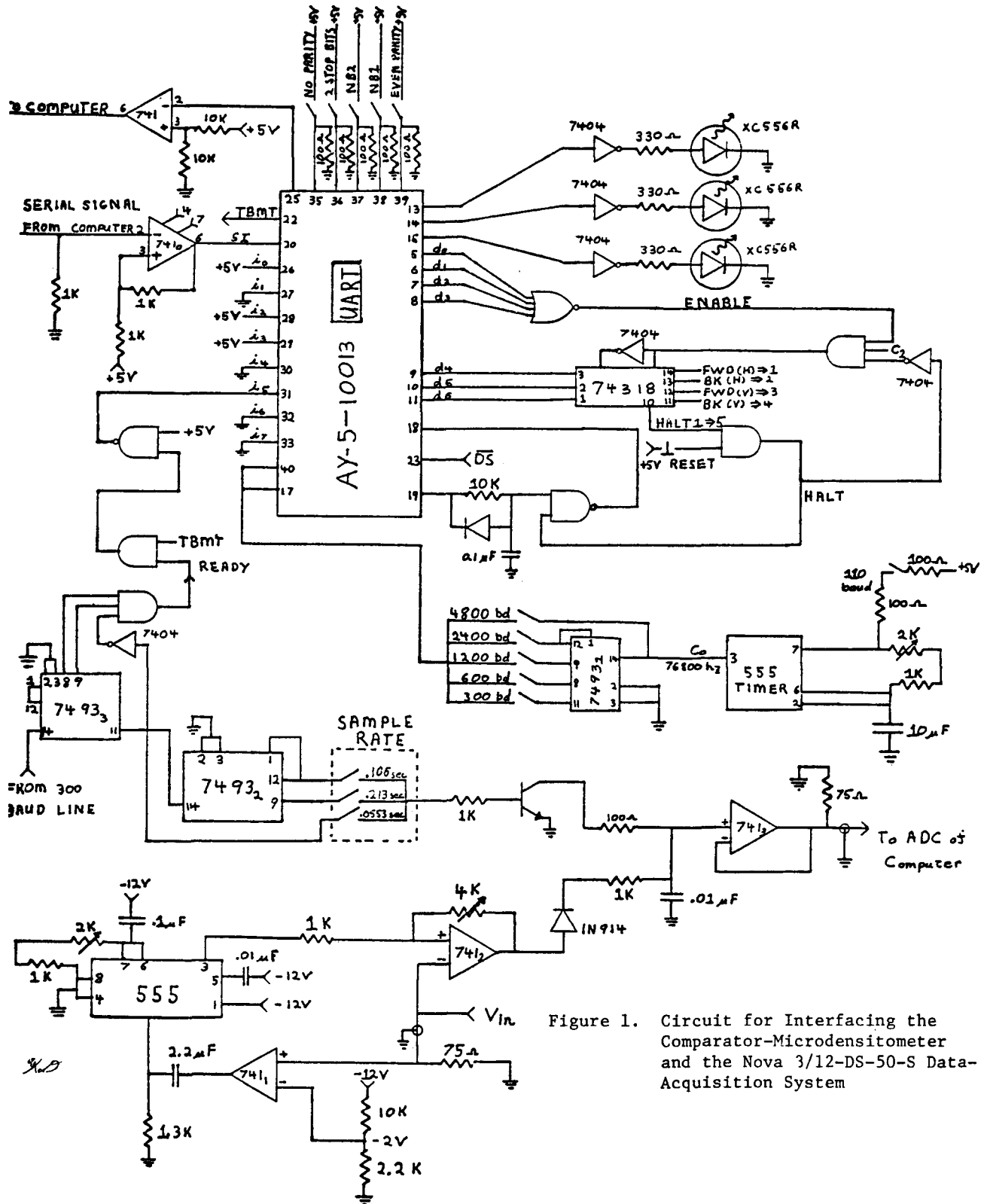


Figure 1. Circuit for Interfacing the Comparator-Microdensitometer and the Nova 3/12-DS-50-S Data-Acquisition System

parity are shown on Figure 1 and are switch selectable. If the control select signals do not match with incoming data then one of the lights on pin 13, 14, or 15 of the UART (Figure 1) will turn on.

The sample rate is selected by the three switches in Figure 1 labelled Sample Rate which select a rate from the 7493₂ and 7493₃ counters (4-bit binary counter). The Ready signal (Figure 1)² is generated in the middle of the pulse from the comparator by the decoder circuit from the same set of counters. This signal is generated to indicate that the analog output signal is stable and can be sampled. The Ready signal also causes an 00001101 to be placed on the UART input lines and a data strobe signal to be generated. This will send an ASCII 13 to the computer to initiate a sample.

If the moving of the vertical and horizontal stepper motors are to be stopped, a halt signal is sent from the computer and interpreted by the 74318 multiplexer as a HALT1 signal. The HALT1 signal causes the monostable (one shot) on the UART pins 18 and 19 to be disabled which does not allow the UART to reset. This will disable all output from the UART until the reset button (Figure 1) is pushed which will reset the UART and reenable the 74318 multiplexer. Thus this part of the circuit in Figure 1 controls the stepper motors which drive the table of the comparator and controls when the computer is to sample the signal from the comparator. After the commands to move the table of the comparator, sampling rate, sample time, and other needed commands have been received, the data from the photographic plate on the comparator table is taken by the computer in the following method. The voltage from the comparator-microdensitometer which results when a peak is being read on the plate is input into the 741₁ operational amplifier (Figure 1). The 741₁ acts as a switch. When the input voltage, V_{in} , drops below -0.15 volts the 741₁ output goes to a positive 12 volts. This charges the 2.2 μ f, 1.3K RC combination. When this RC combination reaches a charge of 8 volts after 3 milliseconds the output of the 555 (monostable multivibrator) goes to 0 volts the 741₂ operational amplifier is enabled. This operational amplifier has a gain of 1 to 4 (variable) and inverts the input signal. This causes a voltage of 0 to 12 volts to appear on the output of the 741₂ (depending on the gain set and the input voltage). This voltage charges the RC combination 1K, 0.01 μ f for a period of 0.2 milliseconds in the center of the sample time. After enough of these samples are obtained to provide a good average voltage the Ready signal is generated by the 7493_{2,3} counters (4-bit binary counters) and a sample can be taken. After the Ready signal is generated, the capacitor 0.01 μ f of the RC combination in question is discharged to prepare for another sample. The output from the 1K, 0.01 μ f RC combination is then impedance matched to the 75 ohm line by the buffer 741₃ to provide an output consistent with the NOVA 3/12 computer analog input.

The overall driver for sampling data by the computer is called CMPTR. The general logic for this is shown in Figure 2. The terms in this flow diagram are defined as follows:

INIT \equiv initialization

INPT \equiv input

MCMND \equiv the user's input command from the VDU

DSTNC \equiv the distance to be moved

(XPOS, YPOS) \equiv the x, y coordinates of the comparator table position
at any instance in time

JDR \equiv the direction of comparator table movement on the horizontal
axis, either forward (JF) or reverse (JR)

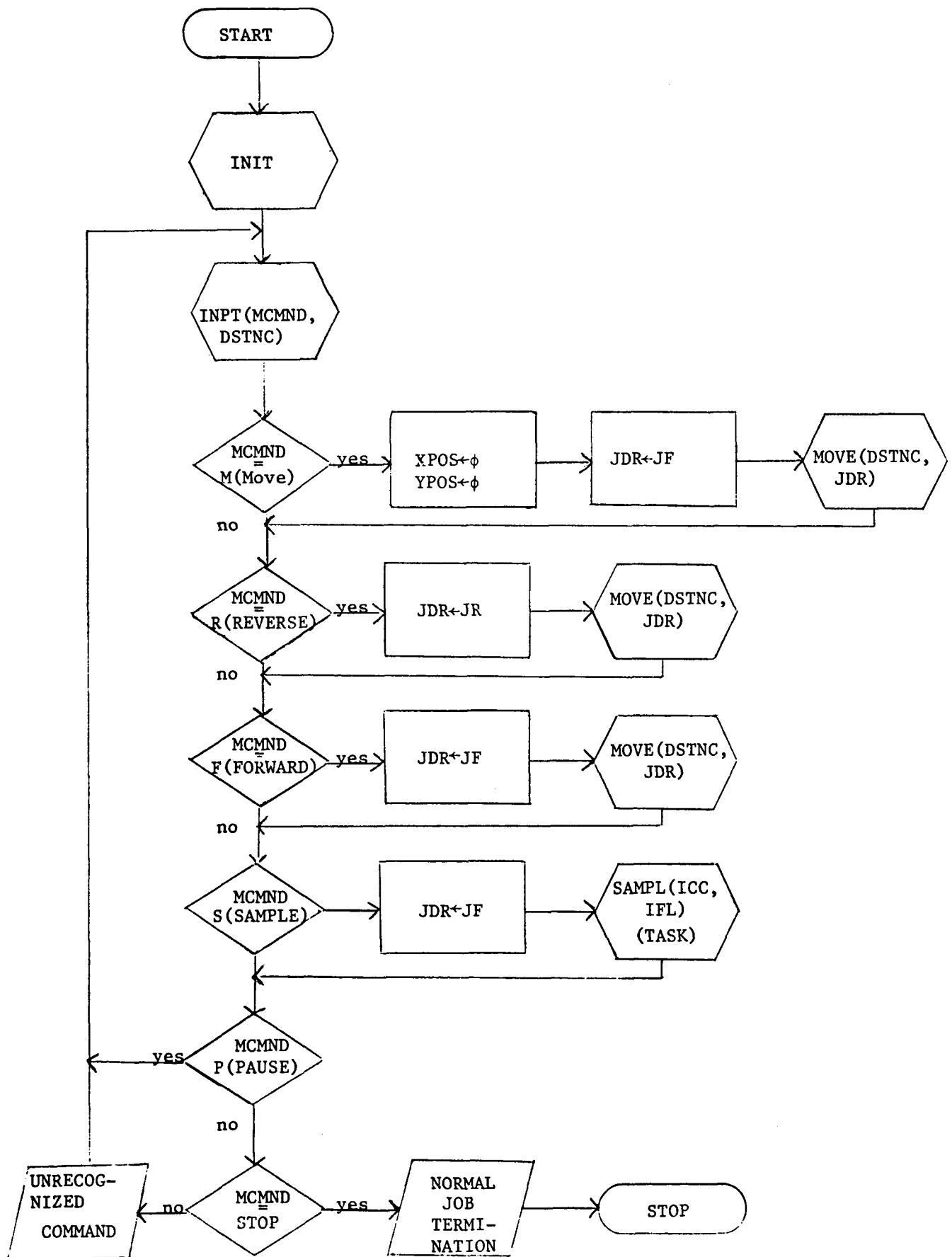


Figure 2. Flow Diagram for the Overall Driver (CMPTR) for Sampling Data

ICC \equiv condition code upon return from sampling routine
IFL \equiv the file on which the sampled data is stored
MOVE \equiv a subroutine to cause the comparator table movement without
sampling
SAMPL \equiv a subroutine to initiate/terminate the sampling process.

Thus an overview of what happens when data are taken from the photographic plate on the comparator is as follows. First, the comparator-microdensitometer's table must be positioned to (0,0) (0 horizontally, 0 vertically) manually. All internal flags are then initialized and the driver seeks a command from the input VDU. The input consists of the command and a distance to move, when appropriate. Positioning is accomplished via a MOVE subroutine which sends the appropriate signals to the hardware logic drivers through the RS-232-C interface. The drivers then cause the stepper motors to move the proper distance in the proper direction. When the input command specifies that sampling is to occur, the overall driver initiates a sampling subtask. This subtask increments the position of the table and causes the analog output from the comparator to be sampled by the AEI ADC (analog to digital converter) input board. At the completion of the sampling, the next command is input and processed.

Work Forecast

- 1) Continue development of FI/MS as a technique for the quantitative analysis of saturated hydrocarbons.
- 2) Finish manuscript pertaining to the effect of aromatic compounds on saturate FI sensitivities and vice versa.
- 3) Further determine the dynamic resolution and the mass measurement accuracy of mass spectra acquired in real time using higher static resolutions.
- 4) Prepare manuscript describing modifications made to mass spectrometer in order to incorporate the replacement electron multiplier and the data system.
- 5) Prepare manuscript describing automation of probe distillation in combination with FI/MS analysis.
- 6) Prepare manuscript describing algorithm MZF for processing high-resolution mass spectral data.
- 7) Continue development of the interface between the NOVA 3/12 and the comparator/microdensitometer.

Task 3 - Use of Mass Spectrometer Facilities to Provide Analytical Data for Other DOE-Sponsored Projects

With the approval of the technical officer, Janet L. Shultz, Pittsburgh Energy Technology Center, DOE, the principal activities under Task 3 have been deferred until installation of the data-acquisition system. However, this project has supported out collaborative characterization research with J. E. Dooley's Separations and Characterization Group at the Bartlesville Energy Technology Center. The present contract has also been used to support the characterization aspects of our collaborative research with Professor B. L. Crynes and his colleagues in the School of Chemical Engineering at Oklahoma State University. During this quarter the high-resolution field-ionization mass spectral data for the remaining asphaltene fractions from the COED coal liquid were completed. The fractions which were analysed during this quarter are FMC Asphaltene Acid Fractions A, B, C, and D and FMC Asphaltene Base Fractions E and F. A static resolution of 9,500 was used in obtaining data for each of these samples. Higher resolution could have been obtained from the instrument; however, because of the large mass ranges of the fractions, approximately 400 amu, a higher resolution setting would have caused the lower intensity peaks to become non-detectable. Thus a trade-off resolution had to be chosen.

Contained in Table VII is a list of samples obtained from the Separations and Characterization Group at the Bartlesville Energy Technology Center. High- and low-resolution field-ionization mass spectral data were obtained for each of the samples listed in Table VII. A static resolution of 10,000 was used in obtaining the high-resolution data. These samples were introduced into the mass spectrometer through the batch inlet system at a temperature of 320°C. The asterisk by the Sample Name in Table VII means that high-resolution electron-impact mass spectral data were also taken on the sample. These data were processed by the Kendrick mass program to give molecular formula assignments for each observed ion. The instrument was set for a static resolution of 13,000 during these runs. These data are now being processed at the

TABLE VII

SAMPLES OBTAINED FROM BETC FOR HIGH-
AND LOW-RESOLUTION FI/MS ANALYSIS

Sample Name	Distillate Temperature Range	Sample Type
Wyodak B*	200-325°C	Polyaromatic Polar Conc.
Wyodak B	200-325°C	Diaromatic Conc.
Wyodak B	325-425°C	Polyaromatic Polar Conc.
Wyodak A	425-540°C	Polyaromatic Polar Conc.
Ill. #6A	325-425°C	Diaromatic Conc.
Pittsburgh*	200-325°C	Polyaromatic Polar Conc.
Pittsburgh*	200-325°C	GPC 283 Fr. 17
Pittsburgh	325-425°C	Monoaromatic Conc.
Pittsburgh	425-540°C	Polyaromatic Polar Conc.
Lignite*	200-325°C	Polyaromatic Polar Conc.
Lignite	325-425°C	Polyaromatic Polar Conc.
Lignite	425-540°C	Polyaromatic Polar Conc.
Western Kentucky	325-425°C	Monoaromatic Conc.
Western Kentucky	325-425°C	Diaromatic Conc.
Western Kentucky	325-425°C	Polyaromatic Polar Conc.
Western Kentucky	425-540°C	Diaromatic Conc.
Western Kentucky	425-540°C	Polyaromatic Polar Conc.
Ill. #6B	425-540°C	Polyaromatic Polar Conc.
Colstrip	425-540°C	Polyaromatic Polar Conc.

Bartlesville Energy Technology Center. Activities during this quarter have also involved a continuation of our detailed characterization of the various fractions obtained from separation of a hydrotreated Greater Canadian Tar Sands oil sample. The results obtained from this detailed analysis will be presented at a later time.

Work Forecast

Support of our collaborative research with J. E. Dooley's group at BETC and with Professor Crynes' group at Oklahoma State University shall be continued. With the completion of the high-resolution field-ionization mass spectral data for the asphaltene fractions, a molecular-ion group-type analysis for the oils and asphaltenes from the COED coal liquid based upon the low- and high-resolution FI/MS data and the high-resolution 70-eV EI/MS data will be completed. The same type of analysis shall also be obtained from the high-resolution low-voltage EI/MS data obtained by T. Aczel and B. Bieber of Exxon Research and Engineering Company in Houston, Texas. Previously acquired infrared spectral data will be used in assigning group-type to each fraction and in corroborating the correctness of separation procedures. Low- and high-resolution FI/MS data and high-resolution 70-eV EI/MS data shall be acquired for the requisite number of GPC fractions in order to permit us to complete the detailed characterization of the Greater Canadian Tar Oil Sands sample.

References

- 1) Quarterly Progress Report FE 2537-3, Dist. Category UC-90d, submitted under DOE contract EX-76-S-01-2537.
- 2) Quarterly Progress Report FE 2537-8, Dist. Category UC-90d, submitted under DOE contract EX-76-S-01-2537.
- 3) S. E. Scheppele, C. S. Hsu, T. D. Marriott, P. A. Benson, K. N. Detwiler and N. B. Perreira, Int. J. Mass Spectrom. Ion Phys., 28, 335 (1978).