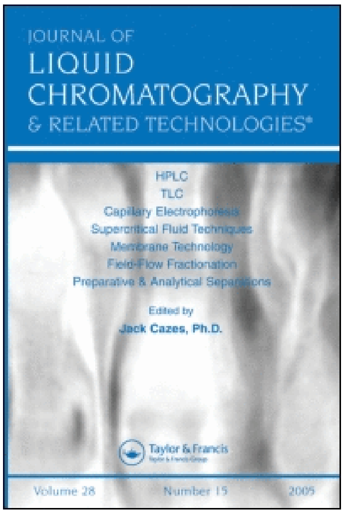

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An Inexpensive, On-Line Data Processing System for Gel Permeation Chromatography

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AN INEXPENSIVE, ON-LINE DATA PROCESSING SYSTEM FOR GEL PERMEATION CHROMATOGRAPHY

By

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ABSTRACT

An inexpensive, on-line data processing system is described for the Waters 200 gel permeation chromatograph. The system consists of (a) interface chassis; (b) microprocessor with ∿3K memory and (c) a console device with cassette tapes for the storage of data and programs.

INTRODUCTION

Gel permeation chromatography is now a routine analytical procedure for the qualitative and quantitative analysis of polymeric materials. It provides an indirect measure for $\overline{M}w$, the weight average molecular weight, $\overline{M}n$, the number average molecular weight and MWD, the molecular weight distribution. All the three parameters are very important in understanding the physical behavior of polymers. The analyses generate raw data that must be laboriously manipulated and calculated to yield results required by the analyst. A number of commercial equipment are available with microprocessors to handle the data. The purpose of this report is to describe an inexpensive on-line data processing system for gel permeation chromatography which can be assembled with some basic knowledge of computer hardware and software. Greggs (1) and MacLean (2) have described data processing systems. The latter uses PEP-2 hardware and software which are proprietary to the Perkin Elmer Corporation.

THE HARDWARE

The hardware consists of three subsystems: (a) the GPC interface, (b) Microcomputer, and (c) the Terminal-Storage unit.

The GPC Interface consists of four subsystems:

1. Buffer-Signal Conditioner or Level Converter.
2. Sample and Hold.
3. Analog to Digital Converter.
4. Clock and Control.

The level converter proved to be the most difficult problem. The analog signal obtained from the refractive index detector of the Waters GPC 200 consisted of a 0-100 millivolt differential DC signal which was superimposed on a 3-5V peak-to-peak AC signal at approximately 60 Hz. A differential input operational amplifier configuration was chosen to eliminate the common mode AC voltage, and to provide a high impedance input buffer. (Figure 1). The amplifier now in use provides an input independence of 10^{11} ohms. This amplifier provides sufficient isolation so as not to effect the signal to the recorder.

After the buffering stage the signal is fed into another operational amplifier providing a gain of 100, to bring the signal into the 0-10V DC range required by the A to D converter.

Due to an imbalance in the differential common mode signal, a high amount of 60 Hz noise was still passed to the buffer stage. This noise was eliminated by providing a 1 second constant for the output amplifier, thus averaging out the 60 Hz noise.

The analog to digital converter board is in two parts, the sample and hold (S & H) and the analog to digital converter (ADC). Connections

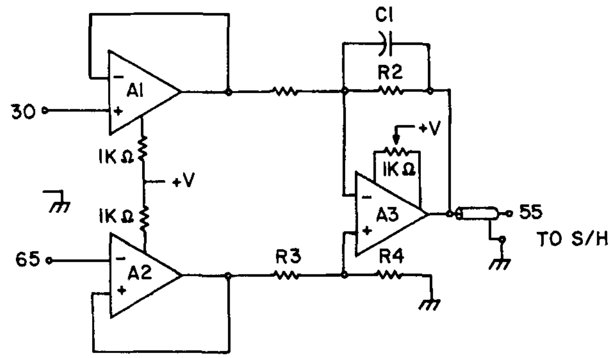

Figure 1. Differential input operational amplifier.

between the buffer amplifier and the sample and hold and the analog to digital converter should be made with a shielded cable.

The sample and hold module (Figure 2) on computer command, holds the value of the input signal constant long enough for the ADC to convert it into digital format. The ADC converts the analog signal into a binary coded decimal (BCD) digital signal on command from the computer (Figure 3).

The clock and control board contains a crystal controlled clock that operates at 3.5794 MHz at an accuracy of .005%. The clock is divided to several frequencies (60 Hz, 10 Hz, 1 Hz, .1 Hz). The .1 Hz frequency is used by the system for determining the sampling rate (Figure 4).

All of the above-mentioned boards can be consolidated into a single board. The component layout is not critical and interconnections are made directly without the use of optoisolators since all power supplies share a common ground reference.

The computer is a unit manufactured by TECHNICO and based on the Texas Instruments TMS 9900 Microprocessor. This is a 16 BIT Processor with a powerful mini computer instruction set, and in the present configuration can be upgraded to mini computer capabilities with "plug-in boards."

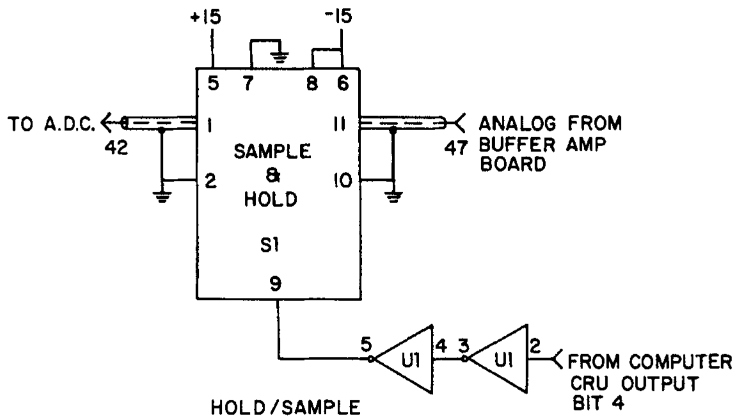

Figure 2. Sample and hold module.

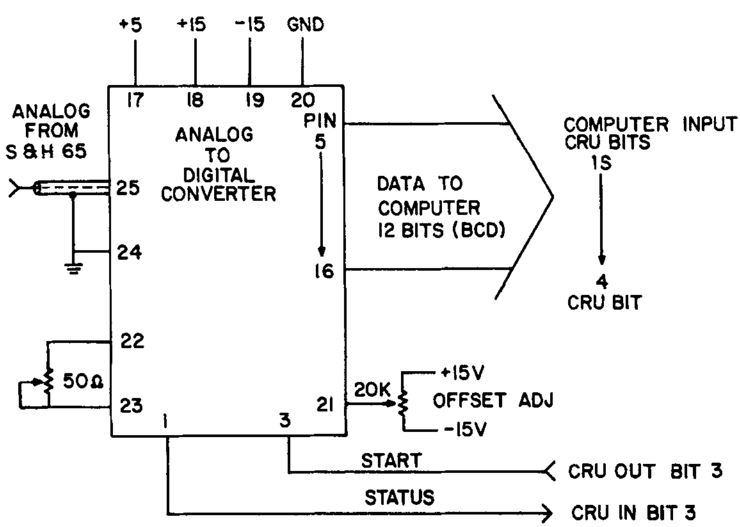

Figure 3. Analog-to-digital converter.

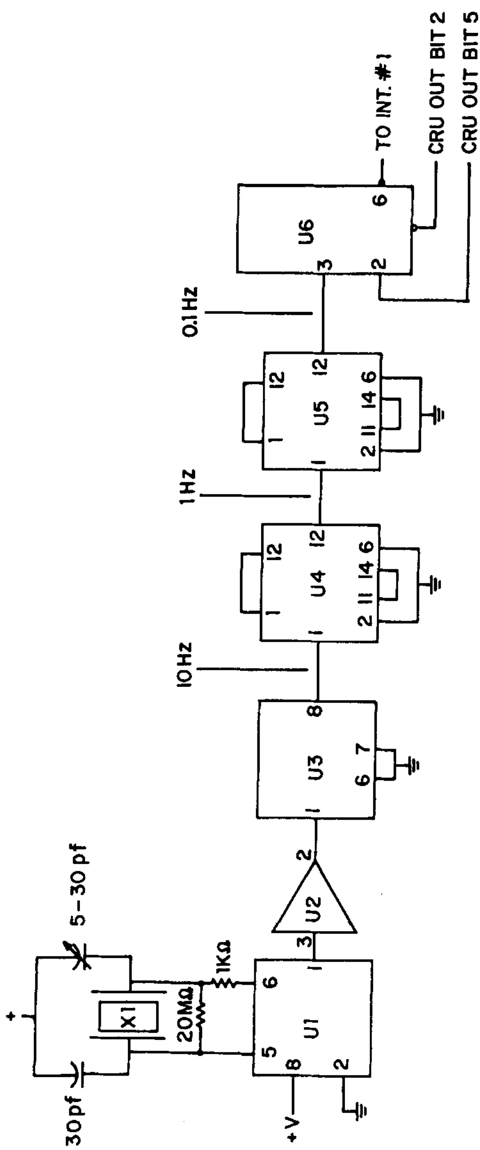

Figure 4. Clock and control board.

The computer is a complete one board unit with all memory and interfaces needed for stand along operation. It is controlled by Texas Instruments Silent 700 terminal with twin cassette tape drives and automatic device control (ADC) option. The ADC allows the computer to control the tape units (on, off, read, write and rewind). The tapes are used for program and data storage during acquisition. All status and warning messages are printed on the terminal during the acquisition process.

TMS 9900 DATA ACQUISITION PROGRAM

The program begins by resetting the A to D converter clock and injection flipflops and placing the S & H in the hold mode. It also initializes various registers, pointers, and outputs as program identification message. In the next step a message goes out requesting the number of counts per sample. On receipt of a character it is converted to binary and the program waits for another character. On receipt of the second character, a check is made to see if it is a carriage return. If not, the character is converted to binary and added to the first number which has then been multiplied by 10. This is now multiplied by 30 (points per count) and stored for later use. If a carriage return was encountered, it means that the first number was multiplied by 30 and stored as above.

The program now requests a command and on receipt of a character determines whether it is a "go," "stop," or an illegal character. If the character is valid, the computer outputs the rest of the command word and branches to the appropriate routine. If the command is "go" the program outputs "working" and enables the inject and interrupt flipflops. The program then returns to wait for a command. If it is a stop command, interrupts are disabled and an "EOT" command is written on tape, next "data scan complete" is put out after which the program restarts.

The interrupt handler takes over if the interrupts are enabled and the clock times out. After entering the handler further interrupts are disabled and the data interrupt flipflog is reset and disabled. Next the program checks to see if an injection has occurred. If it has, a "-1" is printed on the tape to signal injection (all data is positive). The number of points is incremented to reflect the new total for the additional scan. Next the sample and hold is placed in the hold mode and the A to D converter is started. When the converter has completed its task the data is read, converted to A.S.C.I.I., and written on tape. The total number of points is decremented by one and checked to see if the last point has been taken. If the number of points equals Ø, an EOF is written to tape, and the program restarts. If the number of points is not equal to Ø, the sample and hold is set to sample and all the flipflops are enabled. Program control then reverts to the point of interruption. Flow charts provided in Figures 5, 6, and 7 describe the steps involved in data acquisition.

The original data reduction program was written in BASIC. This was modified to allow it to select the desired data out of the stored sample runs. The program requests the data file, checks to see if it exists. Next the program requests the number of counts per sample and the desired scan number. The data is read into the program after it is cleared of noise spikes and the valid counts are found. The program then displays the number of the smallest count and will provide a crude plot if desired, to facilitate picking the desired peak. The peaks' boundary points are entered along with any scale multiplier. A baseline correction is performed and the calculation continues as in the original program.

A typical plot of the GPC chromatogram is presented in Figure 8. The corresponding data sheet is presented in Table 1. A series of narrow distribution standard polystyrenes were run. Table 2 shows Mw, Mn and MWD data obtained by normalizing the distribution curves and using simple integration steps manually. These are compared to values obtained by

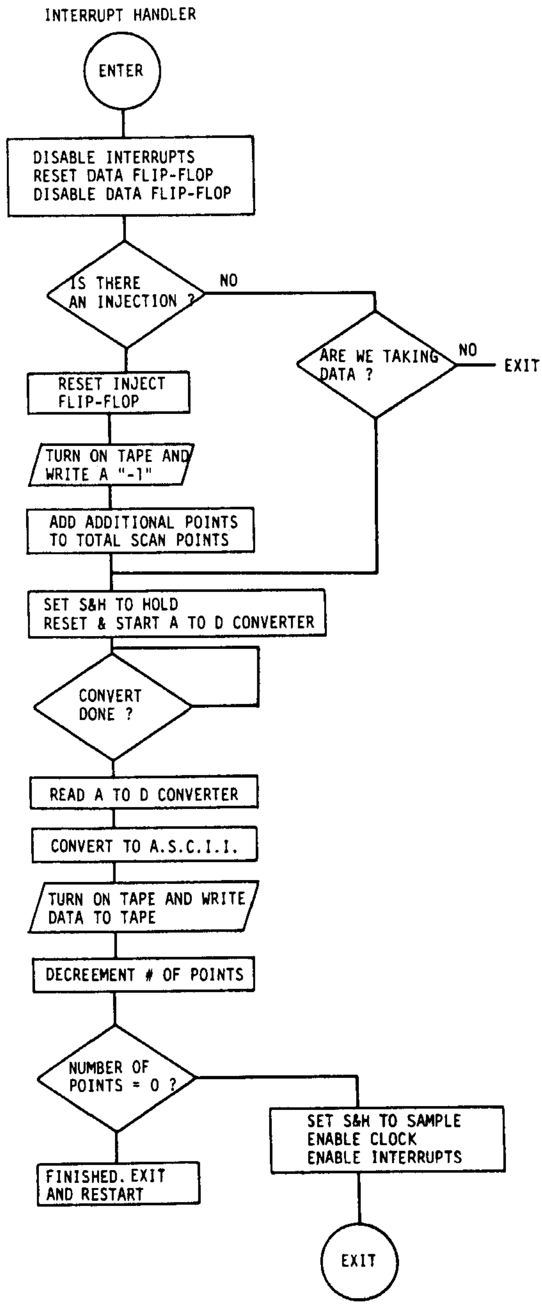

Figure 5. Program flow chart - Interrupt handler.

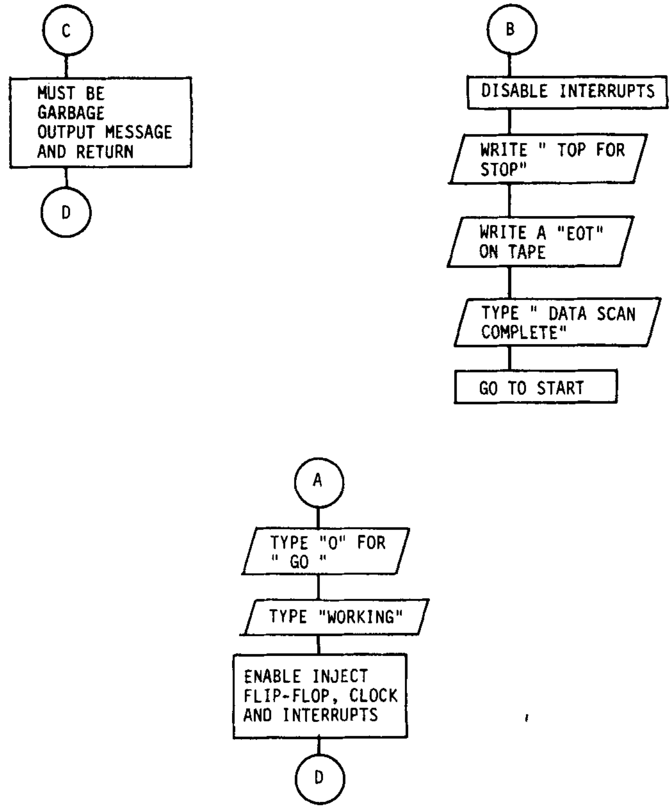

Figure 6. Program flow chart - Auxilliary routines.

the data processor. The agreement is quite good except for the two high molecular weight polystyrenes, presumably due to excessive axial dispersion and skewing effects.

CONCLUSION

A simple, inexpensive on-line data processing system is described for the Waters 200 Gel Permeation Chromatograph. This system can be assembled with only a basic knowledge of computer hardware and software and saves considerable amount of time spent for data manipulation.

Interface chassis and the microprocessor together cost $800.

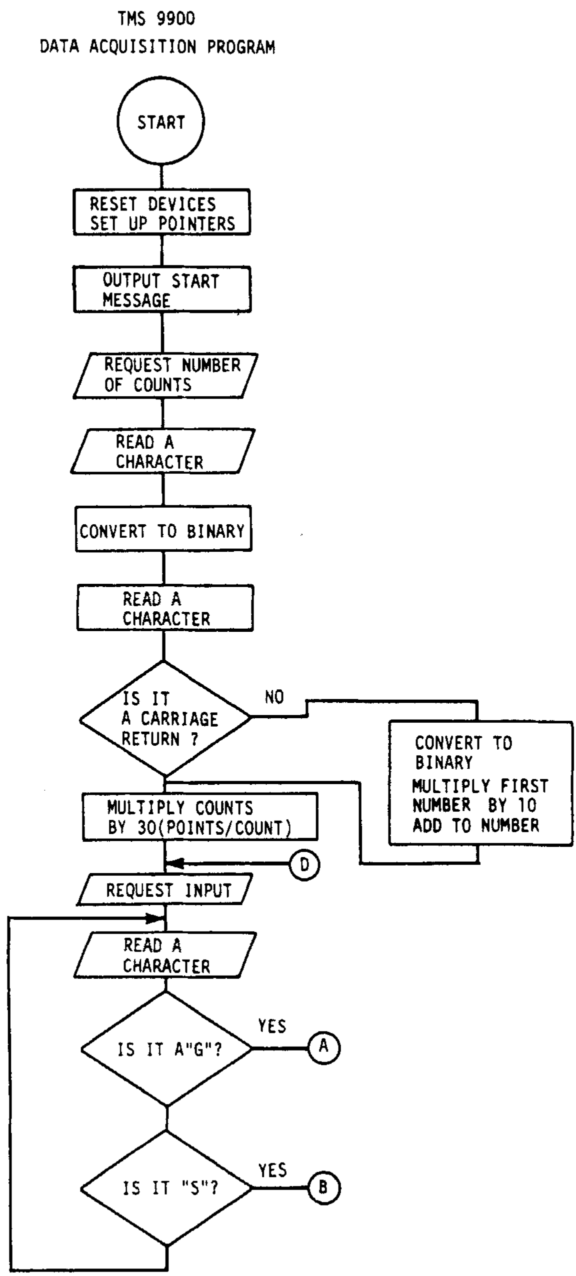

Figure 7. Program flow chart - Data acquisition program.

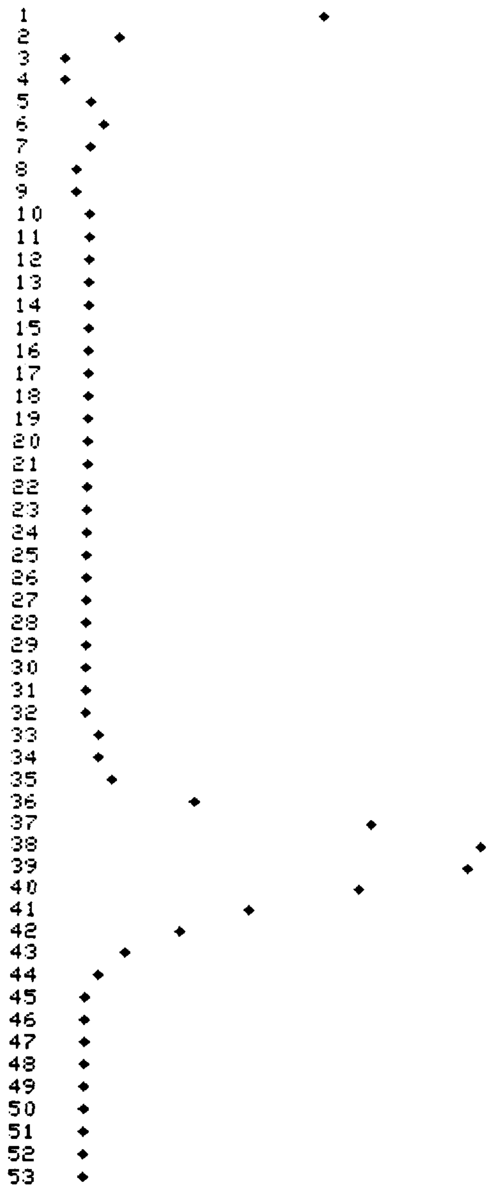
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Figure 8

GEL PERMEATION CHROMATOGRAPHY DATA SHEET

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SAMPLE NO:
?1
ANALYST:
?

COLUMNS: 10 TO 6TH,5TH,4TH,3RD,500,60 SOLVENT: CHCL3

FLOW RATE: 1 ML/MIN ROOM TEMPERATURE

COUNT	AI	HEIGHT	HIXAI	HI/AI
33	16341	0	0	0
34	9609	3.58333	34432.2	3.72914E-04
35	5268	16.1667	85166.0	3.06884E-03
36	3268	91.7500	299839.	2.80753E-02
37	2022	251.333	508196.	.124299
38	1200	345.917	415100.	.288264
39	702	327.500	229905.	.466524
40	405	238.083	96423.7	.587860
41	230	139.667	32123.3	.607246
42	152	81.2500	12350.0	.534539
43	93	33.8333	3146.50	.363799
44	57	9.41667	536.750	.165205
45	35.6000	-2.22045E-16	-7.90479E-15	-6.23721E-18
	TOTALS=	1538.50	1.71722E+06	3.16925

RESULTS:

ANGSTROM WEIGHT AVE MOL SIZE= 1116
ANGSTROM NUMBER AVE MOL SIZE= 485
MOL WGT DIST= 2.30

TABLE 2

GEL PERMEATION CHROMATOGRAPHIC DETERMINATION OF POLYSTYRENE STANDARDS

Material Specification[†]			Manual Measurement			Computer Measurement		
Mw	Mn	MWD	Mw	Mn	MWD	Mw	Mn	MWD
4000	3570	1.12	3895	3280	1.18	4100	3403	1.21
17500	*	*	18368	15621	1.17	16933	14350	1.81
50000	47000	1.06	47191	40508	1.17	48134	40221	1.20
110000	*	*	109962	93111	1.18	107543	95694	1.12
233000	*	*	242064	184787	1.31	233337	190814	1.22
390000	383000	1.02	366827	260300	1.41	347434	26334	1.23
1800000	1780000	1.01	1937700	687600	2.82	1826591	782813	2.33

[†]Data obtained from Pressure Chemical Co., Pittsburgh, Pa.

*Not specified.

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1. A. R. Greggs, B. F. Bowden, E. M. Barrall II and T. T. Horikawa, Separation Sci., 5, 731 (1970).

2. W. MacLean, American Lab., October, 1974.