GLC-8

A COMPUTERIZED SYSTEM
AUTOMATING GAS-LIQUID
CHROMATOGRAPHY

Your attention is invited to the last two pages of this
manual. The Reader's Comments page, when filled in
and returned, is beneficial to both you and DEC. All
comments received are considered when documenting
subsequent manuals, and when assistance is required, a
knowledgeable DEC representative will contact you.
The Software Information page offers you a means of
keeping up-to-date with DEC's software.

Instruction times, operating speeds and the like are in-
cluded in this manual for reference only; they are not to
be taken as specifications.

Documents Referenced (available from DEC's Program Library):

Introduction To Programming, C-18
8K FORTRAN Programmer's Reference Manual, DEC-08-KFXB-D
PDP-8/I System User's Guide, DEC-08-NGCB-D

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PREFACE

GLC-8 is DEC's computerized system for automating the gas-liquid chromatography laboratory. This manual covers the use of GLC-8 from the chromatographer's viewpoint (the scientist, engineer, or laboratory technician), describing the system and how to use it efficiently.

GLC-8 is compatible with virtually any gas-liquid chromatograph or detector (thermal-conductivity, flame ionization, electron capture, etc.), therefore, the manual assumes that you are thoroughly familiar with your chromatograph or detector.

Chapter 1 offers a "bird's eye" view of GLC-8, the system operation and configuration. Chapter 2 is devoted to explaining how you communicate with the system and what GLC-8 does for you when you do. Chapter 3 describes an actual analysis using GLC-8 with a Perkin-Elmer 900 Chromatograph. The appendices contain summarized operating information.

The "heart" of GLC-8 is a set of programs called Monitor, which interact conversationally with the chromatographer.

The PDP-8/I System User's Guide (DEC-08-NGCB-D), furnished with the system, is frequently referenced for console, loading and operating instructions.

Documents referenced:

       PDP-8/I System User's Guide, DEC-08-NGCB-D

       GLC-8 Maintenance Manual, DEC-08-HGAA-D

CHAPTER 1

INTRODUCTION

The science of gas-liquid chromatography has come a long way since it was discovered in 1952. GLC-8 is another step forward.

GLC-8 (Gas-Liquid Chromatography for the PDP-8/I) is a computerized system for automating the gas-liquid chromatography laboratory. With GLC-8, the tedious task of measuring strip-chart recordings is eliminated, analysis becomes exceptionally reproducible, and the result of each analysis is available in a typed report within seconds rather than hours.

The GLC-8 system performs on-line analysis calculations on as many as 20 chromatographs* simultaneously. A simple dialogue between the chromatographer (using the Teletype console) and the GLC-8 Monitor enables him to specify the manner in which the analysis is to be performed, to request a method, run an analysis or calibration, and to receive a typed report of the analysis.

GLC-8 is designed primarily for use with gas-liquid chromatographs; however, it may be used with other analytical instruments (e.g., amino-acid analysers) which use the same method of analysis as the chromatograph. The system will operate on systems utilizing thermal conductivity, flame ionization, and electron capture detectors, and will allow parallel operation of a strip-chart recorder and attenuation switching.

GLC-8 is primarily a special-purpose system, dedicated to chromatographic analyses. However, when the system isn't being used to control analyses it is available as a general-purpose computer for use with such programs as FOCAL, BASIC-8, FORTRAN, PAL III, MACRO-8, or the Disk Monitor System.

1.1   MONITOR SYSTEM

The heart of the GLC-8 system is appropriately named Monitor since it monitors and controls every phase of the analysis. Monitor is composed of many computer programs which reside in core memory (PDP-8/I computer). During each analysis, Monitor performs the following functions.

    a.   Samples each channel at the rate specified in the analysis method.

    b.   Selectively attenuates the effect of channel noise with digital signal filtering, as specified in the method.

    c.   Detects peaks and shoulders.

---

* This number may vary with certain configurations.

d. Integrates the area under the peaks and shoulders.

e. Condenses all this information into a file and stores it on the disk.

When the specified time of the analysis run is reached or when the analysis is terminated, Monitor takes the information accumulated during the analysis and

a. Performs sophisticated baseline corrections.

b. Reallocates the area of fused peaks to give an accurate area measurement for each peak.

c. Adjusts retention times relative to the predefined reference peak in the method.

d. Identifies and names each component or peak.

e. Applies an internal standard, if requested in the method.

f. Applies response factors.

g. Calculates component concentrations.

h. Types out a complete, accurate report of the analysis.

Monitor analyzes and interprets the chromatographic data according to the method specified. In essence, Monitor automatically performs everything except sample injection; there is no manual data reduction or analysis after the method of analysis has been specified.

1.2   AN OVERVIEW

The brief description of an analysis that follows reveals the features and usefulness and flexibility of GLC-8. Questions arising will be answered as you read on.

To start an on-line chromatographic analysis, you have only to type

ANALYZE, 18, 7, :

to inform Monitor that you wish to initiate an analysis on chromatograph number 7 using method number 18. (See Figure 1.) Monitor makes the following checks:

a. Is method 18 written for chromatograph 7?

b. Can the sample rate of the analysis be fitted into the schedule? (No more than 240 data points per second* can be processed at any one point in time.)

c. Is there sufficient disk storage to hold the collected chromatographic data?

d. Is chromatograph 7 available for this analysis (i.e., not busy running another analysis)?

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* This is true for a 60-cycle system. For a 50-cycle system, the maximum rate is 200 samples/second.

```
                                  ENTER,18,0,:
INST, PK CT                 --      7,       20,    :
LNK, FF, SMPR               --      1,        1,         5,    :
RTYP, AF                    --       ,      100,    :
IS TM/SCALE                 --    224,    :
RPKT, RRT, TBEG, TEND       --    224,     1000,     200,        250,      :
F TM, CODE, VAL             --      1,        1,          ,    :
F TM, CODE, VAL             --      1,        4,         1,    :
F TM, CODE, VAL             --      1,        5,          ,    :
F TM, CODE, VAL             --      1,        6,         1,    :
F TM, CODE, VAL             --      1,        9,         1,    :
F TM, CODE, VAL             --    500,        7,          ,    :
F TM, CODE, VAL             --    510,         ,          ,    :
TM, NAME, TOL, RF, CWT      --     15, AIR    ,         5,           ,          ,    :
TM, NAME, TOL, RF, CWT      --     27, CLOHAN,         5,       6785,       192,    :
TM, NAME, TOL, RF, CWT      --     57, PENTL1,         5,       9120,       163,    :
TM, NAME, TOL, RF, CWT      --    103, CYCHOL,         5,      10530,      3963,    :
TM, NAME, TOL, RF, CWT      --    146, PHENOL,         5,       6127,       190,    :
TM, NAME, TOL, RF, CWT      --    224, CYCHON,         5,       9999,      4192,    :
TM, NAME, TOL, RF, CWT      --    320, DICYCL,         5,       5832,       471,    :
TM, NAME, TOL, RF, CWT      --    411, DIC-EH,         5,       7185,       829,    :


                                  ANALYZE,18,7,:
ID, RCYCL                         DEMO,0,:
NORM                              10000:

ANALYSIS

DATE    2   15   69      TIME    1235

INST    7        METHOD    18       NORM

DEMO                              10000
```

COMPONENT	RRT	TIME	PEAK AREA*	TYPE	RF FACT	COMP %	TOL
AIR	.071	16	9033	BB		.000%	- 18%
CLOHAN	.124	28	13464 !	BV	6785	1.929%	- 17%
PENTL1	.253	57	8478 !	VV	9120	1.632%	+ 5%
CYCHOL	.462	104	178102 !	VV	10530	39.603%	- 10%
PHENOL	.653	147	14595 !	VV	6127	1.888%	- 6%
CYCHON	.999	225	198655 !	VV	9999	41.946%	+ %
DICYCL	1.426	321	38148 !	VB	5832	4.697%	+ 8%
DIC-EH	1.831	412	54715 !	BB	7185	8.301%	+ 16%

Figure 1   A GLC-8 Method (top) and Analysis Report (bottom)

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*An exclamation mark (!) raises the typed number to the power of 3. A quote mark (") raises the typed number to the power of 6.

If Monitor finds a "no" answer to any of these questions, it will type a three-character error-message code indicating which condition cannot be met. Monitor would then ignore your request and wait for you to issue another.

If the analysis can be handled, Monitor will ask you to name the analysis and state whether you want to repeat the method of analysis using other samples. If the analysis is to use the internal standard calculation technique, Monitor asks you to enter the sample and internal standard weights.

After you have answered these questions, the READY light on the local operator's console (LOC) associated with chromatograph 7 is turned on. The system is now ready for you to inject the sample. Immediately after sample injection, you should depress the START pushbutton on the LOC; the SAMPling light will illuminate. The system is now running the analysis.

If you wish to cancel the analysis, depress the CLEAR pushbutton on the LOC. This will abort the run and Monitor will return to command mode. If you wish to prematurely terminate the analysis, depress the STOP pushbutton on the LOC. This will generate an analysis report based on the accumulated data.

Unless interrupted, the run will complete itself at the time specified by the method being used. Upon completion of the analysis the COMPute light on the LOC turns on and Monitor performs the following on the accumulated chromatographic data.

a. Baseline correction

b. Resolution of overlapping peaks

c. Calculation of adjusted and relative retention time

d. Identification of peaks

e. Computation of internal standard

f. Adjustment by relative response factors

g. Calculates component concentrations

h. Types a concise, accurate report of the analysis

The typed report lists all identified components, unknown peaks, and not-found components in the chronological order in which they eluted (or should have eluted, in the case of not-found components). The following data is typed for each peak (see Figure 1).

a. Name or UNKnown

b. Relative retention time

c. Retention time

d. Peak area

e. Peak type

f. Response factor

g. Component percent

h. Retention tolerance

Relative retention time is based upon a stated relative retention time of the reference peak. The reference peak is a peak on the chromatogram and is used to scale the time frame (window) of the chromatogram when the method is built.

Peak type demonstrates the basic nature of the peak by denoting how it started and how it ended. For example, in the preceding analysis report, the first three peaks were identified as BB, BV, and VV, in the peak type column of the report; the following occurred:

a. The first peak, BB, started at a baseline and terminated at a baseline.

b. The second peak, BV, started at a baseline and ended in a valley.

c. The third peak, VV, started in fusion with the valley and ended on its reverse slope by fusing with another valley.

Retention time tolerance, the rightmost column in the report, is a diagnostic feature which indicates whether a peak identified with a component had a retention time before or after the component's expected retention time as determined by the selected method. If a component has a tolerance of 100%, it was sitting on the upper bound of its tolerance. This figure constantly reflects the state of column aging.

1.3   SYSTEM CONFIGURATION

The major hardware components of the basic GLC-8 system for simultaneous acquisition and analysis of data are listed below.

a. One PDP-8/I computer with at least 8K words of core memory and an EAE unit (KE8/I) for fast arithmetic operations

b. One 32K disk (DF32)

c. Type AF06-A interface: 64 channel multiplexer, 1×10^{6} dynamic range analog-to-digital converter, 4x line frequency clock (240 cps*), and control for up to 64 LOCs

d. At least one ASR33 Teletype (to a total of nine)

Figure 2 illustrates the GLC-8 system configuration. For additional technical specifications of the system see the GLC-8 Maintenance Manual, DEC-08-HGAA-D.
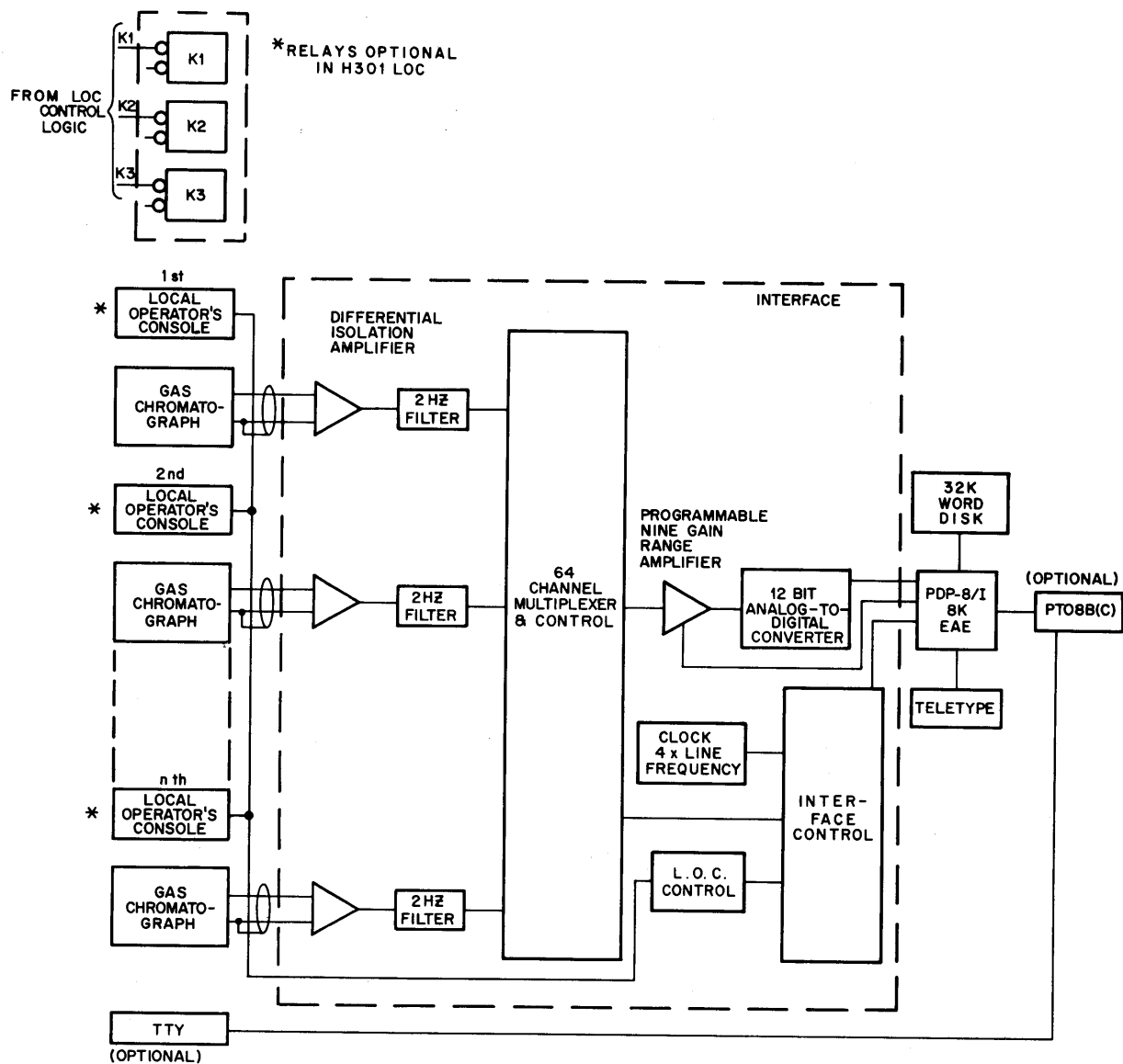
---

*200 cps for 50-cycle systems

Figure 2 GLC-8 System Configuration

1.3.1   Local Operator's Console

A local operator's console (LOC) is associated with and often connected to each chromato-graph. A LOC is shown in Figure 3.

Pushbuttons are used to control the operation of the computer. The purpose of each is as follows.

CLEAR	Depress to cancel the analysis and clear the channel. This button can be used at any time.
START	Depress to start the analysis.
STOP	Depress to terminate the analysis and generate the analysis report.

The indicator lights reveal the status of the analysis, as explained below.

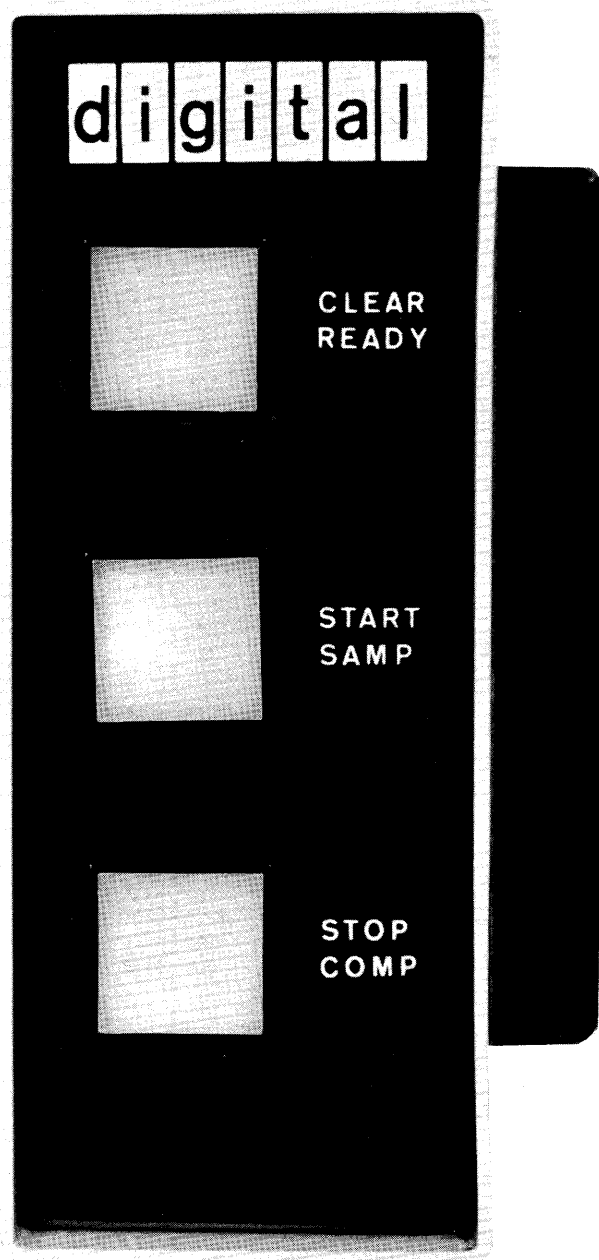|	
READY	Lights when the system is ready for sample injection.
SAMPLE	Lights when START is depressed to indicate that the analysis is in process.
COMPute	Lights when the report is being generated and typed.

Figure 3   Local Operator's Console

1.3.2   Hardware Options

The most commonly added options to a GLC-8 system are Teletypes. The system will support up to nine Teletypes operating independently and simultaneously. When additional Teletypes (after the third) with their PT08s are added, the number of chromatographs which may be operated simultaneously is reduced on a one-for-one basis.

1.4   SYSTEM SOFTWARE

The GLC-8 system operates under control of its Monitor. Monitor is composed of various programs which control the operation of the analog-to-digital (A/D) converter, multiplexer, range amplifier, local operator's console, and interprets both Teletype input and output. Monitor's functions can be divided into two major sections:

a. Foreground programs process the chromatographic data, forming and integrating peaks, and storing on the disk parameters characterizing individual peaks, which include

  (1) Sampling and digital signal filtering

  (2) Threshold logic peak detection

  (3) Integration of area under peaks and shoulders

  (4) Peak and shoulder detection

  (5) Storage of peak parameters on disk

b. Background programs are called into action by Monitor when they are needed. There are two groups of background programs: conversation and report generation.

  (1) Conversation programs allow entering, changing, deleting, and reporting analysis methods. Monitor's conversation programs are those which accept and respond to your typed input. They also maintain a library of up to 100 different analysis methods and facilitate the updating and control of stored methods (i.e., printout, entry of new analysis methods, deletion of stored methods, changing of methods, and audit trial dumps).

  (2) Report generator, at the end of an analysis, processes the stored chromatographic data and produces the analysis report on the Teletype.

1.5   SYSTEM INITIALIZATION

The GLC-8 Monitor is loaded into core and activated as explained in Appendix A. When the system is started Monitor assumes control and automatically types

        V1Ø: MONTH,   DAY,   YEAR,   HOUR,   MINUTE

(V10: is the version number of the system program) and waits for you to type the above information in the following format.

AA,BB,CC,DD,EE,

where   AA is the number of the month (1-12)

BB is the number of the day (1-31)

CC is the number of the year (last two digits only)

DD is the hour (in military time, i.e., 24-hour clock)

EE is the number of minutes after the hour (1-59)

For example,

2,18,69,14,30,

which is February 18, 1969 at 2:30 p.m. Monitor examines your input for correct format, and if an error is detected, it ignores that information and repeats the question and waits for you to try again.

When the information has been entered correctly, Monitor sets its "calendar" and "clock" and heads each typed report with the date and time the analysis report was started (see Figure 1). Incidently, Monitor's calendar even recognizes leap years.

1.6   USING THE TELETYPE CONSOLE

Communication between you and Monitor is through the Teletype console. Each Teletype operates independently on a time-sharing basis, giving each user the feeling of having the whole system to himself.

Monitor divides the Teletype paper into two sections: the left half is for Monitor's output and the right half is for your input, except when Monitor is typing an analysis report. Figure 1 reveals the division line.

Initially, Monitor spaces over toward the center of the paper and waits for you to give it a command (referred to as being in command mode). You need only specify the task and indicate how you want it done and Monitor will either go and perform the task or it will ask a series of questions to determine precisely how you want the task performed.

When Monitor spaces over to your side of the paper and pauses, it is in command mode and is waiting for you to type one of its seven commands:

ASSIGN	(allows a chromatograph to be assigned to a specific Teletype)
ENTER	(allows the on-line entry of a method)
CALIBRATE	(initializes a calibration)
ANALYZE	(initializes an analysis)
MODIFY	(allows modification of a method)
DUMP	(allows the print and/or punch of specific method(s))
DELETE	(allows the deletion of specific method(s))

Each command is discussed in detail in Chapter 2 and is summarized in Appendix B.

1.7   MODES OF OPERATION

When the system is operating, Monitor is in any one of seven modes of operation. When Monitor is waiting for you to issue a command, it is in command mode. After a command has been issued correctly, it is in the mode of the command. For example, when Monitor is in command mode and you type

MODIFY,23,3,:

Monitor transfers to its modify mode. Monitor is always in the mode of the operation it is performing except, of course, when it is in command mode. Monitor may be operating simultaneously in as many modes as there are Teletypes -- each Teletype is independent and can be operating in a different mode.

1.8   ERROR MESSAGES

Whenever you terminate a line, using the last comma when initializing or the colon for all other lines, Monitor checks your input for proper format. If Monitor detects an improperly formatted response, it either types a three-character error message, ignores your input, and returns to command mode or it repeats the question. All error messages are explained in Appendix C.

CHAPTER 2
MONITOR COMMANDS

When the GLC-8 Monitor is loaded into core, activated, and initialized (see Appendix A), Monitor spaces toward the center of the Teletype paper where you are to enter a Monitor command, which tells Monitor what you want done (see Figure 1). For example, if you want to build and enter a new method, you type

ENTER,mn,:

where ENTER is the Monitor command, mn is the number to be assigned to the method you plan to build, and the colon terminates the line, returning control to Monitor.

When entering a method using the paper tape reader, the ENTER command should always include a second parameter:

ENTER,mn,1,:

where the second parameter (number 1) causes Monitor to suppress its questions. Questions can be suppressed when entering a method from the keyboard. If an error is made when entering with the questions suppressed, an error message is typed out. Typing a colon directly after the error message causes Monitor to type *P0 and then type the question (the response to which contained the error) and subsequent questions are typed during the entering of the particular method.

A Monitor command and the parameters to a command are terminated by commas.

NOTE

Monitor always interprets no reply as a 0 reply. There-
fore, since each parameter is terminated by a comma, the
following are identical.
--0,75,0,:
--,75,,:
This convention applies throughout GLC-8.

The seven Monitor commands are shown below with the required parameters (entered as decimal integers) and a concise explanation of each. In the following examples, the parameters are coded as follows.

mn	Method number (decimal integer, 0-99)
q	Questions: 0 = type question; 1 = do not type questions*
cn	Chromatograph number (decimal integer, 1-64)
tn	Teletype number (decimal integer, 1-9)
ENTER,mn,q:	Used to build and enter a new method into the method library maintained in disk storage.

---

*If this is accidently entered, typing a second colon restores questions. We suggest that questions be suppressed only when entering methods using the paper tape reader.

ASSIGN,cn,tn,:	Assigns subsequent reports of chromatograph cn to be typed on Teletype tn.
ANALYZE,mn,cn,:	Method mn will be used on the analysis of chromatograph cn.
MODIFY,mn,:	Method mn is transferred from disk into core where Monitor types a line-by-line copy of that method, pausing after typing each line to permit you to modify it by entering new parameters.
CALIBRATE,mn,cn,:	Used to perform a calibration analysis on method mn using chromatograph cn.
DELETE,mn,mn,:	Deletes methods mn through mn from the method library; if only one method is to be deleted, the second parameter is not specified.
DUMP,mn,mn,:	A copy of methods mn through mn are typed on the Teletype; if only one method is desired, the second parameter is not specified.

Each of the above commands are explained in detail within this chapter. Monitor commands are summarized in Appendix B.

Monitor commands may be abbreviated to the first four letters of the command because Monitor reads only the first four letters of its commands. For example,

ENTER,   may be abbreviated to ENTE,

CALIBRATE,   may be abbreviated to CALI,

ANALYZE,   may be abbreviated to ANAL,

Thus, a Monitor command may be misspelled and still be interpreted correctly so long as the first four letters are spelled correctly. In other words, ANALYSIS and ANALYSES are interpreted as ANALYZE by Monitor.

2.1   ASSIGN COMMAND

The ASSIGN command is used to establish Teletype/chromatograph assignments and reassignments. When a Teletype is assigned to a chromatograph the analysis reports from that chromatograph will be typed on the assigned Teletype.

To assign or reassign chromatograph 12 to Teletype 2, you have only to type

ASSIGN,12,2,:

Monitor accomplishes the assignment and returns to command mode. The assignment will remain valid until reassigned using another ASSIGN command.

A Teletype can be assigned to any number of chromatographs. However, a chromatograph can be assigned to only one Teletype at any one time. If any attempt is made to assign a chromatograph to more than one Teletype, Monitor will recognize the last Teletype number entered and ignore the others.

Unless otherwise assigned, all chromatographs are assigned to Teletype 1.

Reassignment can be made at any time, even when the chromatograph is busy. If, in the example above, chromatograph 12 had been running an analysis when the reassignment was made, the reassignment would be accomplished and the analysis report of the analysis running on chromatograph 12 would be typed out on Teletype 2.

2.2   ENTER COMMAND (BUILDING A METHOD)

To perform an analysis you need a method. The method has two primary functions:

a.   To define the technique by which the sample is to be analyzed.

b.   To specify the expected components in the sample.

Methods are built during a question and answer session between Monitor (asking questions) and you (answering). The method building session begins after you type the command ENTER and the number to be assigned to the method. The ENTER command is typed when Monitor is in command mode. To build method number 13, you may type

ENTER,13,:

If a method numbered 13 is already on the disk, Monitor types the error message *U2 immediately after the colon. (See Appendix C for a list of all error messages and their explanations.)

However, assuming the command is correct and a method number 13 is not already on the disk, Monitor would return to its side of the paper and ask the following question.

INST, PK CT

and wait for you to specify the chromatograph (INST, for instrument) number (1-64) to which this method will be assigned, and the maximum number of peaks (PK CT, for peak count) expected in the analysis (1-200). Each method is assigned to a specific chromatograph, except for method 0 which can be used with any chromatograph (see Chapter 3).

The peak count number is needed as a safeguard against getting page-after-page of "unknowns" if noise spikes start registering as peaks. Therefore, a realistic peak count number should be used.

Monitor types two minus signs to invite you to type in parameters to its question; these two minus signs can be considered as a question mark (?).

If the method is to be assigned to chromatograph number 3 with a peak count of 25, you should type

INST, PK CT   --3,25,:

If you notice a typing error before typing the colon, for example,

INST, PK CT   --13,35,

you should type two minus signs and then type the correct parameters. When Monitor types two minus signs it is inviting you to enter some data with which to control the analysis. However, when you type two minus signs (the first minus sign prints, the second minus sign is read by Monitor but not printed on the Teletype) you are telling Monitor to ignore everything enclosed by the double minus signs. For example,

INST,PK CT   --13,35,-3,25,:

and Monitor ignores 13,35, and accepts 3,25,:.

When you type a colon Monitor checks your input for validity before it asks the next question. When Monitor does not detect an error it proceeds to the question shown below.

UNK,FF,SMPR   --

By UNK, Monitor wants to know how to treat unknowns. You should type 0, 1, or 2 in response to this question, where

0 = Apply a response factor of 0

1 = Apply a response factor of 1

2 = Apply the response factor of the last identified peak.

Since the weight percents printed in the report are calculated using the response factors of each of the components and their peak areas, UNK = 0 allows you to get a printout where unknowns are not included in the percentages.

UNK = 1 or 2 both include these components in the weight percentages. UNK = 2 is helpful when the general response factor changes during the run as a more or less linear function of time.

By FF (filter factor), Monitor wants you to specify how much smoothing is to be done to the chromatograph signal before testing for peak start, end, shoulders, etc. A number from 0 to 7 should be your reply, determined using the following algorithm:

$$\text{SS new} = (\text{new unsmoothed signal}) + (1-2^{-FF}) \times (\text{SS old})$$

where SS is smoothed signal and FF is filter factor (0-7).

Filter factor aids in the detection of peaks. For most analyses, a filter factor of 0 or 1 is satisfactory. When very small peaks are expected, the filter factor should be raised either by raising the filter factor value when building the method or by the use of function code 11, which allows one to vary the filter factor during the analysis. This improves to signal to noise ratio and improves the sensitivity to small peaks. Filter factors of from 4 to 7 will aid the detection of slow small peaks, but should not be used unless these are expected in the run. A high filter factor should not be used with sharp quick peaks.

By SMPR, Monitor wants to know the sampling rate. The sample rate should be only as much as is needed to achieve the desired standard of repeatibility and to detect small peaks. Usually 3.75 or 7.5 data points per second is more than ample. Therefore, to determine the correct sampling

rate for a given sample and chromatograph, use the strip-chart recording to determine the length of the fastest peak or shoulder of interest and divide that length into 20, and then select the next highest sampling rate from the following table.

SMPR	Sample Rate (Samples/Second)	
	60 cps	50 cps
1	03.75	03.125
2	07.5	06.25
3	15.0	12.5
4	30.0	25.0
5	60.0	50.0

For purposes of this discussion, we will assume your response to UNK, FF,SMPR is as shown below.

UNK,FF,SMPR   --1,1,2,:

Next, Monitor types

RTYP,AF   --

where RTYP means report type and AF means area factor. Your reply to RTYP must be one of the following.

0 = Area normalization type calculation
1 = Internal standard type calculation
2 = External standard type calculation

With RTYP = 0, an internal standard peak is specified in the normalization type calculation to select one peak about which the response factor of all other peaks are scaled in calibration. The response factor of this peak is always 1.0.

Your reply to AF can be any decimal integer from 0 to 100. Area factor is a control of the degree to which GLC-8's area reallocation is used (see Appendix D). Area factors of 100% mean that the full area reallocation algorithm is used; 50% means that only 50% of the area reallocation is used; 0 reduces the area reallocation to the equivalent of dropping the perpendicular technique. As the factor is increased (from 0 to 100), the percentage of the area reallocation from the smallest peak to the next largest adjacent peak increases.

If Monitor detects an input error, the question is repeated. When Monitor is satisfied with your reply to RTYP, AF, it types the next question.

IS TM/SCALE   --

Your response to this question depends on the type of calculation specified in the preceding question. If the report type specified is normalization (0) or internal standard (1), your response should be the retention time of some peak in the chromatogram. If all peaks are unknowns, a dummy internal standard peak should be set to one second, at which time, of course, no peaks will be seen. The time of the

internal standard peak must be identical to one of the components listed in the compound table. If the report type specified is external standard (2), the scale factor is used to scale down peak areas. The scale factor is an exponent (10^{-SF}), and a value of 0-7 is normally sufficent.

Monitor's next question is:

RPKT, RRT, TBEG, TEND   --

where RPKT and RRT, respectively, represent adjusted and relative retention times of the reference peak, and TBEG and TEND represent the beginning and ending of the eligibility search-time zone, respectively. The reference peak is the largest peak found within the eligibility time zone. Its adjusted retention time should be identical to one of the components listed in the compound table (see Chapter 3). If the peak is not found in the search zone, relative retention times of all peaks will be reported as zero.

If all peaks are unknowns, a dummy reference peak can be set to one second. This fulfills the requirement that a reference peak be specified.

Your parameters to RPKT and RRT must be within the range 1-4095 seconds, and TBEG and TEND must be within the range 0-4095.

Your response could be

RPKT, RRT, TBEG, TEND   --1,1000,1,1,:

which satisfies the requirement. The relative retention time, 1000, is equivalent to 1.000.

Building a method may, from this lengthy discussion, seem complicated and time consuming. However, the lengthy discussion only represents the services performed by Monitor and the system. Your printout to this point might appear as follows.

```
INST, PK CT            ENTER,13,:
LNK, FF, SMPR          --3,25,:
RTYP, AF               --1,1,2,:
IS TM/SCALE            --0,100,:
RPKT, RRT, TBEG, TEND  --1,:
                       --1,1000,1,1,:
```

This represents about one or two minutes of your time.

Conversation proceeds to the next question, below.

F TM, CODE, VAL   --

where F means function and

      TM   = the time at which the function is implemented (0-4096 sec.)

      CODE = a code defining the function (see Table 1)

      VAL   = a numerical value applicable to most functions

Your reply to TM is taken as seconds and must be within the range 0-4095. The function codes and applicable values should be taken from Table 1.

Table 1
Functions

Function Code	Explanation	Allowable Value	Default Value
0	**Terminate Run.** This is always the last function in the method; e.g., --2016,0,0,: at 2,016 seconds end analysis and type report. Maximum run length is 4096 seconds.	null	
1	**Start Peak Search.** Commence searching for peaks; e.g., --2,1,0,: start accumulating data at 2 seconds into the run.	null	
2	**Stop Peak Search.** Suspend search for peaks, continue ignoring peaks until the next function code 1 is activated; e.g., --1,1,0,: --25,2,0,: --50,1,0,: This sequence sets a time window from 25 to 50 seconds from the beginning of the run during which data is ignored.	null	
3	**Autobase Threshold.** This controls the degree of stability which the signal must exhibit before Monitor declares it to be a true baseline. For a signal to be declared baseline it must not exceed the amplitude threshold (code 4) for the length of time specified by the autobase (code 3): e.g., --1,3,10,: --540,3,45,: At 1 second the autobase time is set to a value of 10 seconds, and at 540 seconds the autobase time is reset to 45 seconds.	1-255	12 seconds
4	**Amplitude Threshold.** This is the first stage sensitivity for detecting peaks. It is a measure of the departure from baseline that the new peak must undergo in order for the system to consider the rise as a new peak. A threshold factor of 1 is most sensitive; e.g., --58,4,1,: --125,4 4,:	0-12	1

Table 1 (Cont)
Functions

Function Code	Explanation	Allowable Value	Default Value
4 (cont)	Increase the amplitude which a peak must exhibit to be recognized as a peak from a value of 1 to a value of 4 125 seconds into the run.		
5	Slope Threshold. This is analogous to amplitude threshold with regard to shoulder sensitivity. A sensitivity of 5 is very sensitive, 40 is quite insensitive. Zero has the special meaning of ignore shoulders; e.g., --1,5,10,: --205,5,0,: --410,5,40,: At 1 second, set shoulder sensitivity to be quite sensitive; at 205 seconds, ignore shoulders until 410 seconds; then set shoulder sensitivity to be insensitive.	0-255	
6	Time Filter Period. This is the anti-noise spike control, protecting the system against false peaks and shoulders. When the signal rise penetrates the amplitude threshold, the system monitors the rise, treating it as a probationary peak. At the conclusion of the time filter period (code 6), the peak rise must be above the baseline by a value greater than the threshold specified by code 4 and the slope must not have gone negative. In this time period the peak must have accumulated an area equal to or greater than that specified by code 9. If the time between a shoulder's two inflection points does not exceed the time filter period, the false shoulder is rejected. Therefore, the larger the filter period the greater the protection. However, too large a period will result in a loss of peaks of short duration and of shallow shoulders. This parameter is stated in 1/2 seconds. See Appendix D.	1-15 (1/2 sec)	1
7	Peak Termination Time: This code protects the system from being confused by a falling baseline at the end of a large peak. With this code, one can set a time-after-crest at which a peak is arbitrarily terminated. This is also very useful when applied just before the analysis termination function (code 0). It assures that a peak which is eluting at run termination time is included in the report. (If the peak had not gone back into baseline at run termination, the peak can be terminated using code 7 with a value of 0 just before run termination.) e.g., --409,7,0,: --410,0,0,:	0-255	4095

Table 1 (Cont)
Functions

Function Code	Explanation	Allowable Value	Default Value
8	Fix Baseline. This function is effective against a drifting baseline. When implemented, the next valley found is called baseline.	null	
9	Area Threshold Factor. This is the third sensitivity control. Area threshold is the area which a peak must have within the time period set with code 6 to be recognized. See Appendix D.	0-15	5
10	Optional Relay Output. This function is used when some automatic control is desired. The value of the octal integer (0-7) is interpreted as a 3-digit binary number in which each digit is 1 or 0, according to whether the corresponding digit is set or reset; e.g., --400,7,0,: --401,10,1,: --500,10,0,: For 100 seconds, between 401 and 500, we have used relay number 1 to flush the column. The value of this function is interpreted in binary as shown below. Octal Value 3-Digit Binary 0 000 1 001 2 010 3 011 4 100 5 101 6 110 7 111	0-7	
11	This function is used to change the current filter factor to the value specified for this function. Filter factor is used to control the system's sensitivity to slowly rising peaks by improving the signal to noise ratio. In general, if the peak's amplitude rises (or falls) less than 3 μV sample (on a x 10 amplifier), the use of a filter factor greater than 0 will be necessary. Since filtering is essentially a smoothing process, it will tend to decrease the system's sensitivity to quick peaks and short baseline segments. Hence, a high filter factor should be used only for that portion of the run where long, small peaks or shoulders are expected. With a filter factor of 5, peaks of 100 μV high with a 200-second duration can be consistently detected.	1-4095	

At designated times during the analysis, certain parameters are changed or the system institutes a functional decision according to the function code and its corresponding value.

Monitor repeats this question until the function code 0 is entered, it then proceeds to the next question. Your response to these questions might be as follows.

```
F  TM,  CODE,  VAL      --1,1,0,:          (1)
F  TM,  CODE,  VAL      --1,3,10,:         (2)
F  TM,  CODE,  VAL      --1,4,1,:          (3)
F  TM,  CODE,  VAL      --1,5,0,:          (4)
F  TM,  CODE,  VAL      --1,6,2,:          (5)
F  TM,  CODE,  VAL      --1,9,1,:          (6)
F  TM,  CODE,  VAL      --3999,7,0,:       (7)
F  TM,  CODE,  VAL      --4000,0,0,:       (8)
```

The parenthesized numbers 1 through 8 are to aid you in following the discussion below.

Analysis commences upon depression of the LOC START pushbutton, and time (in seconds) is taken from that moment. The above analysis would perform as follows:

1. At one second into the analysis accept chromatographic data and accumulate peaks.

2. At one second, the autobase threshold is set to 10 seconds.

3. At one second, the amplitude threshold is set to 1, i.e., very sensitive.

4. At one second, the slope threshold is set to 0, i.e., ignore shoulders.

5. At one second, the time filter (protationary) period is set to one second (value is stated in 1/2 seconds).

6. At one second, the area threshold is set at one second.

7. At 3999 seconds, terminate the peak.

8. At 4000 seconds, cease accepting chromatographic data and commence generating the analysis report using the data accumulated.

Upon recognizing the 0 or null function code in the previous question, Monitor types

TM, NAME, TOL, RF, CWT   --

where

TM   = adjusted retention time of the component NAME (allowable input, 0-4095 seconds).

NAME   = component name (up to six characters, the first must be a letter).

TOL   = time tolerance (in seconds) for searching for the component; plus and minus this number is the time window in seconds encompassing the TM (1-99). The largest peak found within this tolerance is the component NAME. If the tolerance is greater than time, then it will be automatically adjusted to equal time.

RF   = response factor (0-32767)

CWT   = component weight percent in the calibration sample (for area normalization analyses) or component weight in the calibration sample (for internal standard analyses) (0-32767) (see Chapter 3).

Here is where you identify and name all expected components. The component time tolerance is the zone on either side of the stated retention time in which peaks are considered as potential matches for the component. The largest peak is selected in the case of multiple candidates. Other peaks found in this window are called unknowns.

Overlapping component zones (windows) are permitted and are often very useful. Caution must be taken because of the convention mentioned in the above paragraph (underlined sentence).

Response factors are scaled such that 10000 is equivalent to 1, i.e., there is an implied decimal point four places from the right.

If the calibration weight is set to 0, the system does not update the response factor of that component during a calibration run. The value assigned to CWT is taken according to the type of calculation (RTYP) specified. For an area normalization type method (RTYP =0), the value of CWT is taken as the component weight percent in the calibration sample. For internal standard (RTYP =1) and external standard (RTYP =2) methods, the value of CWT is taken as the weight of the component.

Monitor will repeat the above question until your response is a single colon. Each complete response defines one component in the sample. Therefore, if you wish to identify three components in the sample, you should respond correctly to three questions.

If you were building a method to analyze a compound for all unknonws, you would respond as follows.

            TM,NAME,TOL,RF,CWT   --1,PEAKS,1,0,0,:

            TM,NAME,TOL,RF,CWT   --:

This honors the requirement for a reference peak and an internal standard peak, although no peak will be found at 1 second; all peaks will be printed out as unknowns, thereby providing exact retention times for precise method building.

There must always be a component in the compound table whose time corresponds exactly to the time listed for the internal standard and reference peaks.

Upon receipt of the single colon (null input), Monitor terminates the method building session and stores the method on the disk for later use.

The printout of the method we've just built appears below.

```
INST, PK CT                         ENTER,13,:
UNK, FF, SMPR                       --3,25,:
RTYP, AF                            --1,1,2,:
IS TM/SCALE                         --0,100,:
RPKT, RRT, TBEG, TEND               --1,:
F TM, CODE, VAL                     --1,1000,1,1,:
F TM, CODE, VAL                     --1,1,0,:
F TM, CODE, VAL                     --1,3,10,:
F TM, CODE, VAL                     --1,4,1,:
F TM, CODE, VAL                     --1,5,0,:
F TM, CODE, VAL                     --1,6,2,:
F TM, CODE, VAL                     --1,9,1,:
F TM, CODE, VAL                     --3999,7,0,:
F TM, CODE, VAL                     --4000,0,0,:
TM, NAME, TOL, RF, CWT              --1,PEAKS,1,0,0,:
TM, NAME, TOL, RF, CWT              --:
```

This method would be used to report all components as unknowns.

2.3   ANALYZE COMMAND

This is the command used to initiate an analysis. As stated earlier, to perform an analysis you need a method, and each method, except method 0, is assigned to a specific chromatograph. Therefore, the method and chromatograph numbers are the parameters to the ANALYZE command. For example,

ANALYZE,13,3,:

tells Monitor that you wish to analyze a sample using method 13 on chromatograph 3. Immediately after you type the line terminator, the colon, Monitor makes the following checks.

a.   Is method 13 stored on the disk?

b.   Is method 13 assigned to chromatograph 3? (This check would not be made on method 0.)

c.   Is chromatograph 3 free and available for this analysis?

d.   Is there sufficient storage available on the disk to hold the chromatographic data?

e.   Is the system able to handle this analysis (no more than 240 data points per second can be specified at any one time)*?

If Monitor gets a "no" answer to any of the questions above, it types an error message indicating which condition cannot be met (see Appendix C).

If all conditions can be met, Monitor transfers method 13 from disk storage into core memory and asks the following question.

ID, RCYCL

---
*200 points per second for 50 Hz power.

which asks you to identify or name the analysis and to specify whether you want to hold method 13 in core for use with subsequent samples, i.e., whether you plan to recycle or repeat the analysis.

For ID, the first parameter, your response is completely arbitrary if your identifying name begins with an alpha character (a letter) and does not exceed 16 alphanumeric characters. This identifier will appear in the header section of the typed analysis report; therefore, care should be taken to uniquely identify each separate analysis. It is recommended that you identify the analysis according to your filing procedures.

For RCYCL, the second parameter, your response must be either 0 or 1, where

    0 = single analysis; return method to disk storage after this analysis run.

    1 = multiple analyses; until LOC CLEAR pushbutton is pressed, retain method in core for succeeding sample analyses (succeeding samples are LOC controlled).

Assume the following parameters and your printout will be as shown below.

```
                              ANALYZE,13,3,:
ID,  RCYCL                    A GLC-8 DEMO,1,:
NORM                          10000:
```

Monitor scans method 13 to determine the type of calculation (normalization, external standard, or internal standard) before asking the next question.

If it is normalization, Monitor asks NORM, to which you should specify the desired total component weight percent to be reported. A response of 10000 is equal to 100%, so that the sum of the reported component weight percents will total 100; for 9000, the sum will total 90. Your parameter must be within the range 0-40959.

If it is external standard, Monitor asks EX STD. It then takes your response, divides it by 10000, and the resulting number is multiplied by the actual component weight percent of each compound reported. A response of 10000 is normally used. Your parameter must be within the range 0-40959.

If it is internal standard, Monitor asks SAMP WT, STD WT, and waits for you to specify the total sample and internal standard weights to enable scaling during the post-sampling phase of the analysis. Your parameters must each be within the range 0-40959. If the run is a calibration, the values entered for total sample and internal standard weights are not used in the calculation.

When Monitor is satisfied with your responses (if an error is made, Monitor repeats the question), the Teletype reverts to the "free" state (to command mode, available for another command), and the READY light on the LOC associated with chromatograph 3 is lit. The system is now ready for sample injection.

As stated earlier, it is very important that you depress the LOC START pushbutton immediately after sample injection. This is repeated here because a major cause of component-peak misidentification is not only the proportional delay due to column aging but also the delay due to the variation of timing between sample injection and START pushbutton depression.

2.4   CALIBRATE COMMAND

The CALIBRATE command is similar to the ANALYZE command in that Monitor asks the same questions and both commands are used to execute an analysis run and to generate an analysis report.

When Monitor is in command mode the calibration run may be initiated by typing

CALI,13,1,:

where CALI is CALIBRATE abbreviated, 13 is the method number, and 1 is the chromatograph number. The procedures for CALIBRATE and ANALYZE are identical in that they both ask for ID, RCYCL and either NORM, EX STD, or SAMP WT,STD WT.

A calibration calculates the response factors and updates them in the compound table in the method (see Chapter 3).

Response factors of components with zero calibration weights are not updated. If any component with an assigned calibration weight is not found, the calibration run is invalid and the component response factors are not updated in the method. This condition is indicated in the report when the response factor and component percent columns are empty. In normalization analyses the internal standard peak is used to scale all other component response factors. Consequently, its response factor is always 10000 (or 1.000).

2.5   MODIFY COMMAND

The MODIFY command allows on-line modification of a method. When Monitor is in command mode (i.e., when it has spaced toward the center of the Teletype paper) it is waiting for you to type one of its seven commands, in this case the MODIFY command.

Assume you want to change the parameter to RTYP in method 13, which was built earlier using the ENTER command. You would type,

MODIFY,13,:

where 13 is the number of the method to be modified. When you type the colon to terminate the line, Monitor retrieves a copy of method 13 from the disk and types the first line of the method:

INST,PK CT   MODIFY,13,:
                              --3,25,

and waits for you to either change one or more parameters or to type a colon which tells Monitor to leave that line unaltered and type the next line,

<div align="center">

UNK,FF,SMPR   --1,1,2,

</div>

and waits for you to type. RTYP is on the next line so you then type another colon and Monitor types

<div align="center">

RTYP,AF   .--0,100,

</div>

If you want to specify an external standard calculation, instead of the present area-normalization calculation, you then type 2,100,: Monitor would accept the change and type the next line. The printout to this point appears below.

```
                                MODIFY,13,:
          INST, PK CT           --3,25,:
          UNK, FF, SMPR         --1,1,2,:
          RTYP, AF              --0,100,2,100,:
          IS TM/SCALE           --1,
```

Monitor will type the next line in the method and wait for you to type the line terminator. This will continue until you have typed a colon after the last line in the method, whereupon Monitor will return the modified method to disk storage for subsequent retrieval.

However, after all modifications have been made, the typing out of the rest of the method can be hastened by placing a punched paper tape of colons in the Teletype reader and setting the reader switch to START. When Monitor types a line it will accept a colon from the paper tape reader and commence typing the next line, accepting subsequent colons from the paper tape reader until the entire method is typed. (Procedures for punching the paper tape of colons are in Appendix C of the PDP-8/I System User's Guide.)

<div align="center">

NOTE

</div>

If one parameter on a line is to be changed, all parameters on the line must be re-entered. In the case of function and component entries, no entries can be added or deleted, but only changed.

2.6   DUMP COMMAND

A copy of a method or a group of methods can be obtained using the DUMP command. This command enables you to have a copy of one or more methods typed on the teleprinter and, if desired, simultaneously punched on paper tape using the Teletype punch.

If, for example, you want a printed copy of method 13 for your files and also a punched paper tape copy for your method library, you would type (with Monitor in command mode and the paper tape punch ON) the following.

DUMP,13,:

(The Teletype operating procedures in the PDP-8/I System User's Guide instruct you on how to generate leader/trailer tape, when to depress the Teletype punch ON button, and why these actions are performed.)

If you wanted to dump methods 8, 9, 10, 11, 12, and 13, you would type

DUMP,8,13,:

and methods 8 through 13 would be copied inclusively.

When a method to be dumped is busy, e.g., being used in an analysis, Monitor types mn METHOD BUSY (mn is the method number) and returns to command mode. For example if, after dumping methods 8 and 9, Monitor types

10 METHOD BUSY

the dumping session stops, Monitor returns to command mode, and waits for your next command.

The method is copied exactly as it was entered onto the disk, except that Monitor will double space between lines as it types the method.

2.7   DELETE COMMAND

The DELETE command is used to erase one or more methods from the disk. When a method is no longer needed it can be deleted from the disk, allowing more storage for additional new methods.

DELETE requests are written in the following format:

DELETE, 13,:

when only one method is to be deleted, or

DELETE,8,13,:

when a group of methods is to be deleted. If an attempt is made to delete a busy method, Monitor types mn METHOD BUSY (mn is the method number) and returns to command mode, as explained in the previous section (DUMP Command).

CHAPTER 3
USING GLC-8

This chapter is a description of an actual analysis using GLC-8 and a Perkin-Elmer 900 Chromatograph with a chromatogram. Here, we will start from "scratch" by building a method 0, which you need do only once. Actual Teletype output is used, and each step performed during this analysis is illustrated in Figure 11, System Operation, concluding the chapter.

We will assume that the chromatograph and chromatogram are calibrated and ready for use, that the system is loaded and started as described in Appendix A, and that we have initialized with the date and time as explained in Section 1.5.

This run is to establish a calibrated method for future quality control analyses of the same type of sample. It has been previously analyzed manually, therefore, the components are known and can be identified by comparing our strip-chart recording to those obtained from the GLC-8.

3.1   ASSIGN CHROMATOGRAPH AND TELETYPE

First we must assign the chromatograph (number 1) we intend to use to our Teletype (number 2). This is done when we type

```
ASSIGN,1,2,:
```

3.2   ENTER METHOD 0

All other methods must be assigned to a specific chromatograph; the method that is built and numbered 0 is reserved for use with any chromatograph.

In this example we will use method 0 for an area normalization analysis, and it is built using the ENTER command. The specified control parameters (filter factor and functions) are at your discretion, however, we have found the following guidelines to be helpful.

a.   RTYP = 0,   (normalization type calculation)

b.   UNK = 1,   (1.0 response factor for unknowns)

c.   IS TM/SCALE = 1,:   ⎰ dummy internal standard

d.   RPKT, RRT, TBEG, TEND = 1,1000,1,1,: ⎱ and reference peak

e.   The last entered F TM, CODE, VAL = 4000,0,0,:

f.   TM, NAME, TOL, RF, CWT = 1,PEAKS,1,0,0,: (only one entered: this is a dummy reference and internal standard peak to correspond with c and d, above.)

This analysis will run for 4000 seconds or until the LOC STOP pushbutton is pressed. All peaks will be reported as UNK (unknowns) with their area percent listed in the report.

Our method 0 is built as follows because we want to see all components in our compound.

```
                                            ENTER,Ø,Ø,:
    INST, PK CT                             --1,15,:
    UNK, FF, SMPR                           --1,1,2,:
    RTYP, AF                                --Ø,100,:
    IS TM/SCALE                             --1,:
    RPKT, RRT, TBEG, TEND                   --1,1000,1,1,:
    F TM, CODE, VAL                         --1,1,Ø,:
    F TM, CODE, VAL                         --1,3,10,:
    F TM, CODE, VAL                         --1,4,1,:
    F TM, CODE, VAL                         --1,5,Ø,:
    F TM, CODE, VAL                         --1,6,2,:
    F TM, CODE, VAL                         --1,9,1,:
    F TM, CODE, VAL                         --3999,7,Ø,:
    F TM, CODE, VAL                         --4000,Ø,Ø,:
    TM, NAME, TOL, RF, CWT                  --1,PEAKS,1,Ø,Ø,:
    TM, NAME, TOL, RF, CWT                  --:
```

Figure 4   Method 0

If any parameter is not clearly understood, refer to Chapter 2 for a thorough explanation.

3.3   DUMP METHOD 0

When method 0 has been decided upon and built it may be dumped on the Teletype printer and punch for future reference and use. This is done when we type

DUMP,Ø,:

When dumping onto paper tape, depress the Teletype punch ON button, and then type DUMP,0,: (see PDP-8/I System User's Guide for additional information).

When a method is built it is stored on the disk, and a copy of the method is transferred from disk into core memory when it is "called" with any but the ENTER command, as when we type DUMP, 0,:. Method 0 is still on the disk and available for subsequent use.

3.4   ANALYZE WITH METHOD 0

With the chromatograph and chromatogram calibrated for use and method 0 built and stored on the disk, we are ready to run our analysis. First, we call method 0 into core memory, name (identify) the analysis run, and specify whether we wish to hold the method in core memory for use on succeeding runs (recycle).

```
                              ANALYZE,0,1,:
      ID, RCYCL               DEMONSTRATION 1,0,:
      NORM                    10000:
```

Our command and response was correct because we didn't get an error message. Therefore, method 0 is on the disk and available for use on chromatograph 1, since it's the universal method, available to any connected chromatograph. Monitor also accepted our identification and recycle parameters.

Immediately after typing the second colon, above, the READY light on the LOC associated with chromatograph 1 lit, indicating that the system is ready for sample injection.

We inject our sample and immediately press the LOC START pushbutton to start the analysis run.

Chromatograph attenuation may be manually changed at any time without affecting the chromatographic input to GLC-8.

Since method 0 is built to run 4000 seconds, visual inspection of the chromatogram output indicates when the compound has passed through the column (Figure 5). At this time, we manually terminate the run by pressing the LOC STOP pushbutton. This causes GLC-8 to generate and type the analysis report (Figure 6).

Since we are looking for all components of the sample, we did not identify by name any expected peak, therefore, in the analysis report all component peaks are printed as UNK for unknown.

Compare the strip-chart recording with the analysis report and select the peaks of interest. We see from the report that 11 peaks (numbered in parentheses) were detected--some aren't visible in the strip-chart recording. We are interested in only seven of the reported peaks, those numbered 2,3,5,6,7,8, and 9.

The analysis revealed three peaks of no interest. We note that in this case they are all very small peaks. Increasing the minimum peak area (code 9) from a value of one to a value of two should result in disqualification of these unwanted peaks.

3.5   ENTER METHOD 23

We now use the analysis report to build our unique method. The number we assign to this method is arbitrary, as long as the number isn't assigned to a method already on the disk. Ours will be method 23, and it is method X in the flow chart of Figure 11. Method 23 is shown in Figure 7 and is explained below.

Method 23 was built from method 0, therefore, only the changes need be noted; the numbers itemized below correspond to the parenthesized numbers in Figure 7.

1. Looking for the seven peaks of interest only.

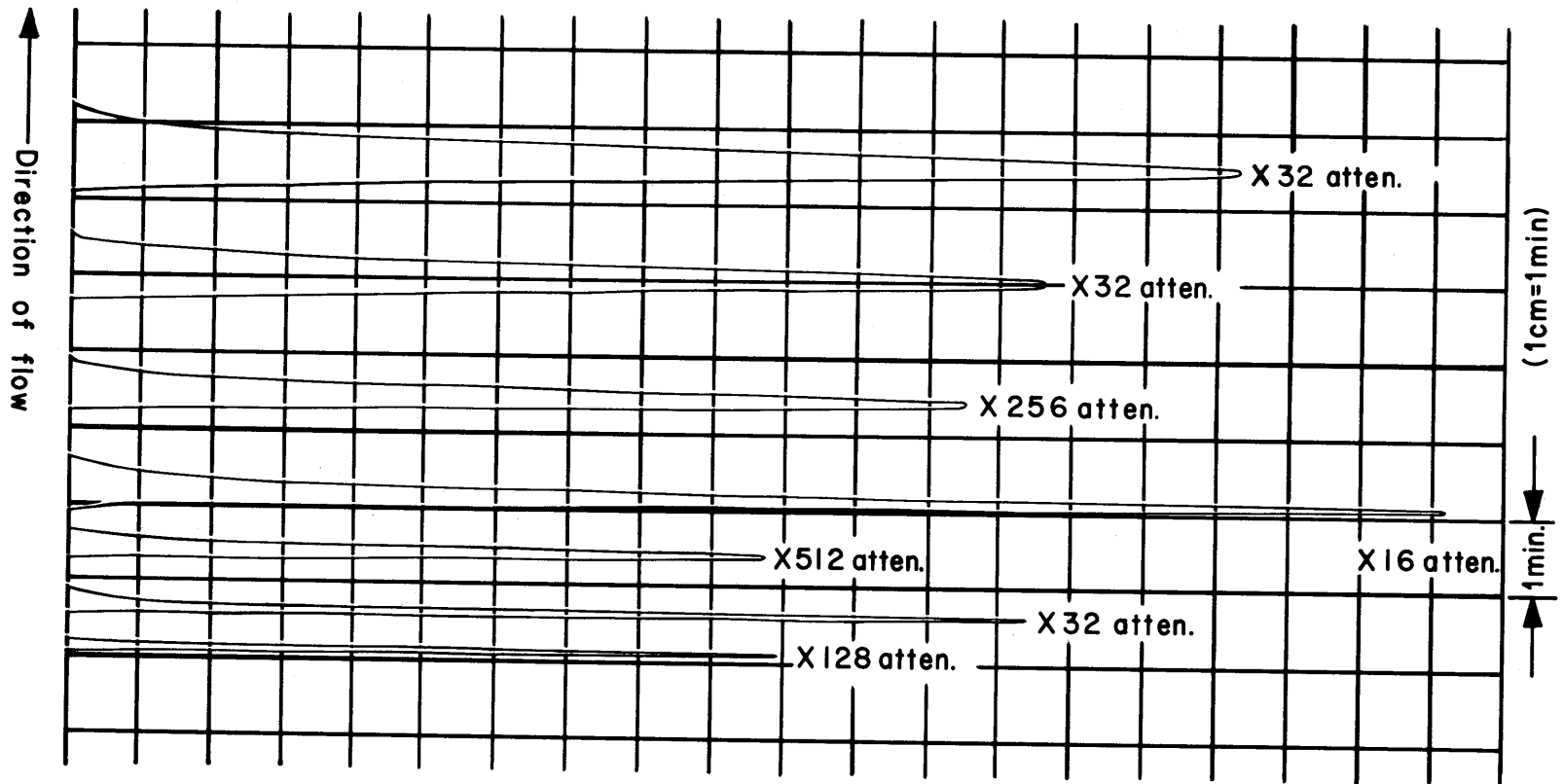2. We chose an arbitrary peak as our internal standard peak, even though this is a normal-ization report.

Figure 5   Chromatogram Recording

```
ANALYSIS

DATE   2   15   69      TIME   1333

INST   1        METHOD           NORM

DEMONSTRATION 1                 10000

COMPONENT   RRT     TIME   PEAK AREA   TYPE   RF FACT    COMP%    TOL

PEAKS

UNK         .000      9      8075       BB     10000     .006%      (1)

UNK         .000     32      6612 !     BB     10000    5.608%      (2)

UNK         .000    107    4065748     BV     10000    3.448%      (3)

UNK         .000    168     93367      VV     10000     .079%      (4)

UNK         .000    192     24032 !     VV     10000   20.381%      (5)

UNK         .000    235      5557 !     VV     10000    4.713%      (6)

UNK         .000    304     36508 !     VV     10000   30.963%      (7)

UNK         .000    349     16712 !     VV     10000   14.173%      (8)

UNK         .000    387     23769 !     VV     10000   20.159%      (9)

UNK         .000    406    547316      VB     10000     .464%     (10)

UNK         .000    510                 BB     10000     .000%     (11)
```

Figure 6   Area Normalization Analysis Report

3. Explained in Section 2.2.

4. Started data acquisition here.

5. Increased the value from one to two to eliminate unwanted peaks.

6. Create baseline point.

7. Terminate analysis run and generate analysis report.

8. This is the component table. The component weights correspond to the weight percents of the components in our known sample.

Now we have built a customized method, identifying the peaks of interest.

```
INST, PK CT            ENTER,23,:
UNK, FF, SMPR          --1,7,:
RTYP, AF               --1,1,2,:                        (1)
IS TM/SCALE            --0,100,:
RPKT, RRT, TBEG, TEND  --192,:                          (2)
F TM, CODE, VAL        --304,1000,280,320,:             (3)
F TM, CODE, VAL        --1,1,0,:                        (4)
F TM, CODE, VAL        --1,3,10,:
F TM, CODE, VAL        --1,4,1,:
F TM, CODE, VAL        --1,5,0,:
F TM, CODE, VAL        --1,6,2,:
F TM, CODE, VAL        --1,9,2,:                         (5)
F TM, CODE, VAL        --409,7,0,:                       (6)
TM, NAME, TOL, RF, CWT --410,0,0,:                       (7)
TM, NAME, TOL, RF, CWT --32,A1,10,10000,767,:  ⎞
TM, NAME, TOL, RF, CWT --107,A2,20,10000,471,:  ⎟
TM, NAME, TOL, RF, CWT --192,A3,20,10000,2087,: ⎟
TM, NAME, TOL, RF, CWT --235,A4,15,10000,562,:  ⎬     (8)
TM, NAME, TOL, RF, CWT --304,A5,18,10000,2941,: ⎟
TM, NAME, TOL, RF, CWT --349,A6,15,10000,1363,: ⎟
TM, NAME, TOL, RF, CWT --387,A7,20,10000,1809,: ⎠
TM, NAME, TOL, RF, CWT --:
```

Figure 7   Method 23 Before Calibration

3.6   CALIBRATE METHOD 23

Our method must now be calibrated to update the response factors. This is done by typing the CALIBRATE command and responses as shown below.

```
                    CALIBRATE,23,1,:
ID, RCYCL           CALI DEMO 3,0,:
NORM                10000:
```

With our input correctly entered, the LOC READY light comes on; the system is ready for our calibration analysis run. We inject our sample and immediately press the LOC START pushbutton and the calibration analysis run commences. After 410 seconds the LOC COMP light comes on and the calibration report shown in Figure 8 is typed.

3.7   DUMP METHOD 23

During calibration, GLC-8 automatically updates method 23 to contain the reported response factors. Method 23 is now complete and may be dumped on the Teletype printer and punch for future reference and use. Method 23 is still stored on the disk and is available for future analyses of the same sample.

We dump method 23 when we type DUMP,23,:, as shown in Figure 9. Notice in the print-out that the response factor parameters have been updated.

```
CALIBRATION

DATE    2   15   69      TIME   1350

INST    1        METHOD    23      NORM

CALI DEMO 3                      10000

COMPONENT    RRT    TIME   PEAK AREA   TYPE   RF FACT    COMP%    TOL

A1          .118     37       8090  !   BB     16784      767    -  41%

A2          .379    118       4990  !   BB     17839      471    -  41%

A3          .646    201      41422  !   BV     10000     2087    -  22%

A4          .781    243       6632  !   VB     16049      562    -  16%

A5         1.000    311      36425  !   BV     15817     2941    +    %

A6         1.144    356      19247  !   VB     14268     1363    +   6%

A7         1.263    393      22503  !   BB     15225     1809    +  14%
```

Figure 8   Calibration Report of Method 23

```
                                   DUMP,23,:--         ENTER,23,1,:
INST, PK CT                     --      1,       7,  :
UNK, FF, SMPR                   --      1,       1,      2,  :
RTYP, AF                        --       ,     100,  :
IS TM/SCALE                     --    192,  :
RPKT, RRT, TBEG,  TEND          --    304,   1000,    280,    320,  :
F TM, CODE, VAL                 --      1,       1,       ,  :
F TM, CODE, VAL                 --      1,       3,     10,  :
F TM, CODE, VAL                 --      1,       4,      1,  :
F TM, CODE, VAL                 --      1,       5,       ,  :
F TM, CODE, VAL                 --      1,       6,      2,  :
F TM, CODE, VAL                 --      1,       9,      2,  :
F TM, CODE, VAL                 --    409,       7,      0,  :
F TM, CODE, VAL                 --    410,        ,       ,  :
TM, NAME, TOL, RF, CWT          --     32,  A1     ,     10,   16784,     767,  :
TM, NAME, TOL, RF, CWT          --    107,  A2     ,     20,   17839,     471,  :
TM, NAME, TOL, RF, CWT          --    192,  A3     ,     20,   10000,    2087,  :
TM, NAME, TOL, RF, CWT          --    235,  A4     ,     15,   16049,     562,  :
TM, NAME, TOL, RF, CWT          --    304,  A5     ,     18,   15817,    2941,  :
TM, NAME, TOL, RF, CWT          --    349,  A6     ,     15,   14268,    1363,  :
TM, NAME, TOL, RF, CWT          --    387,  A7     ,     20,   15225,    1809,  :
--:
```

Figure 9   Method 23 After Calibration

3.8 ANALYZE WITH METHOD 23

Method 23 is now calibrated and ready to control the analysis of our sample. From now on, this is where you'll start when analyzing with method 23; it will always be stored on the disk and available for use merely by typing ANALYZE, 23,1,:.

To perform the analysis of our sample using the calibrated method, we need only type the ANALYZE command and respond as shown below.

```
                          ANALYZE,23,1,:
  ID, RCYCL               ACTUAL ANALYSIS,0,:
  NORM                    10000:
```

The LOC READY light comes on, indicating that the system is ready for sample injection. Immediately after injecting our sample we press the LOC START pushbutton and the analysis commences. After the 410-second run the LOC STOP light comes on and the analysis report is typed by Monitor.

```
ANALYSIS

DATE    2   15   69      TIME   1410

INST    1        METHOD    23       NORM

ACTUAL ANALYSIS                  10000
```

COMPONENT	RRT	TIME	PEAK AREA	TYPE	RF FACT	COMP%	TOL
A1	.119	37	8040 !	BB	16784	7.671%	- 42%
A2	.379	118	5522 !	BB	17839	4.701%	- 38%
A3	.648	201	41045 !	BV	10000	20.872%	- 25%
A4	.780	242	6687 !	VB	16049	5.620%	- 15%
A5	1.000	310	36164 !	BV	15817	29.413%	+ %
A6	1.141	354	19195 !	VB	14268	13.630%	+ 12%
A7	1.264	392	22392 !	BB	15225	18.093%	+ 12%

Figure 10 Actual Analysis Report Using Method 23

Figure 11 provides a flow chart summarizing the analysis performed in this chapter.
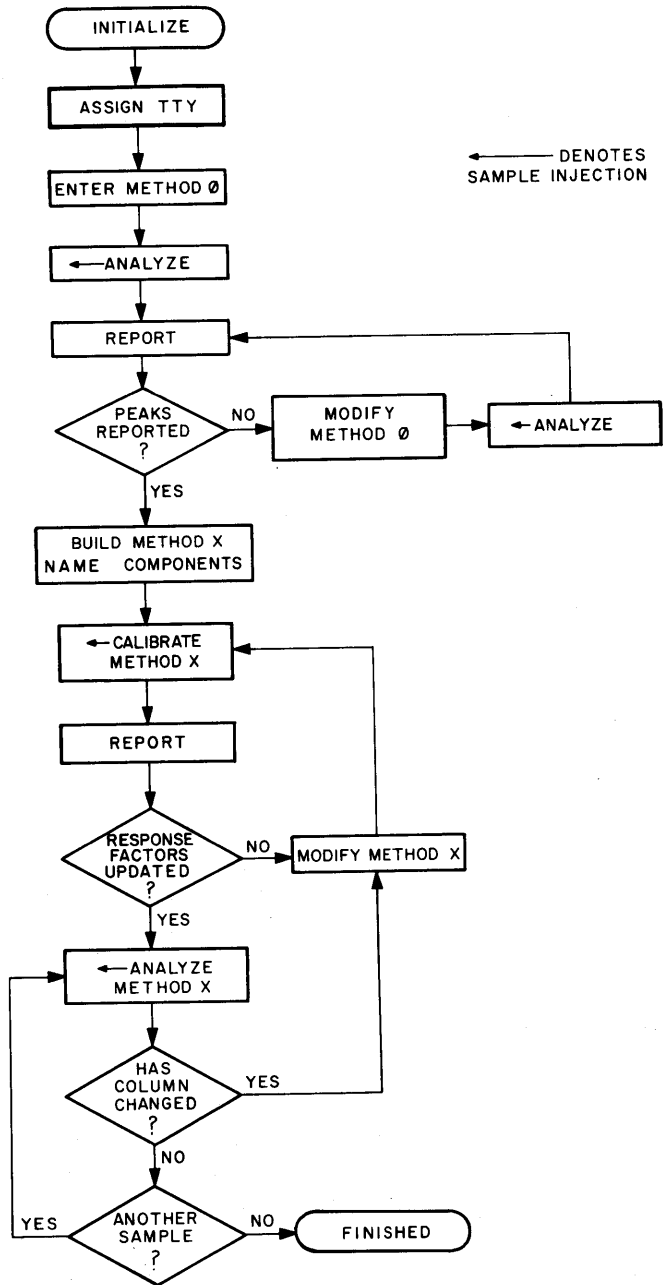
Figure 11 System Operation

APPENDIX A
LOADING AND STARTING PROCEDURES

The GLC-8 program is furnished on a punched paper tape and is loaded into core memory using the paper tape reader, either Teletype or the optional high-speed photoelectric reader. The system program is loaded into core memory, optionally saved on the disk, and initialized for use as outlined below.

The Disk Bootstrap Loader is used to load GLC-8 to and from disk and core.

The GLC-8 system program tape is punched in binary coded format, therefore, it is loaded into core using the Binary (BIN) Loader.

NOTE

For computer and Teletype console operations see
PDP-8/I System User's Guide, DEC-08-NGCB-D.

A.1 LOAD AND START

a. Load the Read-In-Mode (RIM) Loader (see PDP-8/I System User's Guide).

b. Load the Binary (BIN) Loader (see PDP-8/I System User's Guide).

c. Press the STOP switch.

d. Put the program tape into the paper tape reader.

e. Set the switch register (SR) to 7777, press LOAD ADD (if using the high-speed photoelectric reader, reset SR to 3777), and then START switches. The tape will start reading in through the tape reader and stop when about half of the tape has been read in.

f. Check the accumulator (AC), it should not have any bits lit. If it does, the program has not been read in correctly and you must start again at step b above.

g. Press the CONT switch and the tape begins reading in again. When the tape stops GLC-8 is in core.

h. Check the AC, it should not have any bits lit. If it does, start over at step b, above.

i. Press the STOP switch.

j. Set the SR to the starting address of GLC-8 (presently, 6623), press LOAD ADD and then START.

k. GLC-8 will type the version number of the system program and MONTH, DAY, YEAR, HOUR, MINUTE, and wait for your response.

A.2   LOAD ON DISK AND START

The Disk Bootstrap Loader is used to transfer GLC-8 from core onto disk.

a.   After performing steps a through i in section A.1, above, proceed to step b, below.

b.   Put the Disk Bootstrap Loader tape in the paper tape reader and set the reader to START.

c.   Set the SR to 7756, press LOAD ADD and then START switches. Wait until the tape has read in; then proceed to step d.

d.   Press the STOP switch.

e.   Set the disk write-lock switches: DISC 0 and upper 16K to OFF. These switches can be seen by removing the disk cover. (See GLC-8 Maintenance Manual, DEC-08-HGAA-D.)

f.   Set the SR to 7702, press LOAD ADD and then START. The system will be transferred from core onto disk and the PAUSE light will come on.

g.   Press the STOP switch.

h.   Set the disk write-lock switches: DISC 0 and upper 16K to ON.

i.   Set the SR to the starting address of GLC-8 (presently, 6623), press LOAD ADD and then START.

j.   GLC-8 will type the version number of the system program and MONTH, DAY, YEAR, HOUR, MINUTE, and wait for your response.

A.3   LOADING FROM DISK INTO CORE

The Disk Bootstrap Loader is used to transfer GLC-8 from disk into core. With GLC-8 on the disk, perform the following steps.

a.   Load the RIM Loader.

b.   Press the STOP switch.

c.   Put the Disk Bootstrap Loader tape in the paper tape reader and set the reader to START.

d.   Set the SR to 7756, press LOAD ADD and then START switches; wait until the tape has read in, then proceed to step e.

e.   Press the STOP switch.

f.   Set the SR to 7700, press LOAD ADD and then START.

g.   Press the STOP switch.

h.   Set the SR to the starting address of GLC-8 (presently, 6623), press LOAD ADD and then START.

i.   GLC-8 will type the version number of the system program and MONTH, DAY, YEAR, HOUR, MINUTE, and wait for your response.
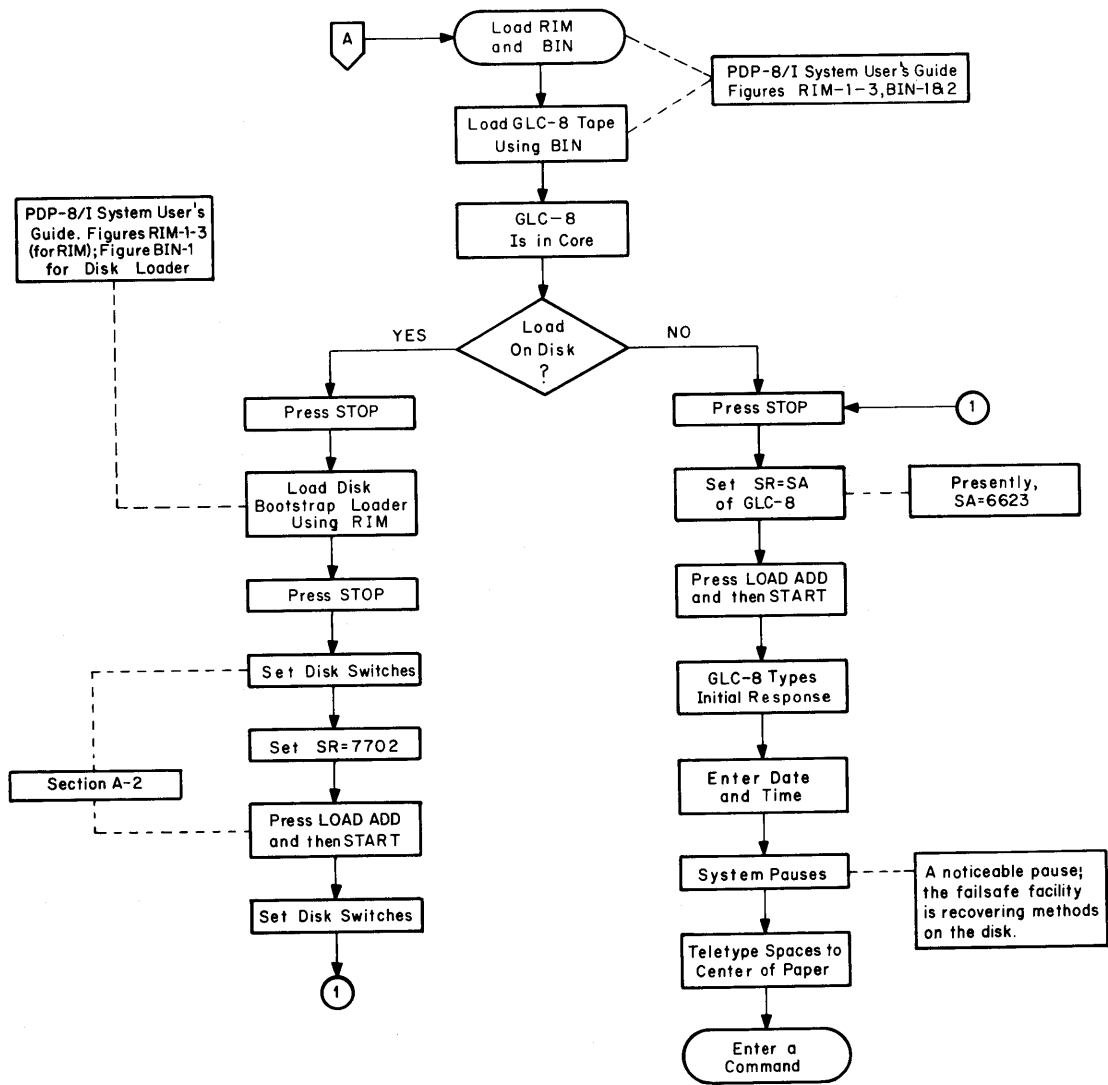
Figure A-1   Loading and Starting Procedures
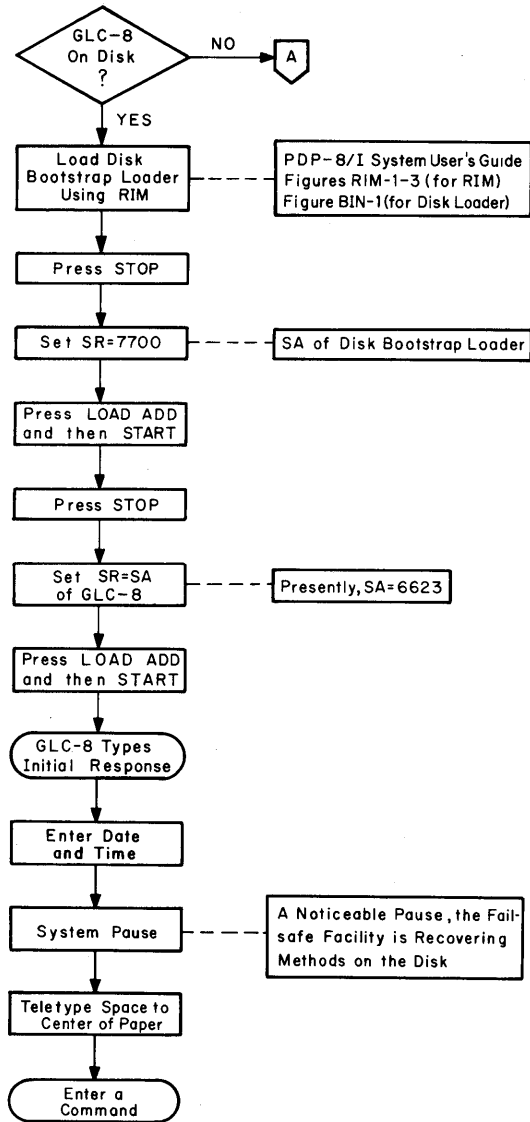
Figure A-2 Loading GLC-8 From Disk Into Core

APPENDIX B
SUMMARY OF COMMANDS, QUESTIONS, AND RESPONSES

B.1   <u>MONITOR COMMANDS</u>

The seven Monitor commands with the required parameters are listed below. The parameters are coded as follows:

    mn = method number (0-99)

    q   = questions (0 = type; 1 = do not type)

    cn = chromatograph number (1-64)

    tn = Teletype number (1-9)

Command	Format	Explanation
ASSIGN	ASSIGN, cn, tn,:	Assigns subsequent reports of chromatograph cn to be typed on Teletype tn.
ENTER	ENTER, mn, q,:	Used to build and enter a new method onto the disk.
ANALYZE	ANALYZE, mn, cn,:	Method mn will be used on the analysis of chromatograph cn.
MODIFY	MODIFY, mn,:	Method mn is typed line-by-line, pausing at the end of each line for parameter modification.
CALIBRATE	CALIBRATE, mn, cn,:	Performs a calibration analysis on method mn using chromatograph cn.
DUMP	DUMP, mn, mn,:	Copies of methods mn through mn are typed on the Teletype and/or punch; for only one method, omit the second parameter.
DELETE	DELETE, mn, mn,:	Deletes methods mn through mn from the method library; for only one method, omit the second parameter.

B.2   <u>QUESTIONS AND RESPONSES</u>

B.2.1   <u>ENTER</u>

Questions	Explanation and Responses
INST, PK CT	Instrument (chromatograph) number (1-64). Peak count -- number of peaks which will stop the run (1-200).

Questions	Explanation and Responses

UNK, FF, SMPR

Response factor applied to unknowns (0-2):
0 = apply response factor of 0
1 = apply response factor of 1
2 = apply response factor of last identified peak
Filter factor -- digital smoothing (0-7):
0 = none; 7 is maximum; 1 is typical
Sampling rate:

	60 cps	50 cps
1 =	03.75	03.125
2 =	07.50	06.250
3 =	15.00	12.500
4 =	30.00	25.000
5 =	60.00	50.000

RTYP, AF

Report type (0-2):
0 = area normalization
1 = internal standard
2 = external standard
Area reallocation factor (for fused peaks)
(0-100):
   0 = dropping the perpendicular
100 = complete GLC-8 algorithm (see Appendix D).

IS TM/SCALE

Elution time of internal standard (1-4095 seconds).
Scale factor to which other components are scaled;
used for external standard (1-4095); 1 is normally
sufficient

RPKT, RRT, TBEG, TEND

Retention time of reference peak (1-4095 seconds).
Relative retention time of reference peak
(1-4095 = 0.001 to 4.095).
Beginning and ending of time window in which
reference peak will be found (1-4095 seconds).

F TM, CODE, VAL

Time at which function is implemented
(0-4096 sec.). Function code (see below).
Value applicable to function (see below).

NOTES

Time (TM) must be entered in an increasing sequence.
Code 0 is entered last

Code	Explanation	Allowable Value	Default Value
0	Terminate run and write analysis report	Null	
1	Start peak search; monitor signal; accumulate peaks	Null	
2	Stop peak search but continue monitoring signals	Null	

Code	Explanation	Allowable Value	Default Value
3	Autobase threshold	1-255 seconds	12 seconds
4	Amplitude threshold	0-12	1
5	Slope threshold; shoulder sensitivity 0 = ignore shoulders	0-255	0
6	Time filter period; validates peaks; anti-noise spike control (value stated in 1/2 seconds)	1-15	1
7	Peak termination time; time-after-crest terminator	0-255 seconds	4095
8	Fix baseline	Null	
9	Area threshold factor	0-15	1
10	Optional relay output	0-7 (octal)	
11	Reset filter factor	1-4095	

TM,NAME,TOL,RF,CWT   Adjusted retention time of component NAME (0-4095 seconds).

Component name (1-6 characters, the first must be a letter).

Time tolerance; search window; + and – number in seconds.

Response factor (0-32767)

Calibration weight (0-32767)

B.2.2   ANALYZE AND CALIBRATE

Questions	Explanation and Responses
ID, RCYCL	Identification (name) of analysis (1-16 characters, the first must be a letter).
	Recycle for next sample (0 or 1): 0 = single analysis 1 = multiple analyses
NORM	For normalization only. Total component weight (1-40959)
EX STD	For external standard only. Response ÷ 10000 x actual component weight percent = comp % reported (1-40959)

Questions	Explanation and Responses
SAMP WT, STD WT	For internal standard only. Total sample weight (1-40959). Internal standard weight (1-40959).

APPENDIX C
ERROR MESSAGES

Error messages are composed of a three-character code. The first character is an asterisk and the next two characters are the error code. In the following list, when the second error code character is shown as an n, the n is actually replaced with the parameter number in error, i.e., N2 means the second parameter is not numeric. Error messages are printed immediately after you type the line terminator, the colon.

Error Message	Meaning
*B3	Chromatograph to which method is assigned is busy
*Cn	Character string not present
*DM	Method deleted in modify since it is impossible to enter an internal standard
*D0	Insufficient disk storage, method is deleted
*D1	Insufficient memory to run analysis
*D2	Insufficient memory to accommodate new method
*I1	Internal standard not entered
*I2	Cannot modify function code zero in analysis method
*I3	Illegal chromatograph/method pair
*M2	Method is missing
*Nn	Numeric response not present
*Pn	More then n parameters expected
*P0	Enter parameters
*S1	Statement not recognized
*S2	Sequence error, third parameter must be equal to or greater than second parameter or zero
*T1	Component or function time not greater than last component or function time
*T2	Insufficient memory to schedule the analysis
*U2	Method number is already entered in method library
*Vn	Value too large
*Zn	Zero is illegal value
mn METHOD BUSY	Method number mn is running an analysis

APPENDIX D
ANALYSIS SEQUENCE

D.1   DURING THE ANALYSIS

During the analysis, GLC-8 accumulates and calculates the chromatograph data as explained below.

D.1.1   Peak Detection

When the system is actively searching for peaks, it maintains a trend analysis not of the digitalized chromatograph output but of a software filtered version of it, i.e., the smoothed signal. The smoothed signal is defined as:

$$SS \text{ new} = (\text{new unsmoothed signal}) + (1-2^{-FF}) \times SS \text{ old}$$

where SS is smoothed signal and FF is filter factor.

A running estimate of what the baseline should be is maintained as a continuous variable. Best estimate of baseline (BEB) is a mathematically computed average of the current baseline amplitude.

If the signal positively penetrates the amplitude threshold, the system tests the validity of this potential new peak while simultaneously integrating the potential peak as though it were valid (see Figure D-1). The system freezes the potential new peak's start time/height vector at the sample period just prior to the period in which the amplitude threshold was penetrated, and it temporarily accumulates the peak area. For a defined time filter period (code 6) the signal is monitored. If the signal decreases during this period, the potential peak is rejected and the system reverts to tracking baseline, thus constituting the noise spike filter feature. At the end of the filter time period, the area is compared with the area threshold (code 9).

1. Amplitude > A
2. Area > A'
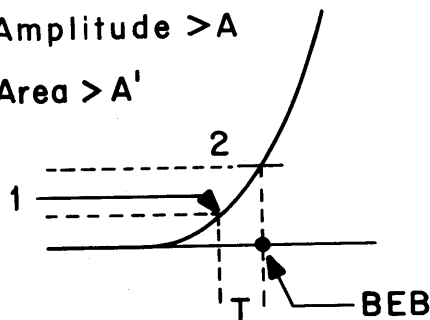
A = Amplitude threshold (code 4)
A'= Area threshold (code 9)
T = Time filter threshold (code 6)
BEB = Best estimate of baseline

Figure D-1   Peak Detection

If the area does not exceed the area threshold, the potential peak is rejected and the system again reverts to tracking baseline (this feature discriminates a true peak from baseline drift).

Peak termination is the inverse of peak detection.

D.1.2 Shoulders

If the slope starts to decrease and if the magnitude of the decrease is in excess of a specified threshold value (see Figure D-2), the first inflection point is established. This threshold is a control variable, called the slope threshold (code 5). The slope threshold can be altered at any time by implementing function code 5.

If the slope again begins to rise, if the magnitude of the rise exceeds the slope threshold, and if the time difference between the two inflections exceeds the time filter period, the peak integrated so far is designated a shoulder and terminated; a new peak is generated, effective from the second inflection.

However, if the elapsed time between the two inflections does not exceed the time filter period, the potential shoulder is rejected as being caused by a noise spike. The reverse is true for trailing shoulders.
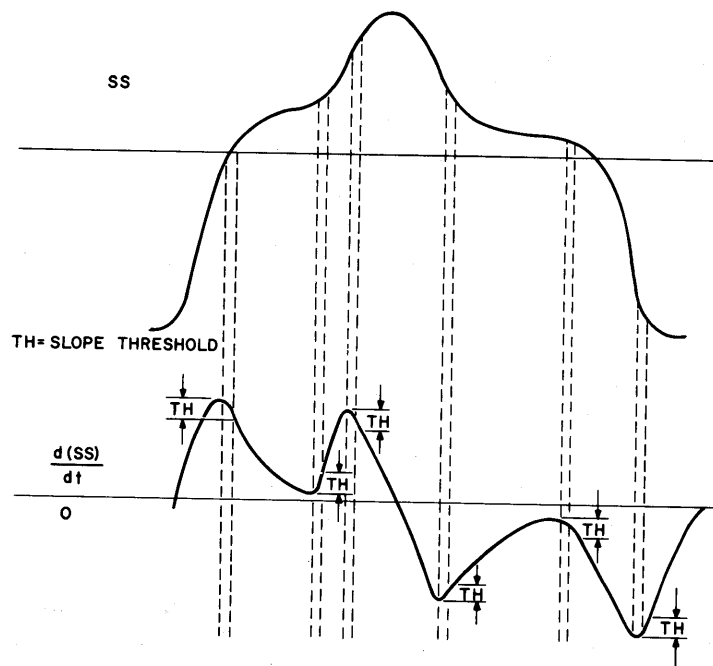
Figure D-2 Shoulders

D.1.3   Storage of Peak Parameters

Using the disk as a chained array, the following information for each peak is stored:

Start time   Start height

Crest time   Crest height

End time   End height

Total Peak area

This can be envisioned as pictured below.
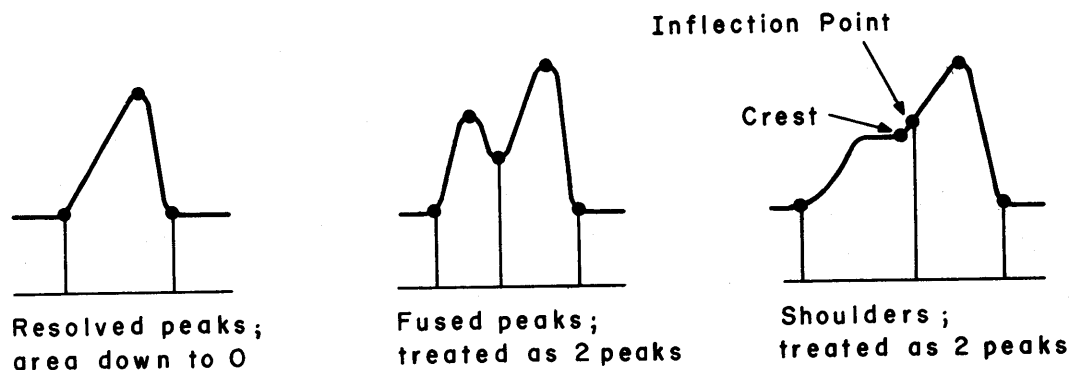
Figure D-3   Peak and Shoulder Detection

D.2   AFTER THE ANALYSIS

Upon completion of the chromatographic analysis, GLC-8 reconstructs the chromatogram and performs analysis calculations as explained below.

D.2.1   Baseline Correction

This operation can be explained in five steps:

1. Start of the first peak and the end of the last peak are called basepoints.

2. A scan is made to establish basepoints as per the autobase time limit (code 3).

3. A scan is made to establish basepoints by fixing the baseline (code 8).

4. A scan is made to establish basepoints by peak termination time (code 7).

5. Starting from the first established basepoint, the program successively calculates the slope between that basepoint and each subsequent valley until it encounters the next established basepoint. The right-hand end (valley) of the line having the most negative (or least positive) slope is flagged as a basepoint. This double-step operation is repeated until the end of the last peak is found. The basepoints are then connected and the area below this baseline is subtracted from each peak area (see Figure D-4).
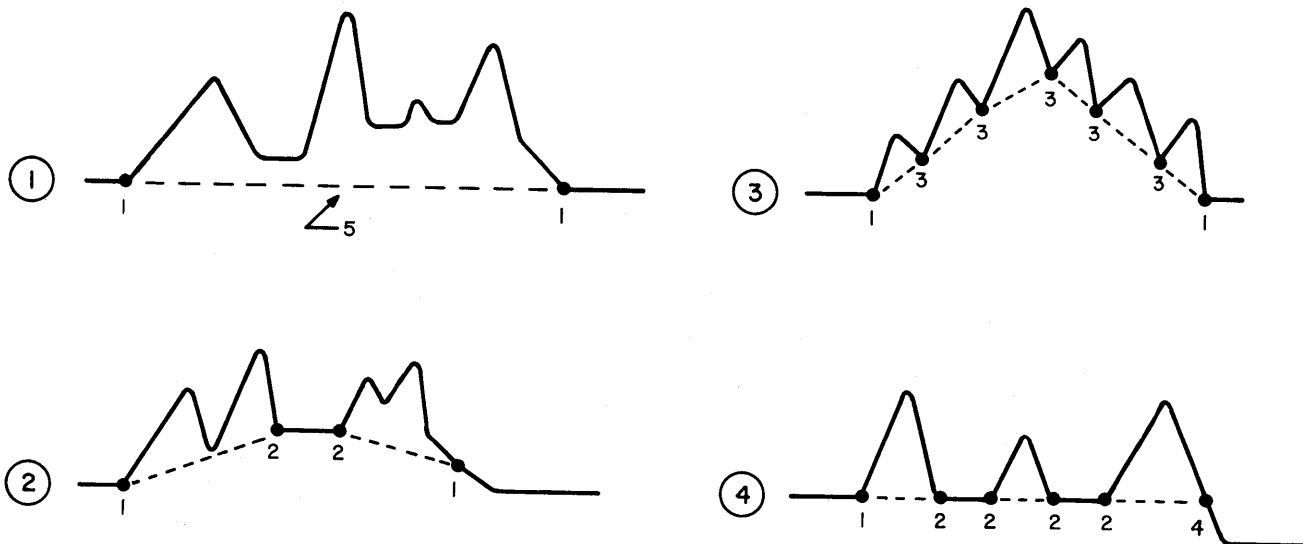
Numbers 1 through 5 correspond to the numbered steps in the discussion above.

------represents the corrected baseline established in step 5 above.

Figure D-4   Baseline Correction

D.2.2   Area Reallocation

Upon completion of baseline correction, the chromatogram is again processed for area reallocation. Area reallocation is the dividing of disputed peak areas when peaks are fused. Disputed area is always relinquished by a smaller peak to those adjacent peaks which are larger. The allocatable area of the smaller peak is that area which lies below a straight line between its start and end points. The fraction of this allocatable area to be relinquished to a larger neighbor is a function of the following.

a.   The area allocation factor (AF) specified in the method.

b.   The relative heights of the smaller and larger peaks (relative peak size).

c.   The relative heights and positions of the peak start and end points (a degree of fusion, amount of skew).

The function (described under c on the preceding page), is used to proportion the allocatable area between the competing requirements of the two larger peaks on either side of the smaller peak, and to apply a weight factor for the relative position of a smaller peak on the tail of a larger peak.

If the area reallocation factor is zero, the area reallocation procedure is equivalent to the technique of "dropping the perpendicular".

D.2.3   Peak Identification

After area reallocation, the chromatogram is compared with the method's component list to identify the individual peaks and to determine each peak weight or volume fraction for analysis calculations, or to update the component response factors in the method during calibration. A peak is identified with one of the listed components of a method, if it is the largest peak whose crest time after reference peak scaling (adjusted retention time) falls within a time tolerance zone of that component. If no reference peak was found, the peak crest time is used for this comparison. If a peak does not fall within the time tolerance zone of any component or is not the largest peak which falls within the time tolerance zone of any component, it is declared an unknown. The component time and tolerance zone are listed in the method.

Peak identification is calculated internally using the following algorithm.

$$\frac{ERT_r}{ART_r} \times ART_i = CRT_i$$

where

ERT_r = expected (adjusted) retention time of reference peak (compound table)

ART_r = actual retention time of reference peak (analysis report)

ART_i = actual retention time of interested peak (analysis report)

CRT_i = calculated retention time of interested peak

Or, using the first peak in the analysis of Section 3.8, Figure 10:

$$\frac{304}{310} \times 37 = 36$$

where the 36-second result (CRT_i) is well within the ± 10 second tolerance specified in the compound table. Therefore, our first peak was properly identified.

If the calculated retention time of an interested peak is outside the specified time tolerance, the peak will not be properly identified, instead, it will be reported as an unknown, as it might even when the actual retention time (ART_i) is within the specified tolerance. For example, if the reference

peak had eluted at 290 seconds (within its specified tolerance) and our first peak at 41 seconds (within its specified tolerance), consider the following:

$$\frac{304}{290} \times 41 = 43$$

where the 43-second result (CRT_i) is outside the specified tolerance. Should this happen, alter the compound table.

Therefore, even when an interested peak is reported as being within the specified time tolerance, the retention time of the reference peak determines whether that peak will be properly identified.

An additional convention applies such that if two peaks, the second being the larger, fall within the tolerance zones of two components, the second peak is assigned the first component and the first is declared an unknown.

D.2.4   Peak Analysis

Final analysis or calibration calculations are made using one of three analysis techniques: normalization, internal standard, or external standard. The technique to be used is specified in the method. On each identified peak, one of the following formulas applies:

<div align="center">

Analysis   Calibration

</div>

Analysis

Normalization

$$W_i = \frac{Ri \; Ai}{\sum Ri \; Ai} \times \frac{NORM}{100}$$

Internal Standard

$$W_i = \frac{Ri \; Ai}{R_{IS} \; A_{IS}} \times \frac{WT \; IS}{WT_{SAMP}} \times 100$$

External Standard

$$W_i = R_i \; A_i \times 10^{-SF} \times \frac{EXT \; SD}{10000}$$

Calibration

Normalization

$$R_i = \frac{CW_i}{A_i} \times \frac{A_{is}}{CW_{is}} \times \frac{100000}{NORM}$$

Internal Standard

$$R_i = \frac{CW_i}{A_i} \times \frac{A_{is}}{CW_{is}}$$

External Standard

$$R_i = \frac{CW_i \times 10^{-SF}}{A_i} \times \frac{10000}{EXT \; SD}$$

where

i = Peak number

IS = Internal standard peak

W_i = Weight or volume fraction in percent

R_i = Response factor of component matched to peak i

A_i = Peak i area

CW_i = Stated weight or weight percent of component, matched to peak i in the calibration standard sample

SF = A method-defined scale down factor

WR = Stated sample/internal standard weights; an internal standard for analysis run.

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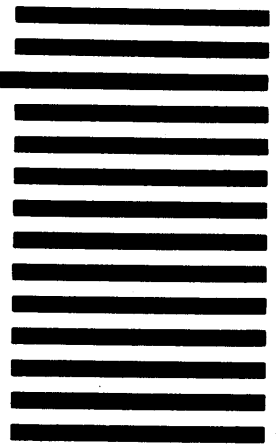
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