PREPARING A METHOD

PREP   METH   ENTER

RUN PARAMETERS
TIMETABLE EVENTS
CALIBRATION FILENAME
INTEGRATION PLOT TYPE
RUN DATA STORAGE
REPORT OPTIONS
POST–RUN LIST OPTIONS

Run Parameters

```
ZERO      [ 0    ]:
ATT 2^    [ 0    ]: 2
CHT SP    [ 1.0  ]:
AR REJ    [ 0            ]:
THRSH     [ 0    ]: 2
PK WD     [ 0.04 ]: .01
```

Timed Event Table

TIMETABLE EVENTS

DELETE CURRENT TABLE [Y/N*]:

TIMETABLE EVENTS

SELECT EVENTS FROM THE FOLLOWING MEN

[IF/EX/ZE/^Z/AT/CS/AR/TH/PW/ST]

TIME: .30

EVENT: PW

VALUE: .05

TIME: .8

EVENT: ST

VALUE:

TIME: .240

EVENT: EX

VALUE: 3

and so on

TIMETABLE EVENTS

IF is integrator function
EX is external event
ZE is plot zero
^Z is control-zero
AT is attenuation
CS is chart speed
AR is area reject
TH is threshold
PW is peak width
ST is stop

LISTING A METHOD

[LIST] [METH] [ENTER]

[LIST] [METH] *filespec* [ENTER]

EDITING A METHOD

[LOAD] [METH] ***filespec***

[EDIT] [METH]

1 = RUN PARAMETERS
2 = TIMETABLE EVENTS
3 = CALIBRATION FILE
4 = INTEGRATION PLOT TYPE
5 = RUN DATA STORAGE OPTIONS
6 = REPORT OPTIONS
7 = POST-RUN LIST OPTIONS
8 = REMOTE DEVICE ACCESS

SECTION TO BE EDITED:

STORING A METHOD

[STORE] [METH] *filespec*

DELETING A METHOD

[DEL] [METH] { *filespec* }

SEQUENCES

and

RUN AUTOMATION

SEQUENCE: A SET OF INSTRUCTIONS THAT DEFINE HOW THE HP3393A PERFORMS MORE THAN ONE AUTOMATED RUN.

PREPARING A SEQUENCE

PREP SEQ ENTER

*PREP SEQ

ALS INFORMATION

1. INET SAMPLER CONTROL [Y*N] :
2. SAMPLER PARAMETERS
   INJ/BOTTLE 1 -->
3. FIRST BOTTLE 1 -->
4. LAST BOTTLE 1 -->
5. PRE-WASH BOTTLE 0 -->
6. # OF PRE-WASHES 5 -->
7. # OF WASHES 5 -->
8. # OF PUMPS 5 -->
9. STROKE 1 -->
10. POST-WASH BOTTLE 0 -->
11. # OF POST-WASHS 5 -->
12. PRE/POST-WASH MODE 0 -->
13. POST-WASH DELAY 0 -->
14. POSITION 0 -->
15. VISCOSITY 0 -->

PREPARING A SEQUENCE

(CONT'D)

16. EQUILIBRATION TIME IN SECONDS [0]:
17. METHOD [*] :
18. SAMPLE INFORMATION TABLE
    BOTTLE OR RUN SAMPLE INDEXED [R/B*]:
19. FIRST BOTTLE [1]: or
    FIRST RUN [1]:
20. BOTTLE # : or RUN # :
21. ISTD AMT :
22. SAMPLE AMT:
23. MUL FACTOR:
24. RECALIBRATION [Y/N*] :
25. LEVEL:
26. NAME
27. REPORT MEMO
28. BOTTLE # : or RUN # :

USING A SEQUENCE

Starting a Sequence

[SEQ] [START]

Aborting a Sequence

[STOP]

LISTING A SEQUENCE

[LIST] [SEQ] *filespec* [ENTER]

```
*  LIST: SEQ

ALS INFORMATION
INET SAMPLER CONTROL ............ YES

EQUILIBRATION TIME IN SECONDS .... 0

METHOD .................... UNSPECIFIED

SAMPLE INFORMATION TABLE
BOTTLE OR RUN SAMPLE INDEXED .... B

19405A SAMPLER/EVENT CONTROL MODULE
 LOOP ADDRESS:  9

 SAMPLER PARAMETERS
  INJ/BOTTLE             1
  FIRST BOTTLE           1
  LAST BOTTLE            1
  PRE-WASH BOTTLE        0
  # OF PRE-WASHES        5
  # OF WASHES            5
  # OF PUMPS             5
  STROKE                 1
  POST-WASH BOTTLE       0
  # OF POST-WASHES       5
  PRE/POST-WASH MODE     0
  POST-WASH DELAY        0
  POSITION               0
  VISCOSITY              0
```

Listing of Default Sequence Parameters

EDITING A SEQUENCE

[EDIT] [SEQ] [ENTER]

1 = ALS INFORMATION
2 = EQUILIBRATION TIME DELAY
3 = METHOD FILE SPECIFICATION
4 = SAMPLE INFORMATION TABLE

SECTION TO BE EDITED:

STORING AND RETRIEVING A SEQUENCE

[STORE] [SEQ] ***filespec*** **[ENTER]**

[LOAD] [SEQ] ***filespec*** **[ENTER]**

.SEQ extension

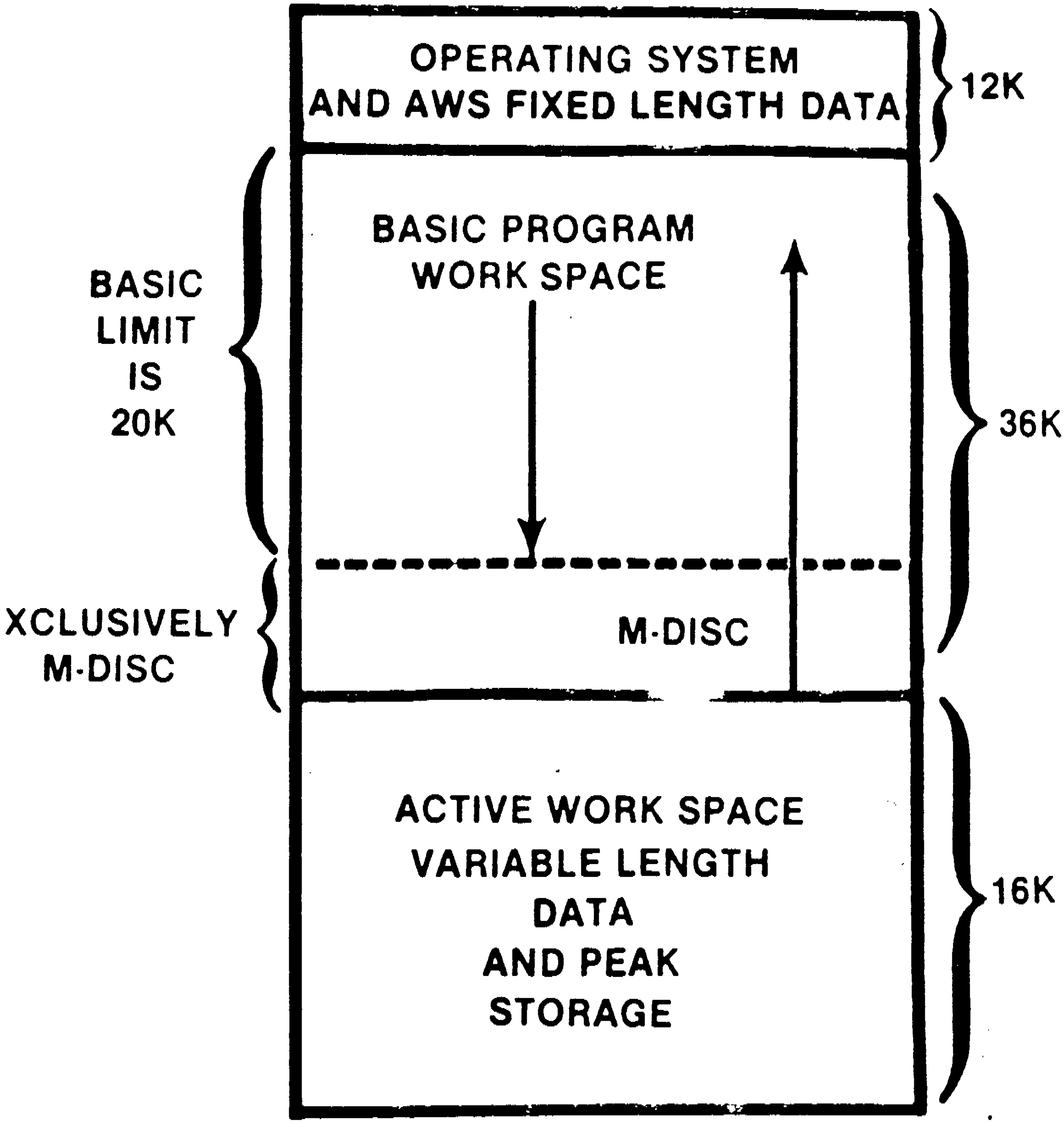
DELETING A SEQUENCE

[DEL] [SEQ] ***filespec*** **[ENTER]**

INTERNAL MEMORY

and

FILE OPERATIONS


OPERATING SYSTEM
AND AWS FIXED LENGTH DATA
12K
BASIC PROGRAM
WORK SPACE
BASIC
LIMIT
IS
20K
36K
XCLUSIVELY
M-DISC
M-DISC
ACTIVE WORK SPACE
VARIABLE LENGTH
DATA
AND PEAK
STORAGE
16K

THE ACTIVE WORKSPACE

FIXED-LENGTH PARAMETERS :

- o Run parameter values
- o Option responses
- o HP-IL configuration table

VARIABLE-LENGTH PARAMETERS :

- o Time Table
- o Calibration
- o Sequences
- o INET configuration table
- o Processed peak data from most recent run

INTERNAL MEMORY DISC

SIGNAL FILE STORAGE

DEVICE SPECIFIER IS **M:**

FILENAME IS **SIGNAL**

EXTENSION IS **.RAW**

.BNC

.BNA

SIG_BUFF DATA BUFFER

M : SIG_BUFF.RAW

FILE : A COLLECTION OF INFORMATION ORGANIZED FOR EASY STORAGE AND RETRIEVAL

FILE SPECIFIER or FILESPEC

completely designates a particular file

D : NAME . EXT

D = DRIVENAME

NAME = FILENAME

. EXT = EXTENSION

VALID FILE EXTENSIONS

.BAA	BASIC program file
.BAS	BASIC program file in memory image format
.DAT	BASIC data file
.MET	Method file
.SEQ	Sequence file
.CAL	Calibration file
.RAW	Unbunched signal data file
.PRO	Processed peak file from an analytical run
.PRA	Processed peak file from an ANALYZE command
.RPT	Report file from an analytical run
.RPA	Report file from an an ANALYZE command
.BNC	Bunched data file from an analytical run
.BNA	Bunched data file from an ANALYZE command

DIRECTORY SYSTEM COMMAND

DETERMINES

1. the file names, types, and length
2. the amount of space taken up by the files present
3. the amount of space left

*DIRECTORY drivename : filename.EXT

*DI AT START UP

```
*DI

VOLUME NAME: M3393        DRIVE: M
DATE: JAN  1, 1901  00:00:13

   FILE NAME         LENGTH   CREATED/VERSION
SIG_BUFF.RAW           2048 01/01/01 00:00:00

                 USED          FREE          MAX
FILES               1            21           22
BYTES            2048         33024        35584
```

CREATE SYSTEM COMMAND

ALLOCATES SPACE FOR A NEW FILE
FOR USE WITH BASIC

*CREATE filespec, size

(Space allocated in 256 byte increments)

COPY SYSTEM COMMAND

MAKES DUPLICATES OF A FILE

*COPY filespec, filespec

RENAME SYSTEM COMMAND

CHANGES THE NAME OF A FILE

*RENAME filespec, filespec

PURGE SYSTEM COMMAND

DELETES A FILE FROM MEMORY

*PURGE filespec

ASSIGN SYSTEM COMMAND

ASSIGNS A BASIC PROGRAM TO A NUMERIC KEY WHICH EXECUTES THE PROGRAM WHEN PRESSED

*ASSIGN keynumber, filespec

TO ASSIGN A BASIC PROGRAM TO A NUMERIC KEY

1. Enter HP3393A BASIC (BA)
2. Create the program.
   (10 PRINT "HELLO")
3. Save the program as a .BAS type file.
   (SAVE M:MYPROG.BAS)
4. Leave BASIC. (EX)
5. Delete the program from the BASIC work area.
   (KEEP PROGRAM IN WORKSPACE [Y*/N]: N)
6. Use the assign command to assign the program to a numeric key. (ASSIGN 1,M:MYPROG.BAS)
7. Press the numeric key to test the result. ([1])

```
*BA

(TYPE "H" FOR HELP)
>10 PRINT "HELLO"
>SAVE M:MYPROG.BAS

>EX

KEEP PROGRAM IN WORKSPACE [Y*/N]:N

EXIT BASIC


*ASSIGN 1,M:MYPROG.BAS

*  HELLO


*
```

FILE MANAGEMENT AND HOUSEKEEPING

PACK SYSTEM COMMAND

CONSOLIDATES STORAGE SPACE ON AN EXTERNAL MAGNETIC DISC DRIVE

*PACK discspec

FORMAT SYSTEM COMMAND

PREPARES A DISC MEDIUM FOR ACCEPTING DATA

*FORMAT discspec,volume,maxfile

volume = Name of Disc

maxfile = large number rounded up to nearest multiple of 16

SYSTEM SYSTEM COMMAND

PROVIDES A LIST OF DEVICES ON THE LOOP WITH THEIR ADDRESSES

*SYSTEM

A LOOP CONFIGURATION LISTING

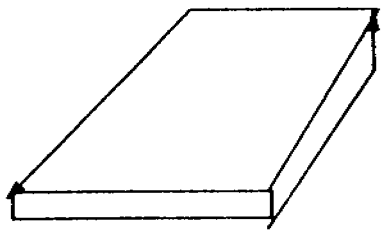
```
             LOOP CONFIGURATION
ADDRESS    DEVICE ID    ACCESSORY ID & CLASS
-------    ---------    --------------------
   8       19405A       70H   INET
   9       HP82169A     43H   Interface
  10       HP2225B      23H   Printer

  1-7      (Reserved for HPIB devices)

DISC NAME   ADDRESS   DRIVE #   VOLUME #
---------   -------   -------   --------
    A          3         0          0
    B          3         1          0


RS-232-C SWITCH SETTINGS
Baud                    4800
Timeout                 15 sec
Handshake Delay         Off
Hardware Handshake      Disabled
```

HP COMPATIBLE DISC DRIVES

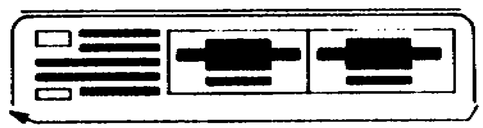
HP 9114B

BATTERY POWERED DISC

HP-IL DISC DRIVES

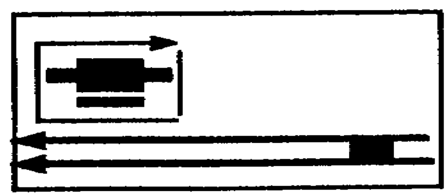


HP 82169A



HP 9122D

HP-IB DISC DRIVES



HP 9153A

10-MBYTE FIXED DISC

XADDRESS SYSTEM COMMAND

PROVIDES LOOP ADDRESS OF EXTERNAL PRINTER

*XADDRESS n

PRINTING TO EXTERNAL PRINTERS

CRTL

LIST

SEQ

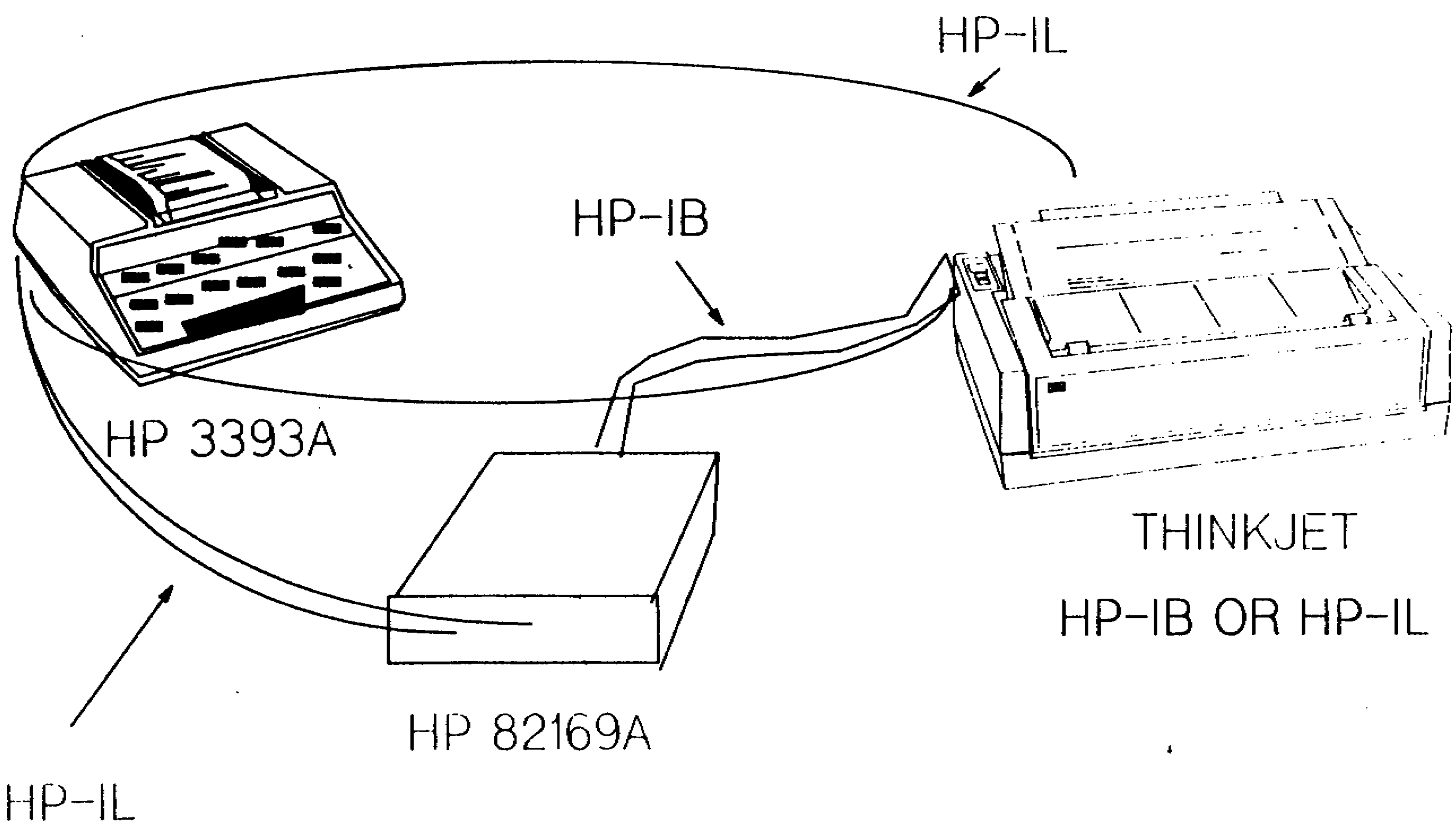
METH

CALIB

ENTER

HP 3393A PERIPHERALS

EXTERNAL PRINTER



READY SYSTEM COMMAND

CHECKS INET SYSTEM READINESS

*READY

SYSTEM IS READY

or

LOOP IS DOWN

BASIC PROGRAMMING on the HP3393A

GETTING INTO BASIC

BASIC ON THE HP3393A

*BA

>

EXTERNAL BASIC

*BX

EXITING BASIC

>EX

KEEP PROGRAM IN WORKSPACE [Y*/N] :

```
10 INPUT X
20 LET Y = 5*(4+X)
30 PRINT X,Y
40 STOP
RUN
```

Where

Statements	=	The content of lines numbered 10,20,30, and 40
Varialbes	=	X and Y
Keywords	=	INPUT,LET,PRINT,RUN STOP
Command	=	RUN
Expression	=	5*(4+X)
Math Operators	=	* and +
End statement	=	STOP; STOP or END Optional statements in HP3393A BASIC

HELP MESSAGES

(TYPE "H" FOR HELP)

MENU:

1 SIMPLE USER VARIABLES
2 USER ARRAY VARIABLES AND FUNCTIONS
3 USER STRING VARIABLES
4 USER LABELS
5 COMMANDS
6 COMMAND PARAMETERS
7 BREAK COMMANDS
8 KEYWORDS
9 SECONDARY KEYWORDS
10 BASIC FUNCTIONS
11 INTEGRATOR FUNCTIONS

ENTER SECTION NUMBER:

DELETE

DELETE or D prints line(s) and then erases

DQ (delete quitely) erases line(s) without printing

EXAMPLES:

DQ 10–70	delete lines 10 <u>to</u> 70
DELETE 10/70	delete and print lines 10 <u>to</u> 70
DQ 10,70	delete lines 10 <u>and</u> 70
DELETE 10,70	delete and print lines 10 <u>and</u> 70
DQ F/L	delete first <u>to</u> last lines in workspace

DEBUGGING COMMANDS

BREAK	—	Set a break point at a specified line number
DEBUG	—	Activate TRACE and BREAK statements
TRACE	—	Print line numbers as each line is about to be executed
STEP	—	Set a break point at each line number

> RESUME

THE BREAK STATEMENT

```
 10  REM:  PROGRAM TO SHOW USE OF BREAK
 20  INPUT X
 30  LET X = ISTDAMT
 40  PRINT "ISTDAMT =  ";X
 50  IF X=2 THEN
 60  BREAK
 70  PRINT "BYE"
 80  ENDIF
 90  IF X<>2 THEN
100  GOTO 20
110  ENDIF
>DEBUG
```

THE BREAK COMMAND

```
>BREAK 30
```

[illegible]

THE DEBUG COMMAND

```
10   TRACE ON
20   REM:  PROGRAM TO CHECK TRACE/BREAK
30   LET X = 1
40   INPUT X
50   LET X = ISTDAMT
60   PRINT "ISTDAMT = ";X
70   BREAK
80   PRINT "FINIS"
>DEBUG
>RUN
```

THE DEBUG STATEMENT

```
5   DEBUG ON
>RUN
```

0072 14

THE TRACE STATEMENT

```
5   TRACE ON
10  ! PROGRAM TO SHOW USE OF TRACE
15  LET X = 1
20  INPUT X
30  LET X = GROUP SUM
40  BREAK
50  PRINT "GROUP SUM = ";X
60  PRINT "DONE"
>DEBUG
>RUN
```

THE TRACE COMMAND

```
>TRACE 10/30
```

GC [illegible]

ARITHMETIC OPERATORS

^ or ** = exponentiate
* = multiply
/ = divide
+ = add
− = subtract

DIV

RESULT OF X1/X2 AS AN INTEGER

SYNTAX: X1 DIV X2

EXAMPLE: 8 DIV 6 returns 1

MOD

REMAINDER WHEN X1/X2

SYNTAX: X1 MOD X2

EXAMPLE: 8 MOD 6 returns 2

MODIFY

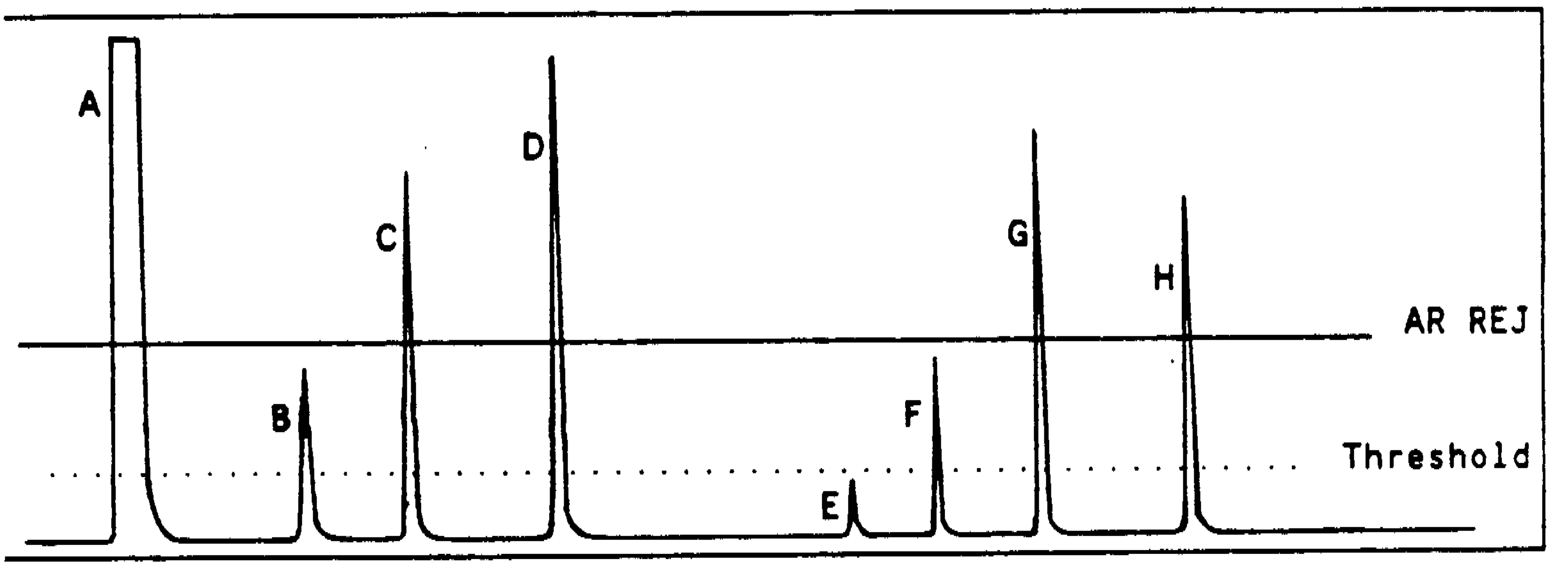
R = REPLACE

D = DELETE

I = INSERT

```
>M10
10  PRINT "DEXIMAL   BINARY    HEX"
              RC

10  PRINT "DECIMAL   BINARY    HEX"

(DELETED OLD LINE 10)
>
```

WORKING WITH PEAK DATA



eak	Chromatogram Number	Saved Peaks Number	AREA% or HEIGHT% & BASIC Peak Number	Calibration Repor Calibration Numbe
A	1	1	1	1
B	2	2	below AREA REJECT	
C	3	3	2	uncalibrated
D	4	4	3	2
E	5	below THRESHOLD		
F	6	5	below AREA REJECT	
G	7	6	4	3
H	8	7	5	uncalibrated

PEAK FUNCTIONS

PEAKNUM

```
140 PRINT PEAK NUM(3)
```

will give 4

NUMPEAKS

```
130 IF NUMPEAKS >20 THEN GOSUB 400
```

The NUMPEAKS value is 5

RT

```
100 LET X = RT(3)+8
```

AREA

```
100 IF AREA (2)<1000 THEN STOP
```

HEIGHT

```
100 IF HEIGHT(I)<123456 THEN GOTO400
```

WIDTH

```
100 IF WIDTH (X-2)>3 THEN GOTO 500
```

SEPCODE$

```
100 PRINT SEPCODE$(5)
```

The SEPCODE for 5 is BB

CHROMATOGRAPHIC DATA FUNCTIONS

MULT	110 PRINT MULT 250 LET X = MULT/100
CALNUM	50 PRINT CALNUM(3) 100 FOR I = 1 TO NUMPEAKS 110 LET C(I)= CALNUM(I) 120 NEXT I
CALAMT	30 PRINT CALAMT(3,1) 110 LET X = 1 + CALAMT(2,1)
CALRT	100 PRINT CALRT(4) 230 IF CALRT(4)<2 THEN 350
RF	80 PRINT RF(2) 120 IF RF(2)<1 THEN REPORT
AMT	100 FOR I = 1 TO NUMPEAKS 110 LET Q(I) = AMT(I) 120 NEXT I
ISTDAMT	250 PRINT ISTDAMT
SAMPAMT	10 PRINT SAMPAMT 50 IF SAMPAMT=0 THEN GOTO 70 60 STOP 70 PRINT "ISTD SAMPAMT =0"

[illegible]

FILE COMMANDS

JOIN: merges a specified file with the current program in the workspace.

SYNTAX: JOIN<d:filename.BAA

EXAMPLE: JOIN K:CH3CHOOH.BAA

LOAD	XLIST
PACK	XMARGIN
FORMAT	XADDRESS

FILE STATEMENTS

CHAIN: used to terminate execution of and clear the currently running program and execute a new specified program.

SYNTAX: CHAIN<d:filespecifier>

EXAMPLE: 200 CHAIN"K:NEWFILE.BAS"

EXTENSION: .BAS

GETCALIB: clears current calibration file from AWS and loads new one.

SYNTAX: GETCALIB "<d:file specifier>"

EXAMPLE: 400 GETCALIB "K:CH3COOH"

GETMETH

GETSEQ

WORKING WITH PEAK DATA

Section 11

PEAK DATA

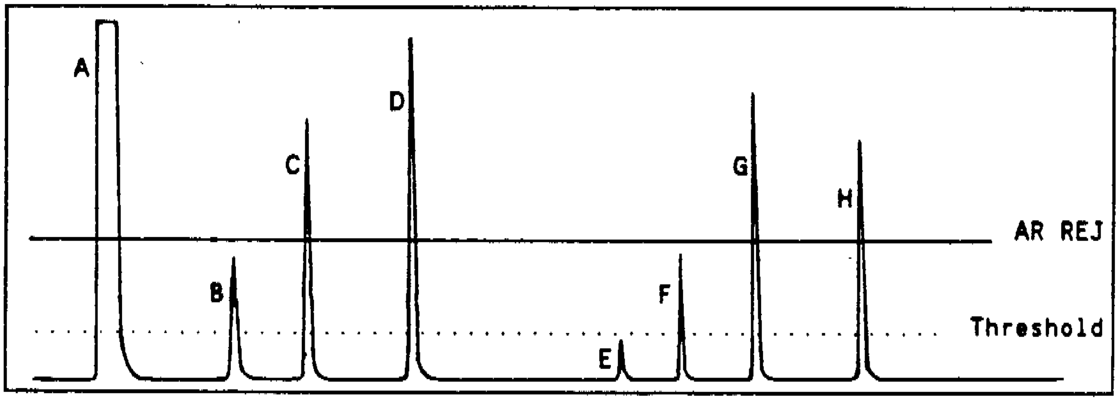
The peak data functions are used to access peak data from BASIC.

Peak Number

In HP3393A BASIC, the peak number refers to the peak that corresponds to a specified calibration number in the printed report (See Table I).

Peaks below threshold (THRSH) and below area reject (AR REJ) are automatically excluded from the HP3393A.

In the following sample chromatogram, all of the peaks are labeled with letters; the threshold level is indicated by the dashed line. Assume that the AREA REJECT has been set to represent an area which is approximately 50% of peak A; and a calibrated method has been selected in which peaks C,F, and H are not calibrated.



NOTE

The RESPONSE FACTOR for uncalibrated peaks is 0 (zero).

Using the above chromatogram, the following table demonstrates the peak numbers assigned to the different stages of data reduction.

TABLE I

Peak	Chromatogram Number	Saved Peaks Number	AREA% or HEIGHT% & BASIC Peak Number	Calibration Report Calibration Number
A	1	1	1	1
B	2	2	below AREA REJECT	
C	3	3	2	uncalibrated
D	4	4	3	2
E	5	below THRESHOLD		
F	6	5	below AREA REJECT	
G	7	6	4	3
H	8	7	5	uncalibrated

As shown in Table I, anything that alters the number of peaks included in the report can also change the peak number in the report.

- In the example shown, if the AREA REJECT value were lowered to zero, two more peaks (B and F) would be included in the AREA% BASIC column and one more peak (B) would be included in the calibrated report peak number column (F is not calibrated).

Channel Number

For the HP3393A integrator, the channel number is a number assigned to a file or an external device and subsequently used to address that file or device.

- The range for channel number selections is from 0 to 99. Channel 0 is defined as the device from which BASIC is being run; channel 0 is always open.

The OPEN# statement is used to assign channel numbers; when the channel number identifies a file, OPEN# will also open the file. The syntax for OPEN# is: OPEN# <channel number>:NAME<file specifier or device address>.

EXAMPLES:

`10 OPEN #20: NAME "B:CHEM.BAS"`	*Assigns channel #20 to file B:chem.bas*
`10 OPEN #12: NAME D$`	*Assigns channel #12 to file identified in D$*
`10 OPEN #12: NAME "8"`	*Assigns channel #12 to device at address 8*

For additional information concerning files, see Sections 1 and 14 of this manual; for information on device addresses, see Section 14.

PEAK FUNCTIONS

The following peak functions are used in HP3393A BASIC.

PEAKNUM This function returns the peak number which corresponds to the specified calibration number. The syntax is: PEAKNUM(calibration number).

EXAMPLE: Use the calibration numbers from Table 1.

```
140  PRINT PEAKNUM(3)
```

will give

```
4
```

NUMPEAKS This function tells you how many peaks have been included in the report (as shown in the Peak Number column of Table I); i.e., the number of peaks in a processed data file. (Reminder: The number of peaks included in the report is determined by the THRSH and AR REJ settings.)

The syntax is: NUMPEAKS.

EXAMPLES:

```
130   IF NUMPEAKS >20 THEN GOSUB 400
```

The NUMPEAKS value for Table I is 5.

RT This function returns the actual retention time (in minutes) of a specified peak. The syntax is: RT (peak number).

EXAMPLE:

```
100   LET X=RT(3)+8
```

AREA This function returns the baseline-corrected area (in 1/8 uV seconds) of a specified peak. The syntax is: AREA (peak number).

EXAMPLE:

```
100   IF AREA (2)<1000 THEN STOP
```

HEIGHT This function returns the baseline-corrected height (at the peak apex) of a specified peak in 1/8 uV. The syntax is: HEIGHT (peak number).

EXAMPLE:

```
100  IF HEIGHT(I)<12345678 THEN GOTO 400
```

WIDTH This function returns the baseline width of a specified peak in minutes. The syntax is: WIDTH (peak number).

EXAMPLE:

```
100   IF WIDTH (X-2)>3 THEN GOTO 500
```

**SEPCODE$** This function returns the peak type separation code (BB, SBB, etc.) for a specified peak in the report. The maximum permissible length for SEPCODE$ is 4 characters.

The syntax is: SEPCODE$(peak number)

EXAMPLE:

```
10  PRINT SEPCODE$(5)
```

NOTE

See section 5 of the HP3393A User's Manual for detailed information concerning peak types.

USING PEAK FUNCTIONS

Unless another calculation method is specified (i.e., height %, normalization, external standard, internal standard), the HP3393A will print an AREA PERCENT report.

Reformatting a Report

The following BASIC program could be used to provide a peak HEIGHT PERCENT analysis report in place of the standard AREA_PERCENT report.

EXAMPLE:

```
10  REM: HEIGHT% PROGRAM
20  LET S=0
30  FOR I = 1 TO NUMPEAKS
40  LET S=S+HEIGHT(I)
50  NEXT I
60  REM: S IS THE SUM OF THE PEAK HEIGHTS
70  FOR J=I TO NUMPEAKS
80  PRINT J,100*HEIGHT(J)/S
90  NEXT J
```

Where line

20 initializes the sum to 0 (zero)
30-60 is a loop used to obtain the sum of the peak heights
70-90 is a loop used to convert each peak height into height% and to print the peak number and the HEIGHT%

This program format can be used for any number of peaks.

Measuring Column Efficiency

The equation for a simple utility program that computes the efficiency of the columns being used is:

```
EFFICIENCY = 5.545*(retention time/width at baseline)^2
```

Where the result will be the number of theoretical plates provided by the column at the current temperature and carrier gas flow.

To measure efficiency, use a test sample which contains only the solvent and one other component. Because there will almost always be some impurities, be careful not to use the wrong peak for the calculation. If there is more than one peak besides the solvent, logically, the largest nonsolvent peak will be that of the component. The retention time of this peak should also be printed to insure positive identification.

EXAMPLE:

```
10  REM: COLUMN EFFICIENCY PROGRAM
20  REM: SCAN PEAK DATA;REJECT PEAKS < 0.5 MIN
30  REM: SAVE R (RT) AND W (WIDTH) FOR
40  REM: LARGEST REMAINING PEAK
50  LET R=0
60  LET W=0
70  LET A=0
80  FOR I=1 TO NUMPEAKS
90  IF RT(I) <0.5 THEN GOTO 140
100  IF AREA(I)<A THEN GOTO 140
110  LET A =AREA(I)
120  LET W=WIDTH(I)
130  LET R=RT(I)
140  NEXT I
150  LET E=5.545*(R/W)^2
160  PRINT R,E
```

Where line

80-140 If the peak retention time is less than 0.5 minutes, it is assumed to be the solvent peak and line 90 transfers to the end of the FOR-NEXT loop to try the next peak

100 Executes only if the retention time is greater than 0.5; the peak is tested to see if it is larger than the peak of area A, if not, then line 100 transfers to the end of the FOR-NEXT loop to try the next peak.

If the current peak is larger than peak A, the width and retention time of the current peak are stored as W and R, respectively. When the loop ends, W and R are the values for the largest nonsolvent peak found. The computation is performed and the results printed.

CHROMATOGRAPHIC FUNCTIONS

Section 12

Using the HP3393A Integrator, five calculation procedures are available for the conversion of peak data into report results. The calculation procedures are divided into uncalibrated and calibrated procedures and will generate the following reports:

- Uncalibrated: AREA% and HEIGHT% reports.
- Calibrated: Normalization (NORM), Internal Standard (ISTD), and external standard (ESTD) reports.

The chromatographic data functions associated with calibration, and the report statements, permit reports to be accessed from BASIC.

Detailed information on the calculation procedures and calibration files and reports may be found in Sections 8 and 9 of the HP3393A User's Manual.

Uncalibrated Procedures

Of the five calculation procedures available, the AREA% and HEIGHT% reports are not calibrated. Either an AREA% or a HEIGHT% report may be selected in response to the OP(4) Pk height mode question in command mode.

AREA% Report: The area percent calculation reports each peak area as a percentage of total peak area in a sample. AREA% is the HP3393A default procedure. The HP3393A will automatically compute and print an AREA% report when:

- Uncalibrated procedures are used and the OP(4) Pk height mode question is not selected.
- A specified calibration procedure is aborted due to an error.

HEIGHT% Report: The height percent calculation reports the height of each peak as a percentage of total peak height in a sample.

Calibrated Procedures

HP3393A calibrated procedures include: normalization (NORM), internal standard (ISTD), and external standard (ESTD).

For these three calibrated procedures, calibration tables are required to tell the HP3393A integrator how to convert areas or heights of peaks into the desired units of measurement. The following steps are necessary for active calibration:

- Analyze a control sample (calibration sample) for components of interest and produce an AREA% or HEIGHT% report (processed peak data).
- Prepare a calibration table and specify the amounts for each component of interest.

CHROMATOGRAPHIC DATA FUNCTIONS

The chromatographic data functions associated with calibration permit you to access information concerning calculation procedures and calibration tables via BASIC. This information can then be used to develop programs to produce specialized report information.

Level number refers to the identification (by number) of the calibration level (i.e., reference to the specific point) in a multi-level calibration table. A maximum of 63 levels may be used.

As already noted in Section 8:

- The peak number must be greater than zero and less than or equal to NUMPEAKS and will be rounded, if necessary, to the nearest integer.

The functions most frequently used to access calibration related information include:

MULT The MULTIPLICATION factor (MULT) may be applied to calibrated and uncalibrated results either to change the scale (as appropriate) or to correct for sample dilution or concentration.

The MULT function returns the value of MULTIPLIER, the constant factor by which all calculation procedures are multiplied. MULT is accessed using OP(7) or OP(3) in command mode unless it is overwritten by a sequence entry. The syntax is: MULT.

EXAMPLES:

```
110  PRINT MULT

250  LET X = MULT/100
```

CALNUM This function is used to access the calibration table for a speci: .i peak number. The syntax is: CALNUM(peak number).

EXAMPLES:

```
50   PRINT CALNUM(3)

100  FOR I=1 TO NUMPEAKS
110   LET C(I)=CALNUM(I)
120  NEXT I
```

If the specified peak is not a calibrated peak, the CALNUM function will return a value of zero.

CALAMT This function returns the value of the calibration amount used for a peak specified by calibration number and peak level within a calibration table. The syntax is:

CALAMT (calibration number*, level number)

The level number must be greater than zero and less than or equal to NUMLEV.

EXAMPLES:

```
30    PRINT CALAMT(3,1)

110   LET X=1+CALAMT(2,1)
```

* The calibration number refers to the CAL# shown in the previous report examples.

CALRT This function returns the value of the calibration table retention time for the specified peak. The syntax is: CALRT(calibration number).

EXAMPLES:

```
100  PRINT CALRT(4)

230  IF CALRT(4)<1.500 THEN GOSUB 350
```

RF This function gives the value of the response factor for a specified peak. The syntax is: RF(peak number).

EXAMPLES:

```
80   PRINT RF(2)

120  IF RF(2)<1 THEN REPORT
```

AMT This function returns the concentration (or amount) of a peak component as determined by the calculation procedure. The syntax is: AMT(peak number).

EXAMPLE:

```
100  FOR I=1 TO NUMPEAKS
110  LET Q(I)=AMT(I)
120  NEXT I
```

The value returned for the AMT function is the reported value (i.e., the result of the calculation procedure times the value of MULTIPLIER).

ISTDAMT This function returns the value of the internal standard amount (ISTD AMOUNT) for a sample. The syntax is: ISTDAMT.

When the ISTD calibration procedure is used, ISTDAMT is set from the OP(3) listing in system command. For sequences, ISTDAMT is set from the OP(7) listing in system command. When the ISTD calibration procedure is used, ISTDAMT is set from the OP(3) listing in system command. The ISTD value may be overwritten by a sequence entry.

EXAMPLES:

```
250  PRINT ISTDAMT
```

SAMPAMT This function returns the value of the sample amount used in internal standard calculations. SAMPAMT is set (for ISTD) using the OP (7) or the OP(3) listing in system command and may be overwritten by a sequence entry. The syntax is: SAMPAMT.

EXAMPLES:

```
10  PRINT SAMPAMT

50  IF SAMPAMT=0 THEN GOTO 70
60  STOP
70  PRINT "ISTD SAMPAMT=0"
```

**TITLE$** This function returns the title selected from the OP(4) Report Options Dialog (replace report title). The title may be up to 42 characters in length. The syntax is: TITLE$.

EXAMPLES:

```
100  PRINT TITLE$

110  IF TITLE$<>"ACETYLSALICYLIC ACID" THEN GOTO 300
```

**PROC$** This function returns the type of calculation procedure used in the current method. The following values are possible: normalization (NORM), external standard (ESTD), internal standard (ISTD), or area percent (AREA%). The default value is AREA%. The syntax is: PROC$.

EXAMPLES:

```
100  PRINT PROC$

110  IF PROC$="NORM" THEN GOSUB 300
```

**REPORT_FILE$** This function returns the name of the current report file in a string of up to 14 characters. The syntax is: REPORT_FILE$.

EXAMPLE:

```
10  PRINT REPORT_FILE$
```

**REPORT_MEMO$** This function returns the report memo from the current sample information table in a string of up to 126 characters. The default value is the OP(7) default sample information report. The syntax is: REPORT_MEMO$.

EXAMPLE:

```
10  REPORT_MEMO$
```

Additional functions associated with calibration in HP3393A BASIC are following. For further information concerning these functions, see the BASIC Reference Manual.

**PROCFILE$** This function returns the name of the current processed data file; The file name can be up to 14 characters in length. The syntax is: PROCFILE$

EXAMPLES:

```
200  PRINT PROCFILE$

300  IF PROCFILE$="SLUDGE.PRO" THEN GOSUB 600
```

CALRF This function returns the value of the response factor for a specified calibrated peak. The value returned is the ratio of the calibration amount to the peak area or height. The syntax is: CALRF (calibration number, level number). The level number must be greater than 0 (zero) and less than or equal to NUMLEV.

EXAMPLES:

```
50   PRINT CALRF(2,1)

100  AMT = CALRF(2,2)*AREA*.317
```

UNCALRF This function returns the value of the response factor for all uncalibrated peaks. The syntax is: UNCALRF.

EXAMPLES:

```
100  PRINT UNCALRF

130  IF UNCALRF=1 THEN GOSUB 400
```

**AMT_LBL$** This function returns the value of the sample amount label for the current sample as shown in the system command OP(4) dialog. The amount label may be up to 10 characters in length; the syntax is: AMT_LBL$.

EXAMPLES:

```
100  PRINT AMT_LBL$

120  IF AMT_LBL$<>"mg/mL" THEN GOSUB 600
```

CALFIT$ This function returns the type of curve fit selected for multi-level calibrations; curve fit is selected using the system command OP(4) dialog. The possible selections are:

S = Single point calibration
P = Point-to-point (multiple straight line segment fit)
L = Linear regression (least squares, straight line fit)
N = Nonlinear (quadratic fit)

The syntax is: CALFIT$

EXAMPLES:

```
200  PRINT CALFIT$

230  IF CALFIT$="N" THEN GOTO 260
```

CALTYPE$ This function returns the type of calibration peak. The possible return values are:

R = reference peak = internal standard peak = reference and internal standard peak = no
S entry for other
&

The syntax is: CALTYPE$(peak number)

EXAMPLES:

```
100  PRINT CALTYPE$(4)

120  IF CALTYPE(1)="R" THEN GOSUB 230
```

**GROUP_NAME$** This function returns the name assigned to a specified group of peaks from the GROUP PEAKS section of the calibration table; the GROUP_NAME$ may be up to 16 characters in length. The syntax is: GROUP_NAME$(group number).

EXAMPLE:

```
10  PRINT GROUP_NAME(2)
```

GROUP_SUM This function returns the value of the sum total of the calculated peak amounts from a specified group in the GROUP PEAKS section of the calibration table. The syntax is: GROUP_SUM(group number).

EXAMPLE:

```
10  SUM = GROUP_SUM(2)
```

ISTDNUM This function returns the calibration number of the internal standard. The syntax is: ISTDNUM.

EXAMPLE:

```
120  PRINT ISTDNUM
```

**NAME$** This function returns the name of a calibrated peak. The maximum length for the peak name is 16 characters. The syntax is: NAME$(calibration number).

EXAMPLES:

```
140  PRINT NAME$(6) 300  IF NAME$(5)="C2H5OH" THEN GOTO 400
```

**METHOD_NAME$** This function returns the file name for the most recently run method. A null string will be returned if the most recently run method is not from a file. The maximum length for the file name returned is 14 characters. The syntax is: METHOD_NAME$.

EXAMPLE:

```
350  PRINT METHOD_NAME$
```

NUMCALB This function returns the value of the maximum calibration number used in the current calibration table. The syntax is: NUMCALB.

EXAMPLES:

```
80   PRINT NUMCALB

130  FOR J= 1 TO NUMCALB
```

NUMGRPS This function returns the total number of peak groups in a sample. The syntax is: NUMGRPS.

EXAMPLES:

```
100  PRINT NUMGRPS

250  IF NUMGRPS<>2 THEN GOSUB 750
```

NUMLEV This function returns the number of calibration levels used to determine the calibrated peak. The syntax is: NUMLEV(calibration number).

EXAMPLES:

```
140  PRINT NUMLEV(5)

230  IF NUMLEV(5)=3 THEN STOP
```

NUMPEAKS This function returns the total number of peaks in the processed data file. The syntax is: NUMPEAKS.

EXAMPLES:

```
230  PRINT NUMPEAKS

500  IF NUMPEAKS>10 THEN REPORT
```

SAMPLES, AND FILES

Additional chromatographic functions are available in HP3393A to permit access to information concerning instrument identification, samples, and files from BASIC mode.

Samples

You can access sample information with SAMPNUM, INJ TIME$, SAMPNAME$, and RUNNUM. In addition, the EXT$ function may be used to access information concerning the current position of the external valve (ON or OFF).

SAMPNUM This function returns the number of the bottle from which the most recent injection has been made OR the number of the most recent sample run, depending upon the selected index (sample bottle or current table index) in the current sequence. If no samples have been run, this function will return the current RUNNUM. The syntax is: SAMPNUM.

```
EXAMPLES:

100  PRINT SAMPNUM

250  IF SAMPNUM>5 THEN GOTO 300
```

**SAMPNAME$** This function returns the name of the current sample. The sample name may be up to 12 characters in length. The syntax is: SAMPNAME$.

```
EXAMPLES:

100  PRINT SAMPNAME$

230  IF SAMPNAME$="CHC13" THEN REPORT
```

**INJTIME$** This function returns the date and time of the most recent sample injection in the format: MMM DD, YYYY HH:MM:SS The injection time and date may use up to 17 characters. The syntax is: INJTIME$.

```
EXAMPLES:

110  PRINT INJTIME$
```

Where the date and time will be returned in the format

```
JAN  14,1999 23:48:05
```

RUNNUM This function returns the current run number. The syntax is: RUNNUM.

EXAMPLE:

```
120  PRINT RUNNUM

230  IF RUNNUM=5 THEN REPORT
```

RUNNUM is used with SET to set the current run number. The syntax is: SET RUNNUM<run number>.

EXAMPLE:

```
100  SET RUNNUM 6
```

**EXT$** This function returns a string value indicating the current position (ON or OFF) of the external valve. The syntax is: EXT$(event number) To get the current position of the valve associated with the specified event number.

The valid range for event numbers is from 1 to 8.

EXAMPLE:

`100  PRINT EXT$(6)` *Will give the current position for the valve associated with event number 6; i.e., will give ON or OFF.*

SET EXT may be used to control the valve position (see SET in this Section).

Files

Current file names can be accessed using PROCFILE$, RUNFILE$, REPORTFILE$, and METHOD_NAME$.

**PROCFILE$** Returns the name of the current processed data file; the file name can be up to 14 characters in length. The syntax is: PROCFILE$

EXAMPLES:

```
200  PRINT PROCFILE$

210  IF PROCFILE4="SLUDGE.PRO" THEN GOSUB 600
```

**RUNFILE$** Returns the name of the file containing unprocessed area slice data for the current run. The file name may have a maximum length of 14 characters. The syntax is: RUNFILE$.

EXAMPLES:

```
500  PRINT RUNFILE$
520  IF RUNFILE$="CHEM.RAW" THEN
530  GOSUB 700
```

**REPORT_FILE$** Returns the name of the current report file in a string of up to 14 characters. The syntax is: REPORT_FILE$.

EXAMPLE:

```
10  PRINT REPORT_FILE$
```

**METHOD_NAME$** This function returns the file name for the most recently run method. A null string will be returned if the most recently run method is not from a file. The maximum length for the file name returned is 14 characters. The syntax is: METHOD_NAME$.

EXAMPLE:

```
400  PRINT METHOD_NAME$
```

Instrument Identification

**IDENTIFIER$** This function returns the identification of the analytical instrument hooked up to the HP3393A. The syntax is: IDENTIFIER$.

EXAMPLE:

```
100  PRINT IDENTIFIER$
```

The instrument identifier is set using the IDENTIFIER command in system command mode.

REPORTS

The AREA_PERCENT and the REPORT statements are used to access reports in BASIC.

The AREA_PERCENT Statement

The AREA_PERCENT statement is used to generate either an AREA% or a HEIGHT% report from BASIC mode. When a calibration procedure has been selected, the AREA_PERCENT statement will generate an AREA% or HEIGHT% report using the same data parameters specified in the current calibration procedures. The syntax for AREA_PERCENT is: AREA_PERCENT

EXAMPLES:

```
100  AREA_PERCENT

140  IF X<>3 THEN AREA_PERCENT
```

- If you select OP(4) in command mode, and respond Y to the Pk height mode question, the following entry in BASIC will provide a HEIGHT% report for the current chromatogram when the program is run:

EXAMPLE:

```
150  AREA_PERCENT
```

- If the response to the OP(4) Pk height mode question IS N, an AREA% report will be provided.

NOTE

When no entry is made in response to the OP(4) Pk height mode question, an AREA% report will automatically be provided.

The REPORT Statement

The REPORT statement can be used to generate both uncalibrated and calibrated reports in BASIC. The REPORT statement will print a copy of the report specified in the current method (AREA%, HEIGHT%, ISTD, ESTD, or NORM). AREA% is the type of report generated when no other report type is specified or when errors occur in calibration specifications and the response to the OP(4) Peak Height question is N. The syntax is: REPORT.

EXAMPLES:

```
130  REPORT
```
To get the current report generated by the specified calculation procedure in the current method.

```
200  IF Y=1 THEN REPORT
```

When the response to the OP(4) Pk height mode question is Y, and no calibration procedure is specified, or a fatal error occurs, a HEIGHT% report will be generated in response to the AREA_PERCENT and the REPORT statements.

EXAMPLES:
```
100  AREA_PERCENT

200  REPORT
```

If you had selected a calibration procedure, you would use:

`100  REPORT` *to get a copy of the calibration report.*

`200  AREA_PERCENT` *to get a copy of an AREA% report without changing the parameters.*

RUN PARAMETERS AND INET

Section 13

In Sections 9 and 11, calculation procedures and peak data and calibration functions were introduced and discussed. In keeping with the discussion of parameters and procedures affecting the chromatogram, this Section will review the remaining keywords available in HP3393A BASIC specifically developed for changing run parameters and accessing chromatographic data.

Using SET as a Command and as a Statement

SET permits the assignment of values to the run parameters from BASIC mode (when used as a command) and from within BASIC programs (when used as a statement). Values must be set before a run; run parameters CANNOT be changed from BASIC mode during a run. Time programming the run parameters is discussed in Section 7 of the HP3393A User's Manual.

When used without SET, the run parameters serve as functions and will return the current value of the specified parameter.

When assigning values to the run parameters, the assigned values (numeric expressions) may be enclosed in parentheses or used without parentheses.

EXAMPLES:

`SET ATT2 -8` *or* `SET ATT2(-8)`	*Shows SET used as a command to set the attenuation to -8.*
`210 SET THRSH 2` *or* `210 SET THRSH(2)`	*Shows SET used as a statement to set the threshold to 2.*
`210 PRINT AR_REJ`	*Shows the area reject parameter used without SET to return the current AR_REJ value.*

SET can also be used with the RUNNUM function to set the current run number (see RUNNUM).

RUN PARAMETERS

The ZERO, ATT2, and CHT_SP parameters affect plot presentation in plot mode during the real time run as well as the re-integration of stored signal data, while AR_REJ, THRSH, and PK_WD parameters alter the quantitation. See Section 5 of the HP3393A User's Manual for a complete discussion of the run parameters. All of these parameters may be set or revised from BASIC mode using the SET statement.

NOTE

If you set a value outside of the range established for each run parameter, the following error message will be printed:

```
OUT OF RANGE SET PARAMETER IN LINE ___
```

ZERO ZERO is the position on the HP3393A printer/plotter chart where the chromatographic baseline is plotted. The ZERO function returns the current value of the chart offset. The ZERO range is from -6 for the left margin to 100 for the right margin. The syntax is:

ZERO	*To get the current value for ZERO.*
SET ZERO<numeric expression>	*To set the value for ZERO.*

EXAMPLES:

`110 PRINT ZERO`	*Prints the current value of ZERO*
`120 IF THRSH>3 THEN SET ZERO 10`	*Sets ZERO to 10 if the condition is met (THRSH>3).*
`SET ZERO 5`	*Sets the ZERO value to 5 from BASIC mode (SET command).*

ATT2 The ATT2 function returns the value of the current attenuation setting. The range for the attenuation setting is from -8 to 36. The syntax is:

ATT2	*To get the current attenuation setting.*
SET ATT2<numeric expression>	*To set the value for ATT2.*

EXAMPLES:

`100 PRINT ATT2`	*Prints the current ATT2 setting.*
`200 SET ATT2(-3)`	*Sets the attenuation to -3 from within a BASIC program*
`SET ATT2 -3`	*(SET statement).*
`SET ATT2 3`	*sets the attenuation to 3 from BASIC mode*
`SET ATT2(3)`	*(SET command).*

CHT_SP Chart speed determines the rate at which the paper will advance when the HP3393A is plotting a signal. The paper advances in units of cm/min except when unigrams are selected. For unigrams, the paper advances in units of mm/peak. See the HP3393A User's Manual for additional information. The CHT_SP function returns the value of the current chart speed setting. Chart speed settings can range from 0.0 to 30.0. The syntax is:

CHT_SP	*To get the current chart speed setting.*
SET CHT_SP<numeric expression>	*To set the value for chart speed.*

EXAMPLES:

`210 PRINT CHT_SP`	*Prints the current chart speed setting.*
`230 SET CHT_SP 15`	*Sets the chart speed to 15 from within a BASIC program (SET statement).*
`SET CHT_SP 5.2`	*Sets the chart speed to 5.2 from BASIC mode (SET command).*

AR_REJ Area rejection is the area count value below which peaks will not be reported or stored in the processed peak file. The AR_REJ function returns the current value of the peak area rejection. The range for AR_REJ is from 0 to 2147483647 area counts. The syntax is:

AR_REJ	*To get the current area reject value.*
SET AR_REJ <numeric expression>	*To set the value for area rejection.*

EXAMPLES:

`300 PRINT AR_REJ`	*Prints the current area rejection value.*
`350 SET AR_REJ 7789`	*Sets the area rejection to 7789 from within a BASIC program (SET statement).*
`SET AR_REJ 45373`	*Sets the area rejection to 45373 from BASIC mode (SET command).*

PK_WD The PK_WD function returns the current value of the peak width. The range for peak width is from 0.01 to 2.50 min. The syntax is:

PK_WD *SET PK_WD<numeric expression>*	*To get the current peak width setting. To set the value for peak width.*

EXAMPLES:

`100 PRINT PK_WD`	*Prints the current peak width value.*
`200 SET PK_WD 1.22`	*Sets set the peak width to 1.22 from within a BASIC program (SET statement).*
`SET PK_WD 0.84`	*Sets the peak width to 0.84 from BASIC mode (SET command).*

THRSH Threshold is the number representing the minimum height that a peak must have to be recognized as a peak of interest by the HP3393A. The THRSH function returns the value of the current peak height threshold setting. THRSH values range from -6 to 28. The syntax is:

THRSH	*To get the current threshold setting.*
SET THRSH <numeric expression>	*To set the value for THRSH.*

EXAMPLES:

`130 PRINT THRSH`	*Prints the current threshold setting.*
`180 SET THRSH 20`	*Sets the threshold to 20 from within a BASIC program (SET statement).*
`SET THRSH -4`	*Sets the threshold to -4 from BASIC mode (SET command).*

LABORATORY

EXERCISES

LAB 5-----REPORT FORMATTING

OBJECTIVE
This lab shows ways in which reports are formatted and manipulated. You will learn how to:

*suppress the report
*replace report title and amount
*produce an extended report
*store the report
*have the report printed externally
*add extra parameters to the report

There are several ways to manipulate reports with the HP 3393A. Reports can be stored to disc, printed on external printers, and formatted by the user. Options 4 and 5 are the report options and they will be covered in this lab.

EXERCISE 5.1
ENTER THESE COMMANDS:

[LOAD] [METH] *A:REPORT* [ENTER]
AN A:REPORT.BNC [ENTER]

This is the way a regular report for a calibrated run with grouped peaks looks. Now try some formatting.

OPTION 4
Option 4 does actual formatting; it allows changes to be made to contents of a report.

[OP()] (4) [ENTER]

REPORT OPTIONS
Suppress local report [Y/N*]: [ENTER]
Peak height mode [Y/N*]: [ENTER]
Replace report title [Y/N*]: *Y* [ENTER]
Report title: (YOUR NAME)*'S REPORT* [ENTER]
Replace amount label [Y/N*]: *Y* [ENTER]
Amount label: *WEIGHT%* [ENTER]
Report uncalibrated peaks [Y/N*]: [ENTER]
Extended report [Y/N*]: [ENTER]

AN A:REPORT.BNC [ENTER]

Observe the report for this run. Notice differences between it and the regular report that you produced at the beginning of this lab. There is now a title below the "RUN #" field, and the amount column has WEIGHT% at the top. Here is another variation:

[OP()] (4) [ENTER]

REPORT OPTIONS
Suppress local report [Y/N*]: [ENTER]
Peak height mode [Y/N*]: [ENTER]
Replace report title [Y/N*]: [ENTER]
Replace amount label [Y/N*]: [ENTER]
Report uncalibrated peaks [Y/N*]: [ENTER]
Extended report [Y/N*]: *Y* [ENTER]

AN A:REPORT.BNC [ENTER]

Notice the difference the extended report makes, such as having peak names on the report. It is also important to note that title and amount header specified in the first run are still there. From now on, they will be there unless you specify for them to be replaced.

EXERCISE 5.2

OPTION 5
Now let's try doing some formatting with Option 5. Option 5 gives the choice of storing the report, sending it to an external device, or adding some additional parameters. If you have access to an external HP-IL printer, or an HP-IB printer with an HP-IL to HP-IB converter, try the option to send reports to it.

[OP()] (5) [ENTER]

POST RUN REPORT OPTIONS
Store post-run report [Y/N*]: *Y* [ENTER]
Device [M*]: [ENTER]
External post-run report [Y/N*]: [ENTER]
List run parameters [Y/N*]: *Y* [ENTER]
List time table [Y/N*]: *Y* [ENTER]
List calibration table [Y/N*]: *Y* [ENTER]
List remote method [Y/N*]: *Y* [ENTER]

AN *A:REPORT.BNC* [ENTER]
DIR [ENTER]

Notice the extra parameters at the end of the report. If you did not get any parameters for "remote method" it is because you have no INET instruments on the loop. If you did, their setpoints would be listed via that option. Also, at the end of the run, the integrator paused to put the report into the file SIGNAL.RPA on the M drive. .RPA is the extension for reports.

Delete the report file from memory:

PU *REPORT.RPA* [ENTER]

REVIEW QUESTIONS

1. How many runs would contain the report title and amount label if you specified them in Option 4?

2. What extra values does the extended report include?

3. Where can the post-run report be sent?

4. What is the extension for a report?

E: (1) [ENTER]
NT: *ST* [ENTER]

E: (3) [ENTER]
NT: *IF* [ENTER]
UE: (-3) [ENTER]

E: (3) [ENTER]
NT: *^Z* [ENTER] (^ is shift + 6)

: [ENTER]

time table events. Abbreviations stand for integrator function, , control z, attenuation, chart speed, area reject, threshold, peak The new function, control z, is used only in unigram mode to aseline, and it only affects plot presentation.

ACE CURRENT CALIBRATION [Y/N*]: [ENTER]

GRATION PLOT TYPE

R PLOT TYPE [S/F*/U/N]: [ENTER]

DATA STORAGE
signal data [Y/N*]: [ENTER]
processed peaks [Y/N*]: [ENTER]

RT OPTIONS
ss local report [Y/N*]: [ENTER]
ieight mode [Y/N*]: [ENTER]
e report title [Y/N*]: [ENTER]
e amount label [Y/N*]: [ENTER]
uncalibrated peaks [Y/N*]: [ENTER]
led report [Y*/N]: [ENTER]

RUN REPORT OPTIONS
ost-run report [Y*/N]: [ENTER]
[M*]: [ENTER]
al post-run report [Y/N*]: [ENTER]
n parameters [Y*/N]: [ENTER]
ne table [Y*/N]: [ENTER]
libration table [Y*/N]: [ENTER]
mote method [Y*/N]: [ENTER]

E] [METH] *TESTMETH* [ENTER]
[ENTER]

d extension is
ace, press:

at time you can
O BE EDITED:"
ou would like to

ising the dialog.
ving keystrokes.

REVIEW QUESTIONS

1. What key sequence starts dialog to create a method?

2. What is the extension for a method?

3. What key starts the key sequence for inputting a method into active workspace?

LAB 7-----SEQUENCES

OBJECTIVE

This lab explains the use of sequences, with or without an automatic sampler. It shows how to set up, edit, store, retrieve and start sequences. After completing this lab you should be able to:

*set up a sequence, either for manual injections or for an automatic sampler
*edit a sequence
*store and retrieve a sequence
*start a series of runs controlled by a sequence

The PREP SEQUENCE dialog contains many variations for controlling a sequence. Sequences can be created for INET samplers, for samplers which supply bottle numbers, or for controlling a series of runs. The method and an optional equilibration time can be specified. In the next three exercises, we'll create an example of each possible sequence.

EXERCISE 7.1

The following dialog sets up parameters for a sequence using an INET sampler. **If there is no S/ECM or HP 7673A Microdrop automatic sampler on the loop, you will not be able to do this exercise. Read through this section and go on to Exercise 7.2.**

ENTER THESE COMMANDS:

[PREP] [SEQ] (no enter)
ALS INFORMATION
INET SAMPLER CONTROL [Y*/N]: [ENTER]

19405A SAMPLER/EVENT CONTROL MODULE
LOOP ADDRESS: 9

SAMPLER PARAMETERS
INJ/BOTTLE 1 --> [ENTER]
FIRST BOTTLE 1 --> [ENTER]
LAST BOTTLE 1 --> [ENTER]
PRE-WASH BOTTLE 0 --> [ENTER]
 # OF PRE-WASHES 5 --> [ENTER]
 # OF WASHES 5 --> [ENTER]
 # OF PUMPS 5 --> [ENTER]
STROKE 1 --> [ENTER]
POST-WASH BOTTLE 0 --> [ENTER]
 # OF POST-WASHES 5 --> [ENTER]
PRE/POST-WASH MODE 0 --> [ENTER]
POST-WASH DELAY 0 --> [ENTER]
POSITION 0 --> [ENTER]
VISCOSITY 0 --> [ENTER]

(If you are using an HP 7673A, the dialog above will be slightly different.)

EQUILIBRATION TIME IN SECOND [0]: [ENTER]

METHOD [M:TESTMETH.MET]: [ENTER]

SAMPLE INFORMATION TABLE
BOTTLE OR RUN SAMPLE INDEXED [R/B*]: [ENTER]
BOTTLE # : *1* [ENTER]
ISTD AMT: [ENTER]
SAMPLE AMT: [ENTER]
MUL FACTOR: [ENTER]
RECALIBRATION [Y/N*]: [ENTER]
NAME: [ENTER]
REPORT MEMO: [ENTER]

BOTTLE # : [ENTER]

Notice that in the sample table a multiplication factor, name, and report memo can be assigned to each individual sample. As an extra note, the choice of using bottle or run indexing is available, but it is not practical to use run indexing with an INET sampler since you already have control over bottle numbers.

EXERCISE 7.2

In this exercise we will create a sequence which could be used with a non-INET instrument which provides bottle number inputs.

ENTER THESE COMMANDS:

[DEL] [SEQ] [ENTER]
[PREP] [SEQ]

ALS INFORMATION
INET SAMPLER CONTROL [Y*/N]: *N* [ENTER]

EQUILIBRATION TIME IN SECONDS [0]: [ENTER]

METHOD [M:TESTMETH.MET]: [ENTER]

SAMPLE INFORMATION TABLE
BOTTLE OR RUN SAMPLE INDEXED [R/B*]: [ENTER]
FIRST BOTTLE [1]: [ENTER]
LAST BOTTLE [1]: [ENTER]

BOTTLE # : *1* [ENTER]

ISTD AMT: [ENTER]
SAMPLE AMT: [ENTER]
MUL FACTOR: [ENTER]
RECALIBRATION [Y/N*]: [ENTER]
NAME: [ENTER]
REPORT MEMO: [ENTER]

BOTTLE # : [ENTER]

After this sequence is created, the sampler moves to the bottle specified in "FIRST BOTTLE", then bottle numbers within the specified FIRST BOTTLE/LAST BOTTLE range activate the sample table parameters. Bottle numbers can be entered into the sample table which do not fall into the first bottle/last bottle range, so that later on, the range can be changed to run those samples. You are free to create as much of the sample table as you want, then run parts or all of the table at any time.

EXERCISE 7.3

This variation sets up a sequence based on run numbers, which can be used for an automatic sampler with no bottle number output, or for valved injections. If the run number is found in the sample table, sample table parameters will be used in that run.

ENTER THESE COMMANDS:

[DEL] [SEQ] [ENTER]
[PREP] [SEQ] (no enter)

ALS INFORMATION
INET SAMPLER CONTROL [Y*/N]: *N* [ENTER]

EQUILIBRATION TIME IN SECONDS [0]: [ENTER]

METHOD [M:TESTMETH.MET]: [ENTER]

SAMPLE INFORMATION TABLE
BOTTLE OR RUN INDEXED [R/B*]: *R* [ENTER]
FIRST RUN [1]: [ENTER]
LAST RUN [1]: [ENTER]

RUN #: *1* [ENTER]
ISTD AMT: [ENTER]
SAMPLE AMT: [ENTER]
MUL FACTOR: [ENTER]
RECALIBRATION [Y/N*]: [ENTER]
NAME: [ENTER]

REPORT MEMO: [ENTER]

RUN #: [ENTER]

Run indexing works the same as bottle indexing, except that the HP 3393A is looking for the run number. The run number can be set using the run number command shown in Lab 1.

STORING AND LOADING

Storing and loading sequences works the same way as methods and calibration tables. Just use the STORE or LOAD key, followed by the SEQ key, along with a disc specifier (if necessary), and the filename. If a calibration table exists when a method is stored, it will be stored also. By being able to name them in a different file, it is possible to use the method as a template and "overlay" other calibration tables or sequences.

RUNNING A SEQUENCE

To let the integrator know you will be using the sample table (sequence) instead of doing single runs, the method of starting the first run is different. Instead of pressing [START], press [SEQ] [START] and the sequence will begin, using sample table values for the bottle or run numbers it finds. As in the HP 3392A, if there is no value in the sample table, the integrator looks in Option 7. If there is no value there, it looks in the calibration table. Each successive run starts automatically after the appropriate equilibrium time. Thus, using a sequence for manual runs is not feasible since you don't really know exactly when to inject.

SEQUENCE/METHOD CHAINING

To switch to another method during a sequence requires chaining sequences, since the method is specified within the sequence. Sequence chaining is possible through BASIC programming.

REVIEW QUESTIONS

1. What are the three types of sequencing available on the HP 3393A?

2. What parameters can be specified in the sample table?

3. What happens if a bottle or run number is placed in the sample table which is not in the first/last bottle range?

LAB 10-----BASIC

OBJECTIVE
To demonstrate use and features of the HP 3393A BASIC, and to show a few examples of possible applications. After completing this lab you should be able to:

*access and exit BASIC mode
*enter, edit, and run a BASIC program
*do some simple debugging
*stop a program during execution

BASIC is a very simple, easy to learn language; the chromatographic keywords and access to different parts of the instrument network make it a powerful tool that should be utilized. Exercise 1 helps you get started.

EXERCISE 10.1
First, to get into BASIC mode, type:

BA [ENTER]

After messages, the prompt ">" appears, and the HP 3393A is waiting for you to enter a BASIC program. Start by typing this program:

```
10 REM A REM STATEMENT IS FOR REMARKS
20 REM BEGINNER'S PROGRAM
30 FOR I=1 TO 10
40 PRINT "MY NAME IS (YOUR NAME)"
50 NEXT I
60 END
```

LIST [ENTER]
RUN [ENTER]

The program should have printed "MY NAME IS ________" 10 times.

EXERCISE 10.2
To show you the editing capabilities, try replacing the name in line 40. Enter the following:

M {40} [ENTER]
40 PRINT "MY NAME IS VIVIAN"

The integrator prints out the line specified and is now in modify mode. There are three commands in this mode: insert, delete, and replace.

To use one of these commands, **space the left margin of the printhead over to the first character to be modified**, type the first letter of the command, and proceed according to what the command designates. Try deleting and inserting:

SPACE OVER 22 SPACES TO LOCATE THE LEFT MARGIN OF THE PRINTHEAD UNDER THE FIRST LETTER OF YOUR NAME.

DELETE YOUR NAME BY TYPING A "D" UNDER EACH CHARACTER OF YOUR NAME:

```
40 PRINT "MY NAME IS VIVIAN"
                      DDDDDD  [ENTER]
40 PRINT "MY NAME IS "
```

You just deleted your name using the delete command, so line 40 should be printed without your name. Every time you press ENTER the new line is displayed; if no editing was done, you will return to normal BASIC mode. Now insert your name back into the statement.

SPACE OVER 22 SPACES, PLACE AN "I" UNDERNEATH THE QUOTATION MARK, AND INSERT YOUR NAME:

```
40 PRINT "MY NAME IS "
                      IVIVIAN  [ENTER]
40 PRINT "MY NAME IS VIVIAN"
```

NOW TRY A REPLACE; TYPE AN "R" UNDER THE FIRST LETTER OF YOUR NAME FOLLOWED BY A NAME:

```
40 PRINT "MY NAME IS VIVIAN"
                      RVALERIE  [ENTER]
40 PRINT "MY NAME IS VALERIE"   [ENTER]

40 PRINT "MY NAME IS VALERIE"
[ENTER]

L  [ENTER]
```

The replace function starts at the character indicated by "R" and overwrites characters, as compared to the insert command which inserts and moves everything else over. You should have returned to BASIC mode after pressing return and obtained a list of the program.

Another more common method used to edit lines is to type the whole line over again, starting with the line number. This succeeds in replacing the whole line.

Now that you're more familiar with BASIC, we'll try some programming with chromatographic data.

EXERCISE 10.2

To have data with which to work , exit BASIC and enter the following commands:

EX [ENTER]

DELETE PROGRAM IN ORDER TO FREE STORAGE
[Y/N*]: [ENTER]
[LOAD] [METH] *A:BASIC* [ENTER]
AN *A:SAMPLE.BNC* [ENTER]
BA [ENTER]

Now that you're back in BASIC mode, enter this program using autonumbering mode. This program will produce a mini-report.

AUTO [ENTER]

FIRST NEW LINE NUMBER: {10} [ENTER]

STEP: {10} [ENTER]

(USE BREAK OR CONTROL-Y TO END)

```
10 REM PROGRAM TO FORMAT A REPORT
20 PRINT "NAME","               AMOUNT" (15 spaces)
30 FOR I=1 TO NUMCALB
40 PRINT NAME$(I),AMT(PEAKNUM(I))
50 NEXT I
60 END
```

[BREAK] [ENTER]

RUN [ENTER]

The result should look something like this:

STARTING JAN 1, 1901 02:01:05

NAME	AMOUNT
METHYL UNDECANOA	224.162
NICOTINE	224.352
HEPTANOIC ACID	441.542

Now try modifying the program to enhance the report.

```
11 PRINT "RUN:  ";RUNNUM, "METHOD:  ";METHOD_NAME$
12 PRINT
13 PRINT

RENU  [ENTER]

FIRST LINE OF SECTION TO RENUMBER:    F  [ENTER]

LAST LINE OF SECTION TO RENUMBER:    L  [ENTER]

NEW FIRST LINE NUMBER FOR
RENUMBERED SECTION:   {10}  [ENTER]

NEW STEP:   {10}  [ENTER]

DONE

L  [ENTER]
10 REM PROGRAM TO FORMAT A REPORT
20 PRINT "RUN:  ";RUNNUM,"METHOD:  ";METHOD
_NAME$
30  PRINT
40  PRINT
50  PRINT "NAME","          AMOUNT"
60  FOR I=1 TO NUMCALB
70   PRINT NAME$(I),AMT(PEAKNUM(I))
80  NEXT I
90  END

RUN  [ENTER]

STARTING JAN 1, 1901  02:26:25

RUN:  1          METHOD:  A:BASIC.MET


NAME                        AMOUNT
METHYL UNDECANOA            224.162
NICOTINE                    224.352
HEPTANOIC ACID              441.542
```

This was a simple example of report formatting. The user has access to most of the variables involved in calculations, so most any kind of report can be generated.

Now try some simple debugging features; although this program has no errors, the procedure can be demonstrated.

EXERCISE 10.3

Tracing a program means to print each line number as it is executed. This is a good way to determine exactly where an error occurs. Here is one way to activate trace mode:

1 BREAK [ENTER]
DEBUG [ENTER]
RUN [ENTER]

STARTING JAN 1, 1901 03:59:03

BREAK AT LINE 1
ENTER BREAK COMMAND: *TRACE F/L* [ENTER]

ENTER BREAK COMMAND: *RESUME* [ENTER]

What you did was to place a break point in the program and then trace from the first line to the last. (You can also stop a program manually by pressing the break key.) After the resume command, the program should resume execution, printing each line number as it is executed. The debug command before the run command activated this mode. To turn off trace, either enter "trace off" at the "enter break command" prompt, or modify the program.

RUN [ENTER]

STARTING JAN 1, 1901 04:09:25

(*TRACE LINE 1*)

BREAK AT LINE 1
ENTER BREAK COMMAND: *TRACE OFF* [ENTER]

ENTER BREAK COMMAND: *A* [ENTER]

The last thing is to save the program.

EXERCISE 10.4
ENTER THESE COMMANDS:

SAVE A:PROGRAM.BAS [ENTER]
DIR A: [ENTER]

Your program is now on disc. Exit BASIC mode and clear the disc of the two files you created.

EX [ENTER]
PU A:CALIB1.CAL [ENTER]
PU A:PROGRAM.BAS [ENTER]

REVIEW QUESTIONS

1. What command switches the integrator to BASIC mode?

   System mode?

2. What command allows you to edit a particular line?

3. How do you execute a program in HP 3393A BASIC?

4. What does the autonumber command do? The renumber command?

5. How can you get a printout of your program?

6. What does a break statement do? A trace command?

7. What command stores a program?

5890/3393 OPERATOR TRAINING
COURSE PROGRAM

DAY 1	Walkaround the 5890	
	Injection Systems	
	Oven Temperature Control	
	Laboratory Exercise 1:	Installation, Turn on
	Detectors	
	Keyboard Commands	
	Laboratory Exercise 2:	Instrument Checkout
		Temperature Programming
DAY 2	Caplliary Systems	
	Autosampler, Valving	
	Laboratory Exercise 3:	Capillary Operation
		Demonstartions of Autosampler and Valving Operations
DAY 2 PM	Introduction to the HP3393 Intergrator	
	Installation and Start-Up	
	Getting Familiar with the Keyboard	
	Using the Plot Mode	
	Laboratory Exercise 1:	Familiarize

5890/3393 OPERATOR TRAINING
COURSE PROGRAM

DAY 3	Integration and Reintegration
	Laboratory Exercise 2: Integration & Reintegration
	Timed Events
	Laboratory Exercise 3: Timed Events
	Calculations, Reports, and Calibration
	Laboratory Exercise 4: Calibration
DAY 4	Report Options
	Methods
	Sequences and Run Automation
	Laboratory Exercise 5: Report Options, Methods, Sequences
	Internal Memory and File Operations
	Laboratory Exercise 6: File Operations
	Basic Programming
	Laboratory Exercise 7: Basic Programming

DATE: ______________

COURSE EVALUATION

In order that we may continue to evaluate, update, and improve our courses, we would appreciate it if you would take a few minutes to fill out the following evaluation. Please complete both sides of the form.

COURSE TITLE: ____________________________ INSTRUCTOR: ____________________________

NAME: ____________________________ COMPANY: ____________________________

Please check one for each question.

1. Information received in course will be:

   __________a) a great help in my job

   __________b) of some help in my job

   __________c) of no use in my job

   Comments ____

   ____

2. Technical level of course for me was:

   __________a) about right

   __________b) too technical

   __________c) too elementary

   Comments ____

   ____

3. Course length for me was:

   __________a) about right

   __________b) too long (could have covered more material)

   __________c) too short (tried to cover too much material)

   Comments ____

   ____

Using the following definitions, please check the letter you feel is appropriate to evaluate the following questions 4 through 9.

a) Excellent: outstanding

b) Very Good: consistently above satisfactory

c) Good: satisfactory

d) Adequate: needs additional development to become consistently satisfactory

e) Inadequate: consistently below minimum acceptance level

4. Course materials (manuals, overhead slides, etc) were:

   a)________ b)________ c)________ d)________ e)________

   Comments ____

   ____

5. Instructor's presentation was:

   a)________ b)________ c)________ d)________ e)________

   Comments ____

   ____

6. Instructor's demonstrated knowledge of the subject was:

a)_______ b)_______ c)_______ d)_______ e)_______

Comments ____

7. Lab sessions were:.

a)_______ b)_______ c)_______ d)_______ e)_______

Comments ____

8. The training center's facilities were:

a)_______ b)_______ c)_______ d)_______ e)_______

Comments ____

9. Overall, the course was:

a)_______ b)_______ c)_______ d)_______ e)_______

Comments ____

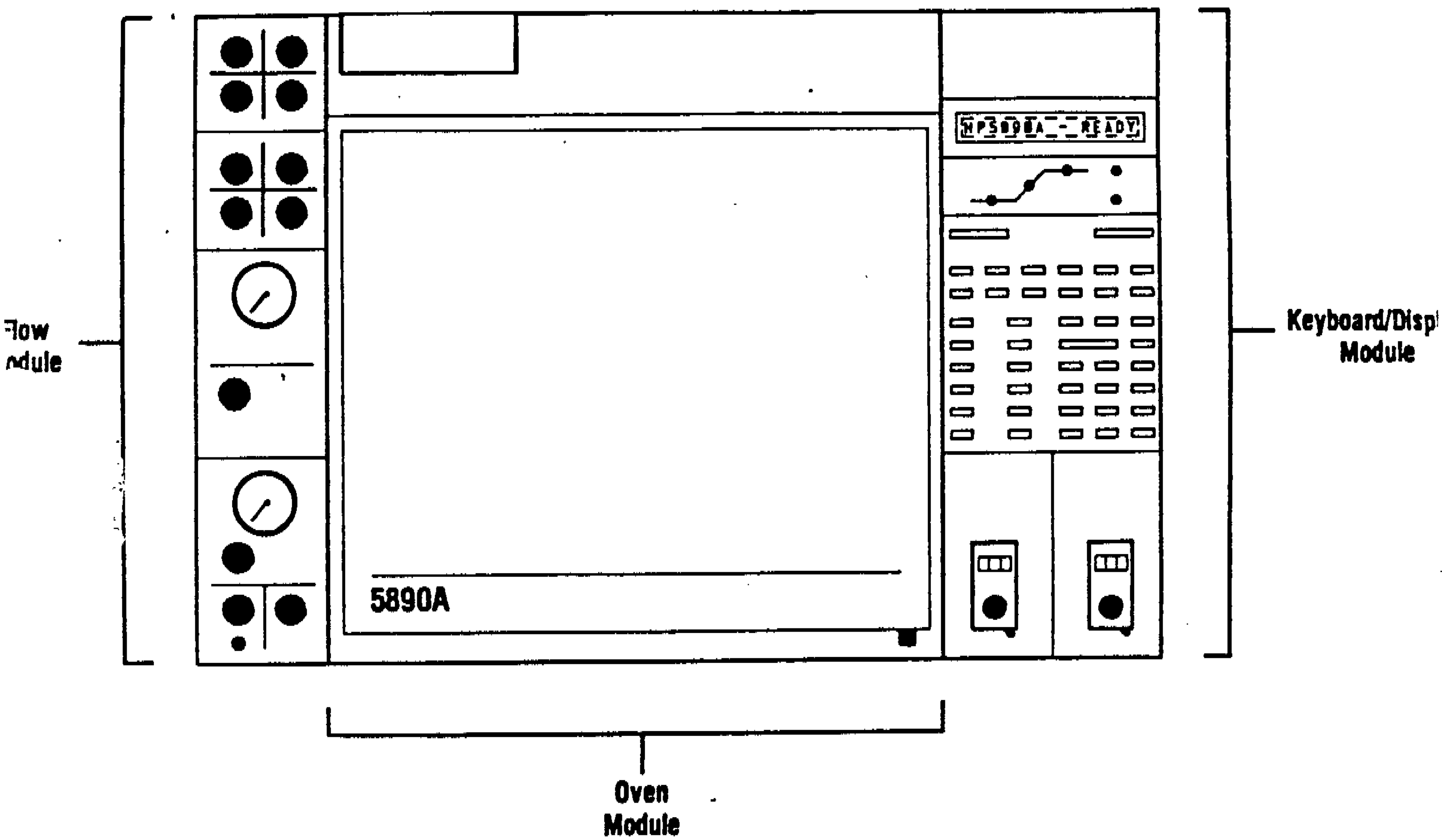
10. What did you like most about the course? ____________________

11. What did you like least about the course? ____________________

12 How could the course be improved? ____________________

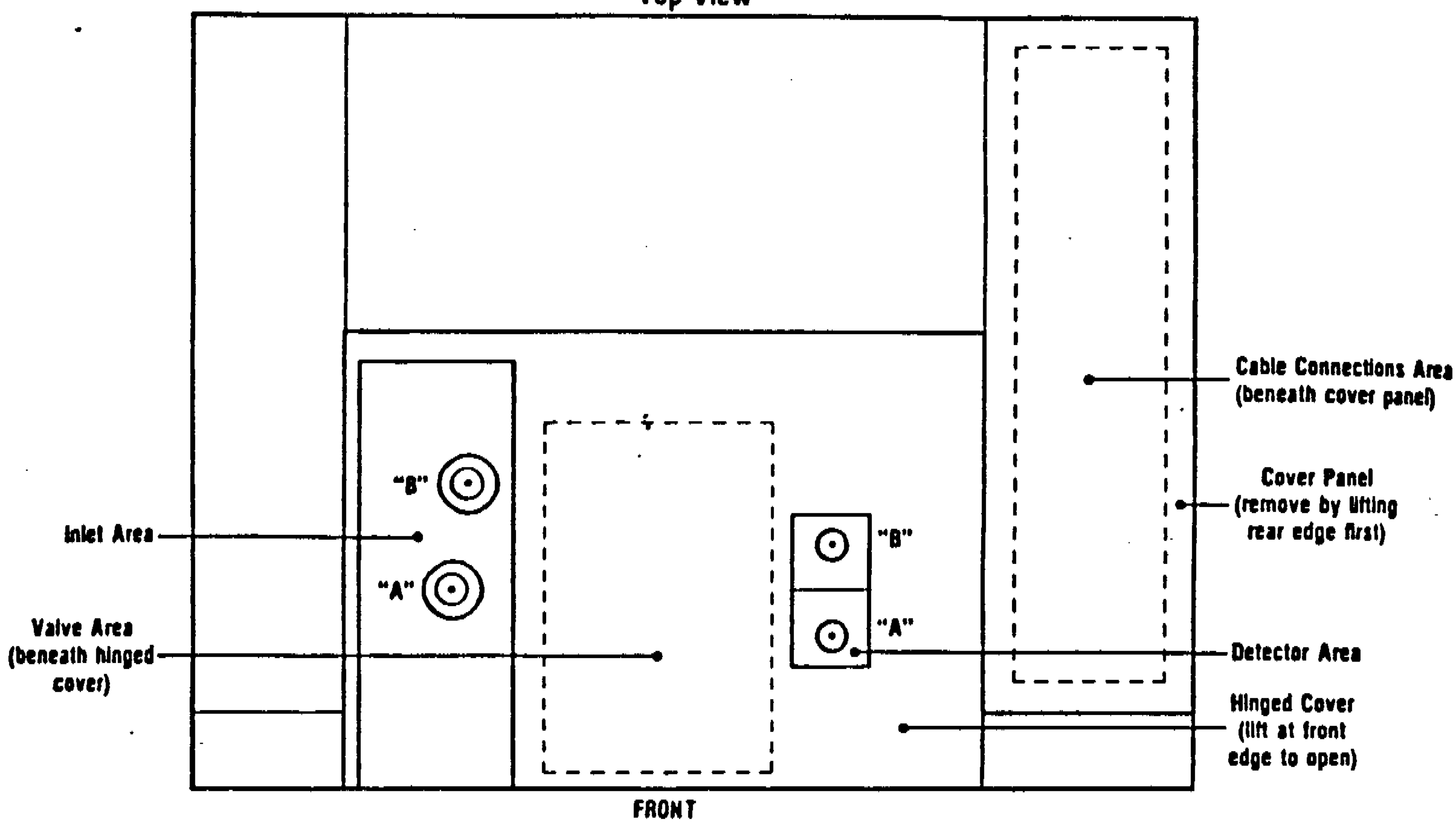
DAY 1

Front View

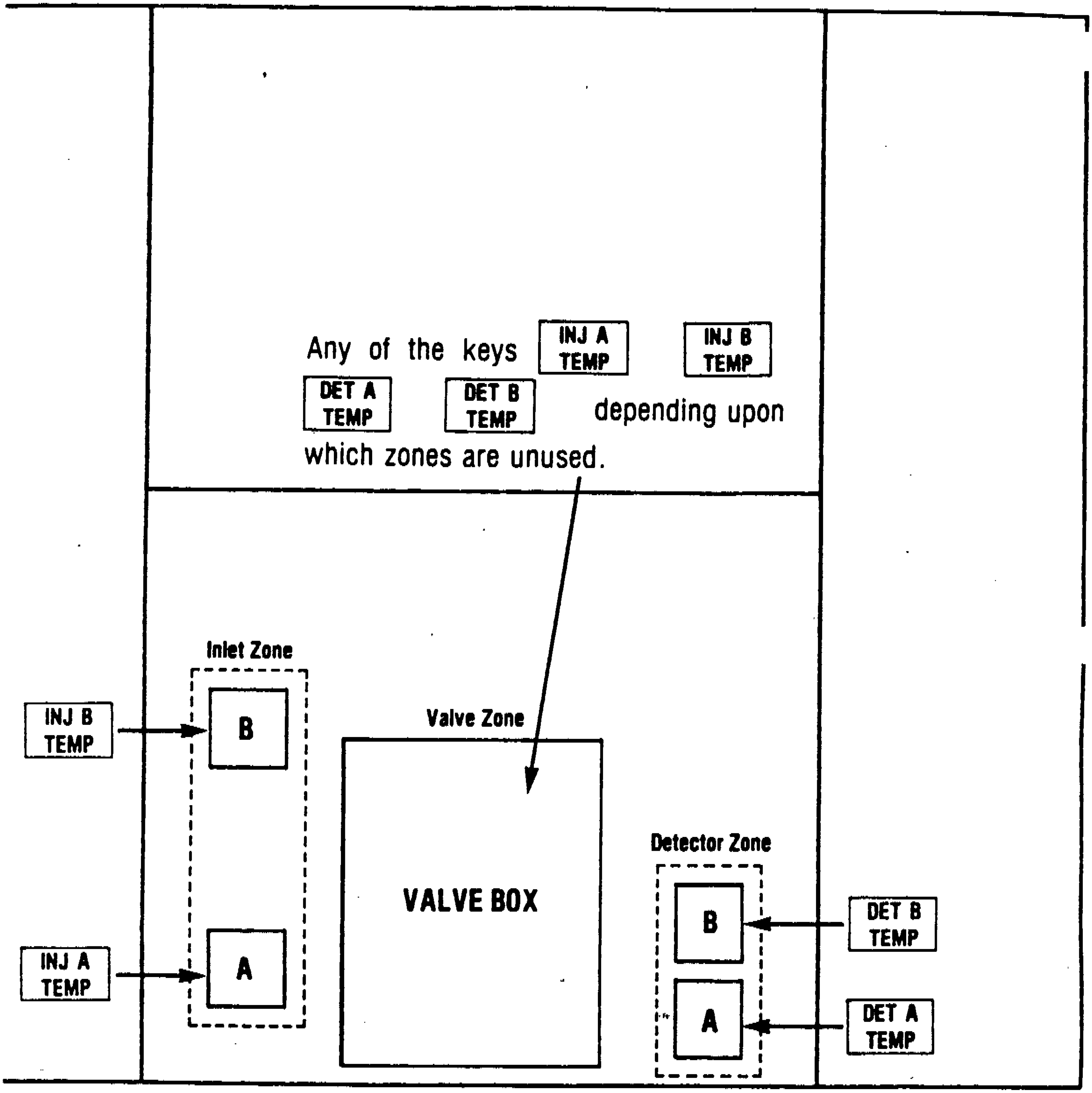


Flow
Module
HP5890A READY
5890A
Keyboard/Displ
Module
Oven
Module

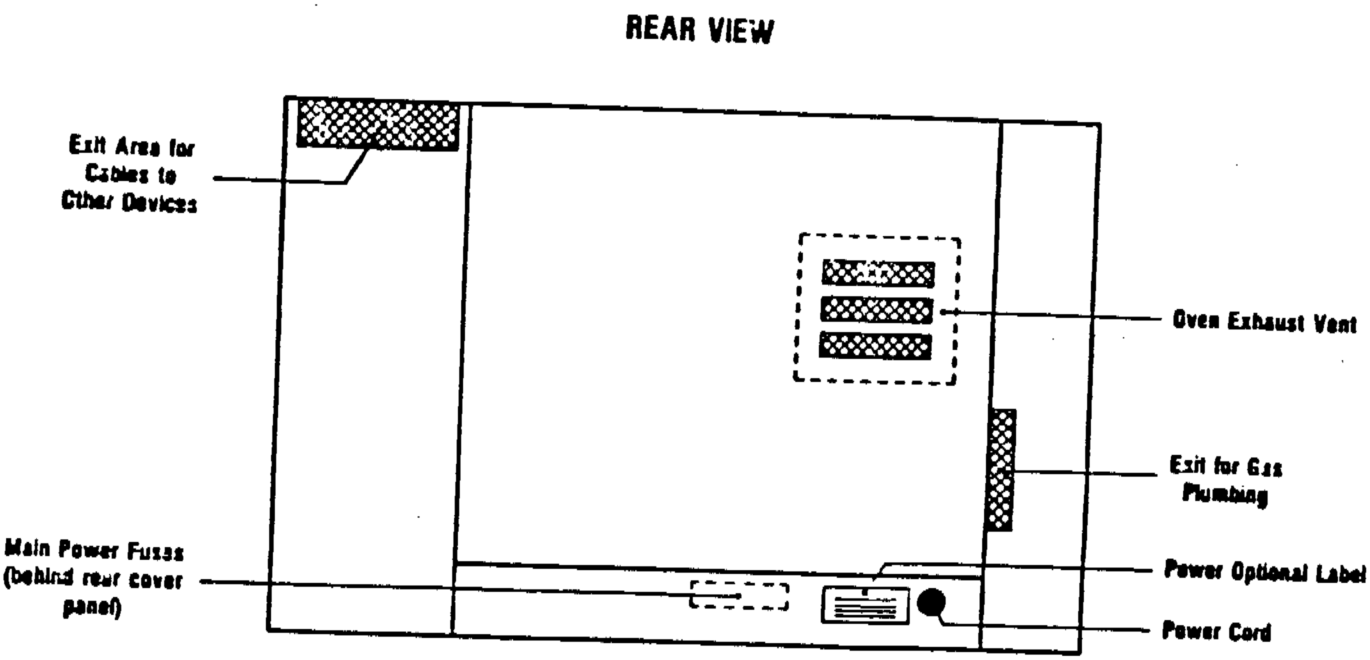
Top View


"B"
"A"
Inlet Area
Valve Area (beneath hinged cover)
"B"
"A"
Cable Connections Area (beneath cover panel)
Cover Panel (remove by lifting rear edge first)
Detector Area
Hinged Cover (lift at front edge to open)
FRONT

Instrument Rear

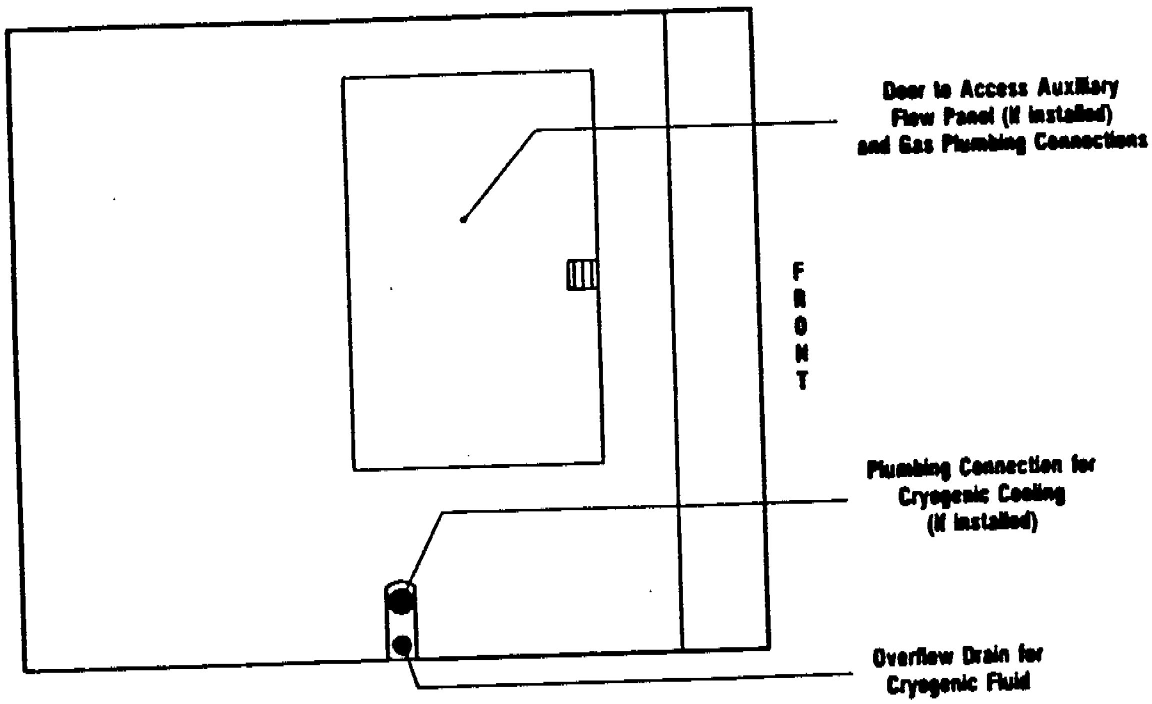


Rear View

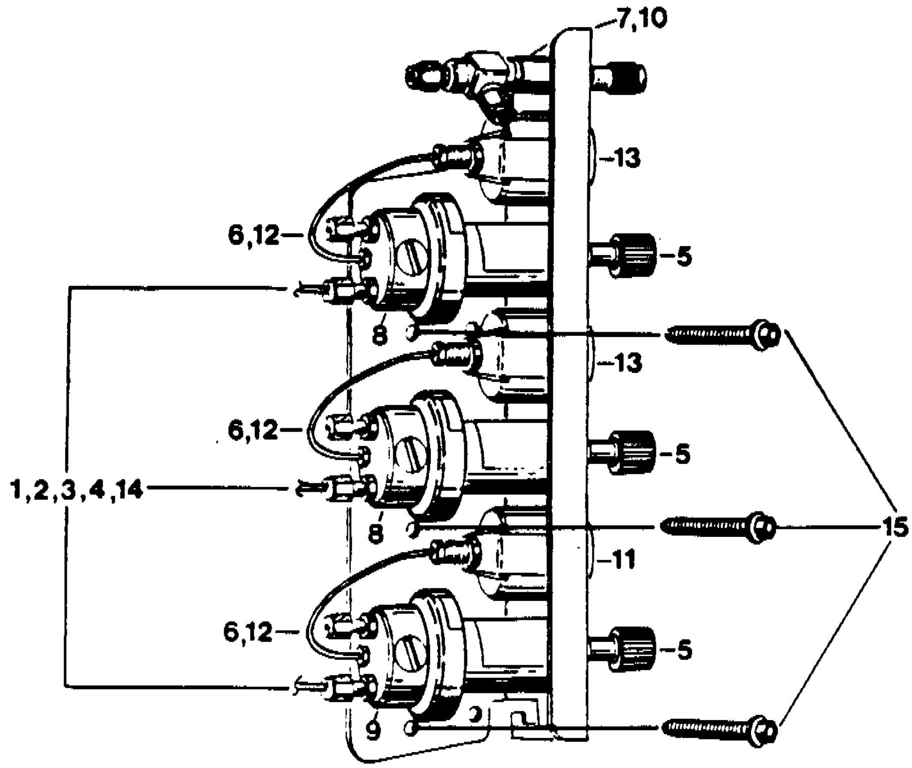


REAR VIEW
Exit Area for Cables to Other Devices
Oven Exhaust Vent
Exit for Gas Plumbing
Main Power Fuses (behind rear cover panel)
Power Optional Label
Power Cord

LEFT SIDE VIEW



Auxiliary Flow Panel



Item	Description	Part No.	Qty.
1	Front Ferrule, 1/8T, Brass*	0100-0032	6
2	Back Ferrule, 1/8T, Brass*	0100-0036	6
3	Nut, 1/8T, Brass	0100-0058	6
4	Male Connector	0100-1115	6
5	Knob, Plastic, Black	0370-3110	3
6	Greased O-Ring*	5080-8770	6
7	Mounting Bracket	19246-00010	1
8	Pressure Regulator, 0-60 PSI	19246-60540	2
9	Pressure Regulator, 0-100 PSI	19246-60550	1
10	Labels, Pressure Sensitive*	19246-90700	1
11	Pressure Gauge, 0-100 PSI	19361-60560	1
12	Jumper Tube, 120 mm long	19361-80020	3
13	Pressure Gauge, 0-60 PSI	19363-60500	2
14	Copper Tube, 1/8 in OD x 17 1/4 in.	5020-8256	3
15	Screw, Tapping 8-16 x 3/4 in.	0624-0546	3

*Not shown.

NOTES

Items 1-4 are supplied with the pressure regulators (8 and 9).

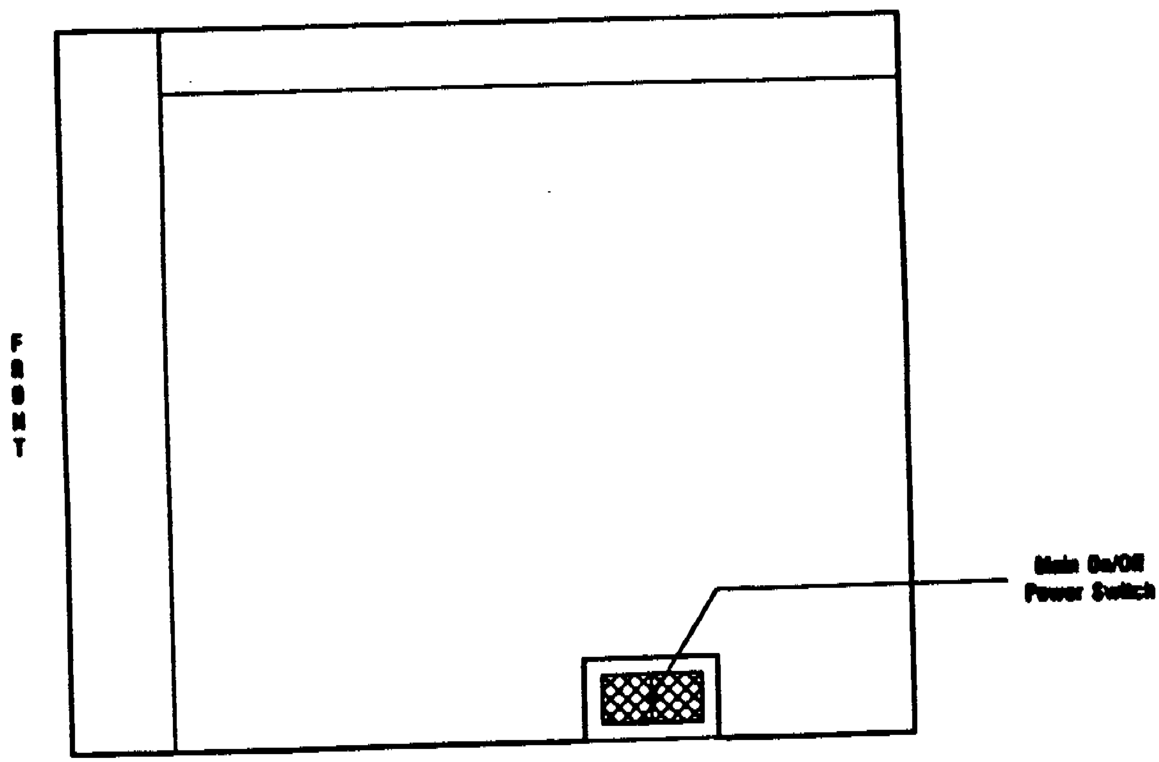
Item 10 is to be applied by the user.

Item 15 is for mounting the assembly.

1132

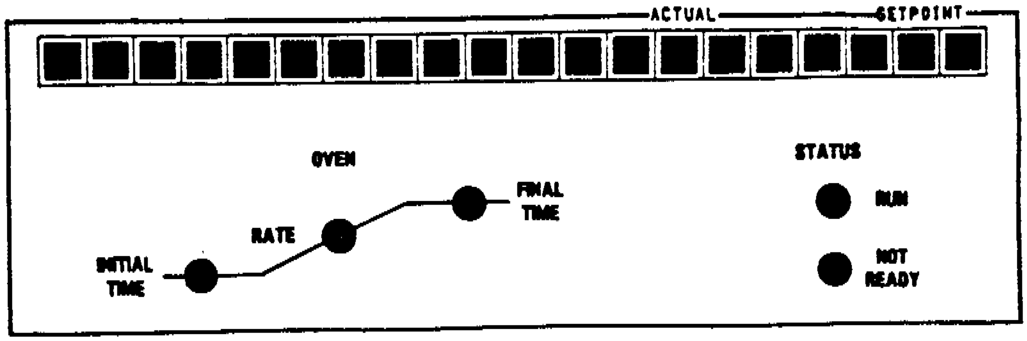
Auxiliary Flow Panel

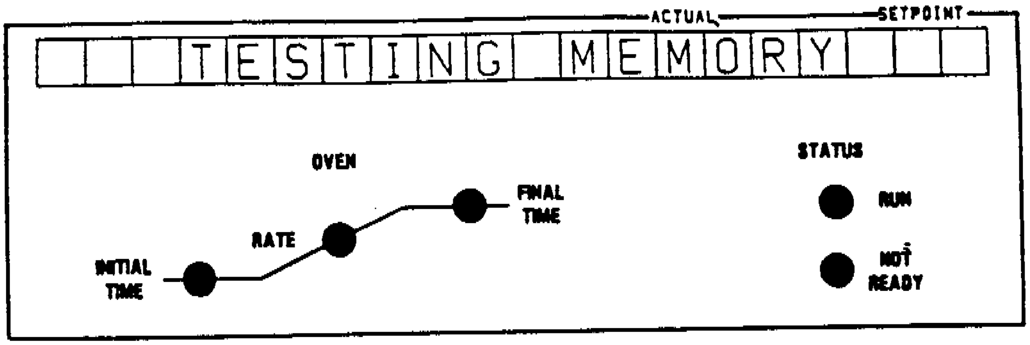
Right Side View

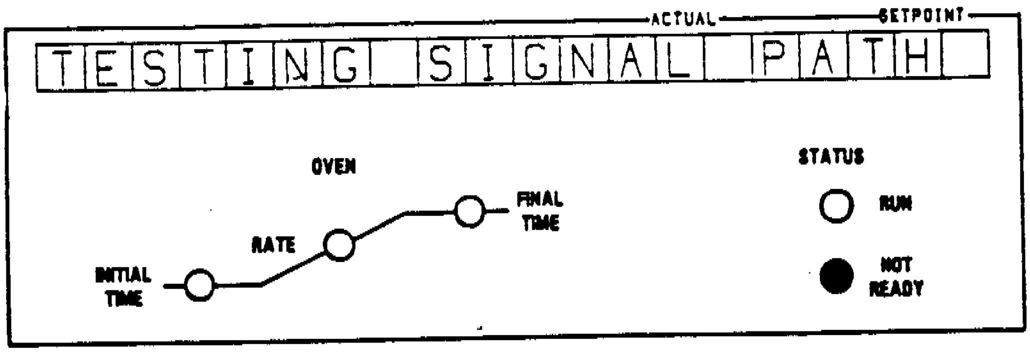


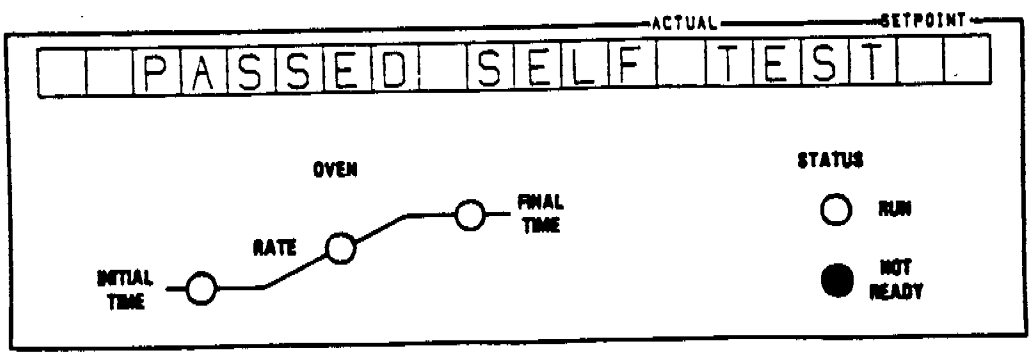
Electronic Performance Verification

NORMAL
"INTEGRITY CHECK" AT POWER-ON

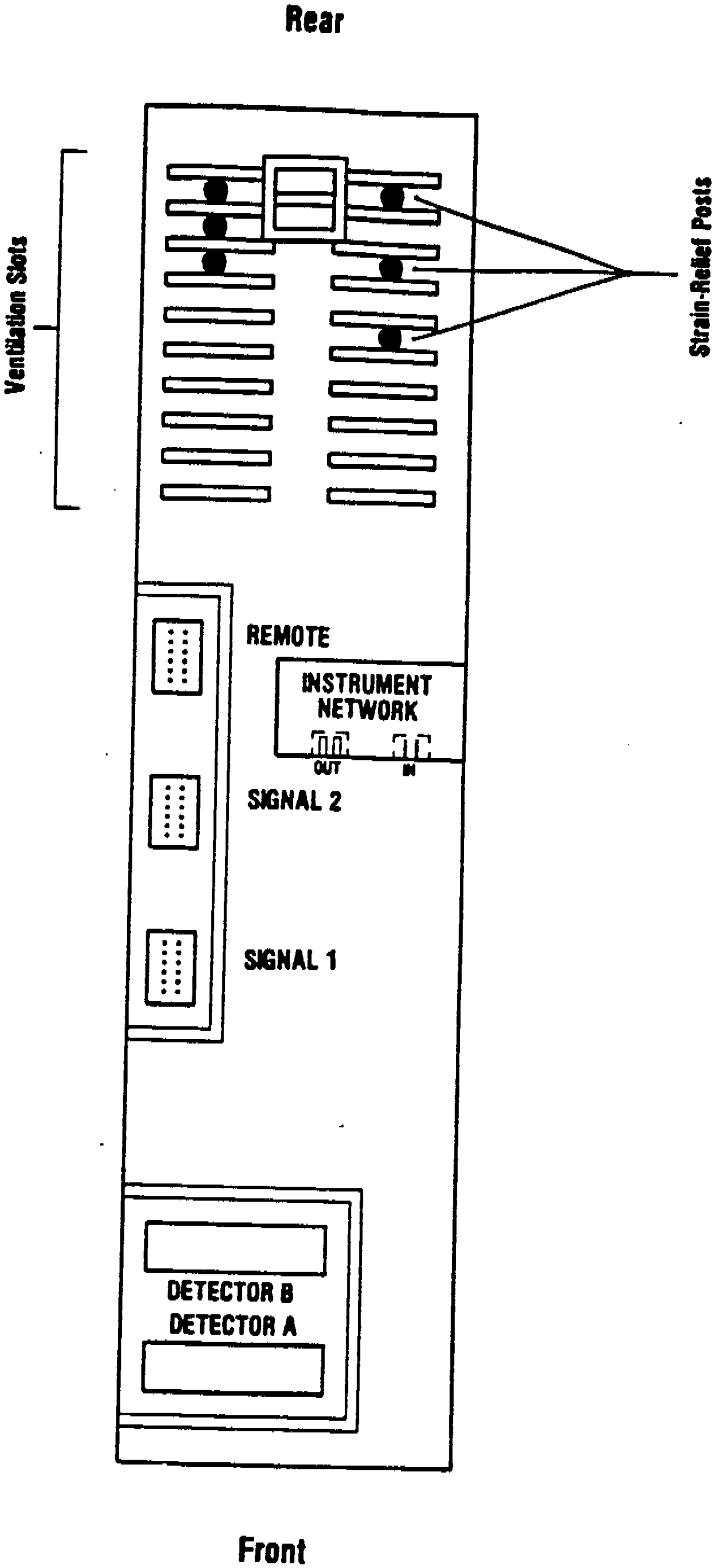




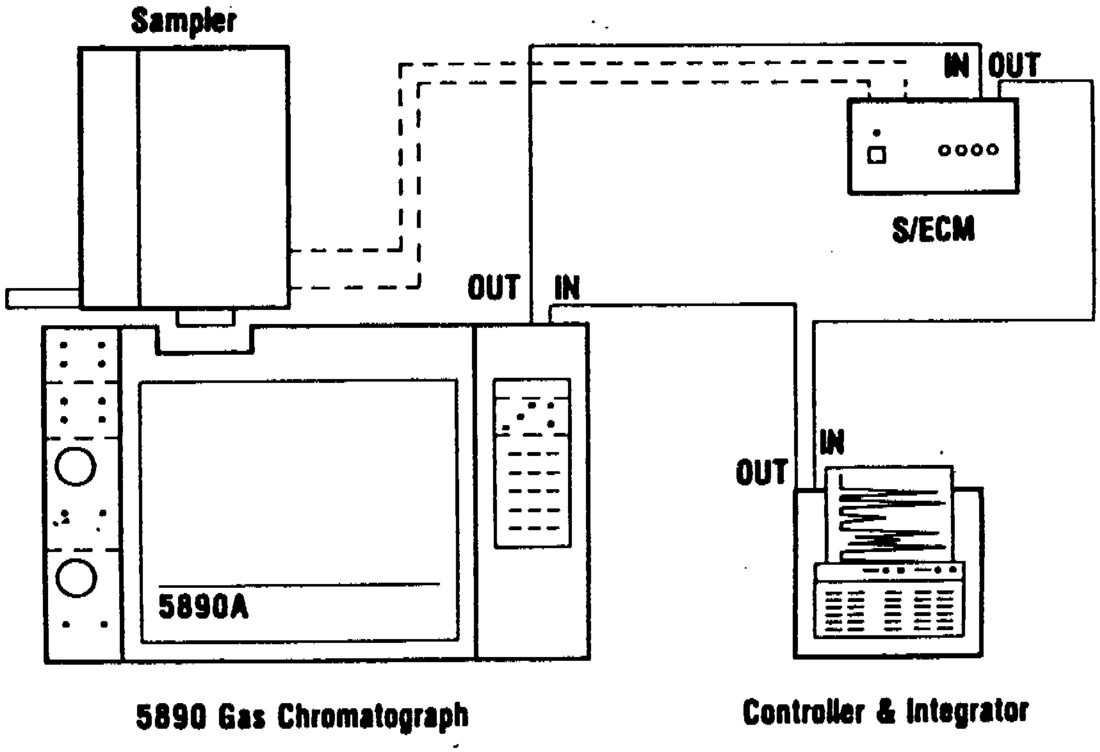




5890 Input/Output Connections



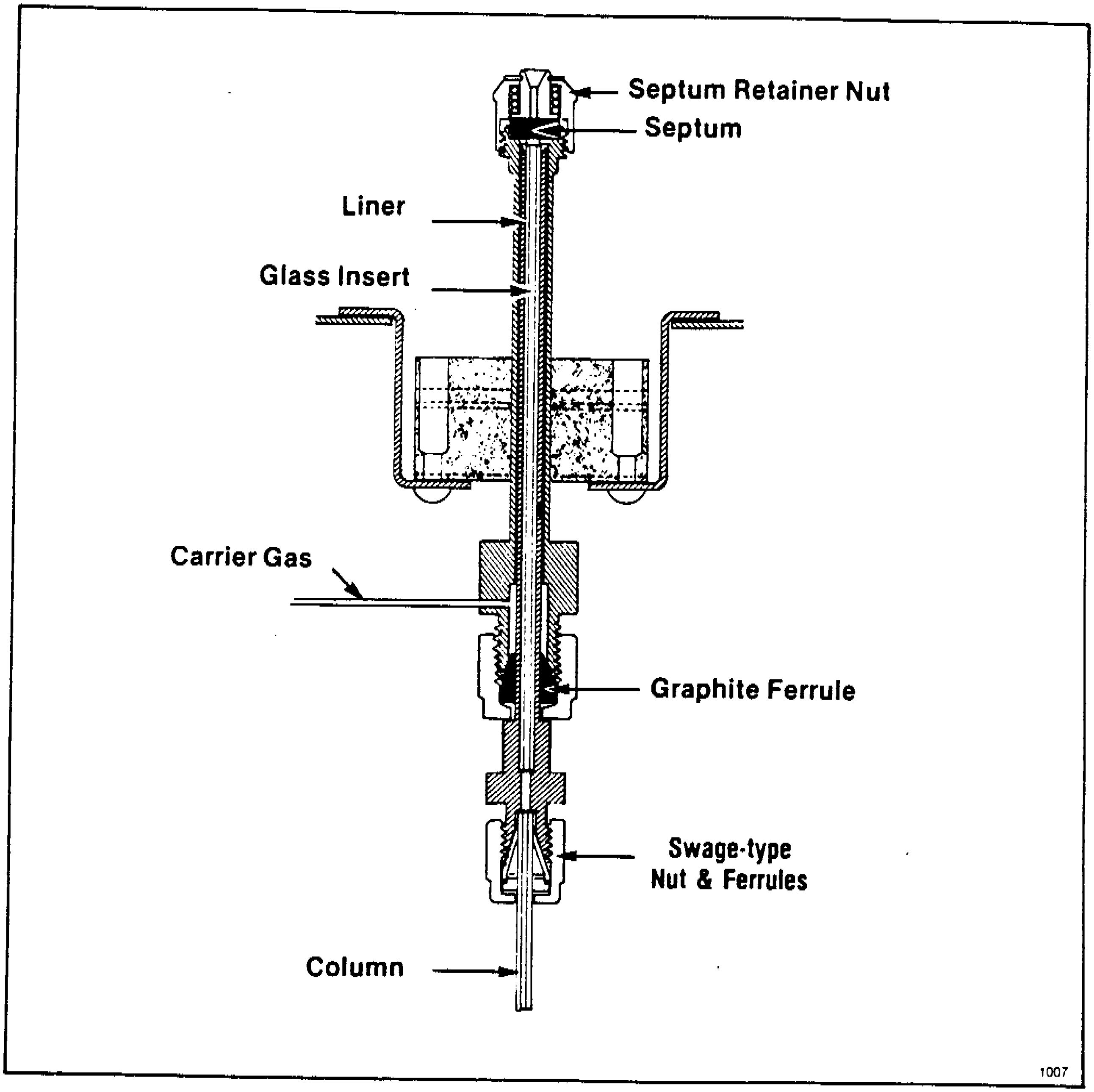
A Simple INET Loop



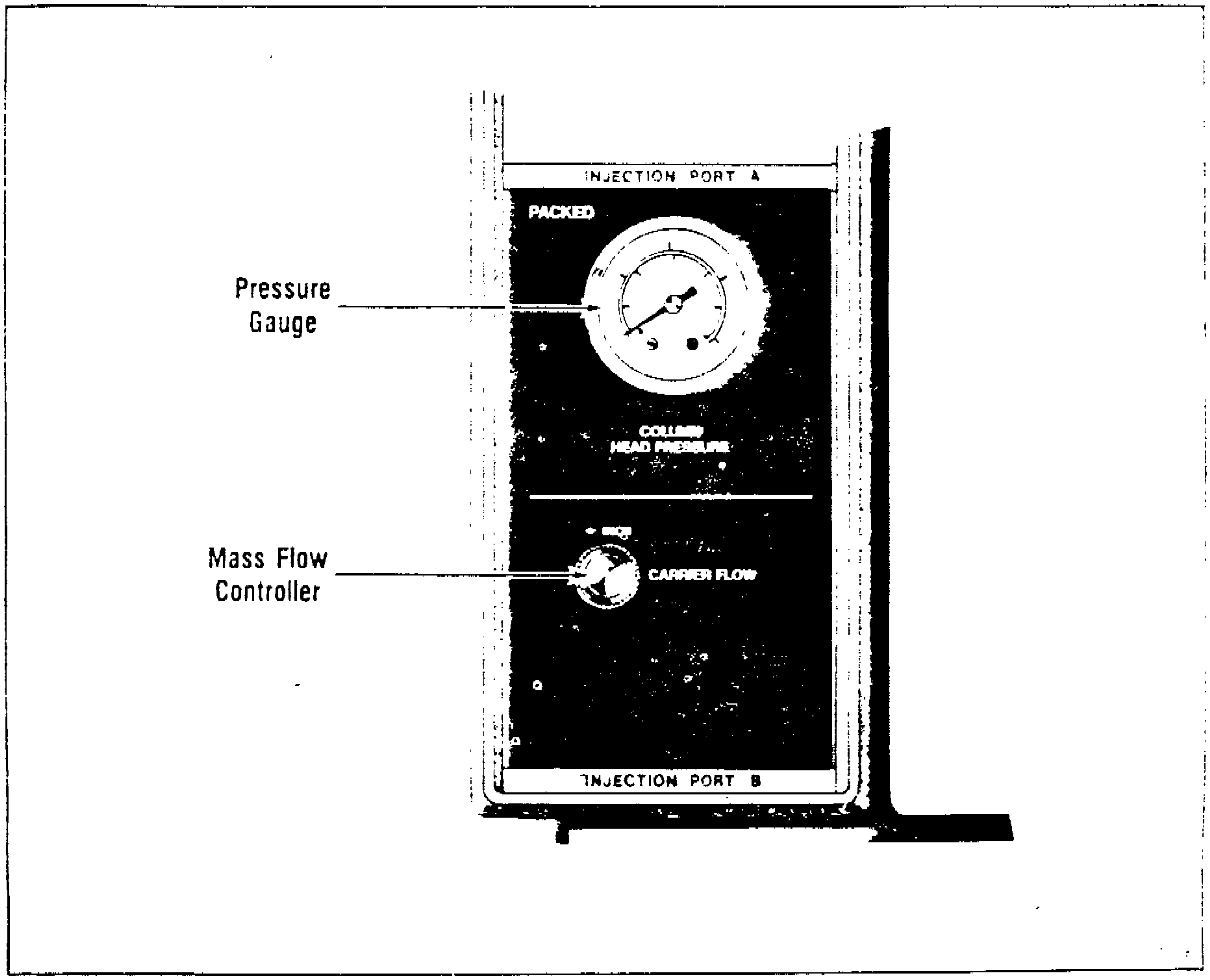
INLET

SYSTEMS

PACKED COLUMN INLET

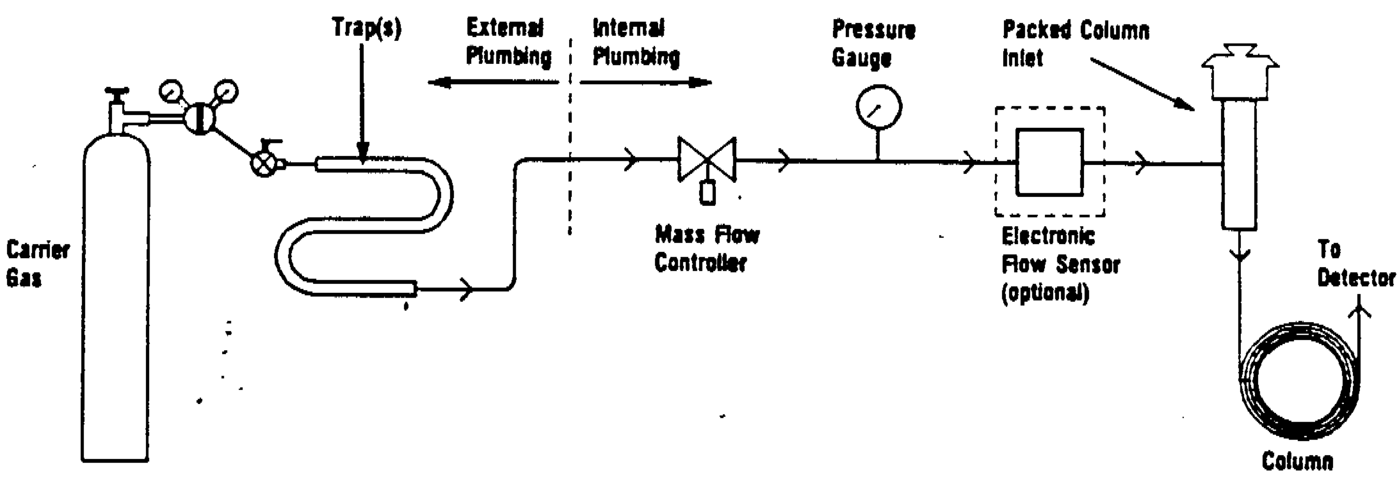


FLOW PANEL CONTROLLING PACKED COLUMN INLET OPERATIONS

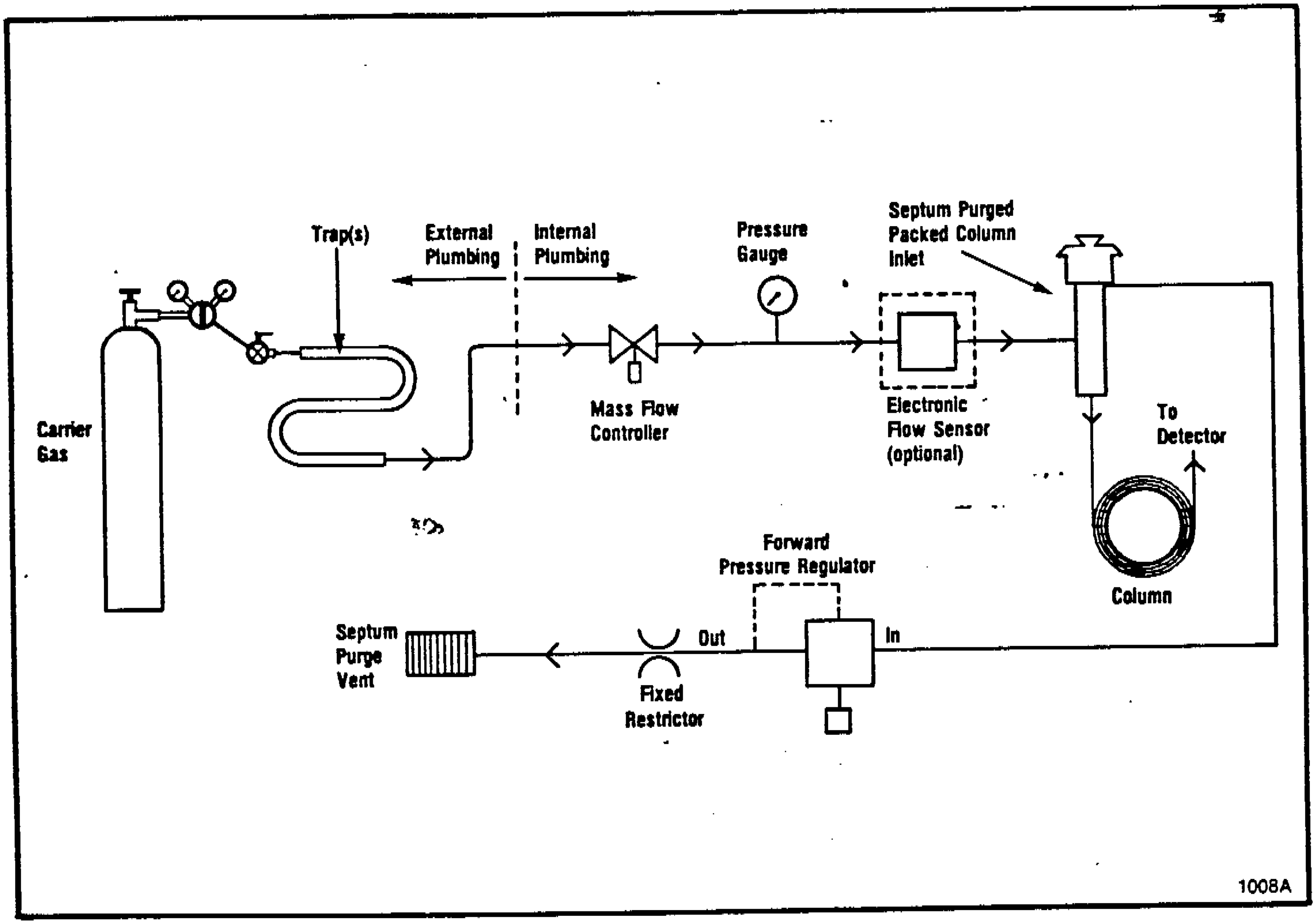


FLOW DIAGRAM: PACKED COLUMN INLET

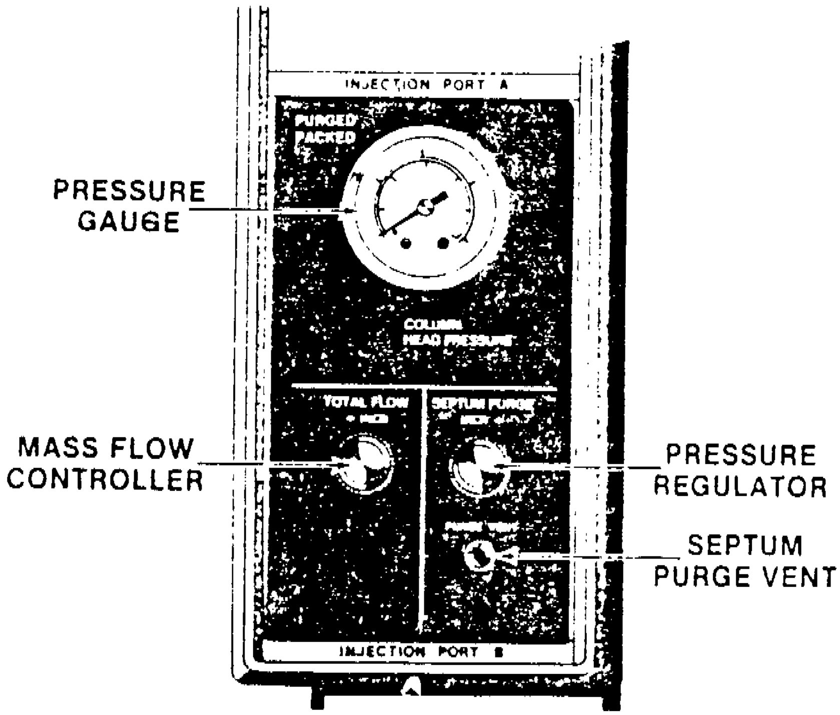
Flow System:
Packed Column Inlet
(with Electronic Flow Sensor)

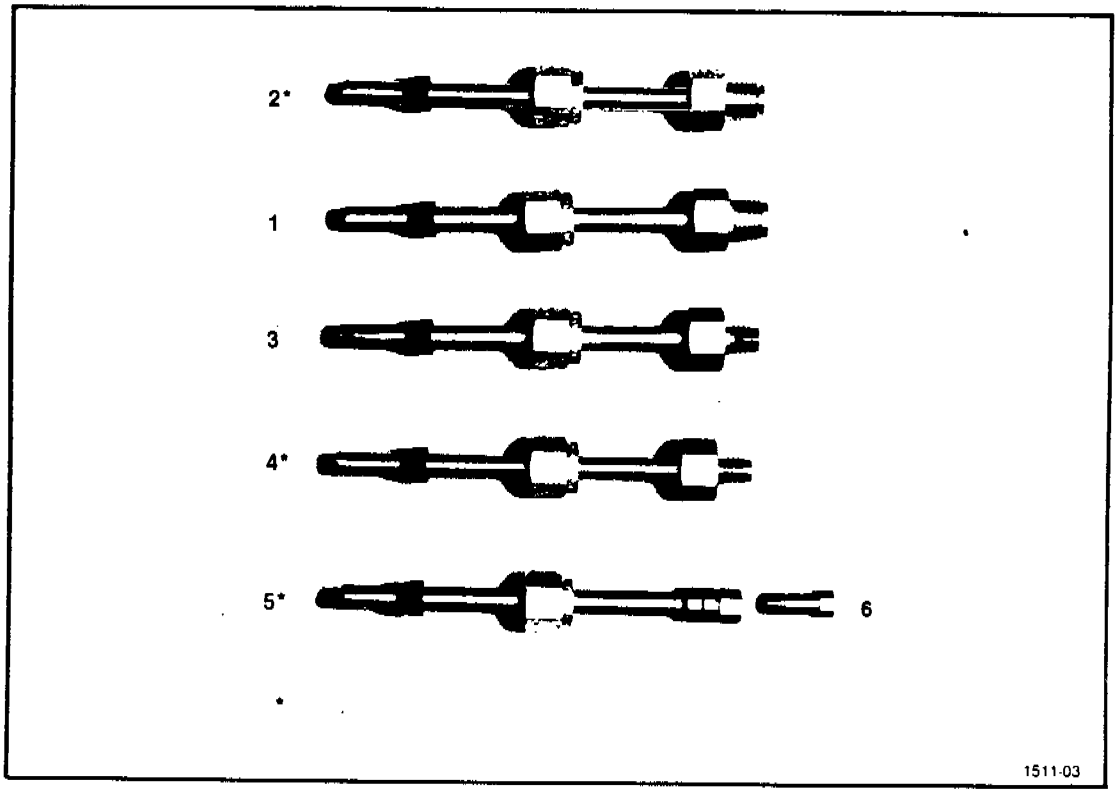


FLOW DIAGRAM: SEPTUM–PURGED PACKED COLUMN INLET



FLOW PANEL CONTROLLING SEPTUM–PURGED PACKED COLUMN INLET OPERATIONS



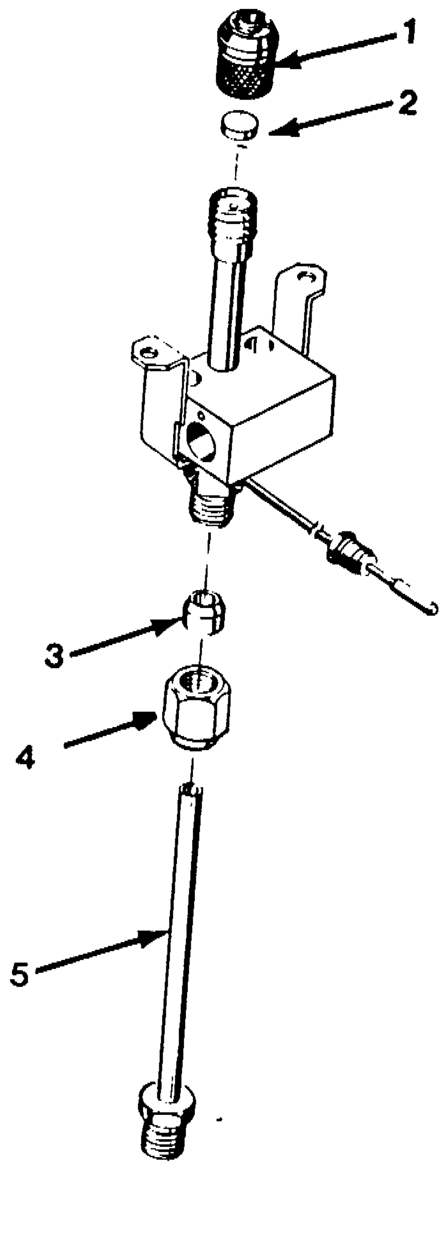


Available Packed Column Inlet Liners

Description	Preferred Column Use	Part No.
1/4-inch swage-type fitting (1)	1/4-inch metal	19243-80520
* 1/4-inch swage-type fitting (2)	1/4-inch metal or glass	19243-80540
1/8-inch swage-type fitting (3)	1/8-inch metal	19243-80510
* 1/8-inch swage-type fitting (4)	1/8-inch metal	19243-80530
* capillary column fitting (5)	"HP Series 530µ" capillary column	19244-80540
capillary column nut (6)		18740-20870

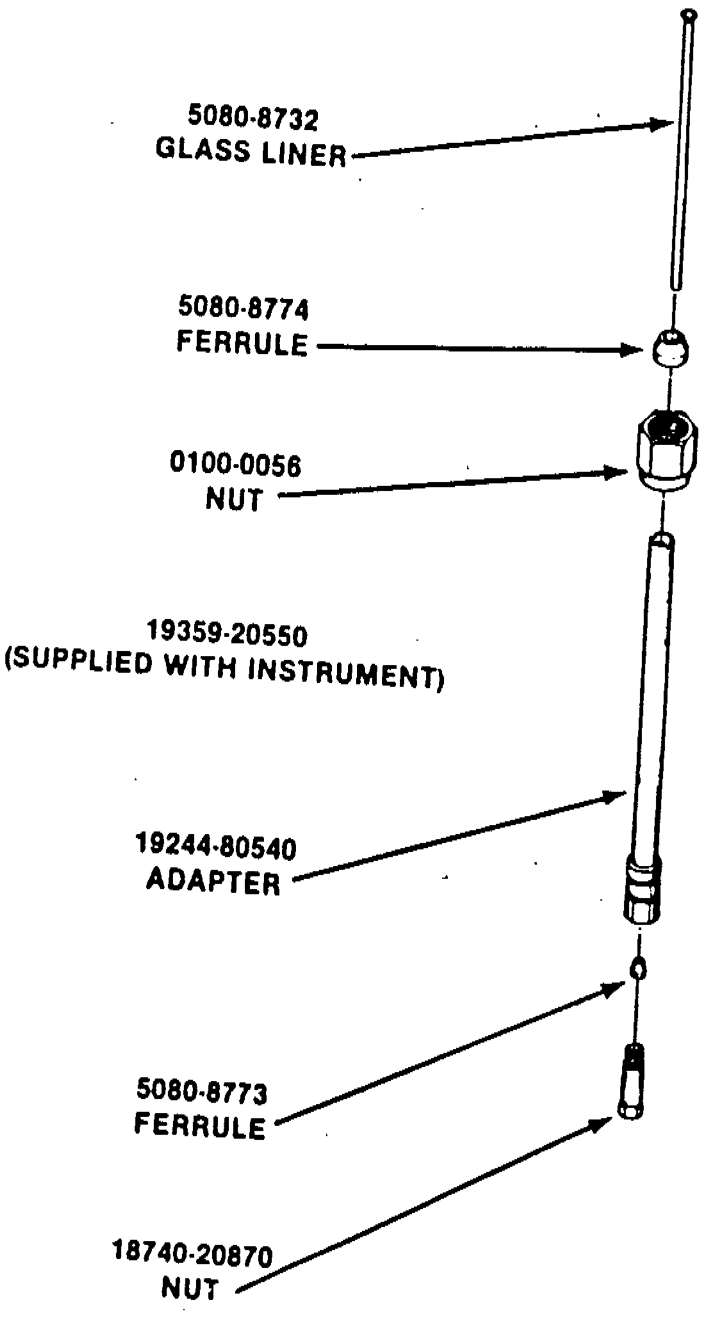
* Requires glass insert, Part No. 5080-8732 (package of 25).

HP 5890A Packed Column Injection Port

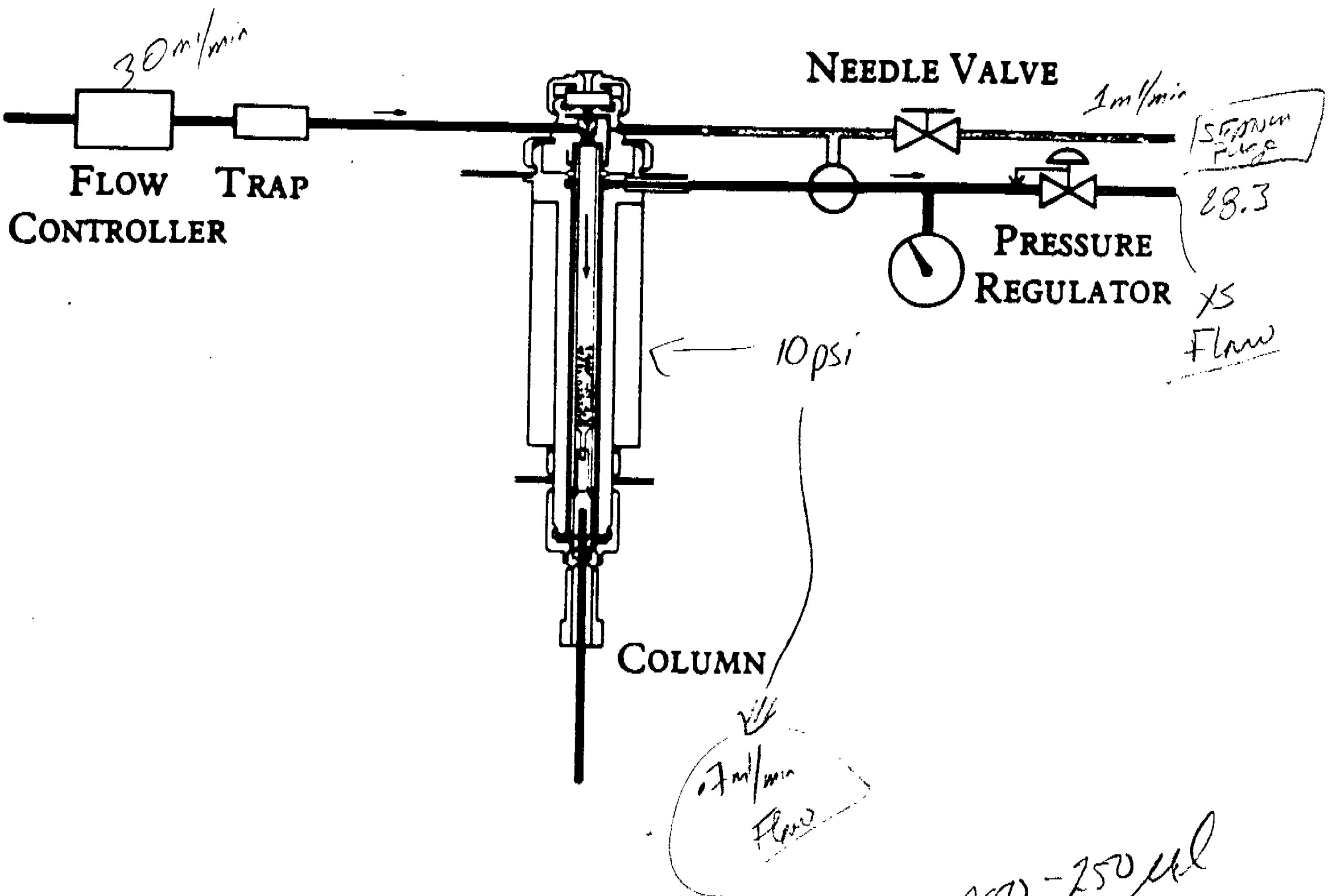


5890 INJECTION PORT KIT FOR SERIES 530 COLUMNS IN PACKED COLUMN INLET

KIT #19095-60500



HP5890A CAPILLARY SPLIT/SPLITLESS INLET



.2mm ID
= needs 10psi for 12 meters

.32 ⇒ 5psi for 12 meter

.35 ⇒ 5psi for 12 meters

INJ Port = 200-250 µl Total Volume

CALIBRATION

and

REPORTS

Calculation Procedures and Reported Results

Formula	Type
Area %	Uncalibrated
Height %	Uncalibrated
Normalization	Calibrated
External Standard	Calibrated
Internal Standard	Calibrated

AREA% REPORT

DEFAULT REPORT

or

WHEN

--- No ISTD peak found in ISTD calib.

--- No matching CALIB peak found even though CALIB table exists.

--- Sample # incorrect (out of range) for an automated run.

--- No CALIB exists.

--- ISTD peak is not matched.

HEIGHT % REPORT

OP #4

* LIST: OP #4

REPORT OPTIONS

Suppress local report . . . . NO

Peak height mode . . . . NO

Report uncalibrated peaks . . . . NO

Extended report . . . . NO

*

A Height % Report

* REPORT

RUN# 9 JUN 24, 1985 11:13:31

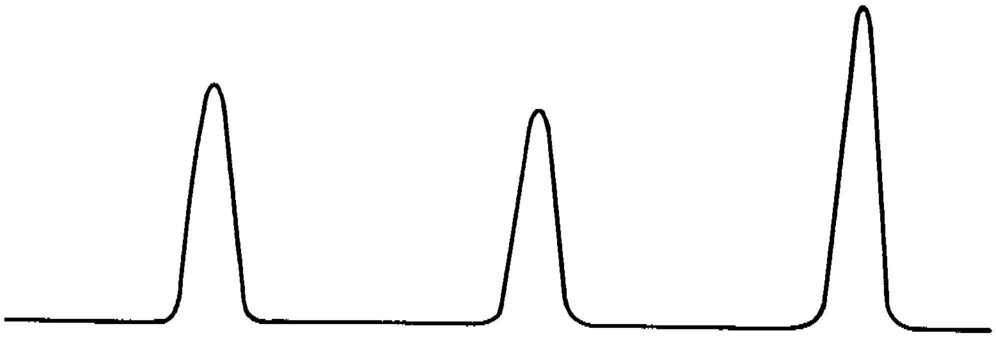
SAMPLE# 170

HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
.126	8236701	SHB	.058	72.50566
.335	820156	TBB	.044	7.21964
.585	659275	BV	.053	5.80344
.660	166038	VP	.030	1.46159
.835	983012	PP	.016	8.65322
1.001	494895 I	PH	.083	4.35644

TOTAL HEIGHT= 11360072.
MUL FACTOR= 1.

Calibration Factors



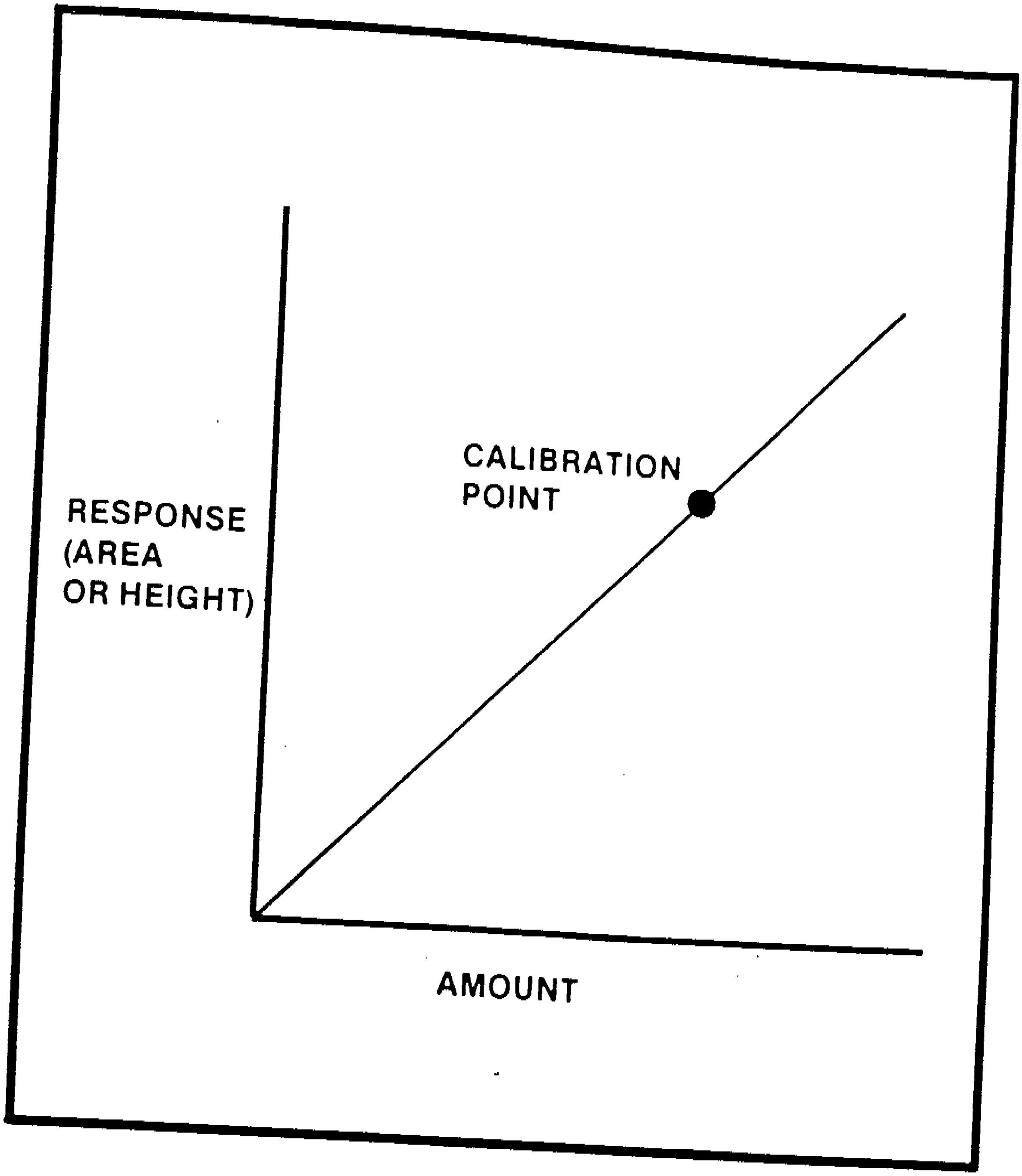
Amount:	100	100	200
Area:	3000	2500	4000

Absolute Response Factor

RF = Amount/Area

- Corrects for detector response
- Independent of:
  - The amount of a component
  - The presence of other components

Single-point Calibration Chart



EXTERNAL STANDARD

To Determine the Amount of i :

$$\frac{\text{AMT STD}}{\text{AREA STD}} = \frac{\text{AMT i}}{\text{AREA i}}$$

$$\frac{\text{AMT STD}}{\text{AREA STD}} = \text{ARF}$$

$$\text{AMT i} = \text{AREA i} \times \text{ARF} \times \text{MUL FACTOR}$$

* REPORT

RUN# 2 JUN 24, 1985 11:00:04

ESTD

RT	AREA	TYPE	CAL#	AMOUNT
.126	27820512	SHB	1	1.300
.335	2121374	TBB	2	3.600
.585	2055233	BV	3	3.099
.660	295681	VB	4	2.099
.835	900307	PB	5R	.870
1.001	2413101	PP	6	.430
1.376	472200	BV	7	.780

TOTAL AREA=3.6078E+07
MUL FACTOR=1.0000E+00

Normalization

$$\%\ \text{of}\ i = \frac{ARF(i) \times A(i)}{\text{sum}\ \{RF(i) \times A(i)\}} \times 100\ \% \times \text{MUL FACTOR}$$

% of i	is the percentage of component i in the sample
ARF(i)	is the absolute response factor for component i
A(i)	is the area or height of component i

A Normalization Report

* REPORT

RUN# 2 JUN 24, 1985 11:00:04

NORM

RT	AREA	TYPE	CAL#	AMOUNT
.126	27820512	SHB	1	10.675
.335	2121374	TBB	2	29.562
.585	2055233	BV	3	25.446
.660	295681	VB	4	17.237
.835	900307	PB	5R	7.144
1.001	2413101	PP	6	3.531
1.376	472200	BV	7	6.405

TOTAL AREA=3.6078E+07
MUL FACTOR=1.0000E+00

RELATIVE RESPONSE FACTOR

$$RRF(i) = \frac{ARF(i)}{ARF(\text{ISTD})}$$

or

$$RRF(i) = \frac{AMT(i) \ / \ AREA\ (i)}{AMT(\text{ISTD}) \ / \ AREA(\text{ISTD})}$$

INTERNAL STANDARD

To Determine the Amount of i :

$$\frac{\mathrm{AMT(ISTD)}}{\mathrm{AREA(ISTD)} \times \mathrm{ARF(ISTD)}} = \frac{\mathrm{AMT(i)}}{\mathrm{AREA(i)} \times \mathrm{ARF(i)}}$$

$$\mathrm{AMT(i)} = \mathrm{AMT(ISTD)} \times \frac{\mathrm{AREA(i)}}{\mathrm{AREA(ISTD)}} \times \frac{\mathrm{ARF(i)}}{\mathrm{ARF(ISTD)}} \times \mathrm{MUL\ FACTOR}$$

$$\mathrm{AMT(i)} = \mathrm{AMT(ISTD)} \times \frac{\mathrm{AREA(i)}}{\mathrm{AREA(ISTD)}} \times \mathrm{RRF(i)} \times \mathrm{MUL\ FACTOR}$$

INTERNAL STANDARD

To Determine the % of i :

$$\%AMT(i) = \frac{AMT(ISTD)}{SAMPAMT} \times 100 \times \frac{AREA(i)}{AREA(ISTD)} \times RRF(i) \times MUL\ FACTOR$$

SAMPAMT = TOTAL AMOUNT OF ORIGINAL SAMPLE

SAMPLE AMOUNT

ISTD% CALCULATION

$$\text{RELATIVE AMOUNT of } y = \frac{\text{ABSOLUTE AMOUNT of } y^{*}}{\text{SAMPLE AMOUNT}} \times 100$$

* THE ABSOLUTE AMOUNT OF y IS OBTAINED FROM THE ISTD CALCULATION.

* OP # 7

DEFAULT SAMPLE INFORMATION
USE SAMPLE TABLE IN MANUAL RUN [Y/N*]:

ISTD AMT [0.0000E+00]:
SAMPLE AMT [0.0000E+00]:
MUL FACTOR [1.0000E+00]:
RECALIBRATION [Y/N*]:
NAME:
REPORT MEMO:

An Internal Standard Report

* REPORT

RUN# 2 JUN 24, 1985 11:00:04

ISTD

RT	AREA	TYPE	CAL#	AMOUNT
.126	27820512	SHB	1	1.300
.335	2121374	TBB	2	3.600
.585	2055233	BV	3	3.099
.660	295681	VB	4	2.099
.835	900307	PB	5&	.870
1.001	2413101	PP	6	.430
1.376	472200	BV	7	.780

TOTAL AREA=3.6078E+07
MUL FACTOR=1.0000E+00
ISTD AMT=8.7000E-01

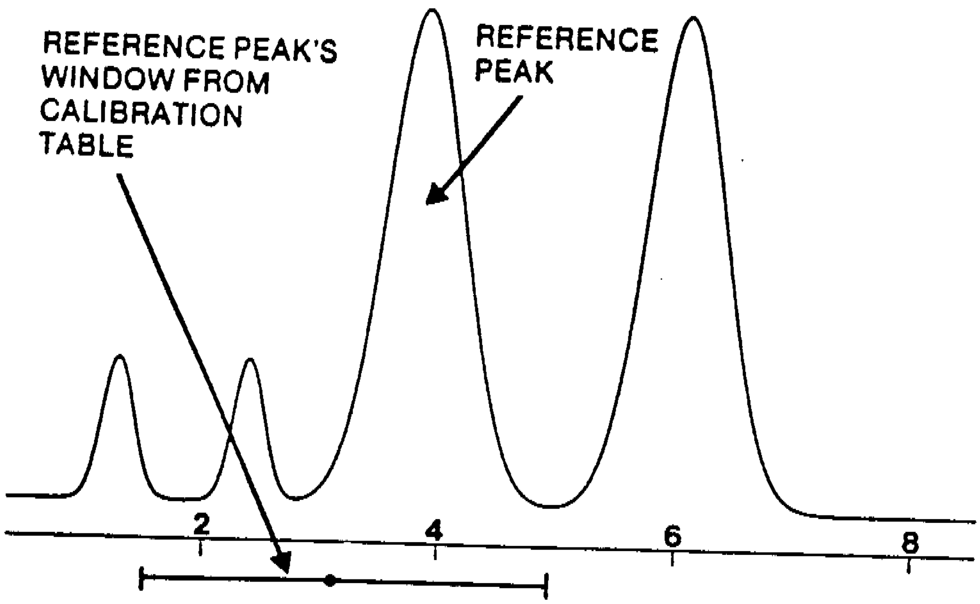
CREATING A CALIBRATION FILE

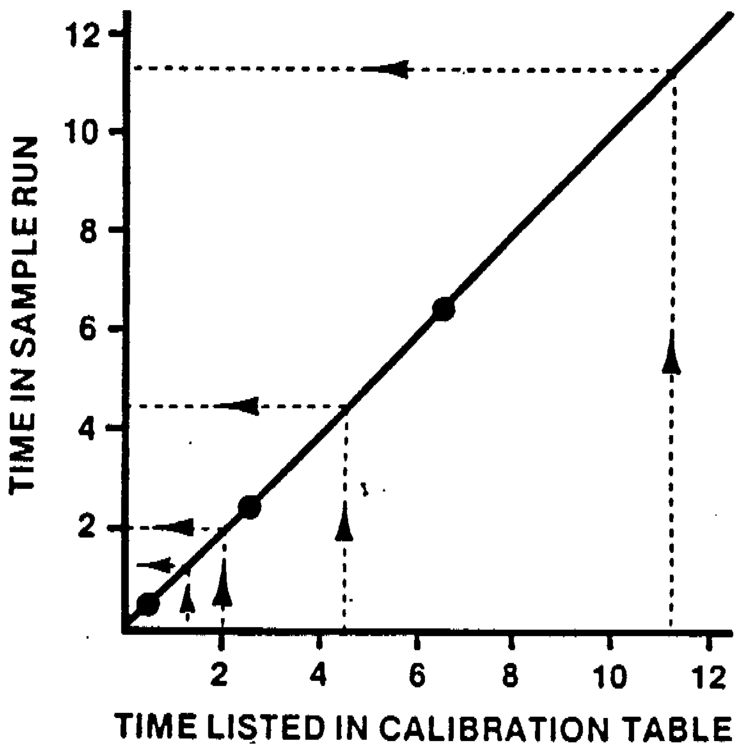
"—" Absolute Value

"—" RT = Reference

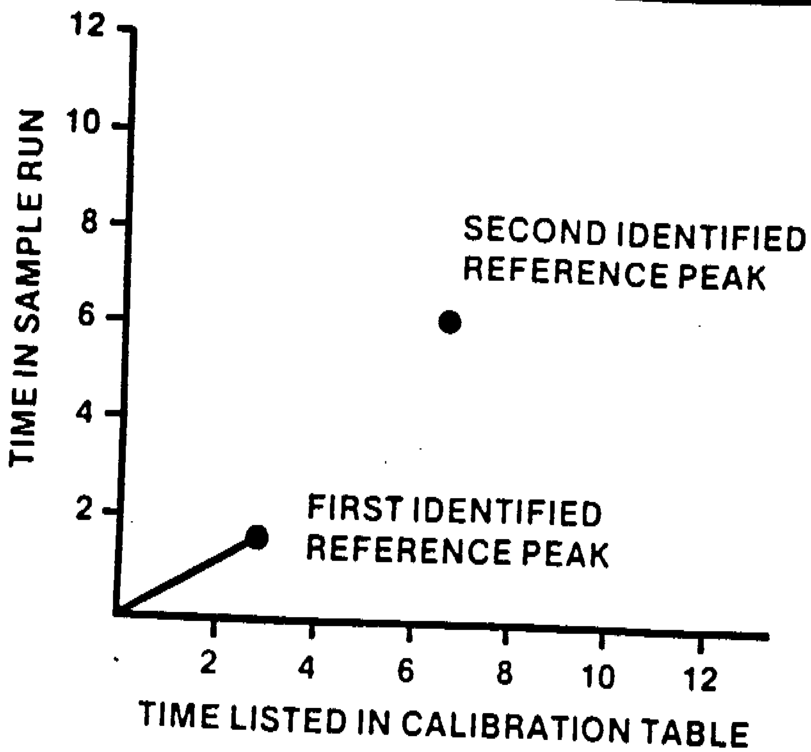
```
CALIB ISTD
REF % RTW: - . 5
    % RTW: 5 .

CAL #     RT        AMT

1:  - .  3 3    :2 . 3
2:  - .  5 8    :2 . 5
3:  . 6  7      :5
4:  1 .  0 0    :1 . 5
5:  - 1  . 1 4  :1 . 3
6:  1 .  3 3    :1 . 0
7:
```

ISTD Number — ISTD CAL # 5

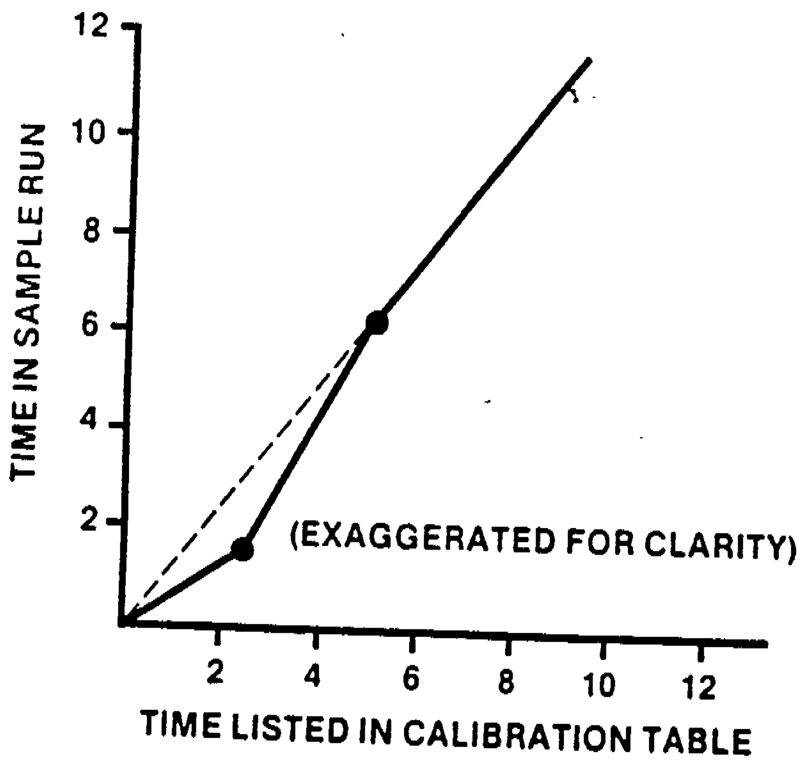

REFERENCE PEAK'S WINDOW FROM CALIBRATION TABLE
REFERENCE PEAK
2
4
6
8



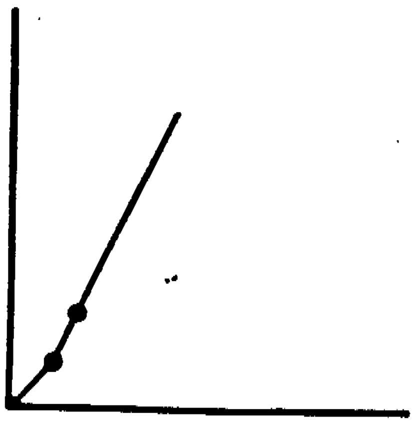
TIME IN SAMPLE RUN
12
10
8
6
4
2
2
4
6
8
10
12
TIME LISTED IN CALIBRATION TABLE



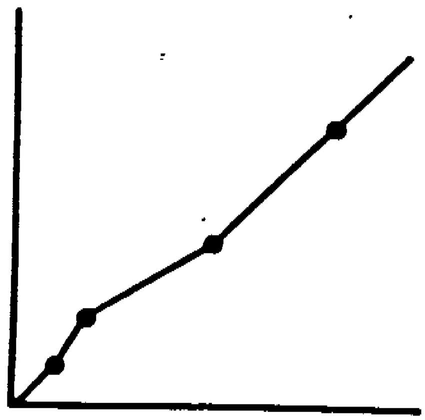
FIRST, PLOT A PEAK . . .



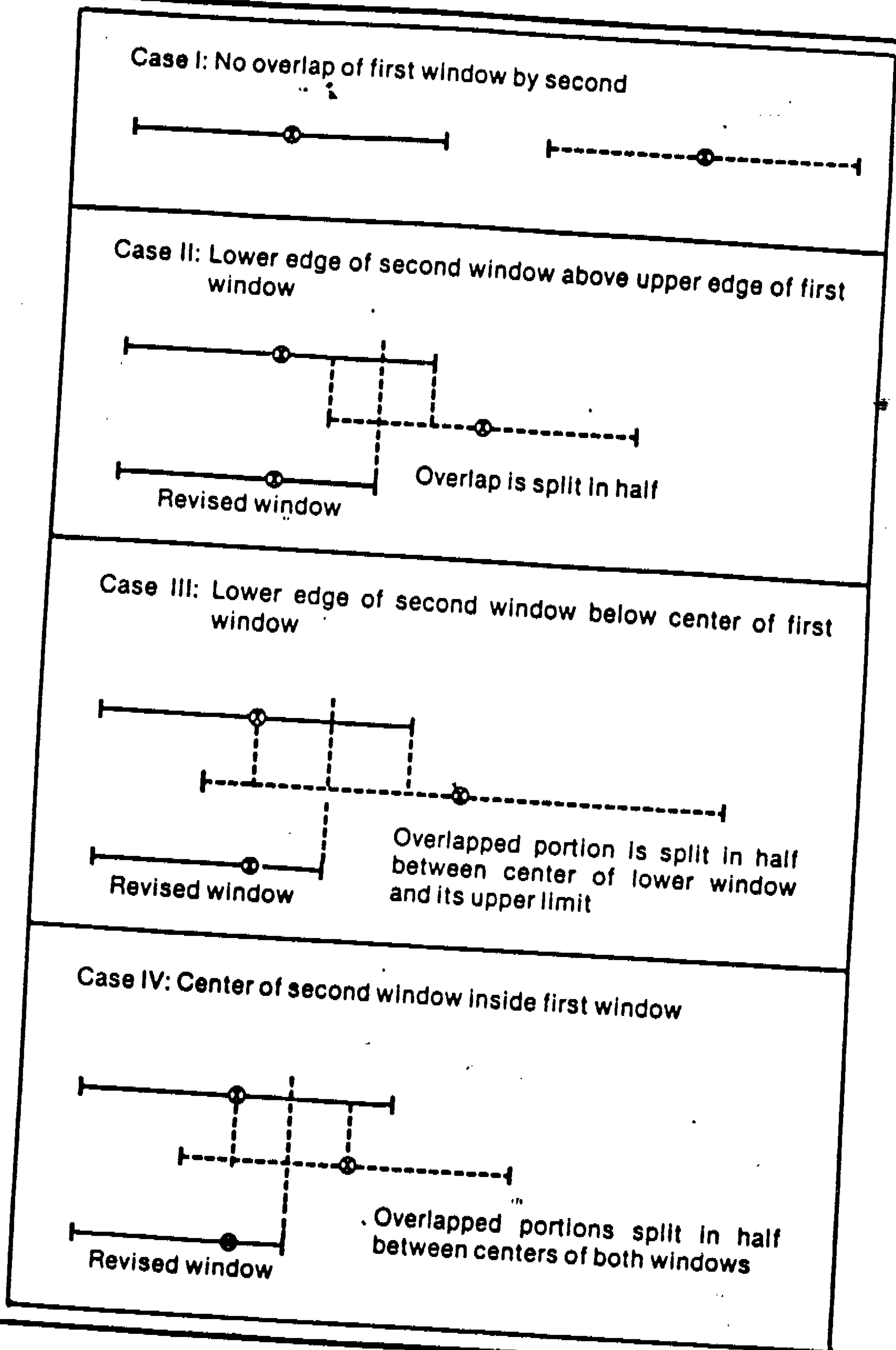
THEN CONNECT THE POINTS . . .



A SLIGHT CHANGE IN AN EARLY REFERENCE PEAK . . .

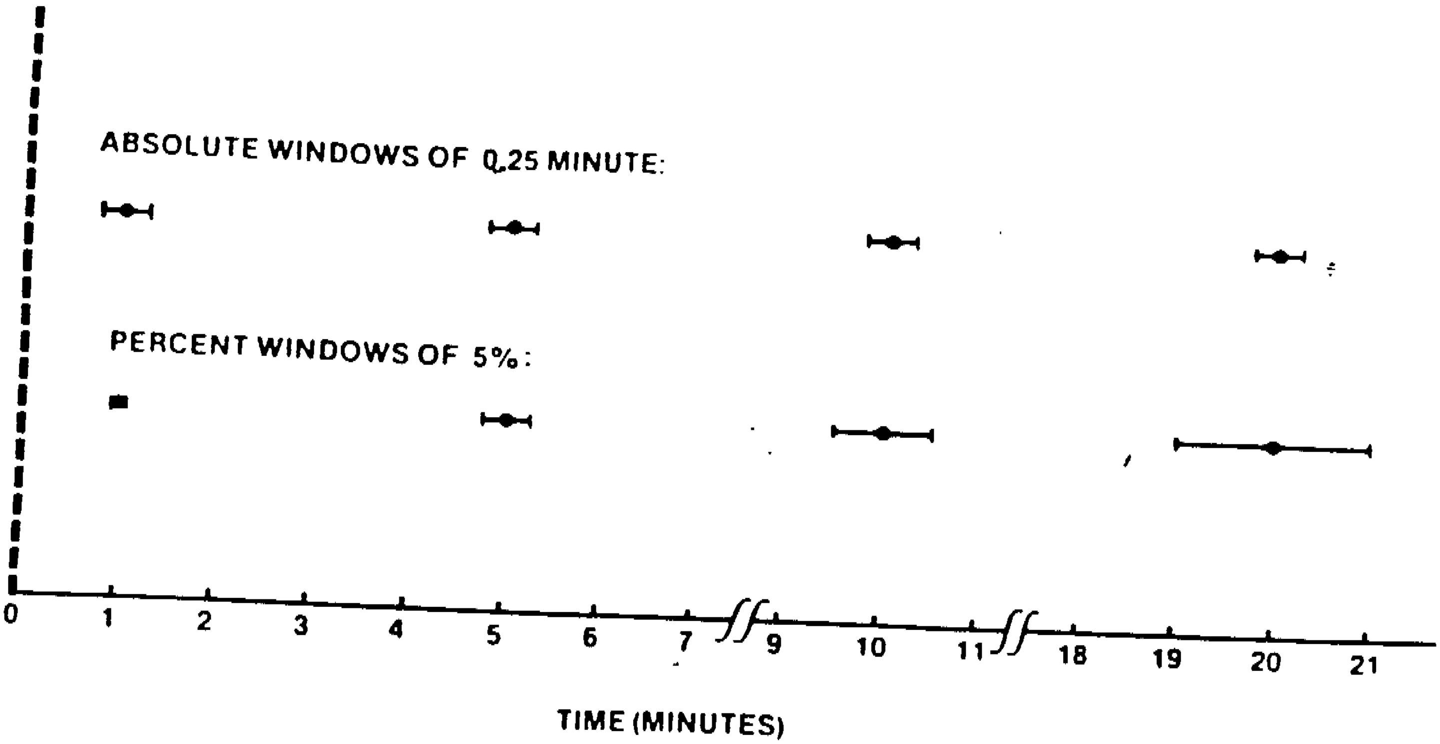


CAN RUIN THE ENTIRE CURVE UNLESS YOU HAVE ADDITIONAL REFERENCES.


Case I: No overlap of first window by second
Case II: Lower edge of second window above upper edge of first window
Overlap is split in half
Revised window
Case III: Lower edge of second window below center of first window
Overlapped portion is split in half between center of lower window and its upper limit
Revised window
Case IV: Center of second window inside first window
Overlapped portions split in half between centers of both windows
Revised window

REF % RTW: [−] [•] [1] [ENTER],

% RTW: [5] [•] [3] [ENTER].



RETENTION TIME UPDATING

- Occurs automatically as report is printed
- If report generation is suppressed – no updating occurs
- Weighted 75% in favor of old times
- Every reference peak must be identified

OP # 3: CALIBRATION OPTIONS

```
CALIBRATION OPTIONS
RF of uncalibrated peaks [0.0000E+00 ]:
Replace calibration fit [Y/N*]: Y

P = point-to-point
L = linear (least square)
N = non-linear (quadratic)

Calibration fit [N/L/P*]:
Disable post-run RT update [Y/N*]:
ISTD peak #:
ISTD AMT [1.0000E+00 ]:
SAMPLE AMT [0.0000E+00 ]:
MUL FACTOR [1.0000E+00 ]:
```

[OP ()] [-] [3] [ENTER].

PREP CALIB ENTER
E = ESTD
I = ISTD
N = NORM

CALIB PROCEDURE (N/E*/I): ENTER

REF % RTW [5.000] : + = %
NON-REF % RTW [5.000] : - = ABSOLUTE
RF BASED ON AREA OR HEIGHT [A*/H] : ENTER

CAL#	RT	AMT	NAME
1:	XX.XXX	XXXXX	16A

ENTER

REF PK CAL #:
ISTD CAL #:
GROUP PEAKS [Y/N*] : Y ENTER

GRP #	CAL #	NAME
1:	(up to 63#'s)	16A

CALIBRATION OPTIONS:

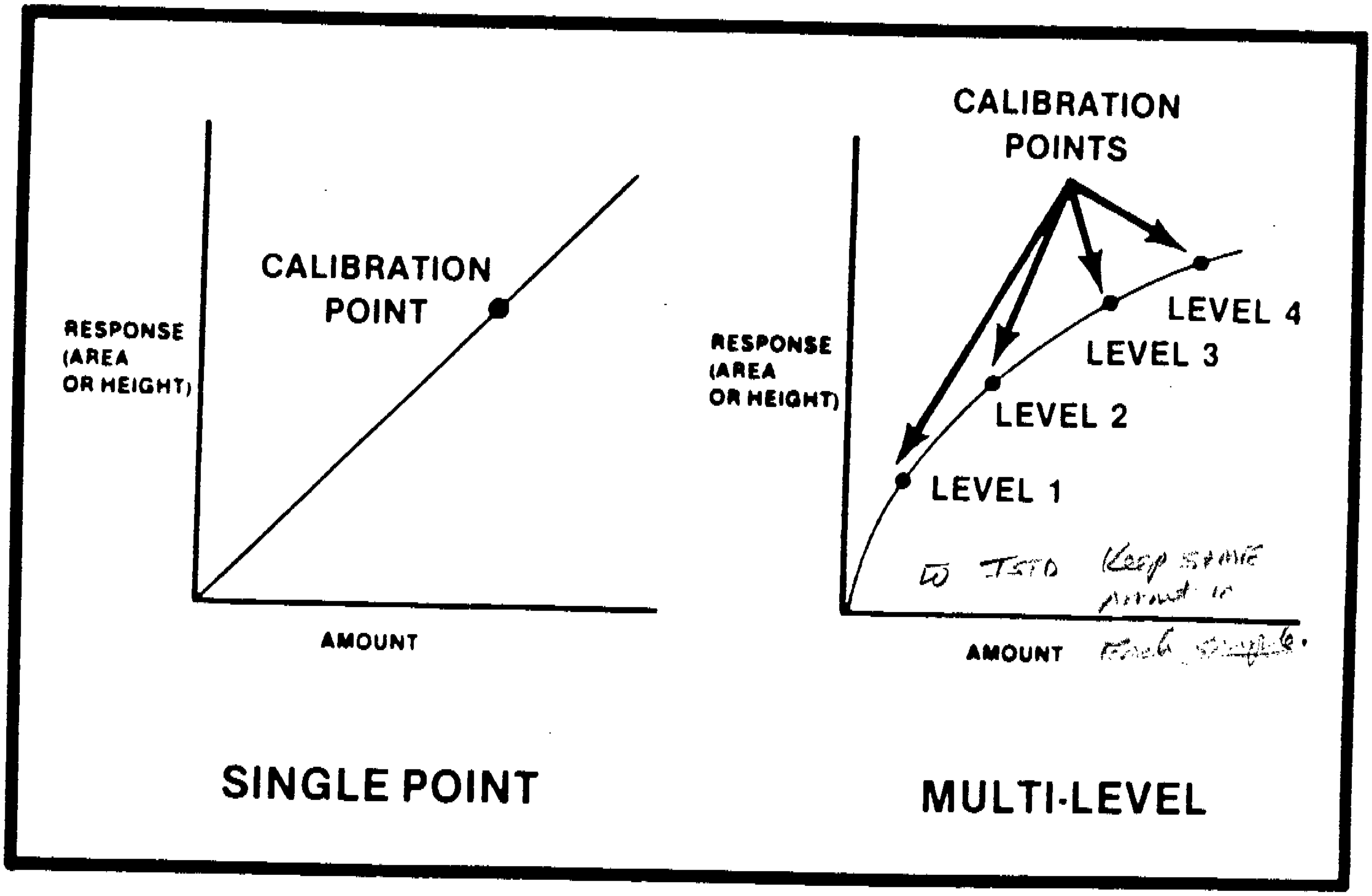
RF of uncalibrated peaks [0.0000E+00] :
Replace calibration fit [Y/N*] :
P = Point-to-point
L = Linear (least square)
N = Non-linear (quadratic)

Calibration fit [N/L/P*] :

Disable port-run RT update [Y/N*] :

ISTD AMT:
SAMPLE AMT:
MUL FACTOR:

Single vs. Multilevel Calibration



[PREP] [CALIB] *level number* [ENTER]

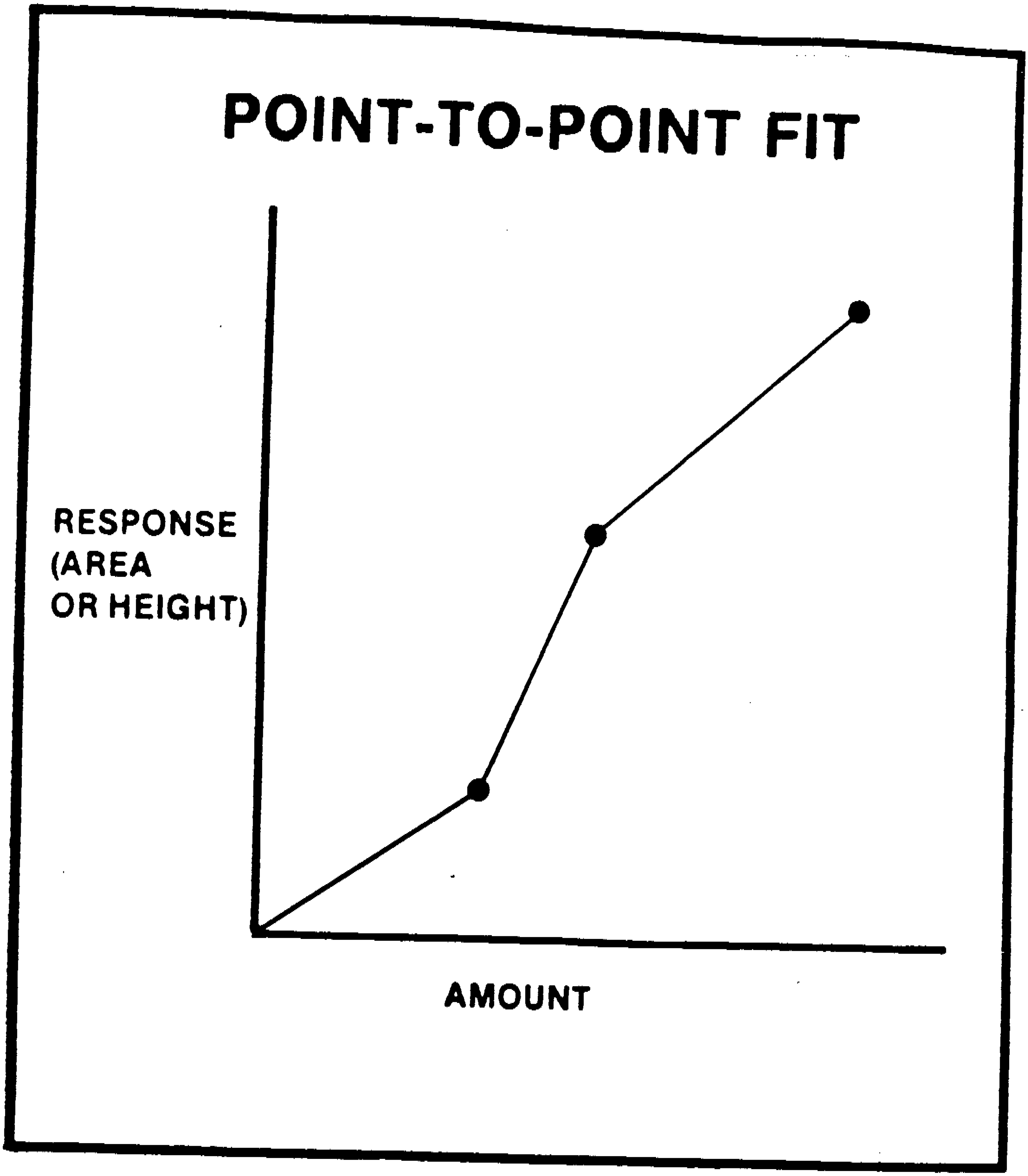
The HP 3393A responds

```
CAL#      RT        AMT
  1   :  rr.rrr  :
```

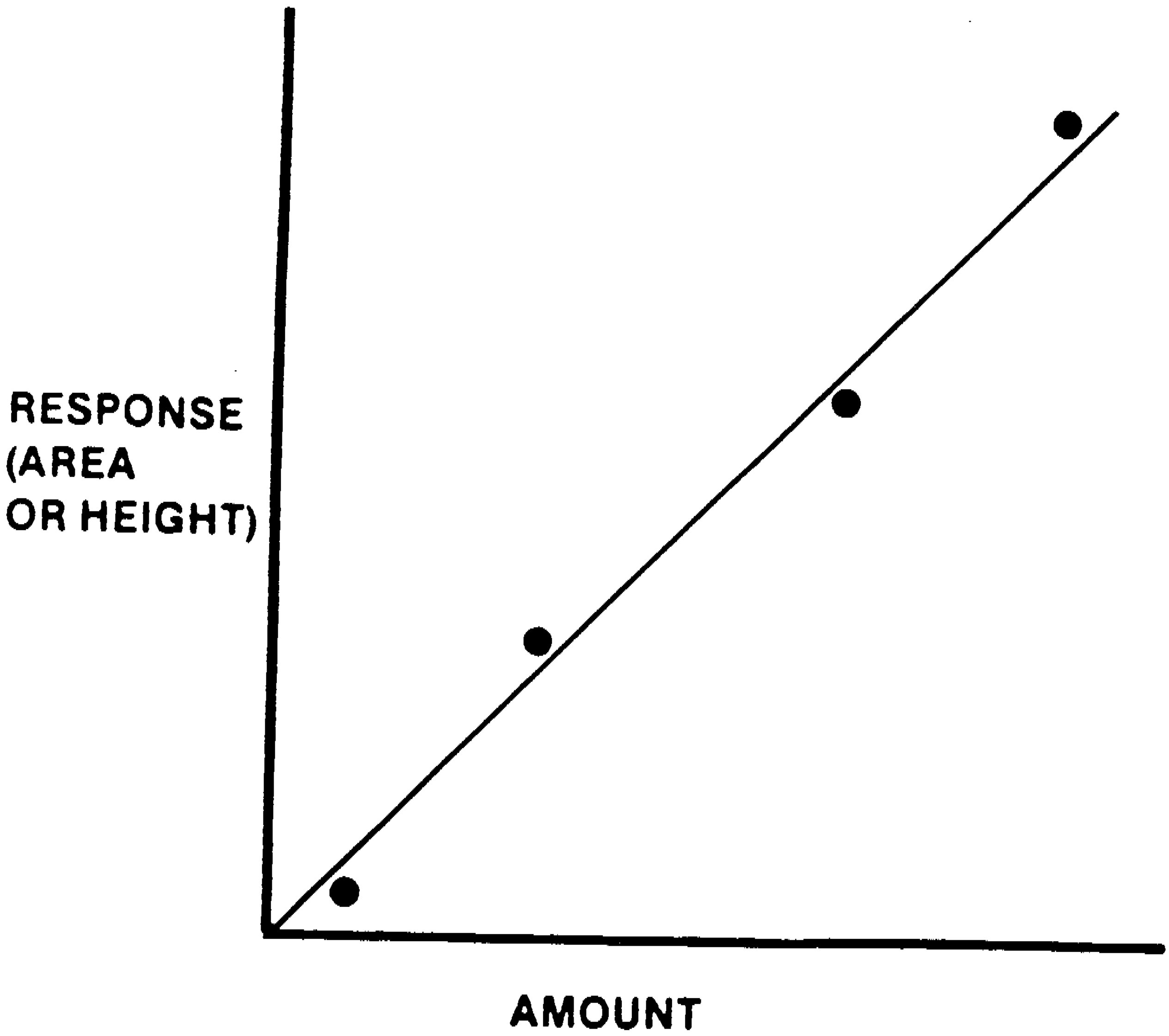
For ISTD calibrations, enter the ISTD amount for each level.

It must be the same for all levels.

Multilevel Curve Fits

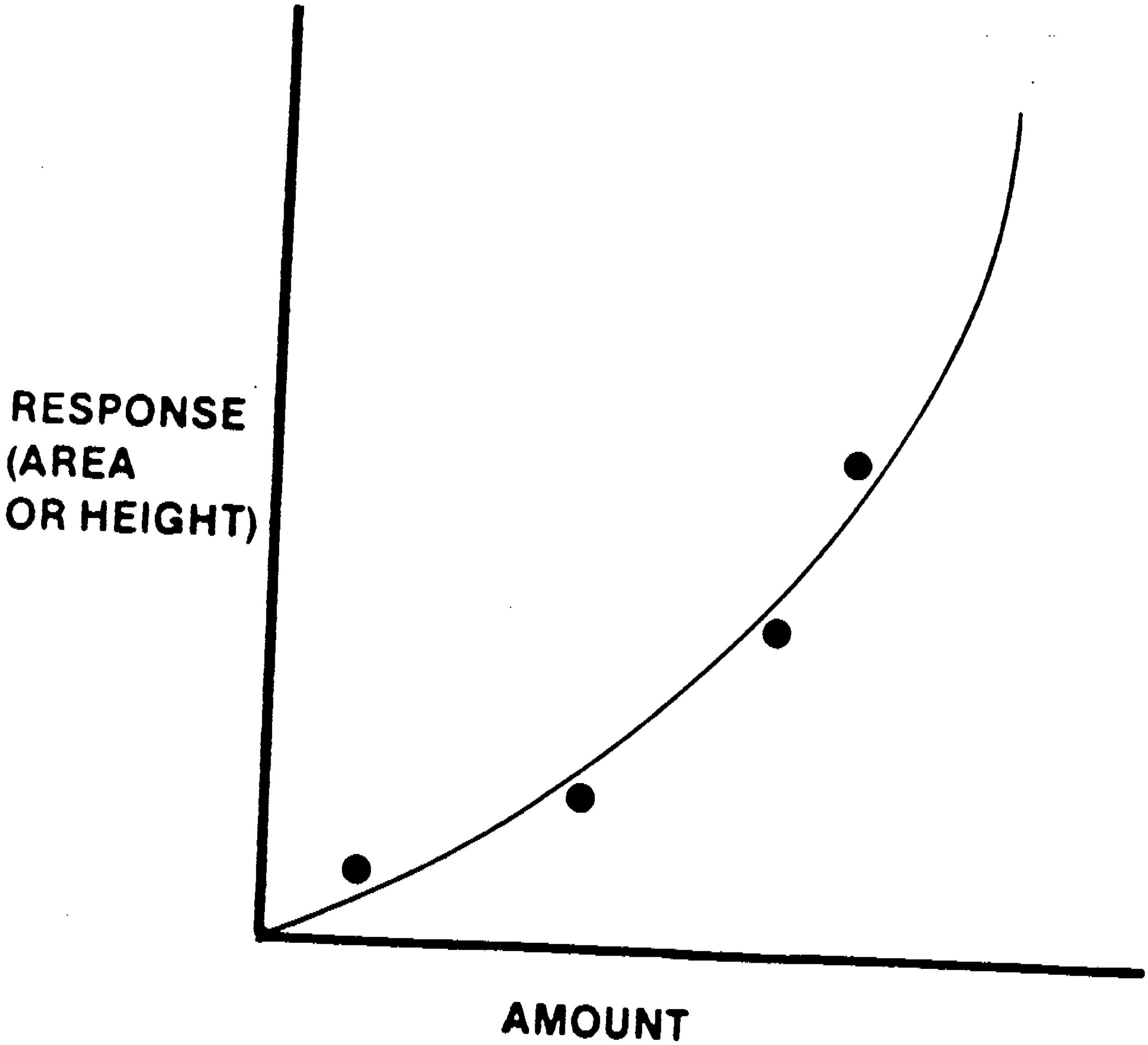


Multilevel Curve Fits



LINEAR REGRESSION

Multilevel Curve Fits



NONLINEAR CURVE FIT
(QUADRATIC)

RECALIBRATION

[CALIB] [ENTER]

- Averages response factors

'he formula used is:

$$ewRF = \frac{RunRF + (Old\ RECALIBRATIONS \times OldRF)}{Old\ RECALIBRATIONS + 1}$$

[CALIB] [–] [ENTER]

- Substitutes new response factors

LIST CALIBRATION FILE

(from **AWS**)

$\boxed{\text{LIST}}$ $\boxed{\text{CALIB}}$ $\boxed{\text{ENTER}}$

LIST CALIBRATION FILE

(from DISC)

$\boxed{\text{LIST}}$ $\boxed{\text{CALIB}}$ filespec $\boxed{\text{ENTER}}$

EDITING CALIBRATIONS

[EDIT] [CALIB] { *level number* } [ENTER]

```
*   EDIT CALIB    @

1 = CALIB PROCEDURE
2 = RETENTION TIME WINDOWS
3 = TABLE ENTRIES
4 = PEAK GROUPS
5 = CALIB OPTIONS

SECTION TO BE EDITED:
```

STORING AND RETRIEVING CALIBRATION FILES

[STORE] [CALIB] *filespec* [ENTER]

[LOAD] [CALIB] *filespec* [ENTER]

extension .CAL

DELETING LEVELS OR ENTIRE CALIBRATION FILES

[DEL] [CALIB] { *filespec* } [ENTER]

DELETE ALL [Y/N*]:

[DEL] [CALIB] { *level number* } [ENTER]

LABORATORY

EXERCISES

LABORATORY EXERCISE 2

INTEGRATION AND REINTEGRATION

PART 1:

1. Enter the following setpoints into the 5890A in preparation for running Sample #3:

   OVEN TEMP 50
   INITIAL TIME 0
   RATE 10
   FINAL TEMP 150
   FINAL TIME 0

   FID DET. TEMP 300
   TCD DET. TEMP 250
   INJ. A TEMP 200
   INJ. B TEMP 200
   SIG 1 to appropriate DET. SIGNAL

2. Enter the following setpoints into the 3393A in preparation for running Sample #3:

   ZERO 10
   CHART SPEED 1
   ATTN 2^ -5 (TCD)
   ATTN 2^ 8 (FID)

   PK WD 0.04
   THRSH -4
   AR REJ 0

3. Select the Plotting format through OP# 1.
   Enter the keycode **OP# 1 ENTER**
   Select a SOURCE plot by responding with PLOT TYPE S

4. Select a Storage Device for storing the data from your run by entering the keycode **OP# 2 ENTER**
   In response to the questions enter the following:

   Store signal data [Y/N*]: Y
   Device [M*]: A
   Bunched or raw data [B/R*]: R
   Store processed peaks [Y/N*]: N

5. Enable START/STOP marks on the peaks by activating Integration Function #8 at time 0 through entering the following keycode:

   TIME 0 IF() 8 ENTER

6. Inject 1 uL of sample #3 and start the run.
   At the end of the run, inspect the chromatogram for gross errors, rerun if necessary. Check with your instructor.

PART 2:

1. In order to replot and reintegrate your data, you need to determine how it is stored on the disc. To find your data file on the A disc drive, call up the display of the directory by entering the system command, **DIR A:**
   Determine which file has stored your raw signal data.

2. Note: Be prepared to start the stopwatch on the 5890A.

Type in the system command **ANALYZE {signal.RAW} ENTER** and start the stopwatch as the replot and reintegration begins. When the process is complete note the elapsed time and compare with the original run time. Discuss this with the instructor.

PART 3:

1. Change OP #1 from SOURCE to FILTERED plot by entering

OP# 1 ENTER

and responding with **F** for filtered plot.

2. Type in **ANALYZE** again and time the replot again.

   Note that this affects only the visual presentation, not the integration.

3. Is the time of the replot different?

4. Look at **Dir A: ; Dir M:**

PART 4:

1. Change OP #2 to indicate the storage of BUNCHED instead of RAW data. Do not change anything else.

2. Enter the **ANALYZE** command and again time the replot/reintegrate.

   Look at the report. Did anything change?
   Does this only change the plot presentation or does it also change the integration?

3. Look at **Dir A: ; Dir M:**

PART 5:

1. Enter OP #2 and prepare to store processed peaks.

2. Type in the command **ANALYZE**, start the timer, and compare the re-plot time, report, and visual presentation to the previous ones.

3. Relist **Dir A:** and **Dir M:** .

   What is being added?

   Have you determined the **filespec** naming code?

PART 6:

1. Call up OP #2 one more time. Now include the storage of EXTENDED peaks.

2. Enter **ANALYZE**, start the timer, compare replot times, and report and visual display.

3. Relist **Dir A: ; Dir M:**
What has been added?

PART 7: (OPTIONAL)

1. Look at the last report which was generated and observe the PEAK WIDTH column. Now set up the following timetable:

   TIME 0 INTG # 8 ENTER (Start/Stop Marks enabled)
   TIME 0 PK WD 0.01 ENTER
   TIME 10 PK WD 0.50 ENTER

2. Type in the command **ANALYZE.**
Look at the effect of the changing PEAK WIDTH on the plot and the integration report.

3. Set in the smallest PK WD setting at TIME 0 and the largerst PK WD setting at TIME 10. **ANALYZE** and note changes in plot and integration.

4. Manually enter the largest PK WD setting from the 3393A keyboard.

   Delete the TIMETABLE by entering the keycode **DELETE TIME ENTER**

   Reenter the command to enable START/STOP MARKS on the chromatogram.

   ANALYZE and observe the results.

   What is actually happeneing to the signal to cause the plot to appear this way?

   Which PEAK WIDTH setting should be optimum for this sample and this column? How could we determine this by looking at the reports?

   Replot the sample at optimum PEAK WIDTH setting.

PART 8:

1. Repeat the steps outlined in PART 7 changing the THRESHOLD setting instead of the PEAK WIDTH setting as you progress through the exercise.

2. Now make several replots in which you change ATTENUATION instead of THRESHOLD. Note the effect on the visual presentation and the report.

3. Finally, make several replots in which you change the AREA REJECT setting instead of THRESHOLD. What difference does this cause in the visual presentation and in the report.

LABORATORY EXERCISE 3

TIMED EVENTS

PART 1:

OBJECTIVE: To learn to change the attenuation automatically during a ru

1. Enter the following conditions into the HP5890A GC:

OVEN TEMP 50	FID DET. TEMP 350
INITIAL TIME 0	TCD DET. TEMP 250
RATE 10	INJ A TEMP 200
FINAL TEMP 150	INJ B TEMP 200
FINAL TIME 0	

   Be certain the SIGNAL 1 is set appropriate for the detector in use.

2. Enter the following setpoints on the HP3393A Integrator:

ZERO 10	PK WD 0.04
CHART SPEED 1	THRSH -4
ATTN 2^ -5 (TCD)	AR REJ 0
ATTN 2^ 8 (FID)	

3. Select the Plotting format through OP #1.
   Enter the keycode **OP #1** **ENTER**
   Select PROCESSED PEAK DATA.

4. Enable START/STOP marks on the peaks by activating Integration Function #8 at time 0 through entering the following keycode:

   TIME 0 IF() 8 ENTER

5. Select a Storage Device for storing the data from your run by entering the keycode **OP #2 ENTER**
   Select the **A** Disc and store procedded peaks.

PART 2:

1. Inject 1 ul of sample #2. Observe the chart. As the printhead reac the valley between the octanol-undecane peaks, press

LIST TIME ENTER

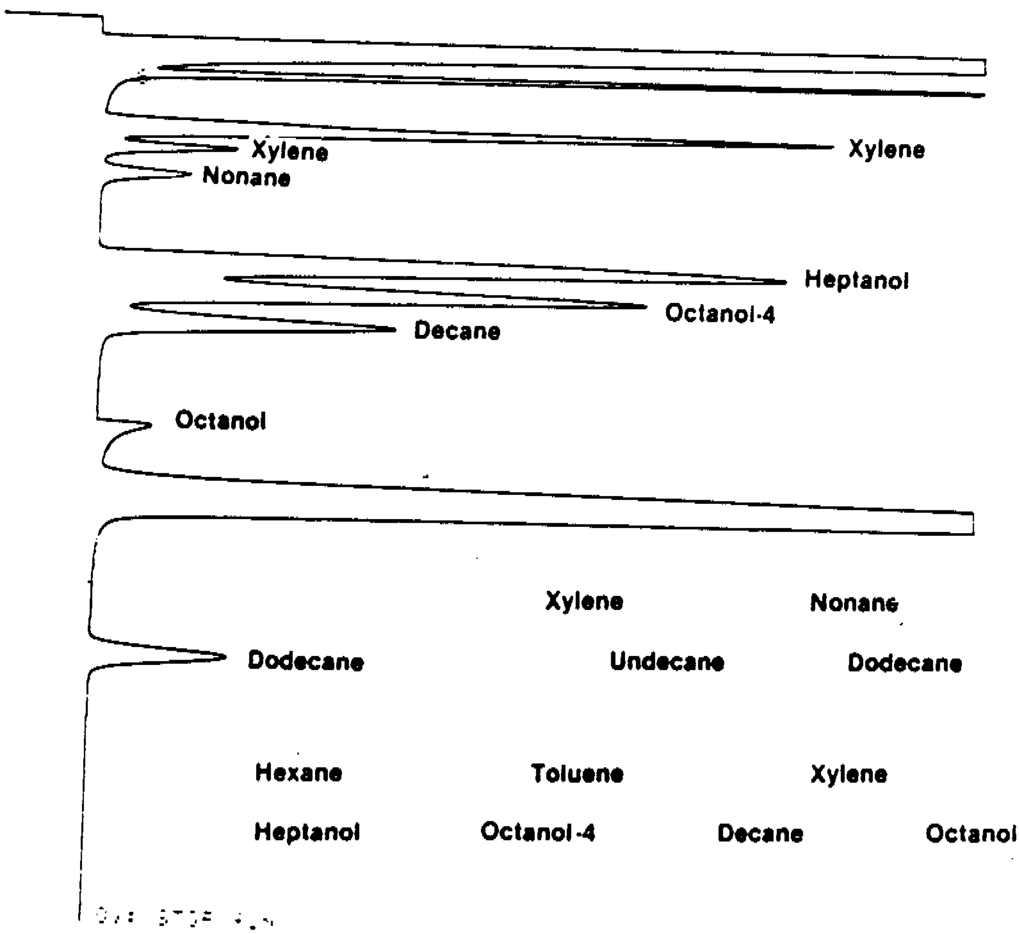
2. Repeat this step between the undecane-dodecane peaks.

3. Enter a **TIME TABLE** to change the attenuation at these two times so undecane peak apex will be on-scale and the dodecane peak larger.

4. Enter a **TIME STOP** to terminate the run just after the dodecane peak

5. Enter the **ANALYZE filespec** Command to reintegrate the previous run and act upon it with the newly created time table.

6. Delete the timetable from **AWS.**



:

IVE: To learn to use integrator functions to change the integrati
during a run.

tain the processed peak data for sample #3 stored during Laborator
ercise 2.

ter appropriate keycodes to:

force a tangent skim integration of the heptanol/decane peaks.
force a tangent skim integration of the cresol/undecane peaks.
reject the solvent peak.

integrate the data and compare the results of peak areas with the
iginal data.

peat the required manipulations to cancel the tangent skimming per
r the solvent peak. Compare the solvent peak and the toluene area
d without tangent skimming.

d evaluate

r sample #
that the s
cise 3, Pa

ion table
ssing

numeric

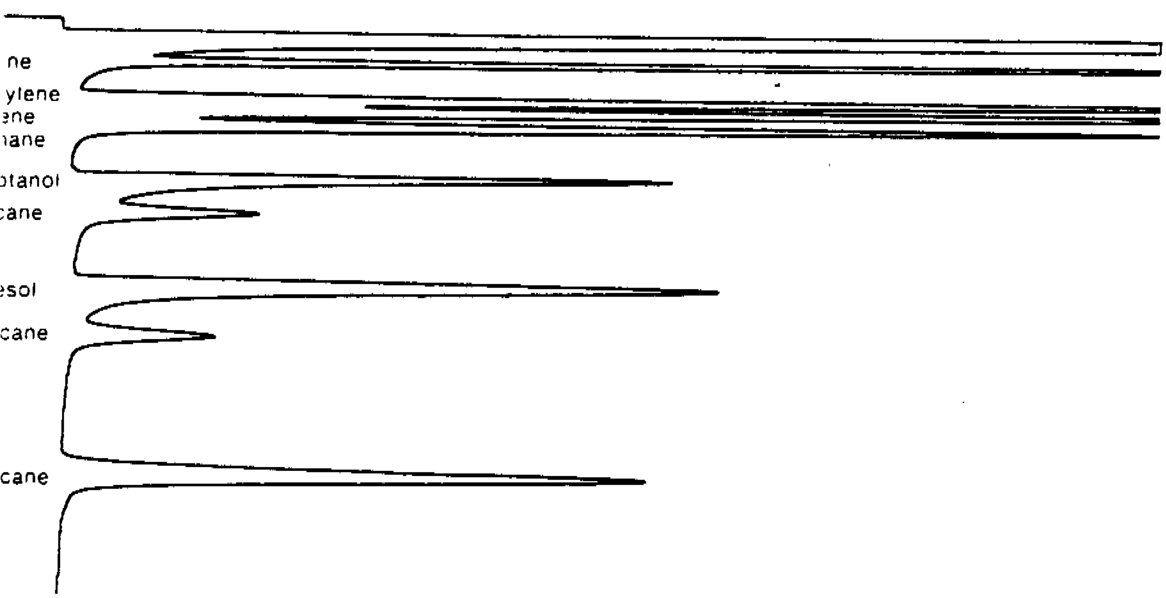
lows.

table.

ure

ie unknown

prior to t
icals.
ke C9-C10
ak.



:ess **EDIT CALIB ENTER** so that this calculated response factor can
: entered. This data will be entered under Section 3, Table Entri-

:ess **REPORT ENTER** to obtain a report with data on the uncalibrate
:ak. Use time events to eliminate the solvent and any other undesi
:aks.

ıve the calibration table and the data for the unknown run on the
.sc on A.

:

w critical is it to inject the same volumes of claibration mixture
ımple in this procedure?

: (OPTIONAL)

:integrate the original processed peak data for sample #3 and estab
calibration table for EXTERNAL STANDARD ANALYSIS.

:integrate the processed peak data for sample #4 and obtain an EXTE
:ANDARD REPORT for the run.

ıve the EXTERNAL STANDARD Calibration table on your disc.

: (OPTIONAL)

:integrate the original processed peak data for sample #3 and estab
calibration table for INTERNAL STANDARD ANALYSIS. Designate C10 a
ıe INTERNAL STANDARD PEAK.

:integrate the processed peak data for sample #4 and obtain an INTE
:ANDARD REPORT for the run.

ıve the INTERNAL STANDARD Calibration table on your disc.

LABORATORY

EXERCISES

LAB 1-----FAMILIARIZE

OBJECTIVE
To familiarize you with the layout of the HP 3393A keyboard and a few new features. After completing this lab, you should know how to:

*list run parameters, integrator functions, options, and methods
*use run parameters knowing their new bounds
*use alpha-numeric keys for typing in information
*use system commands
*access discs available to the system
*enter and exit BASIC mode

FUNCTION KEYS

On the HP 3393A, function keys are across the top row and down the rightmost column. Keywords written in yellow across the top are acessed by pressing the key below it while pressing the shift key.

EXERCISE 1.1

Try the following key sequences:

[LIST] [LIST]
[LIST] [INTG()] [ENTER]
[LIST] <u>[SHIFT] + [OP()]</u> [ENTER]
[LIST] <u>[SHIFT] + [METH]</u> [ENTER]

EXERCISE 1.2

Try the following key sequences:

[ATT 2^] (36) [ENTER]

This is the upper bound for attenuation, which turns off the signal.

[PK WD] {.03} [ENTER]
[LIST] [PK WD]

[PK WD] {.15} [ENTER]
[LIST] [PK WD]

[PK WD] {2.56} [ENTER] ? {2.5} [ENTER]
[LIST] [PK WD]

The HP 3393A uses whatever peak width value is entered. The upper bound on peak width is 2.5.

[THRSH] {28} [ENTER]

The upper bound on threshold is 28. It is large to accommodate the $\sim 10^9$ dynamic range over INET.

SET ALL THE PARAMETERS BACK TO THEIR ORIGINAL STATE:

[DEL] [METH] [ENTER]

This is the default method; loading it restores all variables to their power-on state.

ALPHA-NUMERIC KEYS

One of the most important features on the HP 3393A is the alpha-numeric keyboard. Throughout this tutorial you will learn to use alpha-numeric keys to name and control parameters. Here you will be introduced to the system commands. These are commands which interact with the integrator and other instruments on the loop to perform certain tasks other than those provided by the function keys.

EXERCISE 1.3
TRY THESE SYSTEM COMMANDS:

Type *NOTEPAD* [ENTER]. This puts the integrator in a mode where it acts like a typewriter. (NOTEPAD can be abbreviated by *N* .)

Type in a few sentences to get used to the keyboard.

Typing [CTRL] + [C] switches back and forth between capital and small letters.

The backspace key [bksp] erases the previous character and makes a box on the paper. If you do alot of backspacing in a line, and want to see what it looks like, press [CTRL] + [R] and the line is printed without the backspace marks.

Once you feel comfortable using the keyboard, press [BREAK] to return to system mode (the prompt is an "*").

Type *DATE* [ENTER]

The date is printed out, followed by the time. (Abbreviation is *DA*)

Type *DA* (MM/DD/YY) [ENTER]

The correct date is printed out along with the time.

Type *TIME* [ENTER] (Abbreviation is *TI*)

The time is printed.

Type *TI* (HH:MM:SS) [ENTER]

The correct date and time are printed.

Type *SET RUNNUM* {3} [ENTER]

This command sets the HP 3393A run number.

Type *SYSTEM* [ENTER]

This gives you the loop configuration and RS-232-C switch settings. (Abbreviation is *SY*)

Type *INET_CONFIG* [ENTER] (The _ is [SHIFT] + [-])

This gives the INET configuration for devices on the loop. If there are no INET devices (a disc drive is not an INET device), it gives a "Loop is down" message. (Abbreviation is *IN*)

Type *READY* [ENTER]

The system should respond with "System is Ready". (Abbreviation is *REA*) This means that all instruments are up and the system is ready to acquire data.

Type *DIRECTORY* [ENTER]

This command gives information about file storage and files stored on a certain disc. Since no disc was specified and the default drive is M, the directory gives information about the integrator's internal disc. You should have one file, "SIG_BUFF.RAW", of length 2048 bytes. (Abbreviation is *DIR*)

Type *DIR A:* [ENTER]

This allows you to list the directory for the A disc drive. **The drive name must be followed by a colon, otherwise the HP 3393A looks for a file on the M disc drive.** Depending on how many discs you have, you can use this command with a letter corresponding to each disc drive to find out information about what's on a disc. Your disc should have the volume name "VIV_D5" and contain several files.

Type *BASIC* [ENTER]

This command exits system mode, and enters BASIC mode. The prompt for BASIC mode is ">". At this point you can enter, run, edit, or delete a BASIC program. (Abbreviation is *BA*)

Type *EXIT* [ENTER]

This exits BASIC mode and returns to system mode. System mode is indicated by the "*" prompt. (Abbreviation is *E*)

REVIEW QUESTIONS

1. What is the upper bound on attenuation? 36

   Peak width? 2.5

   Threshold? 25

2. How do you load the default method?

   Clear .6/ Def. [illegible]

   [illegible]

3. What command prints the loop configuration?

   Configure System

4. How would you find out what was on the disc in the A drive?

DAY 3

INTEGRATION

and

REINTEGRATION

PLOT VS RUN MODE

Plot Mode	Presentation is of raw data similar to strip chart recorder.
Run Mode	Signal and plotted chromatogram are filtered. You see what the integrator sees.

	PLOT	RUN
Signal	Unfiltered	Filtered
Integration	No	Yes
Retention Times	No	Yes
Parameters Affecting Chart Presentation	Zero CHT SP ATT 2↑	Zero CHT SP ATT 2↑ PK WD THRSH

3393 INTEGRATION

* SAMPLE RUN (Real Time)

  [START/STOP]

* REINTEGRATION (Stored Data)

  [ANALYZE]

REAL TIME SIGNAL

ANALOG

INET

FILTERING and PROCESSING the SIGNAL

* RAW DATA (Area slices, 20 hertz)
* BUNCHED DATA (Wider Area Slices) — Set By PkWD
* PROCESSED PEAK DATA (Cardinal Points)

  ↳ only 5 points taken

SET UP THE 3393 FOR REAL-TIME INTEGRATION

```
*  LIST: LIST
PEAK CAPACITY:  1244

ZERO   =  0, -11.179
ATT 2^ =  0
CHT SP =  1.0
AR REJ =  0
THRSH  =  0
PK WD  =  0.04
```

PEAK PARAMETERS

KEY (CODE)	Function
PK WD (PK)	Sets data sampling rate and signal filtering for plot and integration
THRSH (TH)	Sets noise rejection for integration
AR REJ (AR)	Sets peak rejection by final area

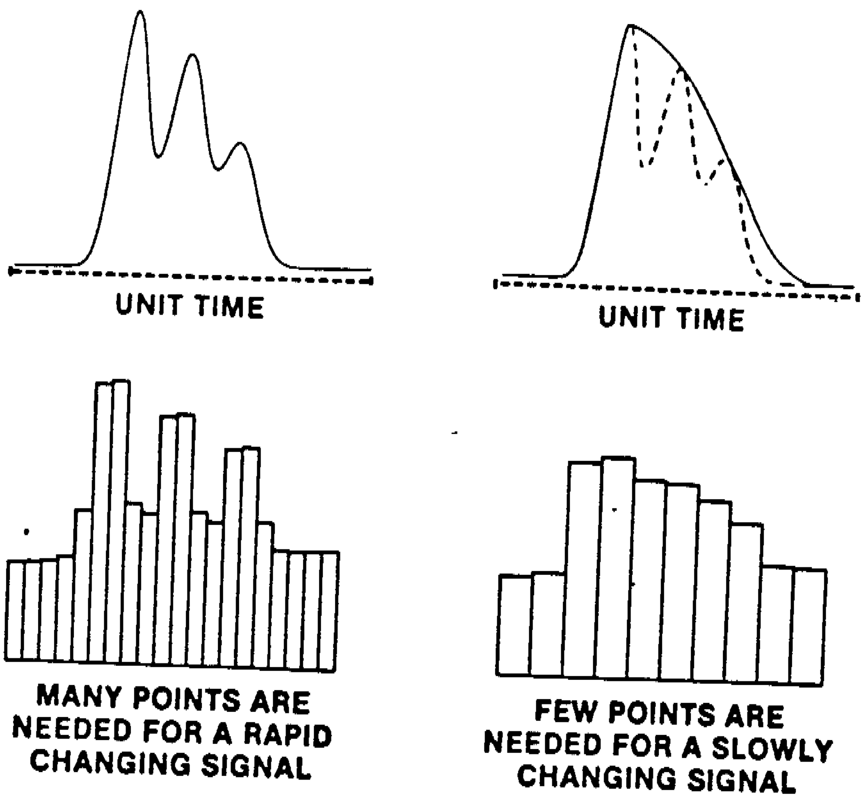
when Attn - Thr = 5% of scale

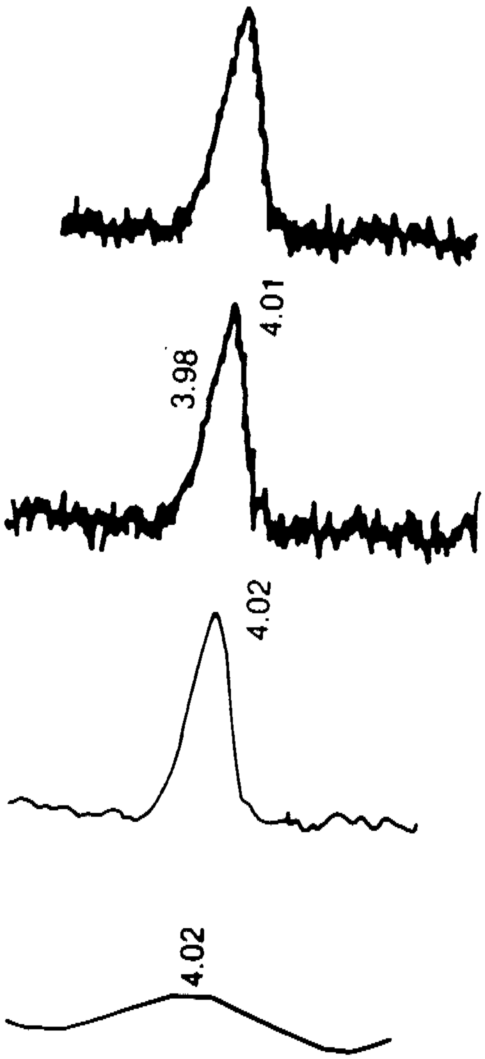
Run TK [illegible] low Attn [illegible]

Peak Width Matches rate of change of the incoming signal to the sampling rate of the integrator.

Controls selectivity in distinguishing peaks from noise by filtering the apparent signal-to-noise ratio.

CHOOSING THE SAMPLING RATE





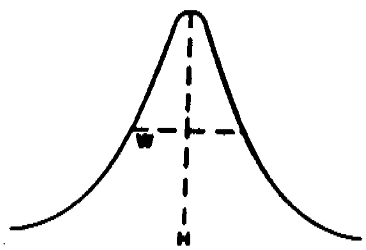
PLOT MODE

PK WD .01

PK WD .04

PK WD .64

DETERMINING PEAK WIDTH



Peak width at half-height

Range available (minutes)

.01 – 2.50

CONFIRMING AN APPROPRIATE WIDTH

RUN# 3 JAN 1, 1901 00:48:37

AREA%

RT	AREA	TYPE	WIDTH	AREA%
.100	11	PV	.013	.00000
.251	247	VV	.016	.00001
.271	603	VV	.040	.00002
.442	1691095	PB	.014	.05515
.523	40030	BH	.009	.00131
.546	3.061E+09	SHB	.096	99.82784
1.946	1402851	BV	.080	.04575
2.230	7995	VP	.113	.00026
2.533	2137181 I	PP	.086	.06970

TOTAL AREA=3.0661E+09
MUL FACTOR=1.0000E+00

Threshold	Controls how much signal information the integrator looks at before making decisions about a peak.

DETERMINING THRESHOLD

- Start with an Autothresh test: THRSH ENTER
- Time period for the test dependent on peak width (4 secs. – 41/2 min.)
- Autothresh value is a starting point.

ENTERING THRESHOLD VALUES

[THRSH] ***threshold*** **[ENTER]**

[LIST] [THRSH]

USING AUTO-THRESHOLD

[THRSH] [ENTER]

HP 3393A in:

- o its READY state (READY LED on)
- o Integrate Mode
- o Plot Mode
- o a reintegration

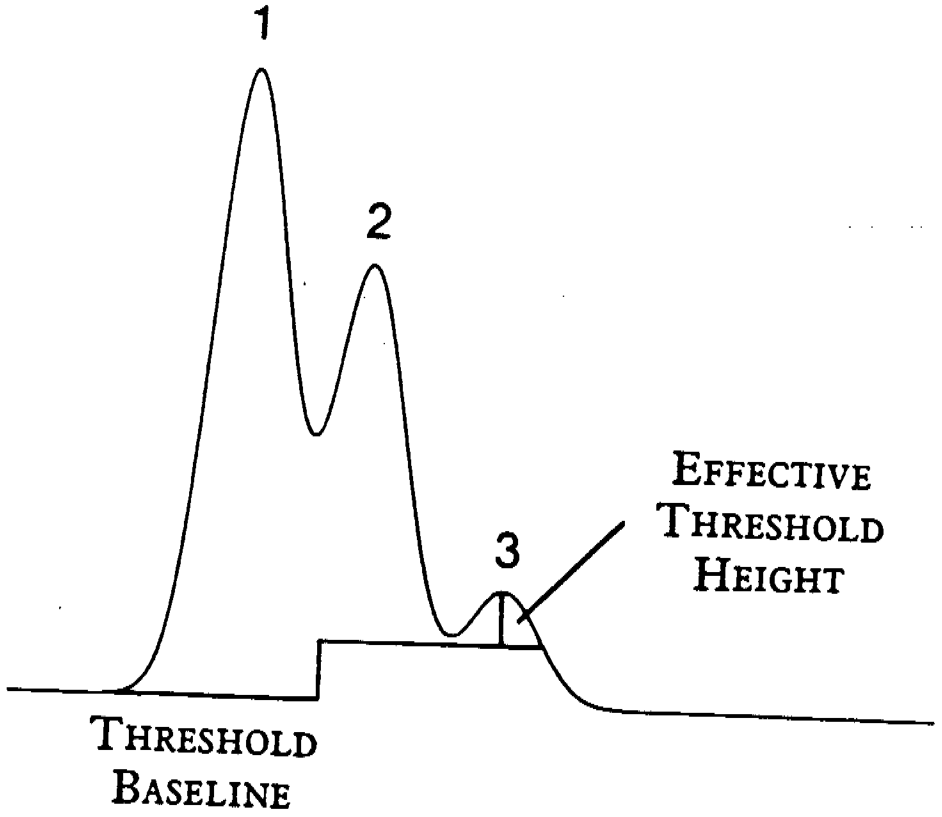
[LIST] [THRSH]

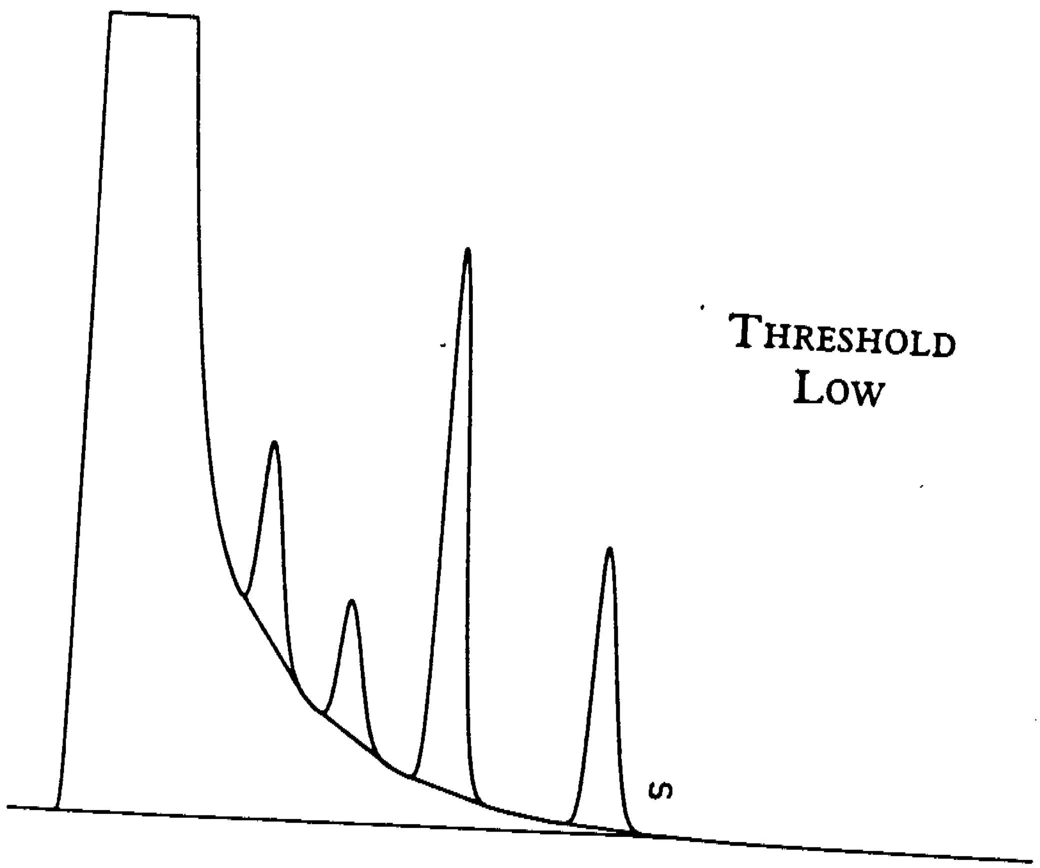
[THRSH] [-] [ENTER]

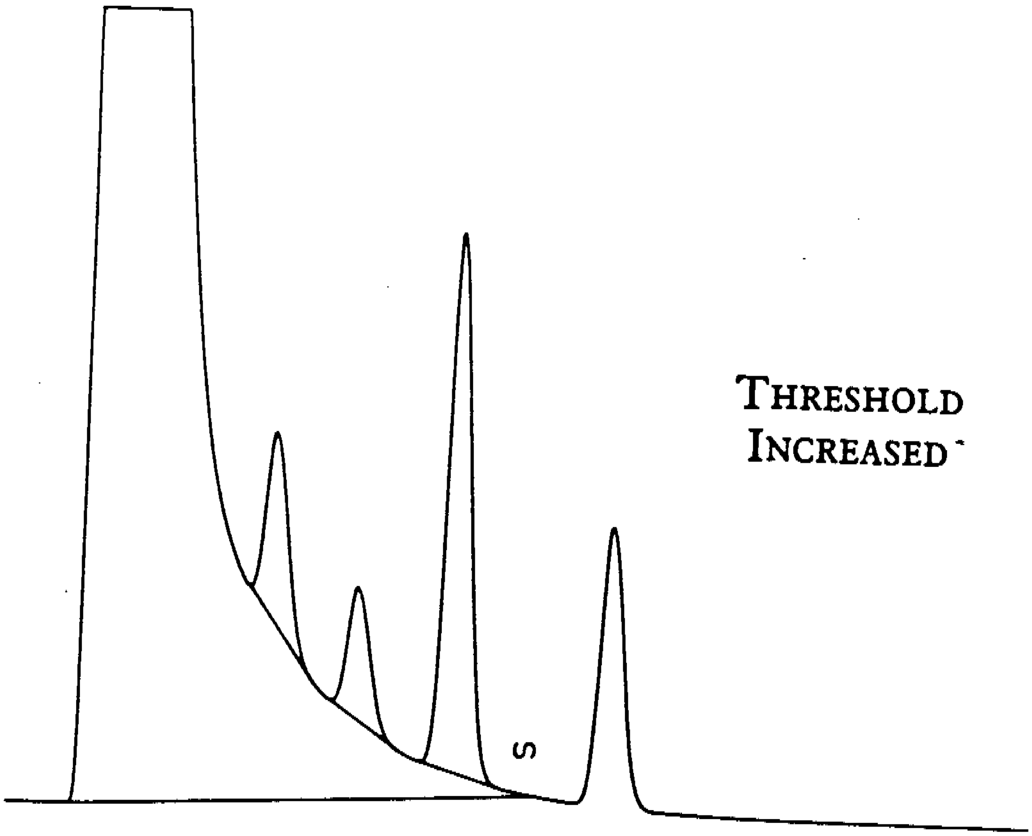
SET THRESHOLD DURING A RUN

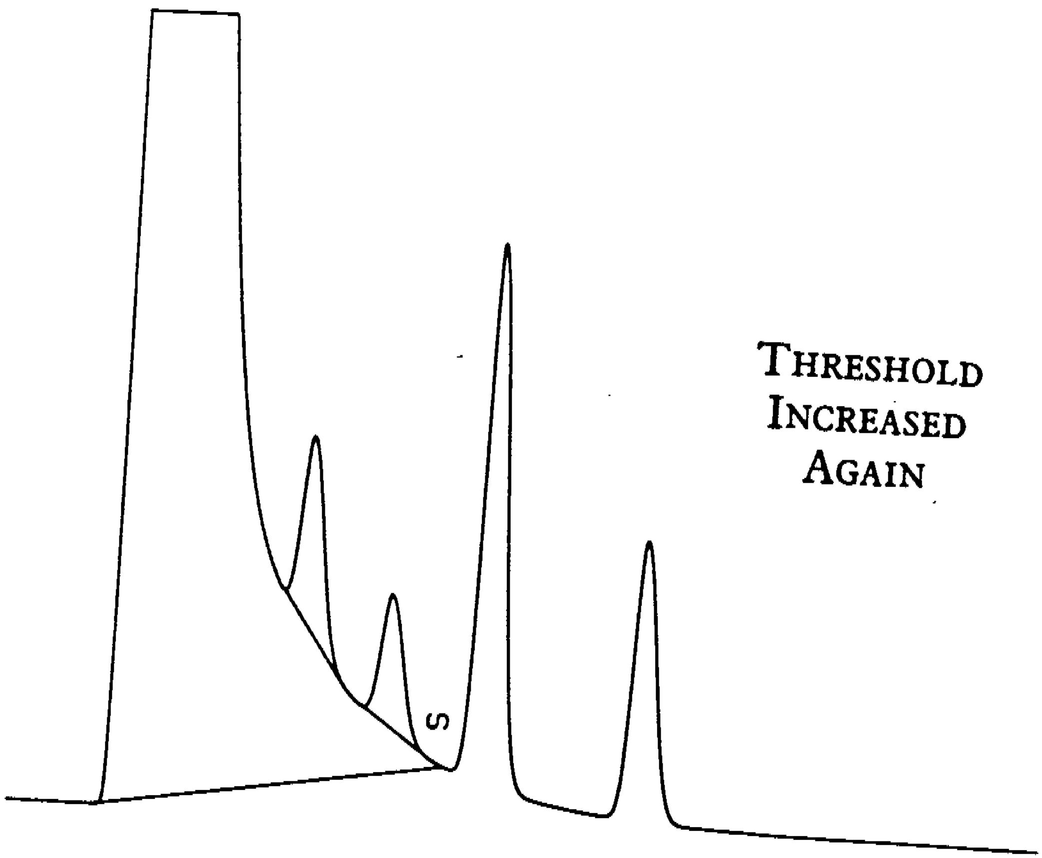
* LIST: INTG # 6 @
MEASURE AND UPDATE THRESHOLD

* LIST: INTG # 10 @
INCREMENT THRESHOLD


1
2
3
EFFECTIVE
THRESHOLD
HEIGHT
THRESHOLD
BASELINE


THRESHOLD
LOW


S
THRESHOLD INCREASED


S
THRESHOLD INCREASED AGAIN

Setting Area Rejection

[AR REJ] *minimum area* [ENTER]

[AR REJ] [1] [E] [6] [ENTER]

[AR REJ] [1] [0] [0] [0] [0] [0] [0] [ENTER]

[LIST] [AR REJ]

SELECTING THE PLOTTING FORMAT: OP # 1

[OP ()] [1] [ENTER]

The HP 3393A responds:

```
*  OP # 1

INTEGRATION PLOT TYPE
(Source/Filtered/Unigram/No Plot)
ENTER PLOT TYPE [S/F*/U/N]:
```

[LIST] [OP ()] [1] [ENTER]

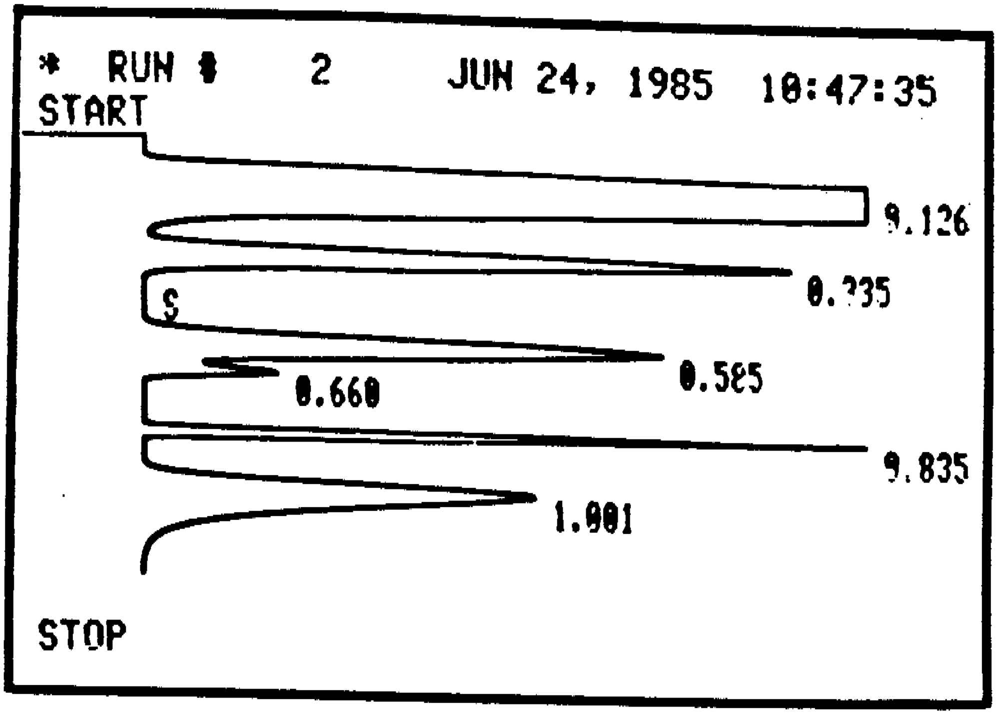
Storing Run Data: OP # 2

[OP ()] [2] [ENTER]

```
*  OP # 2

RUN DATA STORAGE
Store signal data [Y/N*]: Y
Device [M*]:
CAUTION: Previous signal data will be lost
Bunched or raw data [B/R*]: B
Store processed peaks [Y/N*]:
```

[OP ()] [-] [2] [ENTER]

A TYPICAL CHROMATOGRAM



A TYPICAL REPORT

RUN# 3 JAN 1, 1901 00:48:37

AREA%

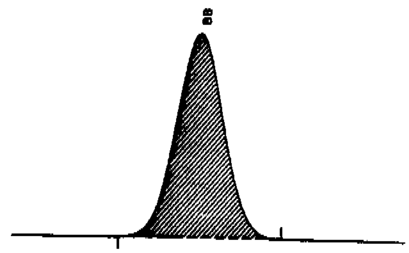
RT	AREA	TYPE	WIDTH	AREA%
.100	11	PV	.013	.00000
.251	247	VV	.016	.00001
.271	603	VV	.040	.00002
.442	1691095	PB	.014	.05515
.523	40030	BH	.009	.00131
.546	3.061E+09	SHB	.096	99.62784
1.946	1402851	BV	.080	.04575
2.230	7995	VP	.113	.00026
2.533	2137181 I	PP	.086	.06970

TOTAL AREA=3.0661E+09
MUL FACTOR=1.0000E+00

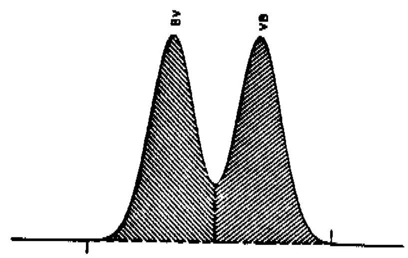
Peak Type Codes

Baseline Codes

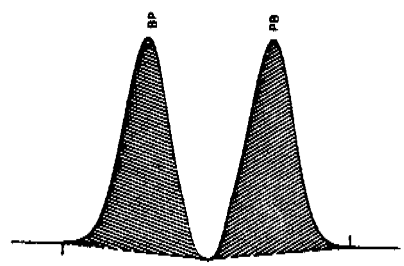
B — Baseline resolution



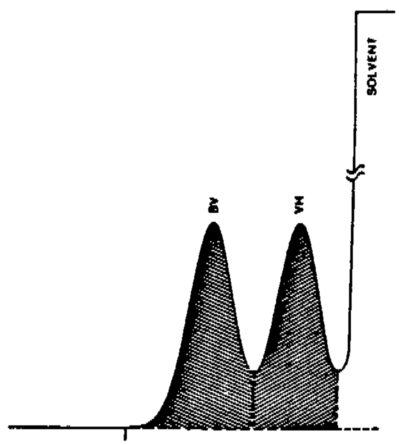
V — Valley point



P — Baseline penetrated, then reset



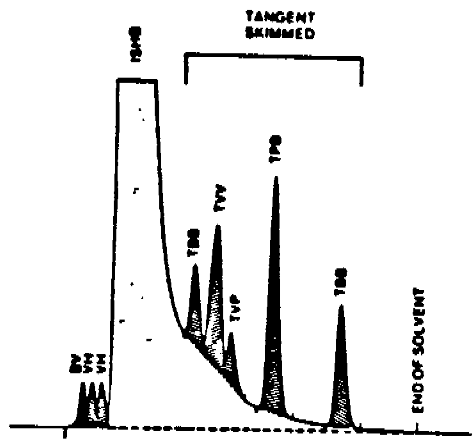
H — Horizontal baseline



PEAK TYPE CODES

SOLVENT CODES

S – Designated solvent
T – Tangent skimmed off the solvent



PEAK TYPE CODES
WARNING CODES

> — Peak over 1 volt

< — Peak under –10 mv

I — Incomplete – peak ended prematurely

D — Distorted – PK WD too narrow

THE REINTEGRATION PROCESS

1. Store Bunched or Raw Data

2. Load New Operating Parameters into AWS

3. Type Analyze [ENTER]

STARTING REINTEGRATION

DEFAULT FILE:

* ANALYZE [ENTER]
* ANALYZE I [ENTER]

STORED FILE:

A: "[illegible]". BNC

* ANALYZE filespec [ENTER]
* ANALYZE filespec, I [ENTER]

SAMPLE INFORMATION DURING REINTEGRATION

ORIGINAL: Stored with Signal Data

NEW:

a) Suppress Post–Reintegration Report with OP# 5

b) Enter New Sample Information with OP# 7

c) Press $\boxed{\text{REPORT}}$

or

$\boxed{\text{AREA\%}}$

* OP # 5

POST-RUN LIST OPTIONS
Store post-run report [Y/N*]: Y
Device [M*]:
HPTB - 1-7
External post-run report [Y/N*]: Y
Printer Address [0]:
List run parameters [Y/N*]: Y
List timetable [Y/N*]: Y
List calibration table [Y/N*]: Y
List remote method [Y/N*]: Y

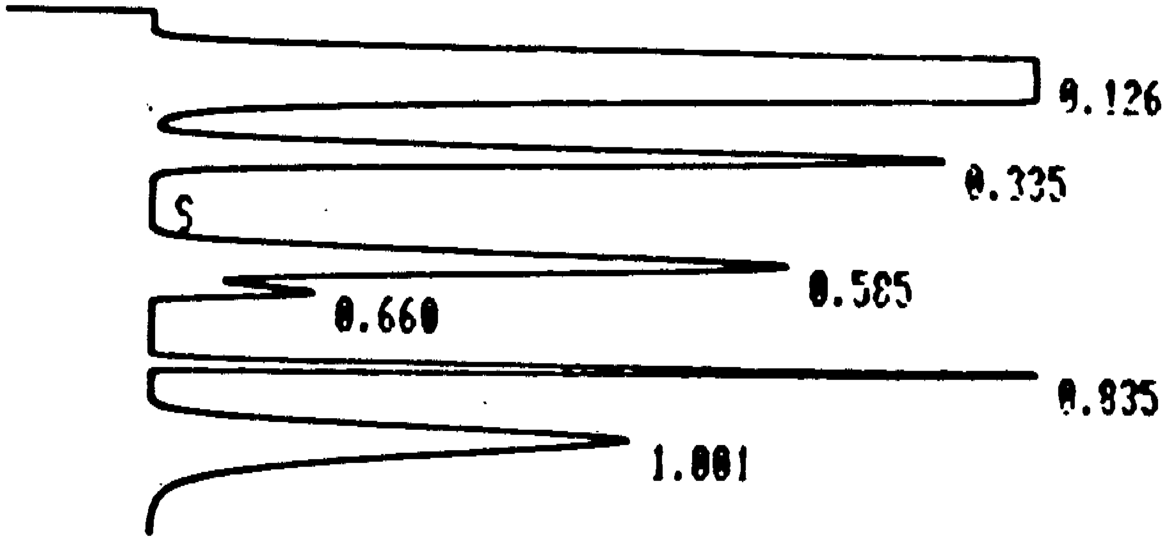
* OP # 7

DEFAULT SAMPLE INFORMATION
USE SAMPLE TABLE IN MANUAL RUN [Y/N*]:

ISTD AMT [0.0000E+00]:
SAMPLE AMT [0.0000E+00]:
MUL FACTOR [1.0000E+00]:
RECALIBRATION [Y/N*]:
NAME:
REPORT MEMO:

Chromatogram Produced by ANALYZE

```
*AN M:SIGNAL.RAW
RUN #    2      JUN 24, 1985  10:47:35
START
```




```
END OF SIGNAL
```

ENDING REINTEGRATION

END of SIGNAL

END of SIGNAL (Aborted Source)

TIMETABLE STOP

STOP

TIMED

EVENTS

TIME PROGRAM

— Run Parameters

— Intg( ) Keys

— Ext Keys

— Stop

CREATE THE TABLE

TIME VALUE FUNCTION VALUE ENTER

LIST TIME

DEL TIME

Time-programmable functions

	KEY	PRINTED CODE	SEE SECTION
RUN PARAMETERS	ZERO	ZE	4
	ATT 2↑	AT	4,6
	CHT SP	CS	4,6
	PK WD	PW	5
	THRSH	TH	5
	AR REJ	AR	5
INTEGRATION FUNCTIONS	INTG ( )	IF	7
STOP	STOP	ST	4,5

THE HP 3393A
&
EXTERNAL EVENTS

USE THE HP 19405A S/ECM FOR:

-4 TRIACS & 4 RELAYS
-BCD INPUTS
-HP 7671/72 CONTROL

EXT() Key: Valve/External Device Control

EXT() *contact#*	Operation	Default Value assigned at Power-On
Contact Closures:		
1	External device control	Open
2	"	"
3	"	"
4	"	"
115 VAC Triacs:		
5	External device control	Off
6	"	"
7	"	"
8	"	"

EXTERNAL EVENT CONTROL

LIST   EXT()   contact#   ENTER

IMMEDIATE EXECUTION

EXT()   contact#   ENTER   =   ON

EXT()   –   contact#   ENTER   =   OFF

TIME–PROGRAMMED   EXT()   COMMANDS

TIME   runtime   EXT()   contact#   ENTER

TIME   runtime   EXT()   –   contact#   ENTER

EXTERNAL EVENT TIME TABLE

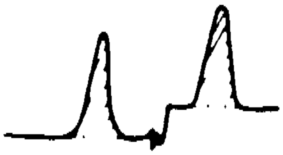
LIST   TIME   EXT()   ENTER

DEL   EXT()   ENTER

012589

INTEGRATOR FUNCTIONS

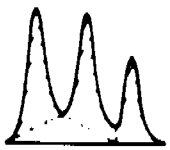
Customize your integration



0 SET BASELINE NOW

0 SET BL NOW

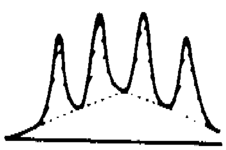
Set baseline immediately



1 SET BASELINE
NEXT VALLEY

1 SET BL NEXT VALLEY

Sets baseline at next valley after apex

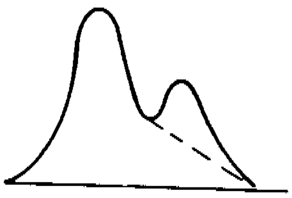


2 SET BASELINE
ALL VALLEYS

2 SET ALL VALLEYS

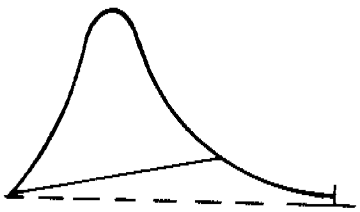
Set baseline at all valleys

INTEGRATOR FUNCTIONS



3 SKIM FROM NEXT PEAK

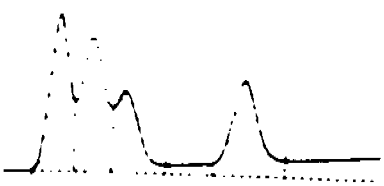
Forces a peak to be declared a solvent. Must be entered prior to apex.



If 3 also lets you extend the data collection for tailing peaks

4 DISABLE AUTO TAN SKIM

Disables auto tangent skimming



5 EXTEND BASELINE
HORIZONTALLY

5 EXTENDED BL HORIZONTALLY

Baseline is reset at penetration points. Not appropriate for ignoring negative deflections.

INTEGRATOR FUNCTIONS

6 MEASURE AND UPDATE THRESHOLD

The applied chromatographic signal or data is monitored, the noise measured, and an optimum threshold value is assigned.

7 DISABLE RT LABEL

Disables retention time labeling

8 TURN ON START/STOP MARKS

Turns on START/STOP marks on chromatogram

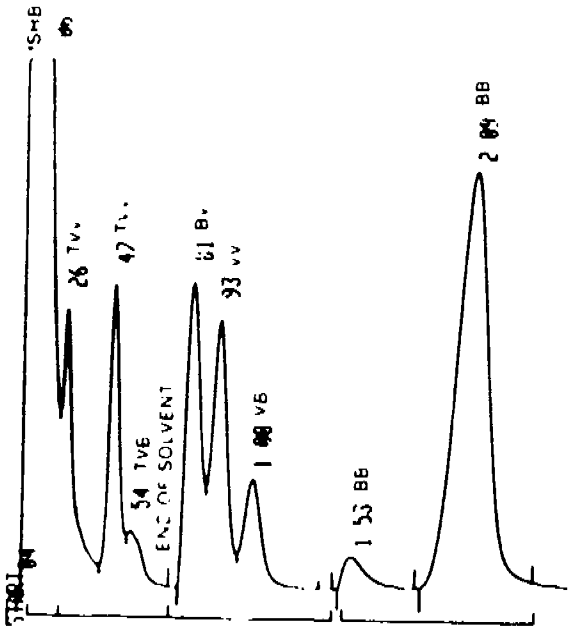
9 DISABLE INTEGRATOR

Inhibits intergration

10 INCREMENT THRESHOLD

Increments the threshold value

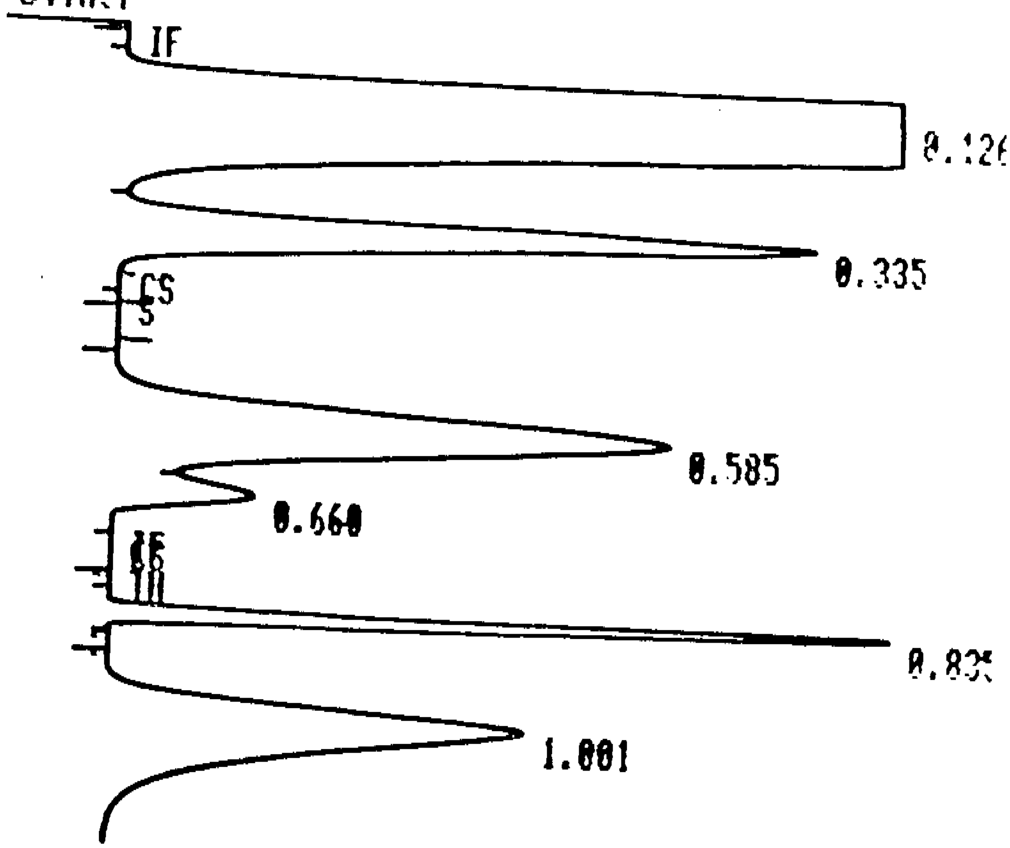
TICK MARKS



LONG —Peak stops or starts on a declared baseline

SHORT —Peak stops or starts at valley point between merged peaks, or tangent point along the tail of a peak declared a solvent

* RUN # 9 JUN 24, 1985 11:13:31



TIME TABLE STOP

Timed Events

* LIST: TIME

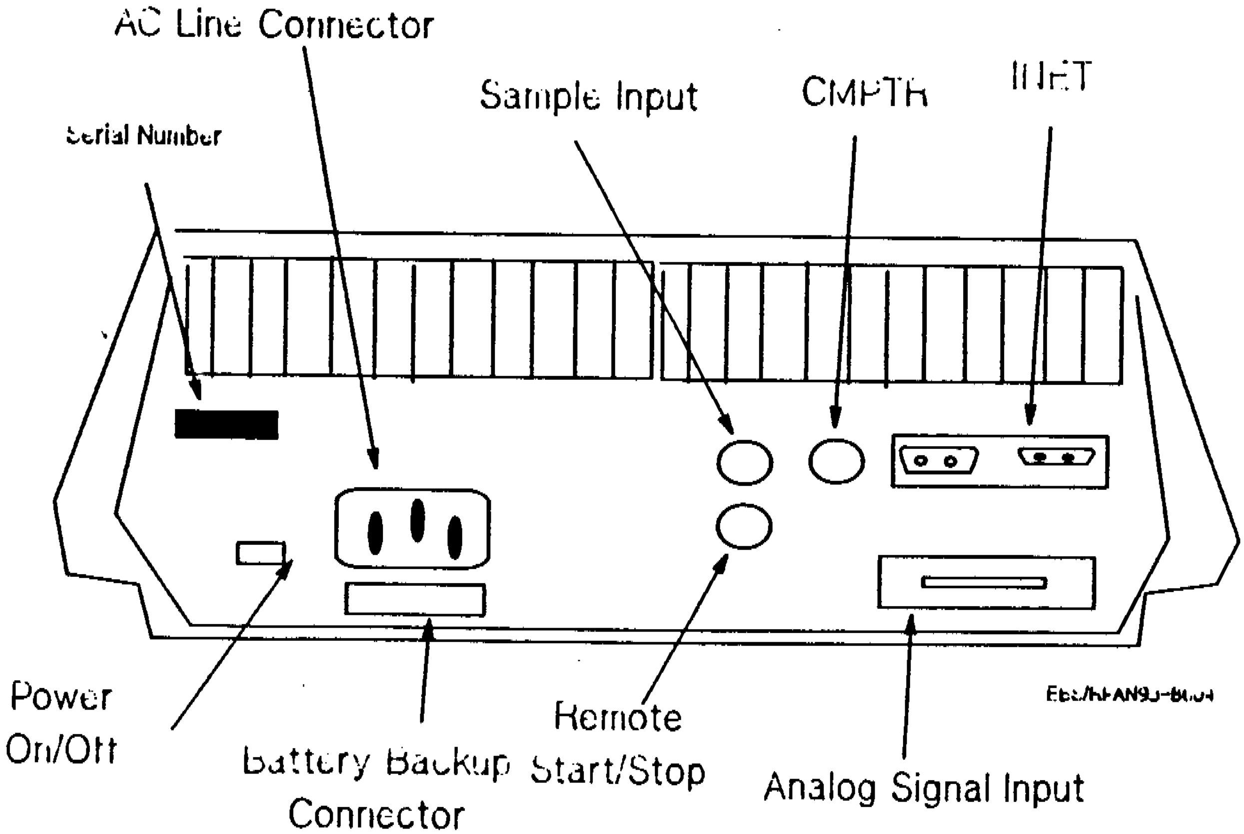
0.000 INTG # = 8
0.400 CHT SP = 7.0
0.700 INTG # = 6
0.710 CHT SP = 5.0
1.200 STOP

USING THE

PLOT

MODE

SIGNALS INTO AND OUT OF
THE HP 3393A



PLOT PARAMETERS

ATT 2↑ KEY (CODE)		Function
ZERO	(ZE)	plot baseline position
ATT 2↑	(AT)	plot height scale
CHT SP	(CS)	paper advance speed

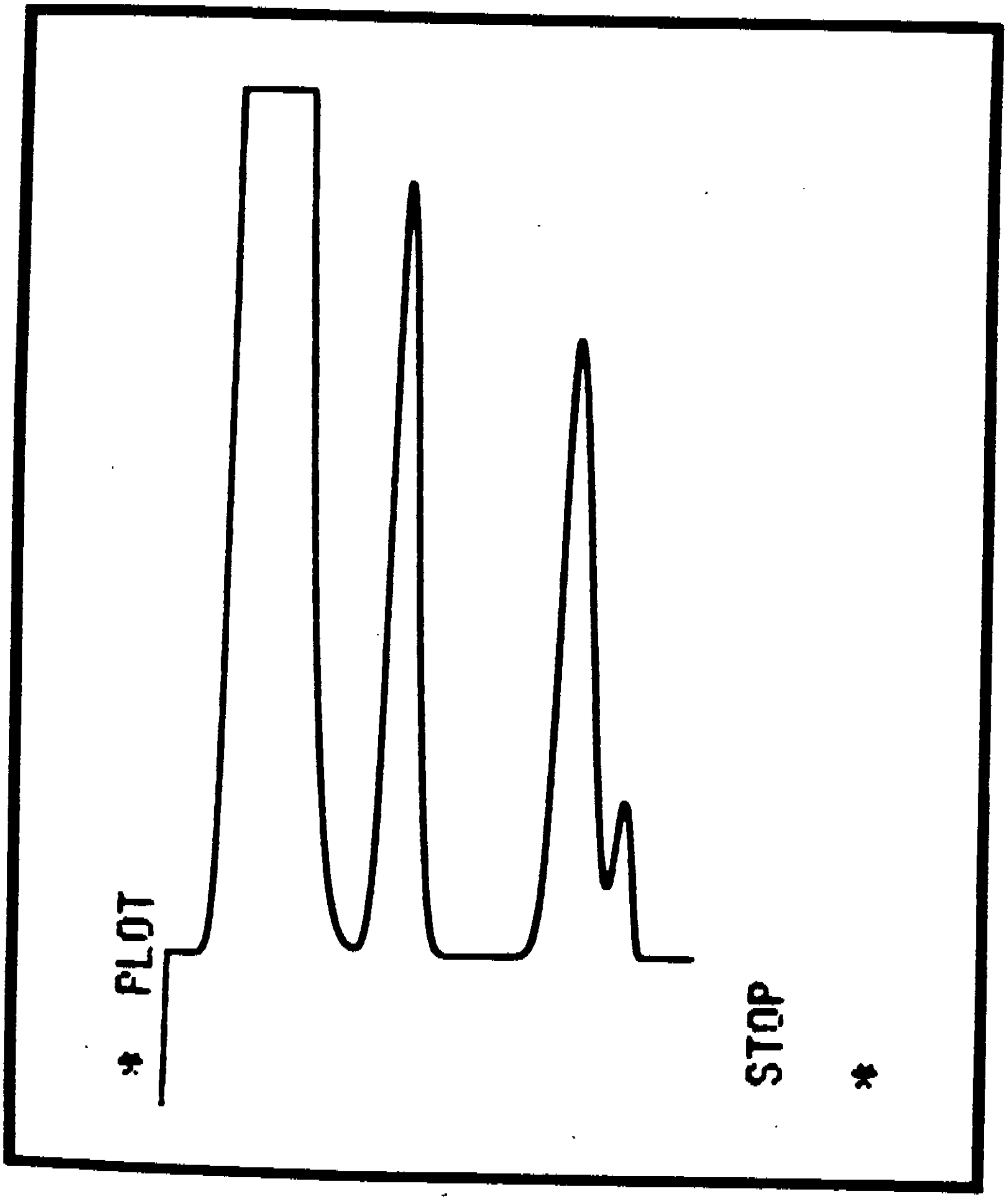
Key Code	mnem	Function	Default Value	Valid Range From	In Minimum Steps (Units) Of
[ZERO]	(ZE)	plot baseline position	0	−6% to 100% of full scale	1% (integer values; ~0.7 mm/unit)
[ATT2]	(AT)	plot height scale	0	−8 to 36	1 (integer values as an exponent of 2)
[CHT SP]	(CS)	paper advance speed	1.0	0.0 to 30.0	0.1 cm/min.

$\boxed{\text{LIST}}$ $\boxed{\text{ZERO}}$ $\boxed{\text{ENTER}}$

ZERO = position, level

position = plot position on the chart

level = voltage value in mv



A Plot In PLOT Mode

DAY

4

REPORT

OPTIONS

OP # 4 : REPORT OPTIONS

$\boxed{\text{OP()}}$ $\boxed{4}$ $\boxed{\text{ENTER}}$

```
* OP # 4
REPORT OPTIONS

Suppress local report [Y/N*] :
Peak height mode [Y/N*] :
Replace report title [Y*/N] :
Report title :
Replace amount label [Y*/N] :
Amount label :
Report uncalibrated peaks [Y/N*] :
Extended report [Y/N*] :

*
```

A STANDARD REPORT

```
*  REPORT

RUN#      9            JUN 24, 1985  11:13:31

                              SAMPLE#  170

ESTD
     RT       HEIGHT TYPE   CAL#        AMOUNT
    .126    8236701   SHB     1          1.000
    .335     820156   TBB     2          2.000
    .585     659275    BV     3          3.000
    .660     166038    VP     4          4.000
    .835     983012    PP    5R          5.000
   1.001     494895 I  PH     6          3.000
```

AN EXTENDED REPORT

* REPORT

RUN# 9 JUN 24, 1985 11:13:31

SAMPLE# 170

ESTD

RT	TYPE	AREA	WIDTH	HEIGHT
.126	SHB	28459952	.058	8236701
.335	TBB	2169522	.044	820156
.585	BV	2102709	.053	659275
.660	VP	302513	.030	166038
.835	PP	920732	.016	983012
1.001	I PH	2467334	.083	494895

RT	CAL#	AMOUNT	NAME
.126	1	1.000	SAMP1
.335	2	2.000	SAMP2
.585	3	3.000	SAMP3
.660	4	4.000	SAMP4
.835	5R	5.000	SAMP5
1.001	6	3.000	SAMP6

OP # 5 : POST-RUN LIST OPTIONS

OP() 5 ENTER

* OP # 5

POST-RUN LIST OPTIONS
Store post-run report [Y*/N] :
Device [M*] :
External post-run report [Y*/N] :
Printer Address [0] :
List run parameters [Y/N*] :
List timetable [Y/N*] :
List calibration table [Y/N*] :
List remote method [Y/N*] :

*

NOTEPAD SYSTEM COMMAND

```
*NOTEPAD

(USE BREAK OR CONTROL-Y TO END)
Testing column conditioning:
First pass -- operator is Dave
*
```

SET SYSTEM COMMAND

*SET RUNNUM 1

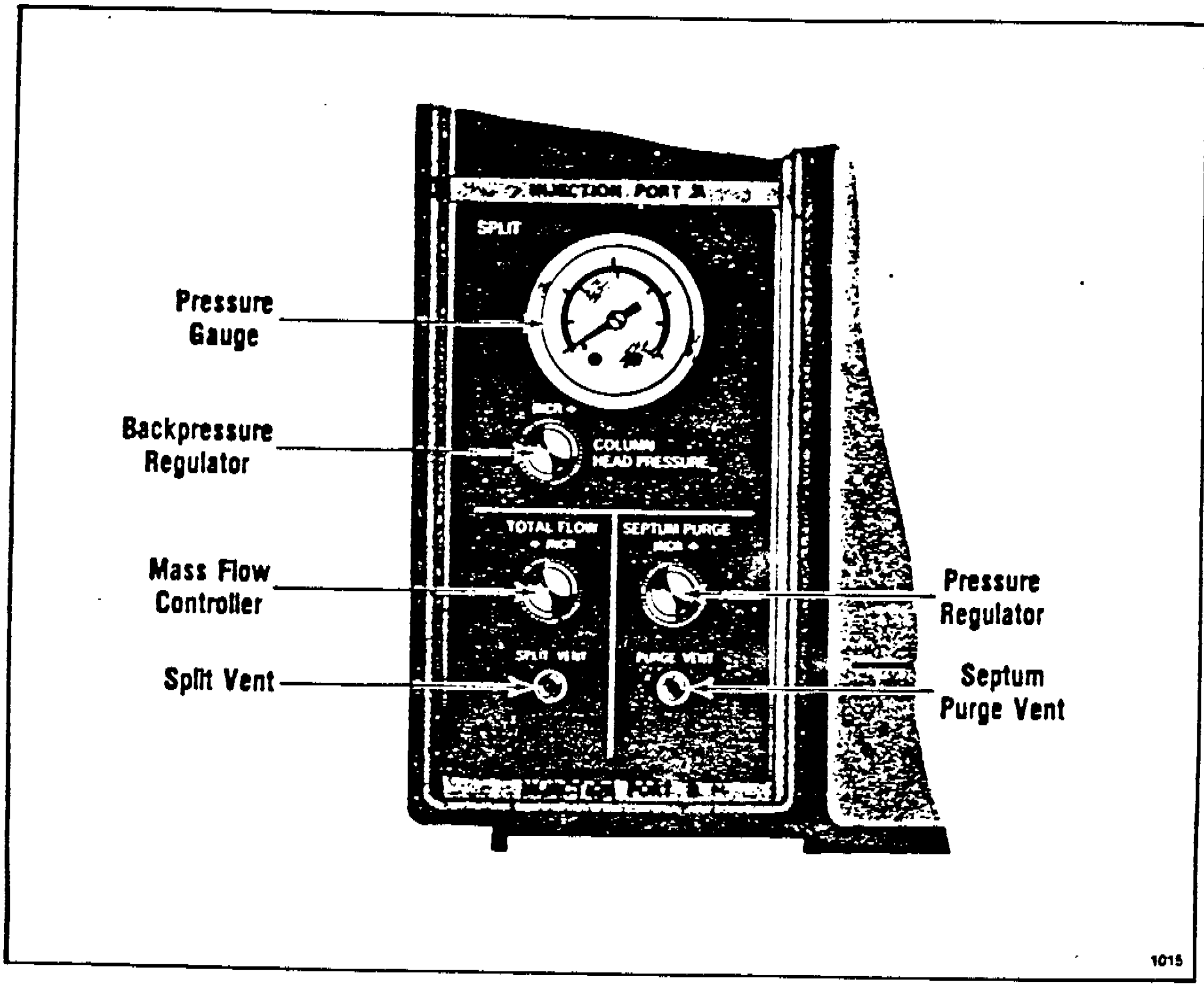
(RUNNUM = desired run number from 1 to 10000)

KEYWORDS USED WITH **SET**

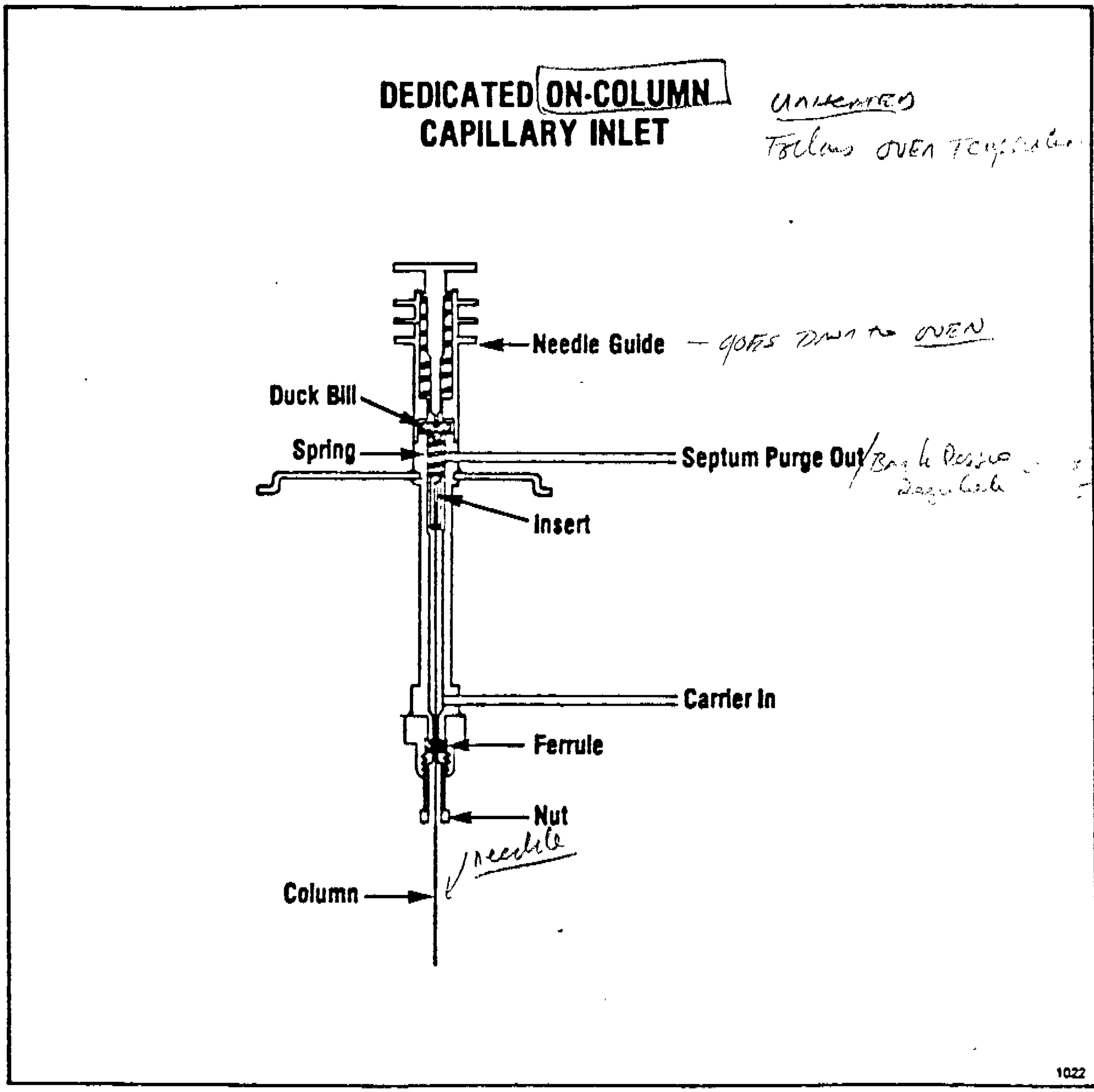
ZERO	THRSH
ATT2	PK_WD
CHT_SP	EXT
AR_REJ	RUNNUM

METHODS

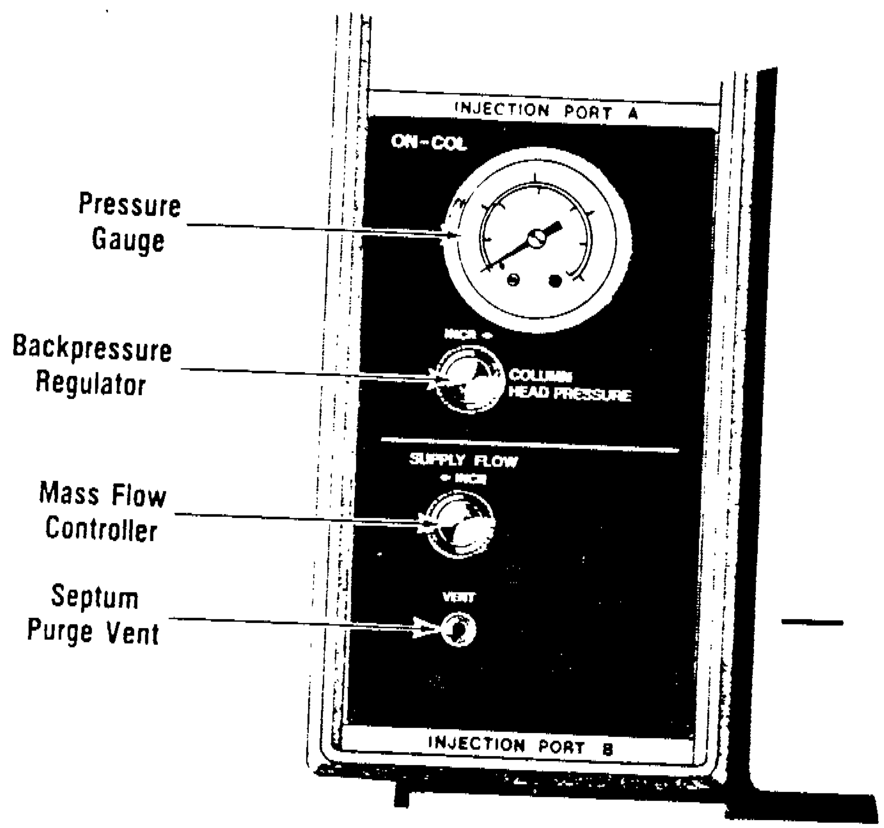
FLOW PANEL CONTROLLING SPLIT OPERATION



DEDICATED ON–COLUMN CAPILLARY INLET

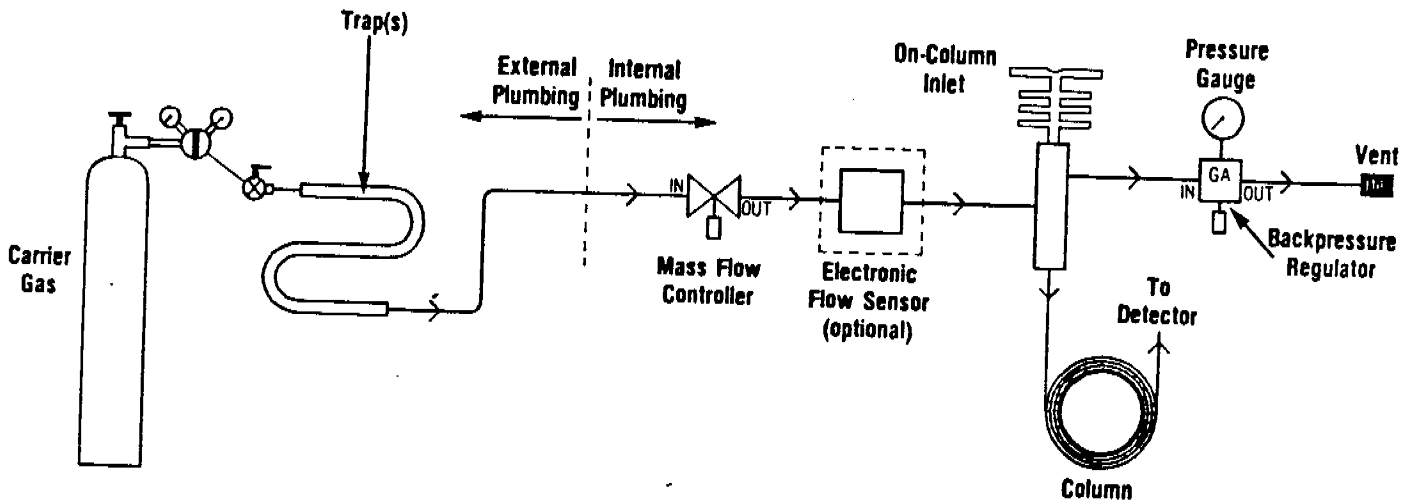


FLOW PANEL CONTROLLING
COOL ON–COLUMN OPERATION



FLOW DIAGRAM: COOL ON-COLUMN OPERATION

Flow System:
Dedicated On-Column Capillary Inlet
(with Electronic Flow Sensor)



TEMPERATURE CONTROL OF INJECTION PORT

												ACTUAL					SETPOINT		
I	N	J		A		T	E	M	P			*					1	5	0

ENTER

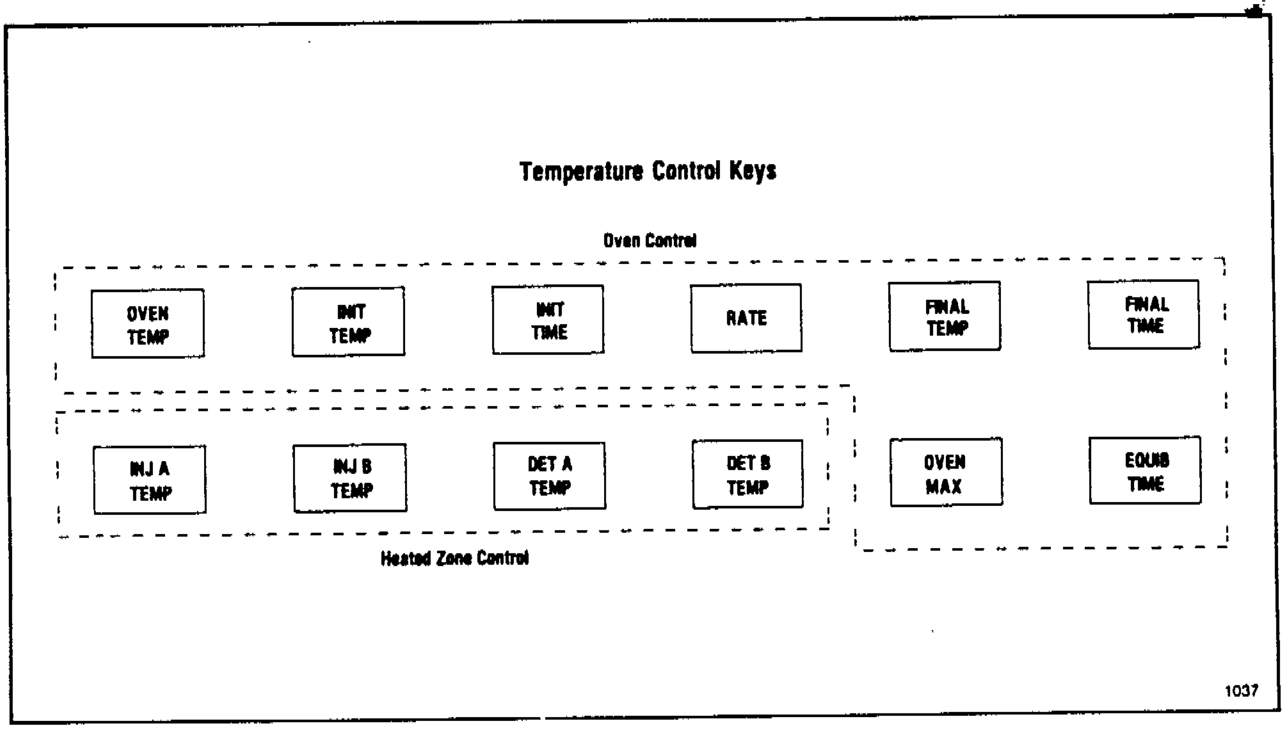
												ACTUAL					SETPOINT		
I	N	J		A		T	E	M	P			4	2				1	5	0

OVEN

TEMPERATURE

CONTROL

Temperature Control



OVEN TEMPERATURE CONTROL

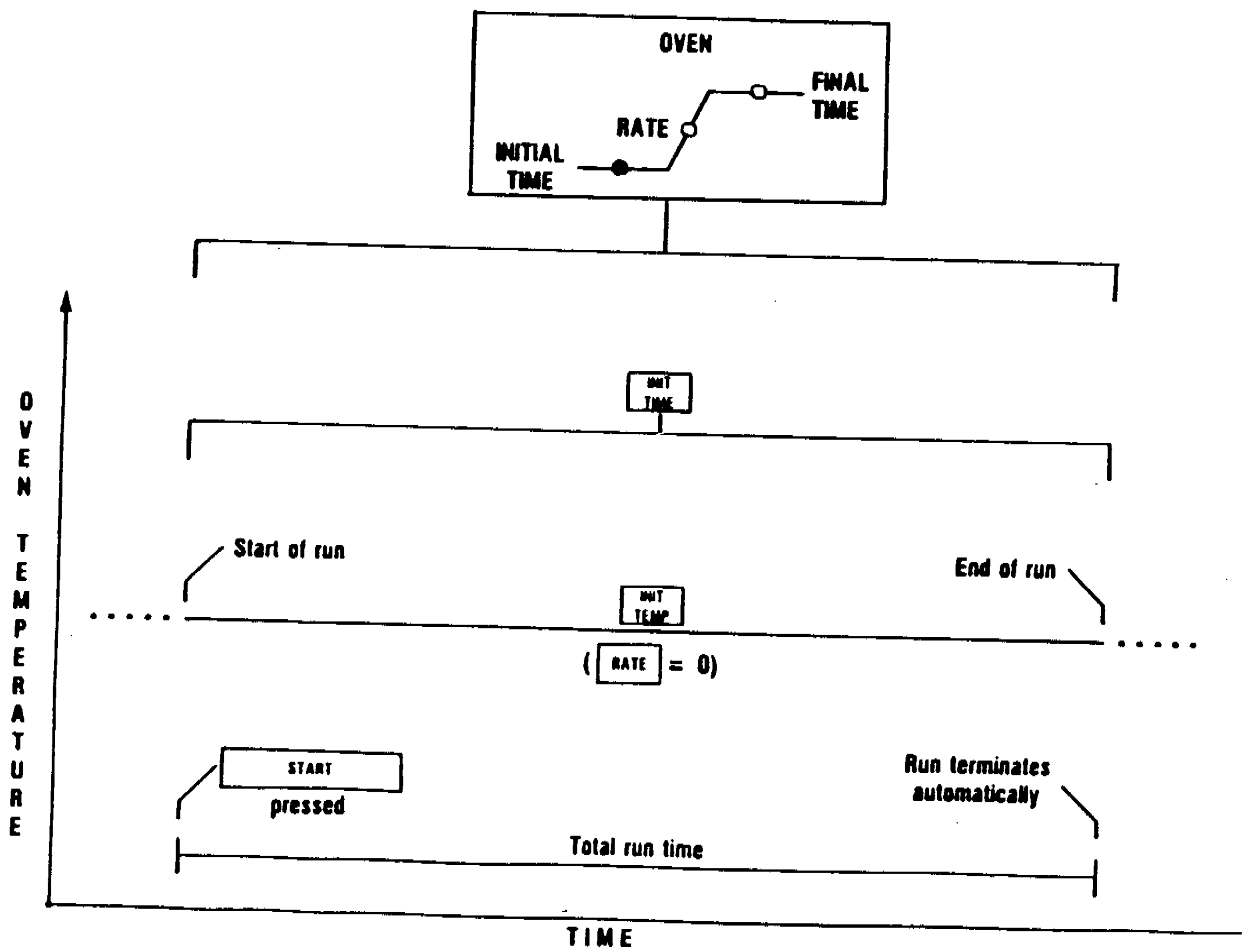
	ACTUAL	SETPOINT
OVEN TEMP	279	350

	ACTUAL	SETPOINT
OVEN TEMP	34	OFF

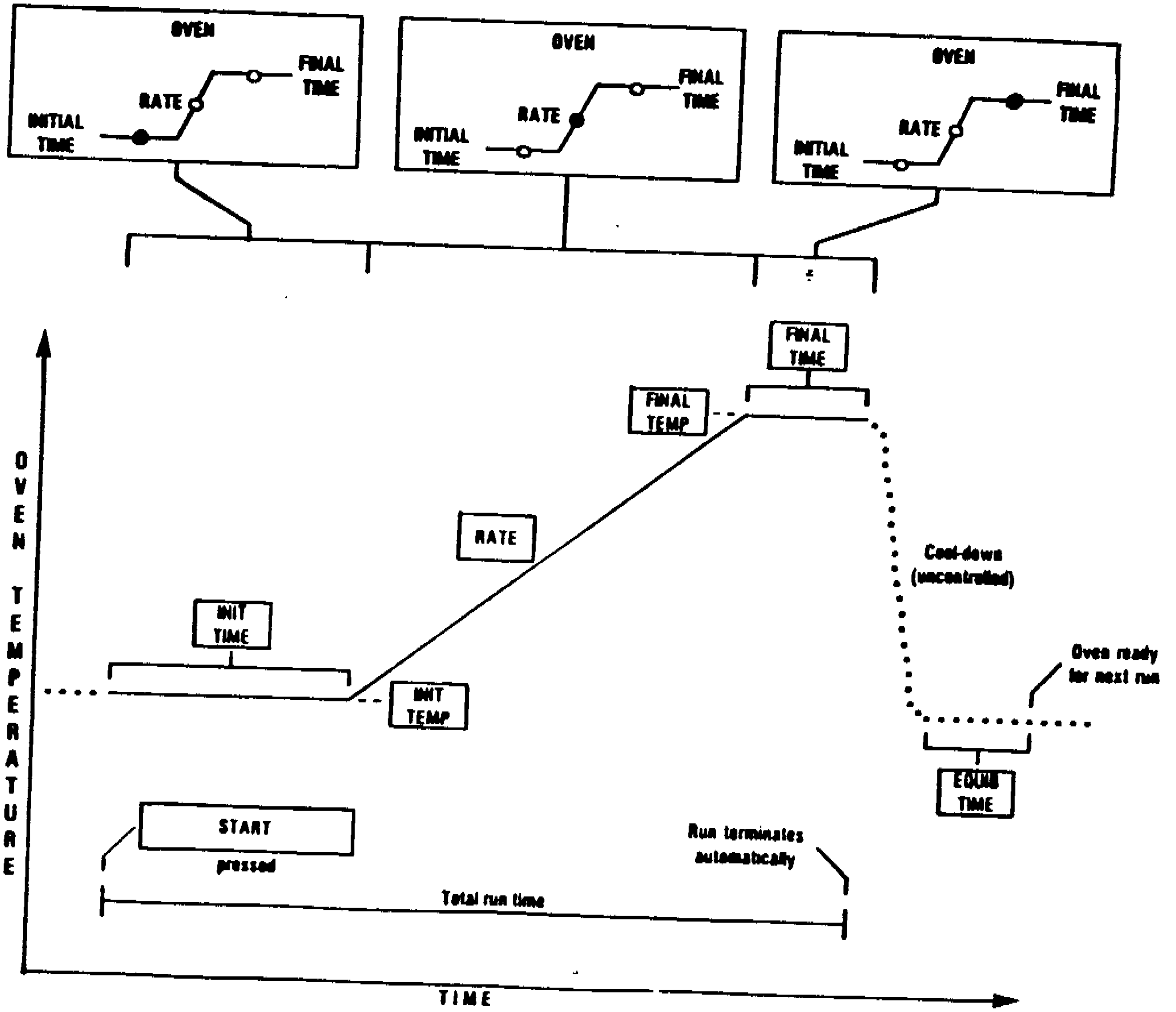
	ACTUAL	SETPOINT
OVEN MAXIMUM		400

0P58901

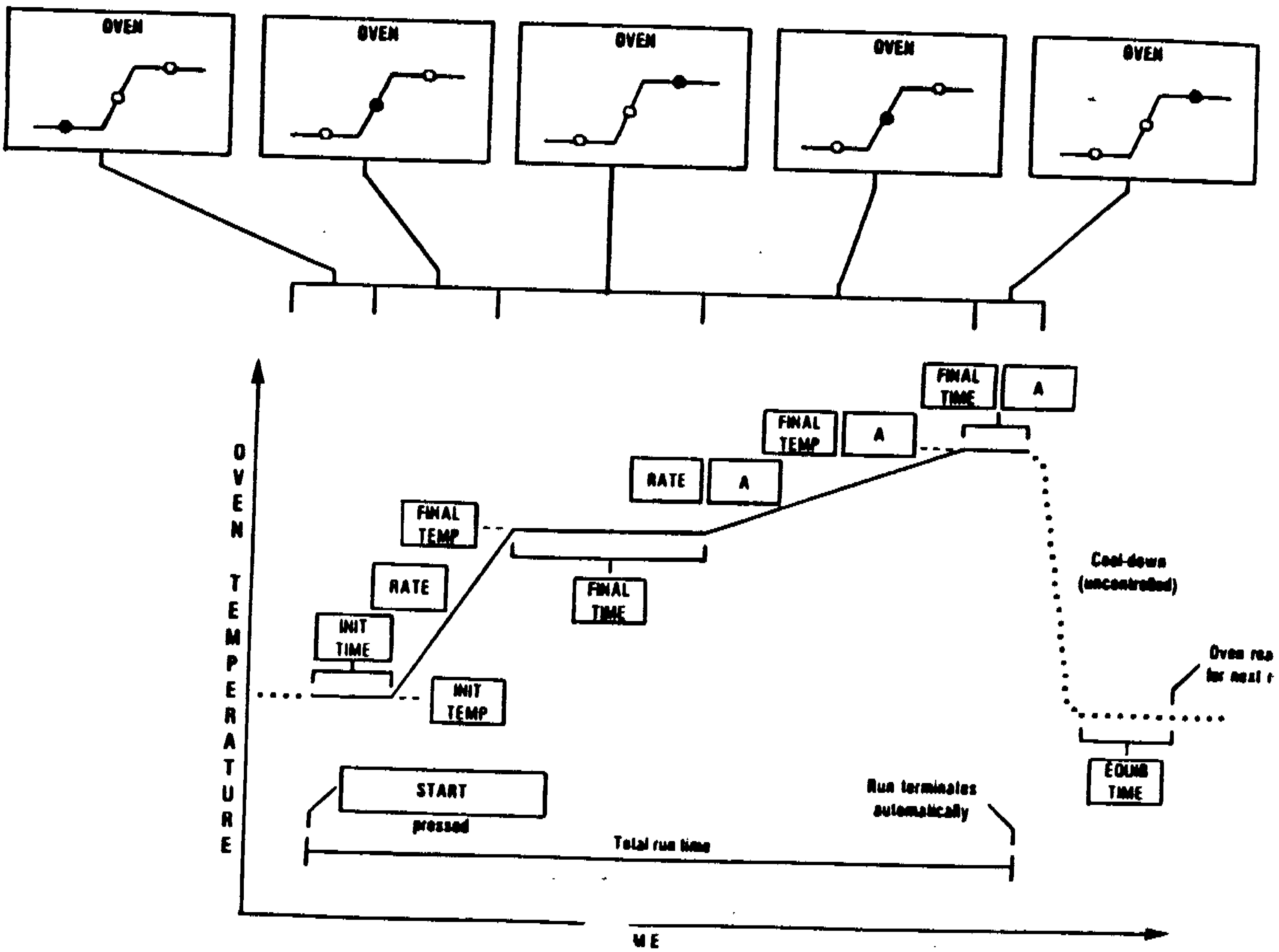
ISOTHERMAL OPERATION



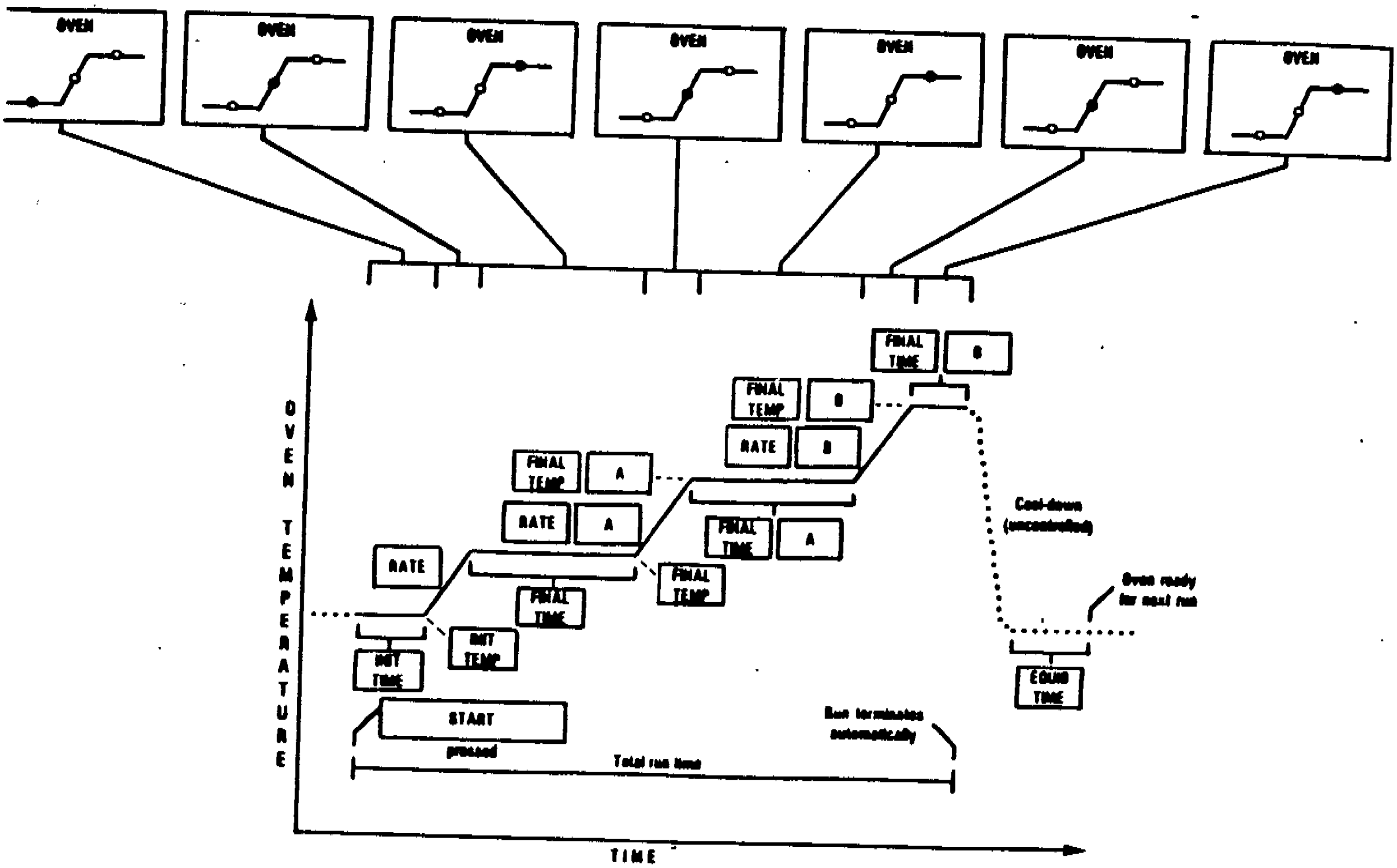
A Single-Ramp Temperature Program



A Two-Ramp Temperature Program



A Three-Ramp Temperature Program



TIME KEY

Outside a Run:

ACTUAL SETPOINT

`NEXT RUN   24.38 MIN`

ACTUAL SETPOINT

`t= 5:10.7  1/t= 0.19`

ACTUAL SETPOINT

`LAST RUN   15.77 MIN`

During a Run:

ACTUAL SETPOINT

`REMAINING  12.23 MIN`

ACTUAL SETPOINT

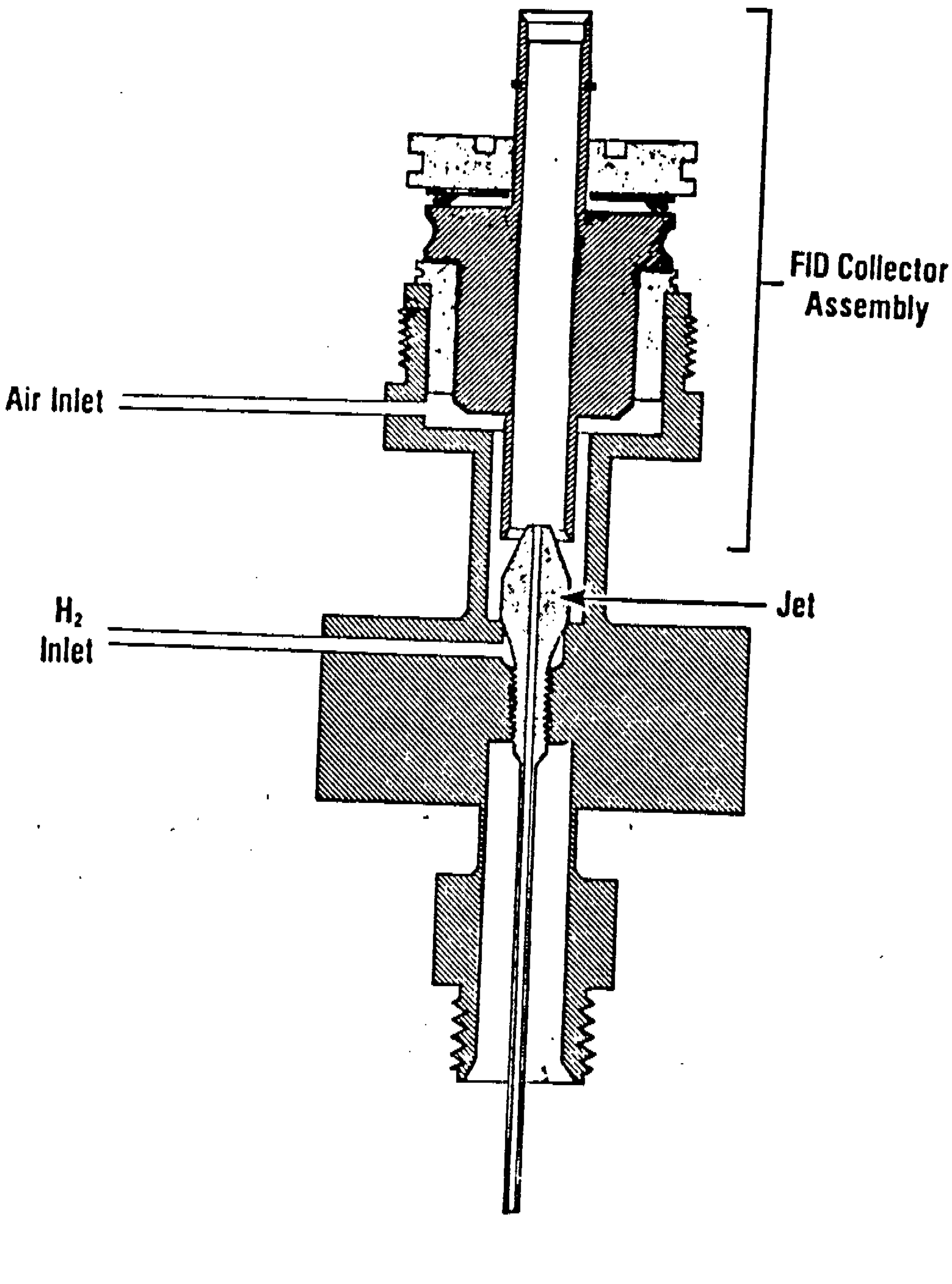
`t= 1:50.7  1/t= 0.54`

ACTUAL SETPOINT

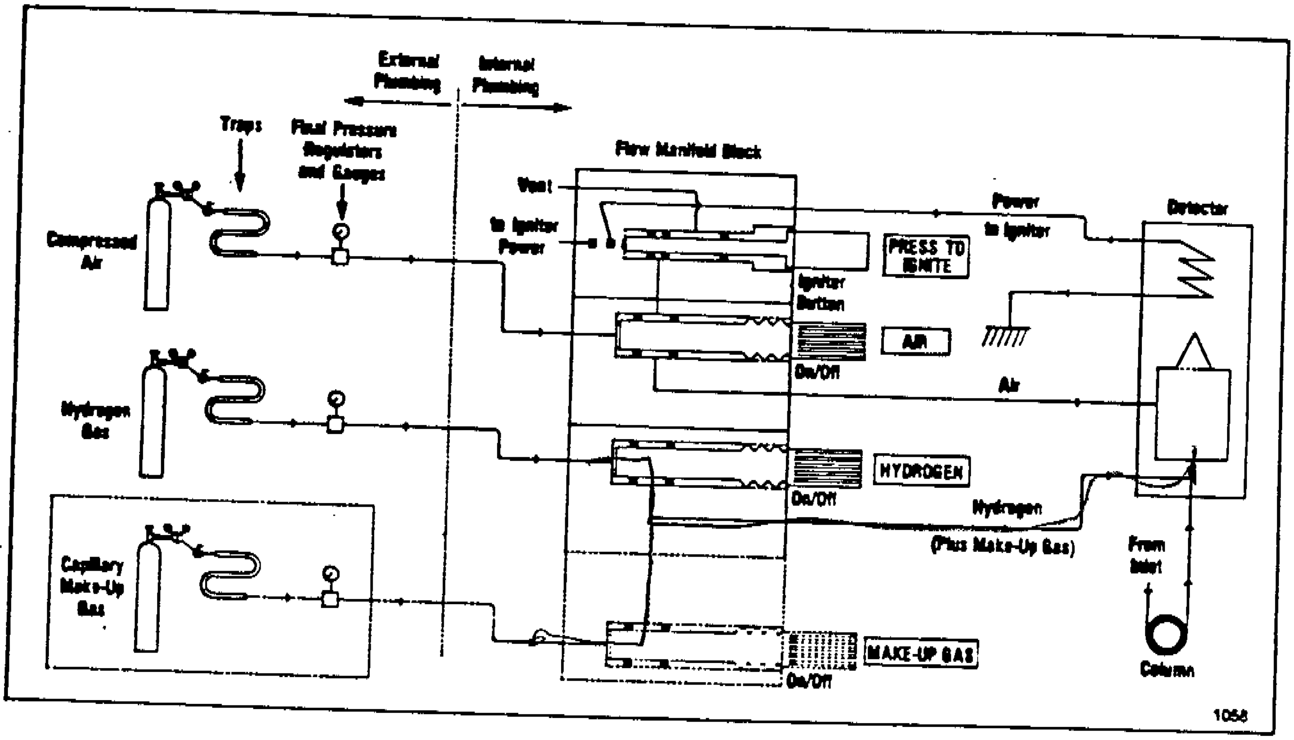
`ELAPSED    12.15 MIN`

DETECTORS

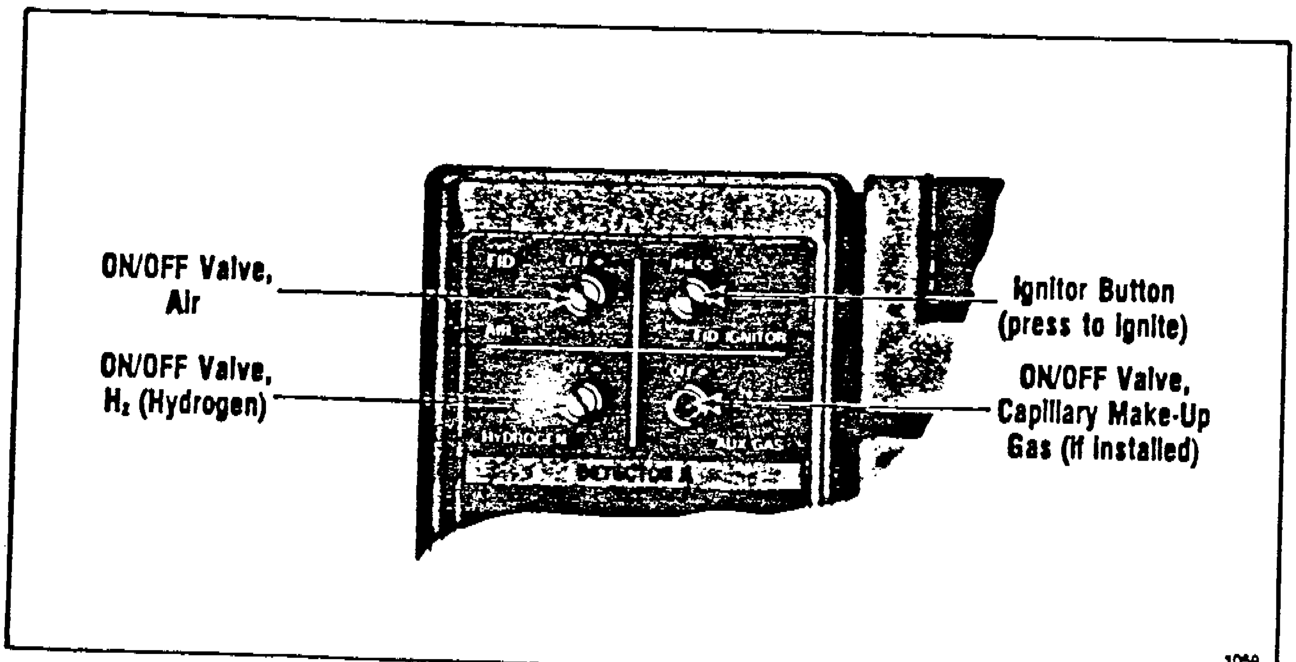
FLAME IONIZATION DETECTOR (FID)



Igniting the Flame

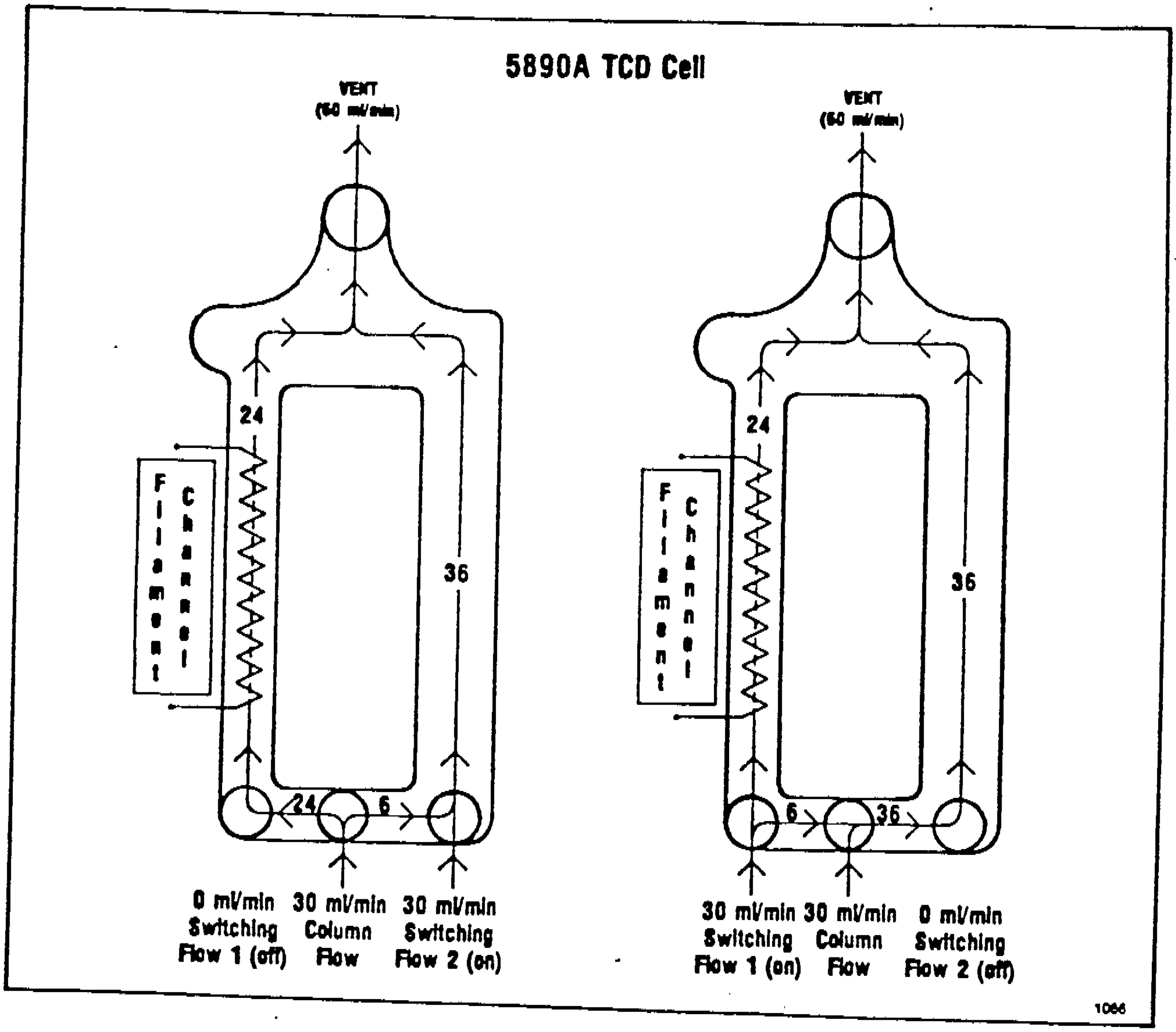


Flow Diagram, Flame Ionization Detector (FID)



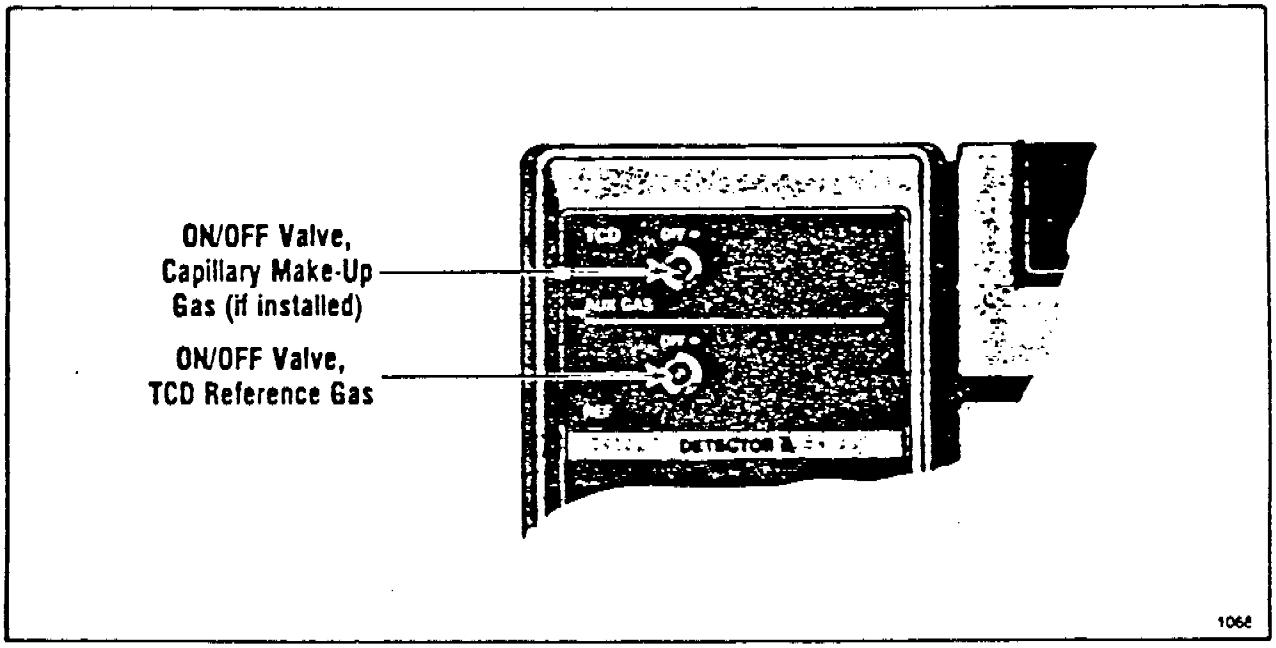
Flow Panel Controlling FID Operation

THERMAL CONDUCTIVITY DETECTOR (TCD)

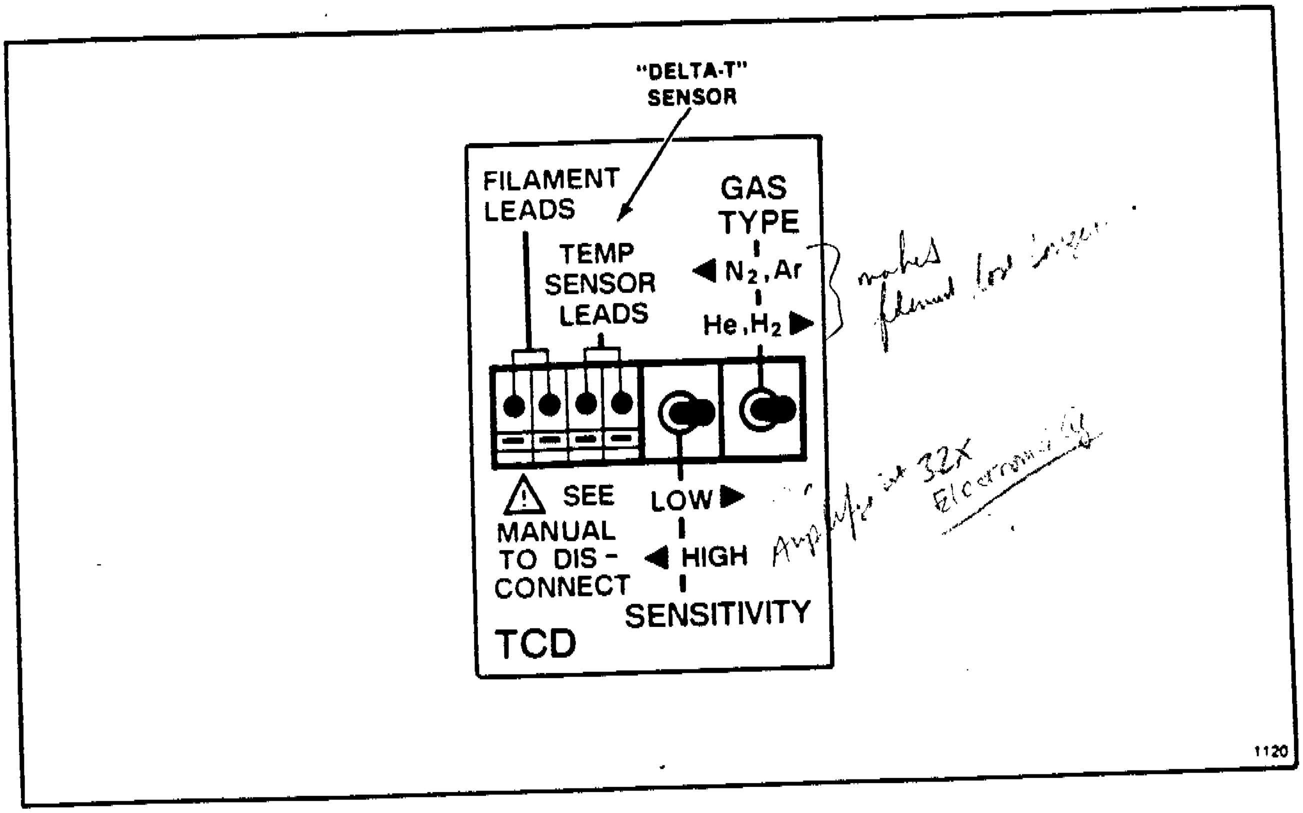


Single-Filament Cell, Thermal Conductivity Detector (TCD)

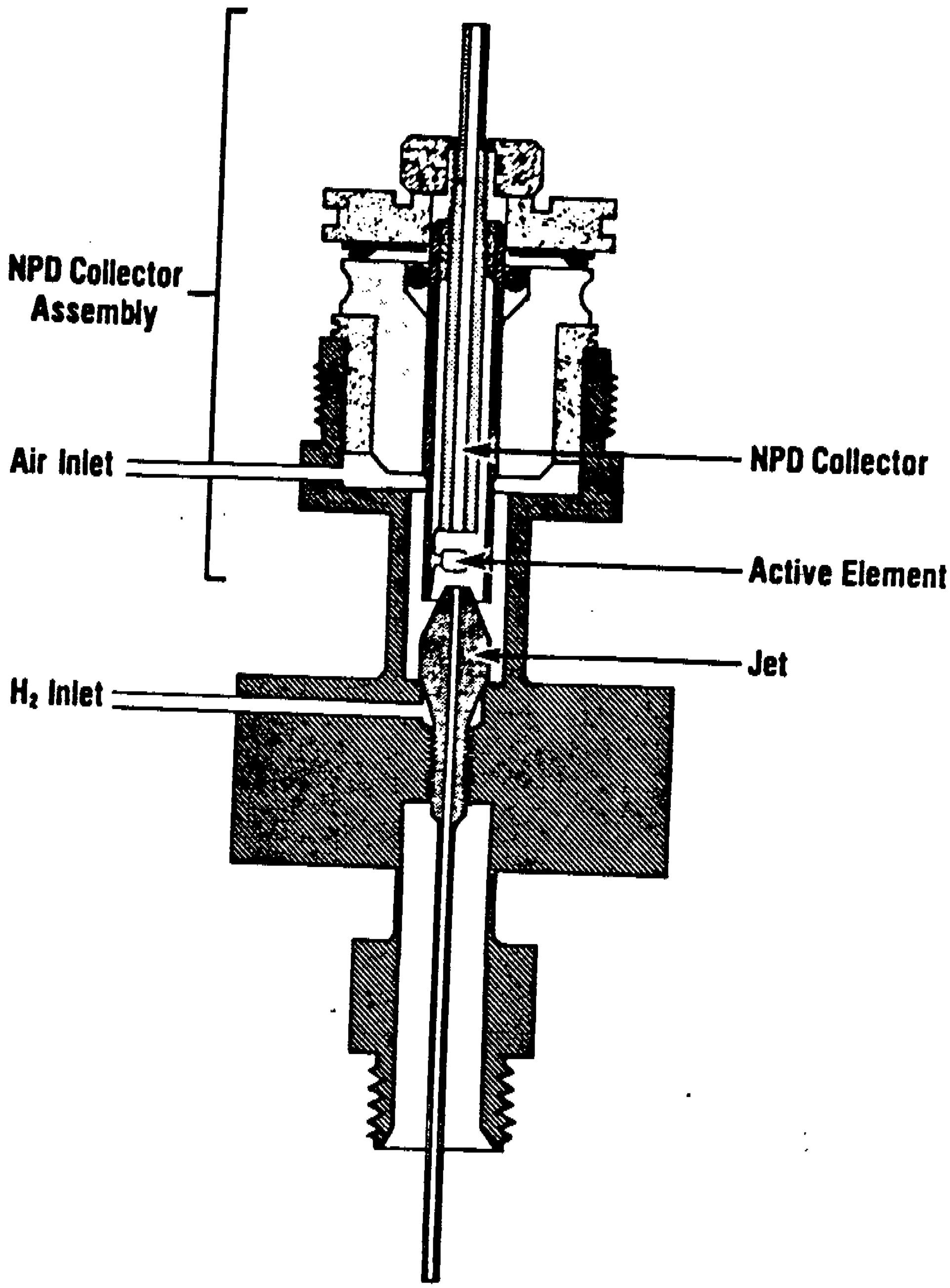
FLOW PANEL CONTROLLING TCD OPERATION



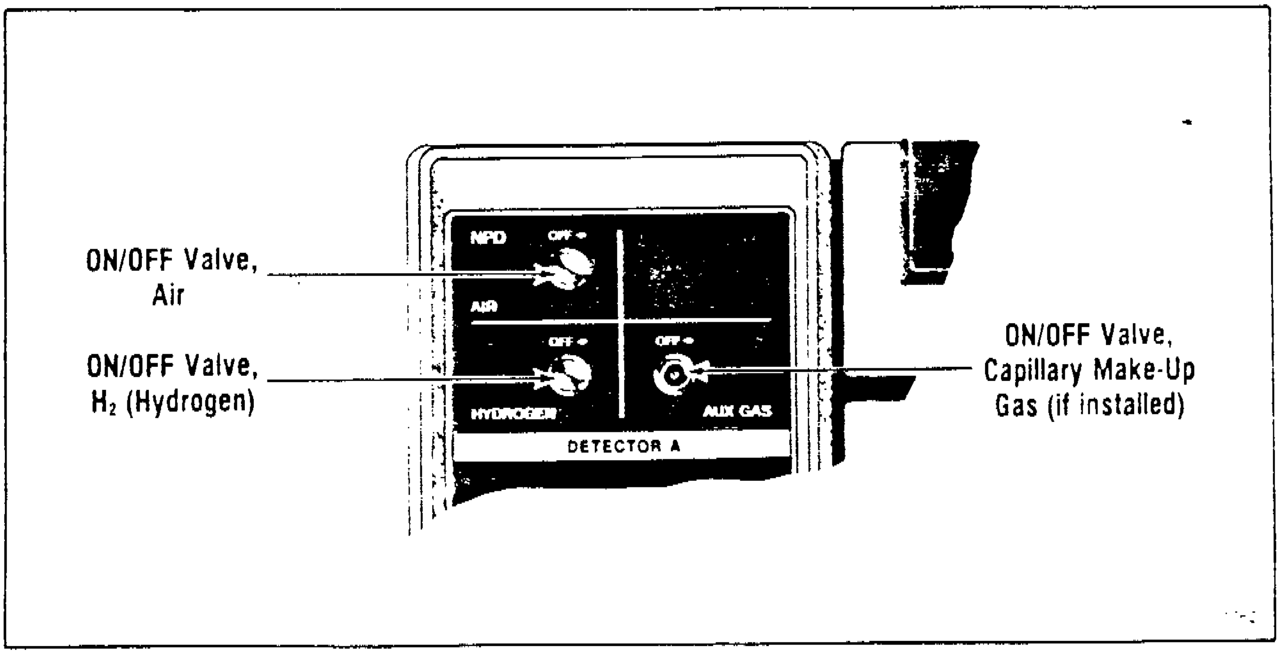
TCD GAS SELECTION
AND SENSITIVITY SWITCHES



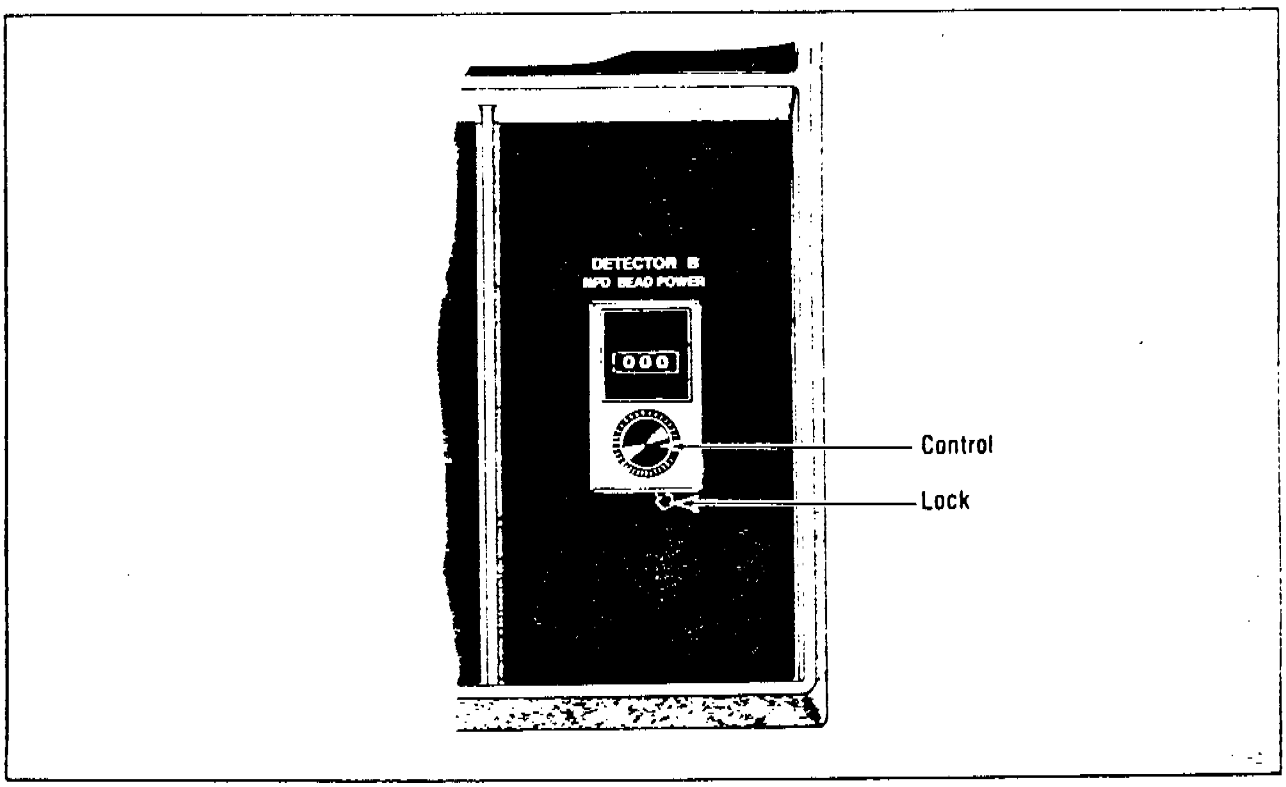
NITROGEN-PHOSPHORUS DETECTOR (NPD)



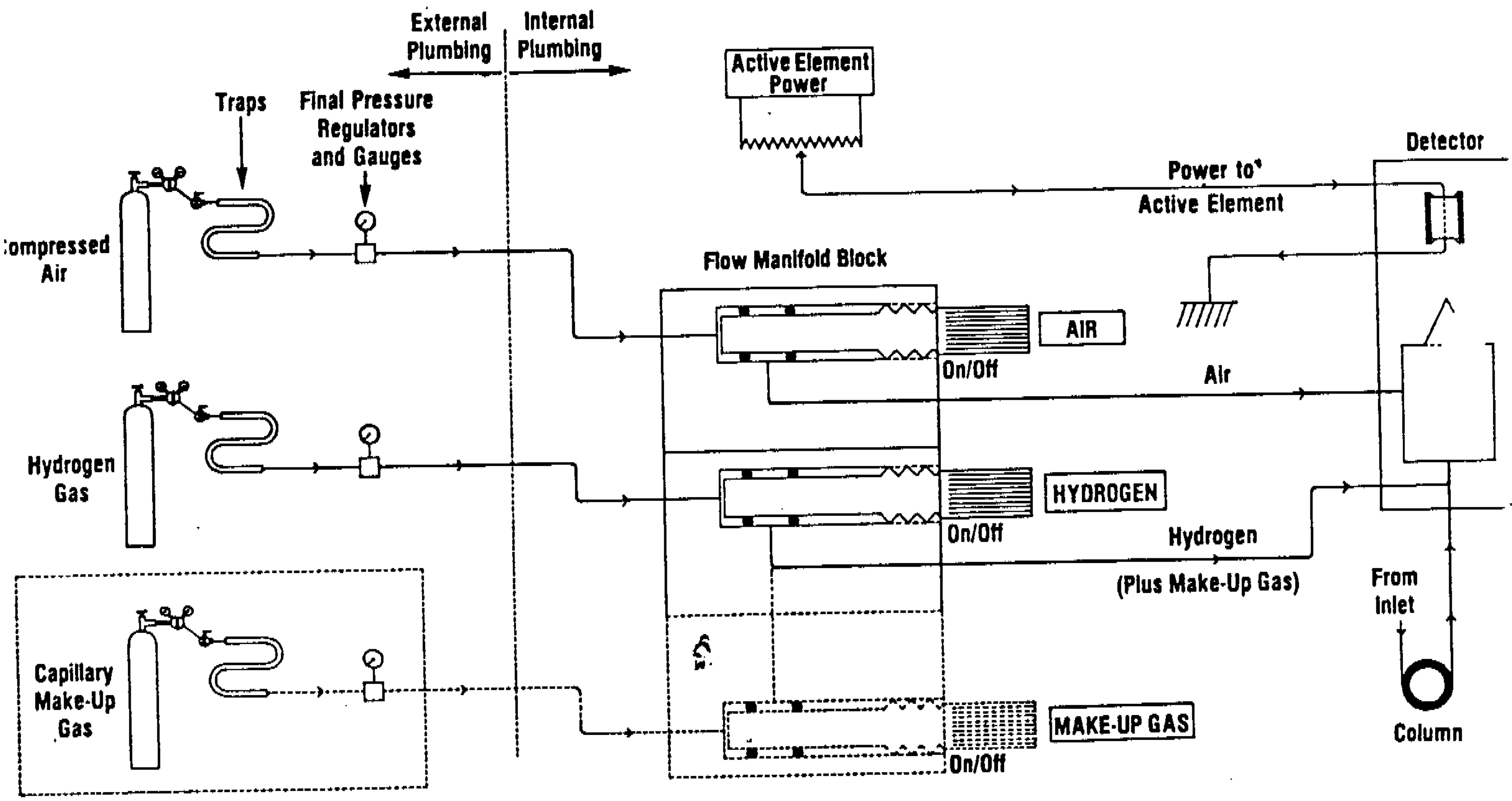
FLOW PANEL CONTROLLING NPD OPERATION



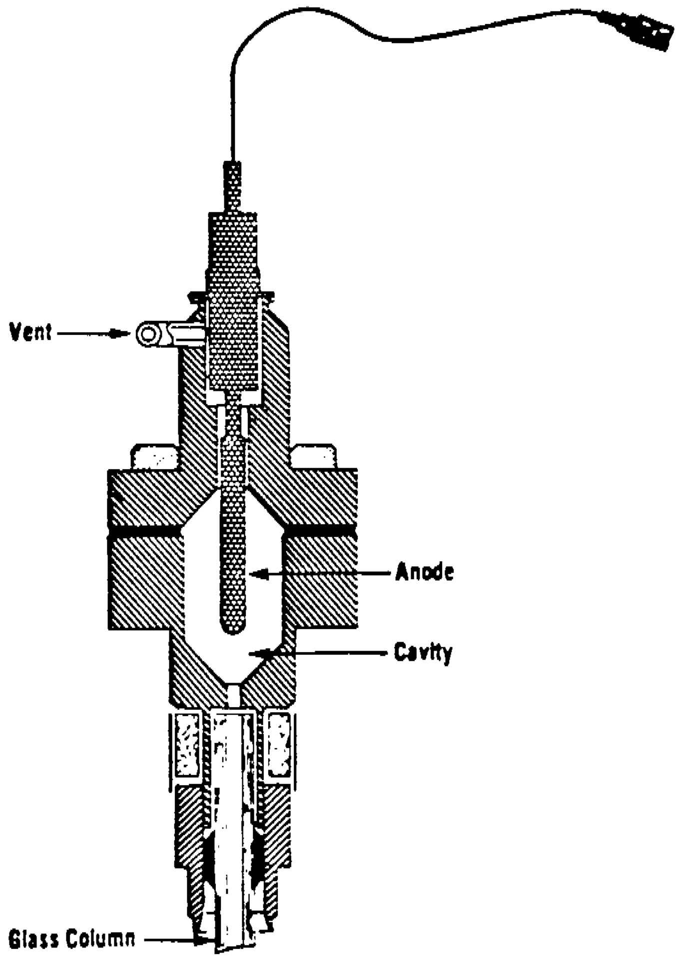
NPD ACTIVE ELEMENT POWER CONTROL



FLOW DIAGRAM: NPD

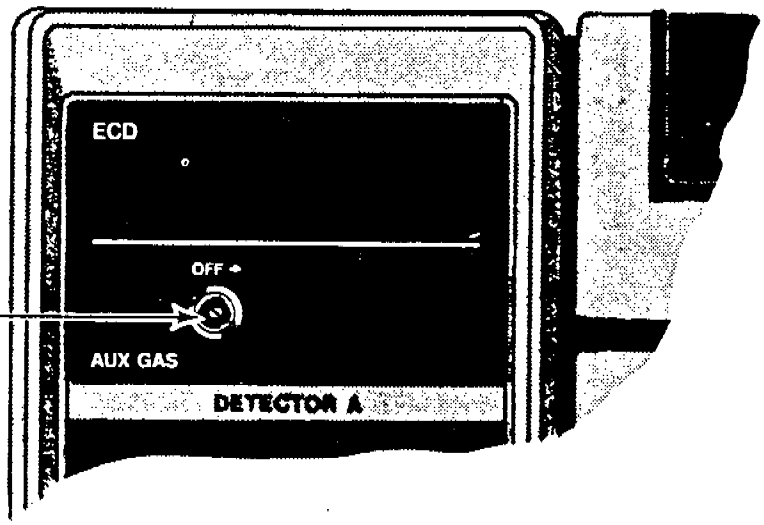


ELECTRON CAPTURE DETECTOR

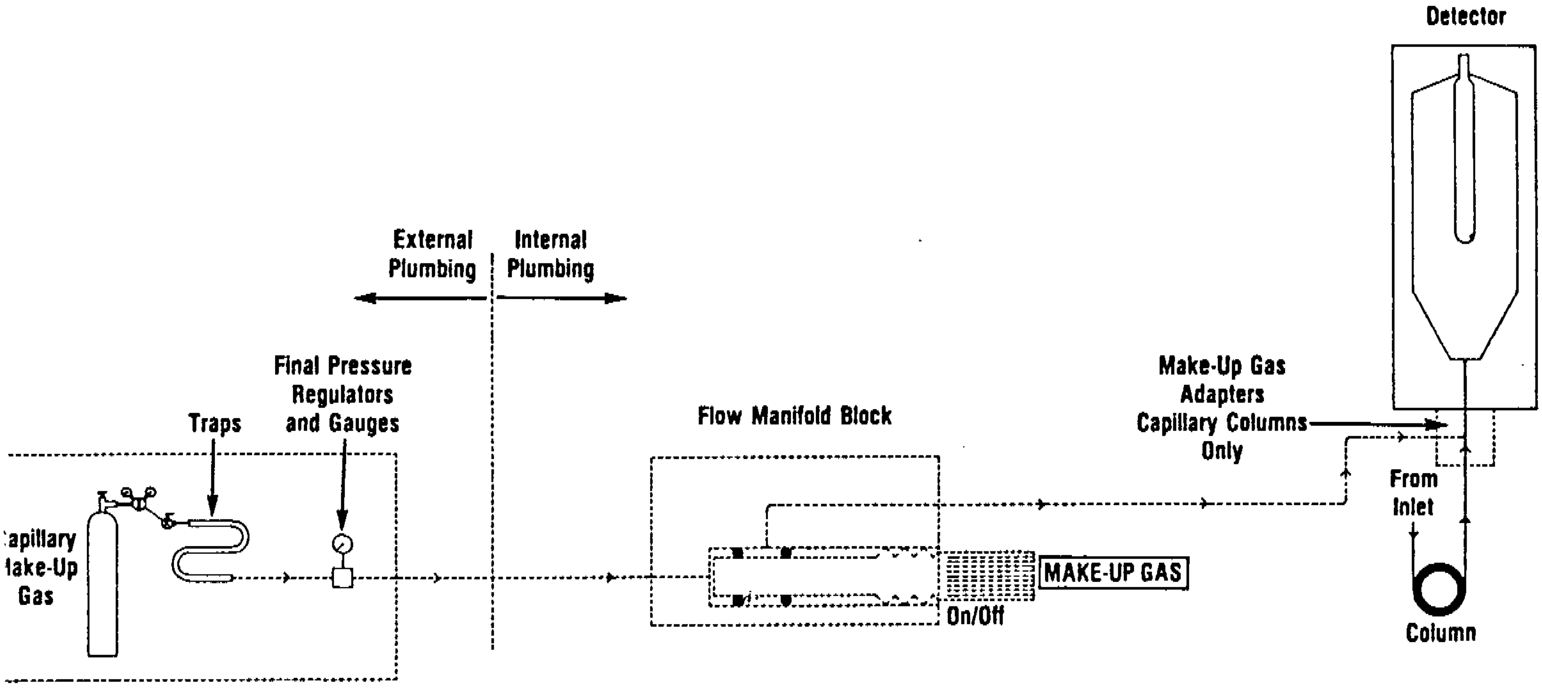


FLOW PANEL CONTROLLING ECD OPERATION

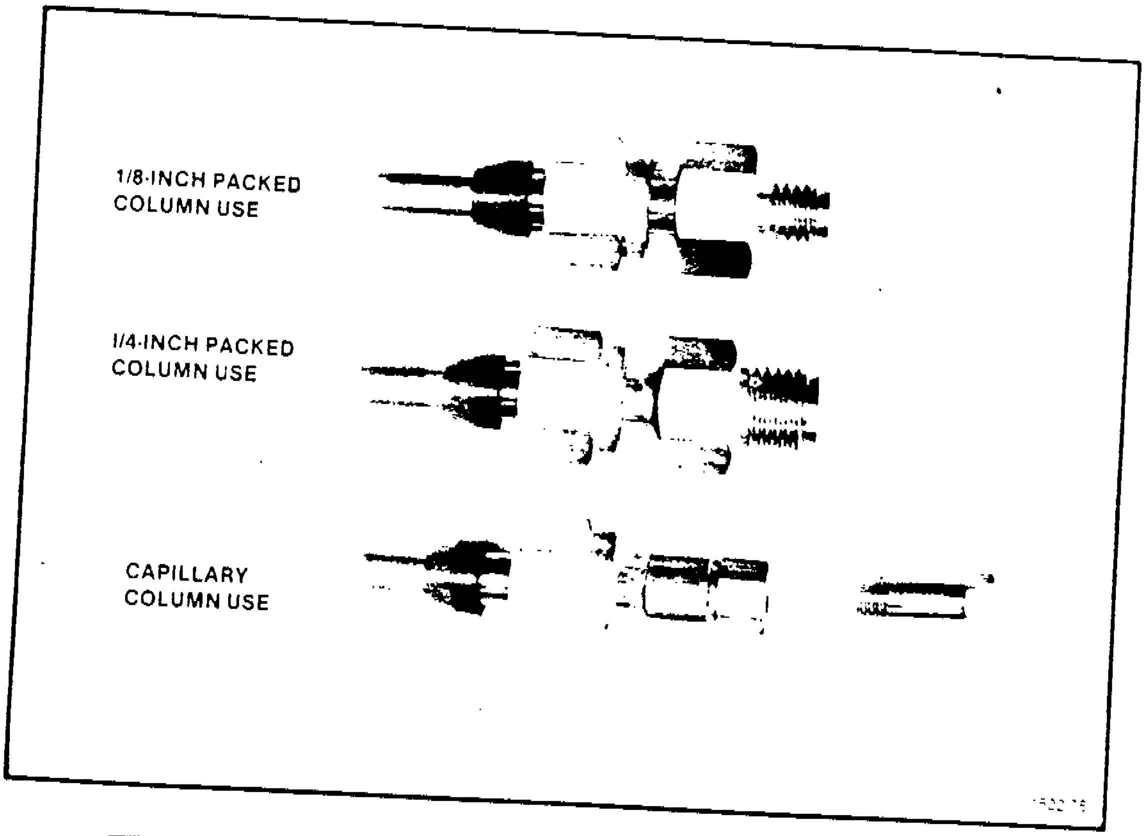
ON/OFF Valve, Capillary Make-Up Gas (if installed)



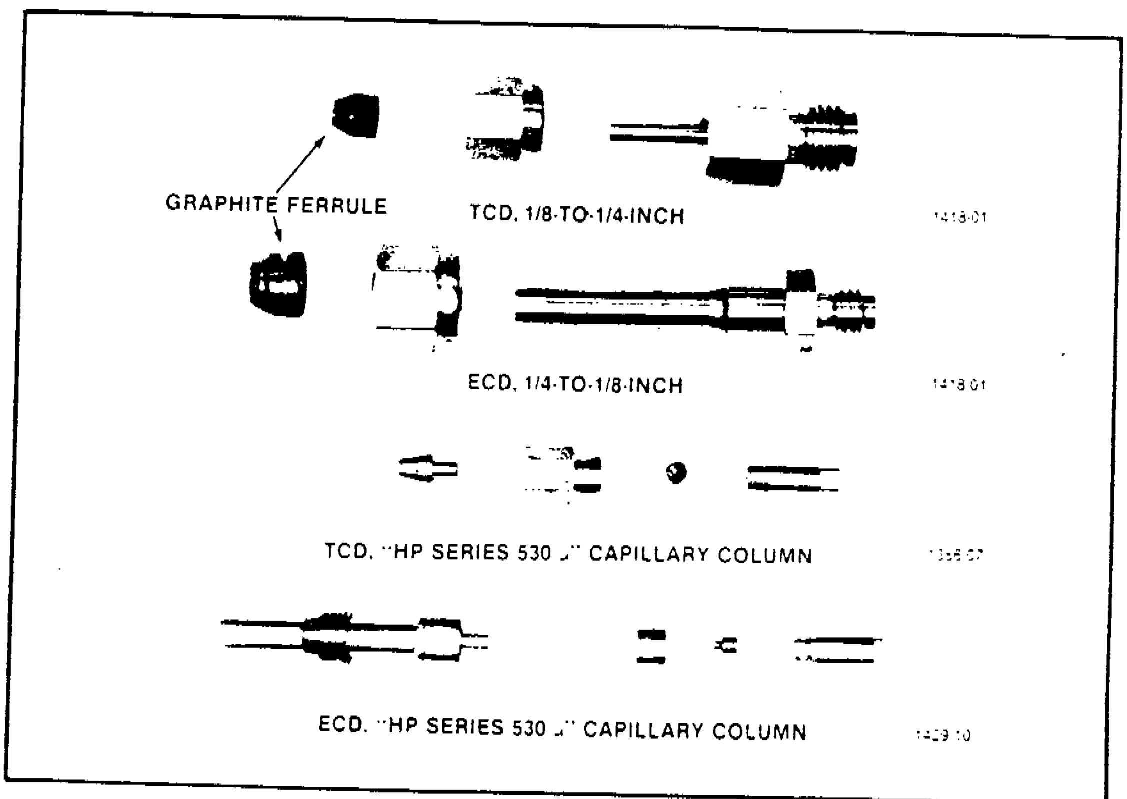
FLOW DIAGRAM: ELECTRON CAPTURE DETECTOR



FID/NPD LINERS/ADAPTERS



ECD AND TCD PACKED AND SERIES 530 COLUMN ADAPTERS



TEMPERATURE CONTROL OF DETECTOR

												ACTUAL						SETPOINT	
D	E	T		A		T	E	M	P			5	0				O	F	F

TEMPERATURE CONTROL OF DETECTOR

													ACTUAL					SETPOINT	
D	E	T		A		T	E	M	P			*					1	5	0

ENTER

													ACTUAL					SETPOINT	
D	E	T		A		T	E	M	P			5	2				1	5	0

DETECTOR STATUS DISPLAYS

ACTUAL SETPOINT

DET A FID ON

ACTUAL SETPOINT

DET A TCD OFF [+]

ACTUAL SETPOINT

DET B NOT INSTALLED

```
              ACTUAL   SETPOINT
SIGNAL 1 RANGE    1Ø

              ACTUAL   SETPOINT
SIGNAL 1 ATTN     1Ø

              ACTUAL   SETPOINT
SIGNAL 1 ATTN 1Ø OFF

              ACTUAL   SETPOINT
SIG 2 NOT INSTALLED 
```

MONITORING DETECTOR OUTPUT

SIG 1 {or SIG 2} A {or B} ENTER

ACTUAL SETPOINT

SIGNAL 1 A

ACTUAL SETPOINT

SIGNAL 1 258.7

ACTUAL SETPOINT

SIG 2 NOT INSTALLED

Displaying Current [ZERO] Setpoint

ACTUAL SETPOINT

```
SIG 1 ZERO     104.5
```

ACTUAL SETPOINT

```
SIG 2 NOT INSTALLED
```

ACTUAL SETPOINT

```
ZERO 1    104.5  OFF
```

ACTUAL SETPOINT

```
ZERO LIMIT = 830000
```

"TYPICAL" DISPLAYS: SIGNAL ASSIGNMENT AND MONITORING

Detector Signal Assignments

ACTUAL SETPOINT

SIGNAL 1 A

Detector Signal Monitoring:

ACTUAL SETPOINT

SIGNAL 1 29.7

Oven/Zone Temperature Assignment:

ACTUAL SETPOINT

SIGNAL 1 OVEN TEMP

Oven/Zone Temperature Monitoring:

ACTUAL SETPOINT

SIGNAL 1 15998

Flow Rate Assignments:

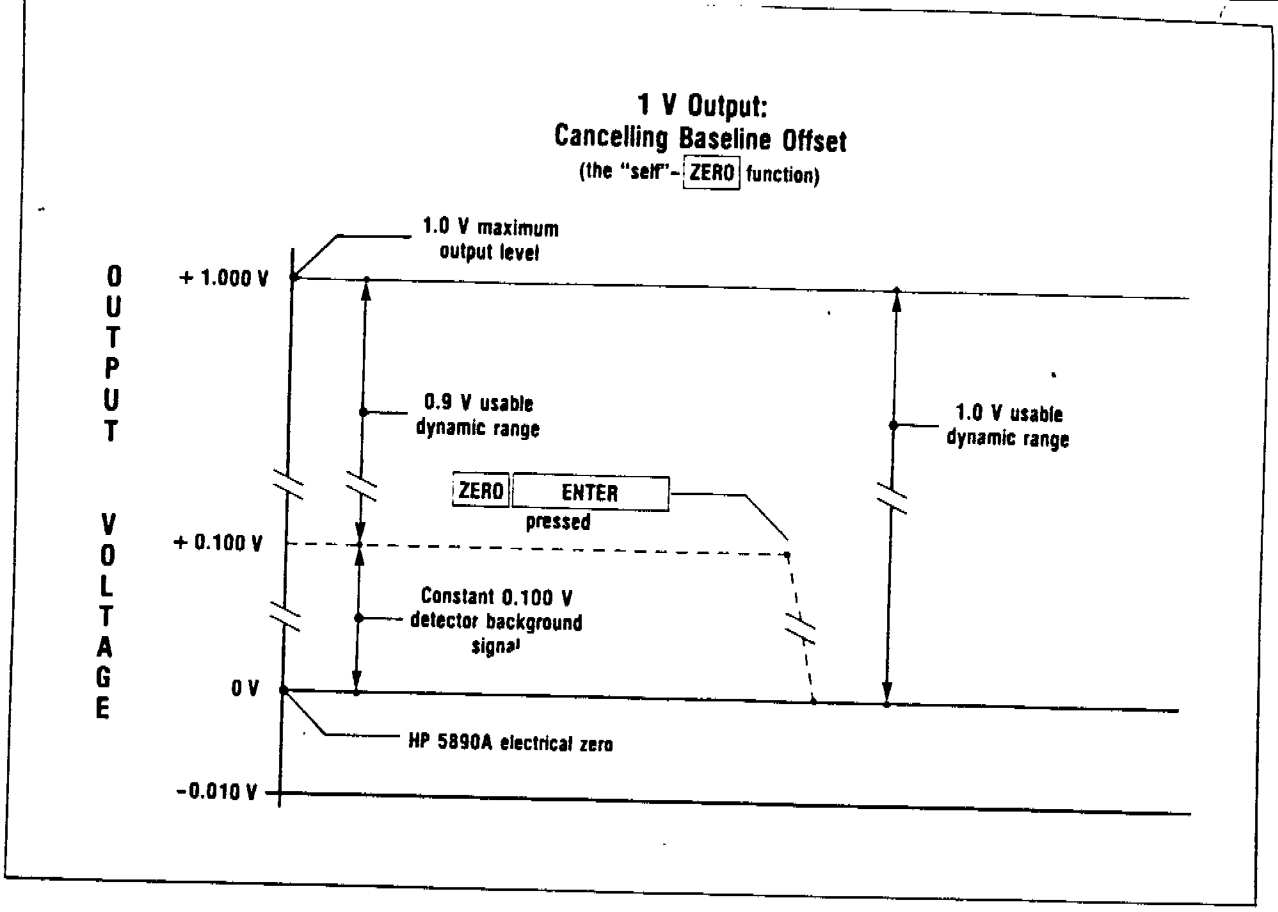
ACTUAL SETPOINT

SIGNAL 1 FLOW A

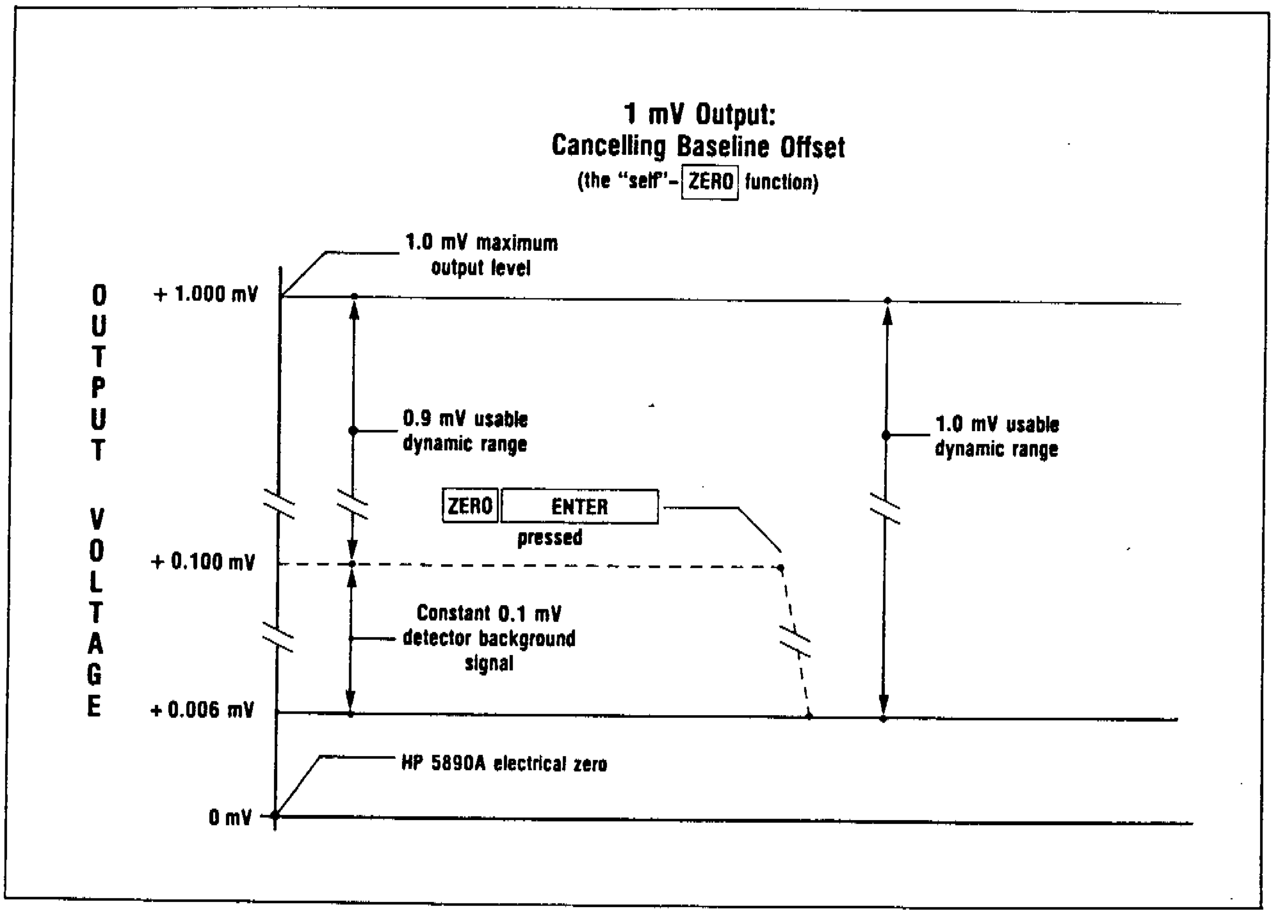
Flow Rate Monitoring:

ACTUAL SETPOINT

SIGNAL 1 30.2



Effect of ZERO on the "+1 V" Analog Output



Effect of ZERO on the "+1 mV" Analog Output

Key	Permitted Setpoints	Affected Output
[RANGE]	0 to 13	BOTH "+1 mV" & "+1 V"
[ATTN]	0 to 10, [OFF], [ON]	ONLY "+1 mV"

[ATTN] functions only for the "+1 mV" output, and acts on the signal AFTER it has been "ranged" by [RANGE].

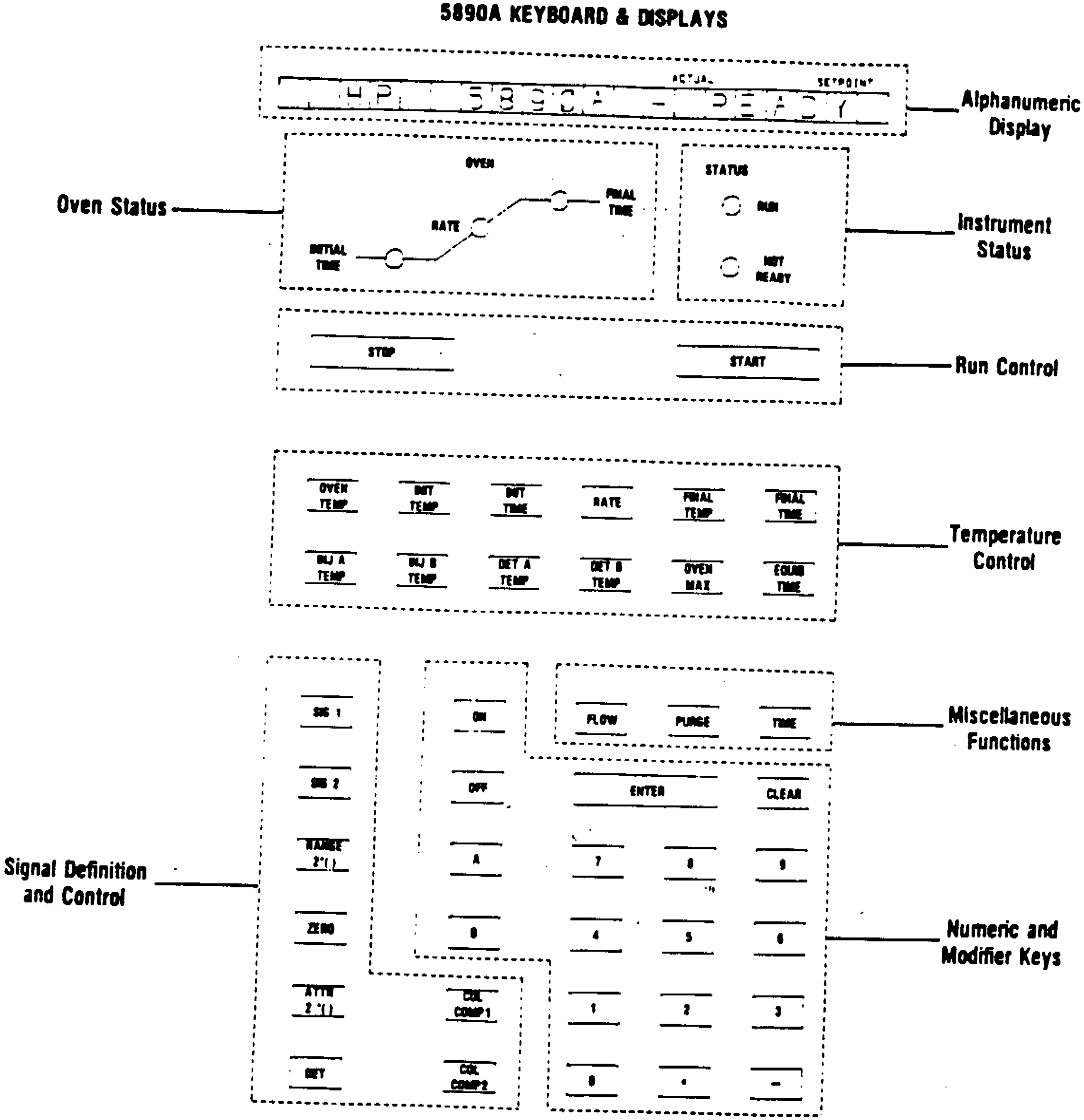
RANGE 2 ()	MAXIMUM DETECTOR SIGNAL PRODUCING +1 V OUTPUT			
	FID & NPD (pA)	TCD(mV,"High" Gain)	TCD(mV,"Low" Gain)	ECD(kHz)
0	1.0×10^{3}	25	800	10
1	2.0×10^{3}	50 max Linear Range?	•	20
2	4.0×10^{3}	•	•	40
3	8.0×10^{3}	•	•	80
4	1.6×10^{4}	•	•	160
5	3.2×10^{4}	•	•	320
6	6.4×10^{4}	•	•	•
7	1.3×10^{5}	•	•	•
8	2.6×10^{5}	•	•	•
9	5.1×10^{5}	•	•	•
10	1.0×10^{6}	•	•	•
11	2.0×10^{6}	•	•	•
12	4.1×10^{6}	•	•	•
13	8.2×10^{6}	•	•	•

E5890

KEYBOARD

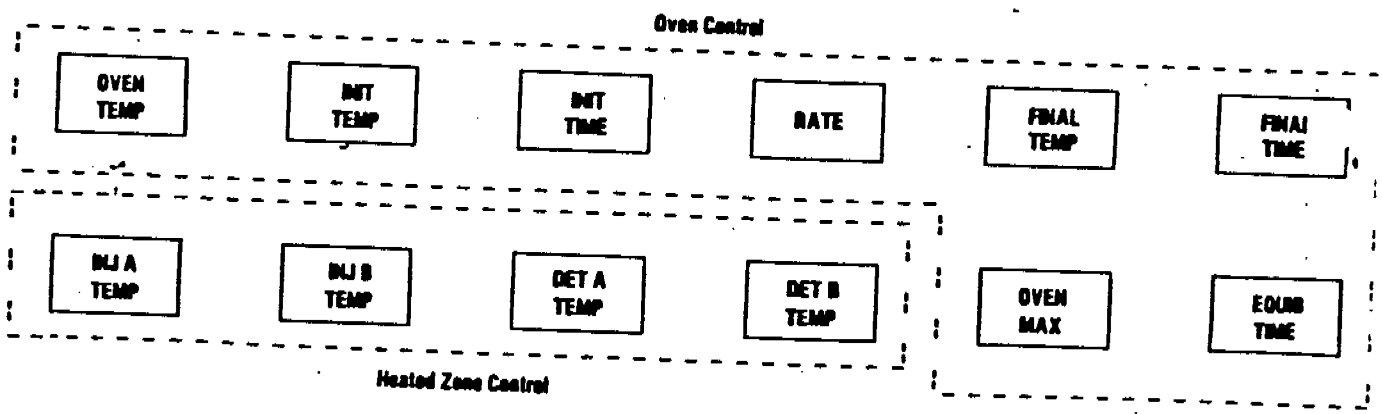
OPERATION

Keyboard and Displays



Temperature Control

Temperature Control Keys



DISPLAYING GAS FLOW RATE

Current flow rate is displayed by pressing:

[FLOW] [A] {or [B]}

													ACTUAL					SETPOINT	
	F	L	O	W		A			2	5	.	4				N	2		

													ACTUAL					SETPOINT	
			N	O		F	L	O	W		S	E	N	S	O	R			

KEYBOARD LOCK/UNLOCK

$\boxed{\text{CLEAR}}$ $\boxed{\cdot}$ $\boxed{-}$ $\boxed{\text{ENTER}}$ $\boxed{\text{ON}}$ / $\boxed{\text{OFF}}$

$\boxed{\text{ON}}$ . . . LOCK

$\boxed{\text{OFF}}$ . . . UNLOCK

065890

ACTUAL SETPOINT

CALIB AND.TEST [Ø-9]

ACTUAL SETPOINT

KEYBOARD LOCK ON

ACTUAL SETPOINT

KEYBOARD LOCK OFF

ASSIGNING A SIGNAL

Key(s)	Notes
[A] or [B]	To output the signal from either detector "A" or detector "B". The message " DET A (or " B ") NOT INSTALLED " is displayed if detector "A" (or "B") is not present.
[A] [−] [B] or [B] [−] [A]	To output a difference signal between two detectors of the same type. The message " DET A (or " B ") NOT INSTALLED " is displayed if detector "A" (or "B") is not present; the message " UNLIKE DETECTORS " is displayed if detectors "A" and "B" are not of the same type.

COLUMN COMPENSATION

Key(s)	Notes
[A] [−] [COL COMP 1] or [B] [−] [COL COMP 1] or [A] [−] [COL COMP 2] or [B] [−] [COL COMP 2]	To output a difference signal between a given detector and column and a stored "blank run" signal for the detector and column. The message " DET A (or " B ") NOT INSTALLED " is displayed if detector "A" (or "B") is not present.

PLOTTING OTHER PARAMETERS

0	To output oven temperature.
1 or 2	To output stored "COMP 1" or "COMP 2" data.
3, 4, 5, or 6	To output, respectively, inlet "A" temperature, inlet "B" temperature, detector "A" temperature, or detector "B temperature.
7 or 8	To output, respectively, carrier gas flow rate "A" or "B".
9	To output a test signal (stored chromatogram) for use in verifying proper operation of a data-receiving device (integrator, chart recorder, etc.).

TEST SIGNAL" OUTPUT

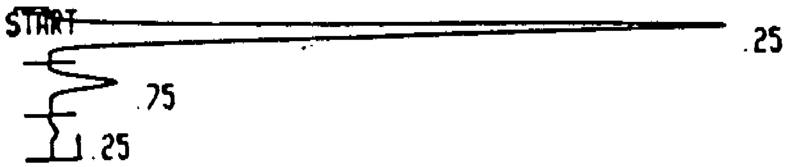
5890A:

RANGE 2↑( )	= 0
ATTN 2↑( )	= 0

1 V Analog Output

LIST: LIST
PEAK CAPACITY: 1159

ZERO = 0, -1.5
ATT 2↑ = 7
CHT SP = 1.0
PK WD = 0.13
THRSH = 0
AR REJ = 0



STOP

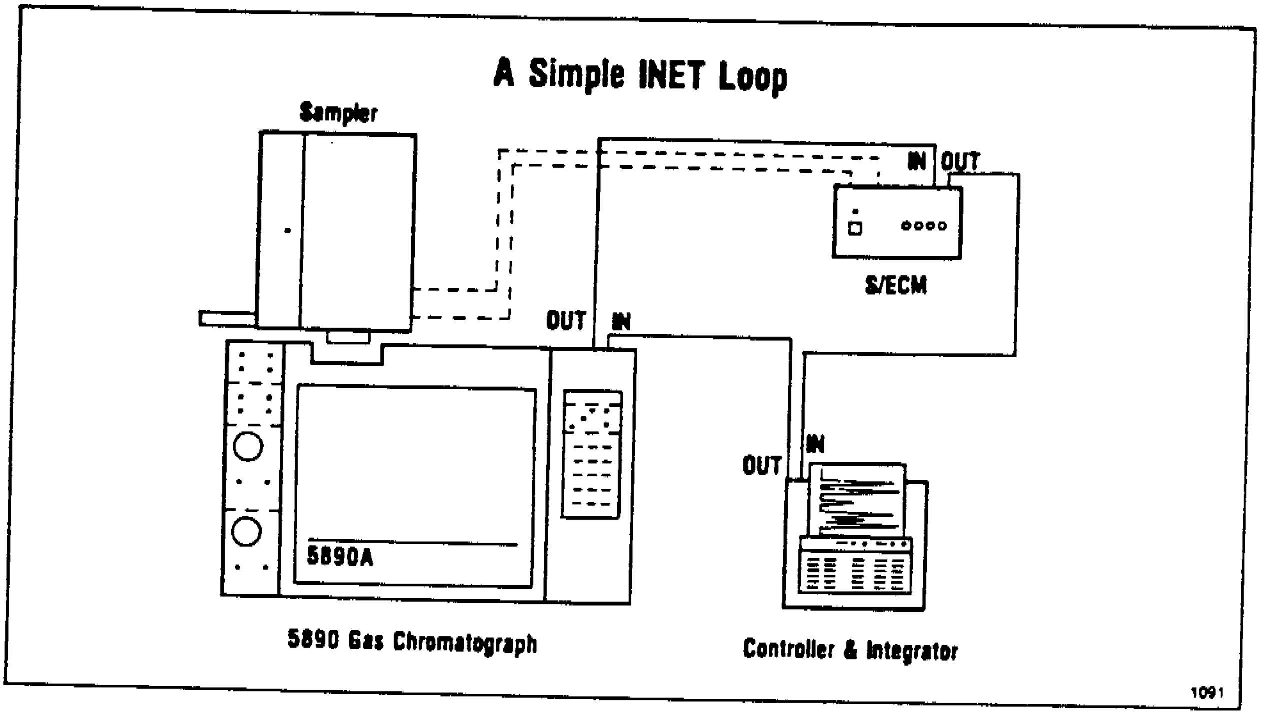
RUN # 8

AREA%

RT	AREA	TYPE	AR/HT	AREA%
0.25	7939400	BB	0.135	90.097
0.75	793530	PB	0.135	9.005
1.25	79094	BB	0.134	0.898

TOTAL AREA= 8812100
MUL FACTOR= 1.0000E+00

INET
The Instrument Network



COLUMN COMPENSATION

INITIALIZATION

COL COMP 1	A	ENTER	
COL COMP 1	.	ENTER	
SIG 1	A	–	COL COMP 1

085890

DAY 2

CAPILLARY

SYSTEMS

Capillary Injection Modes

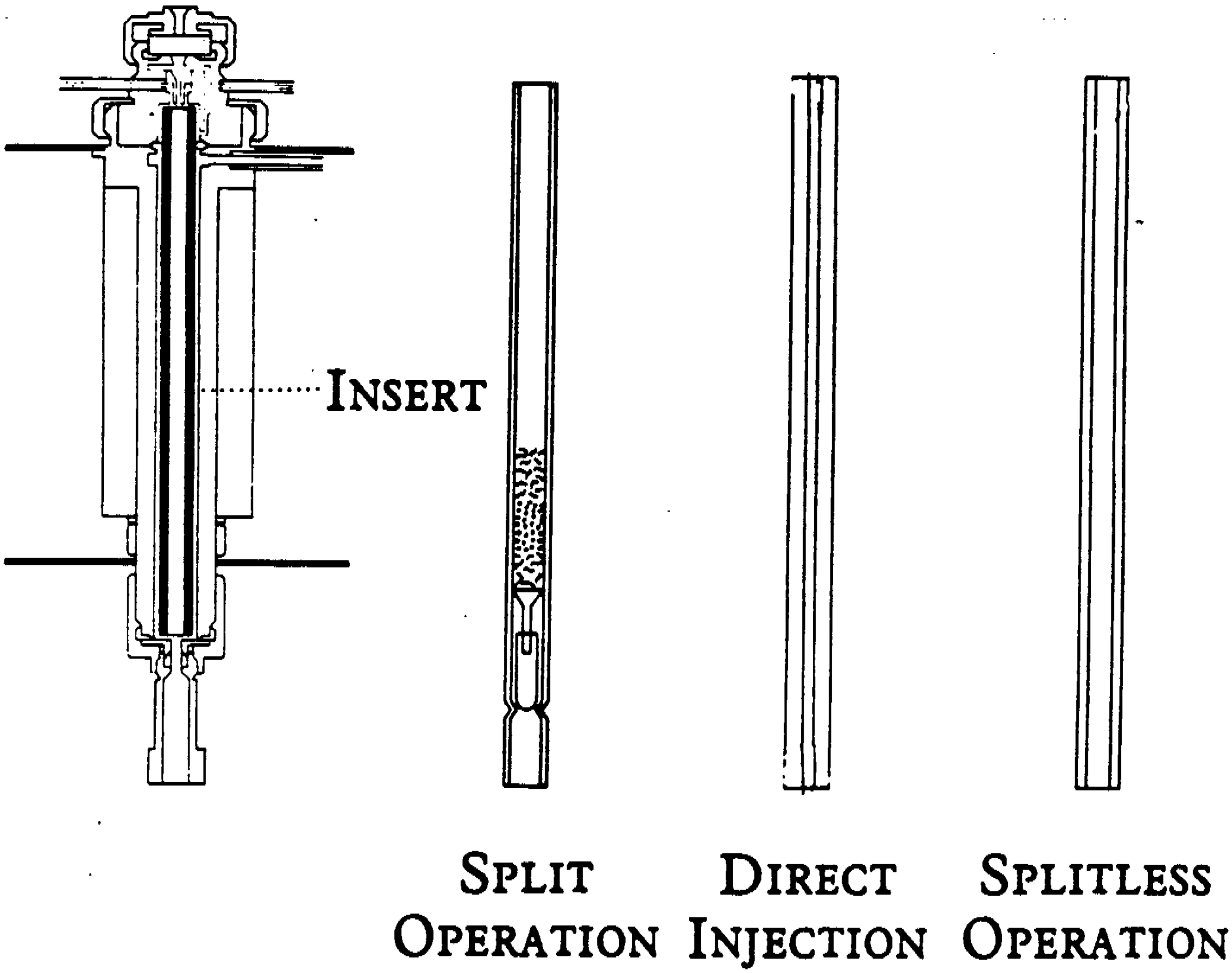
Split

Splitless, Purged
(Grob)

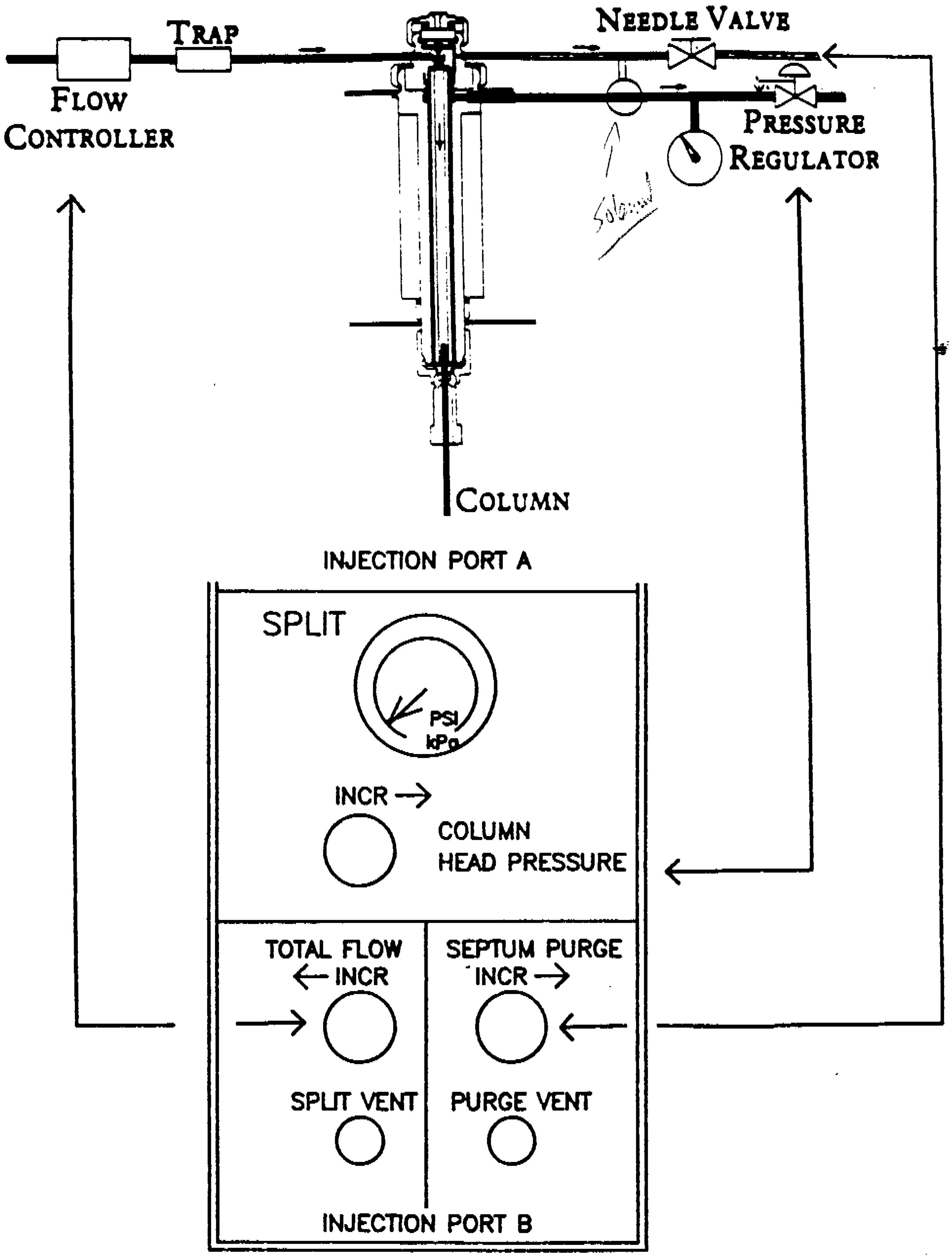
On-Column

Direct Injection
(No Purge or Split)

INJECTION PORT GLASS LINERS FOR THE DIFFERENT INJECTION MODES

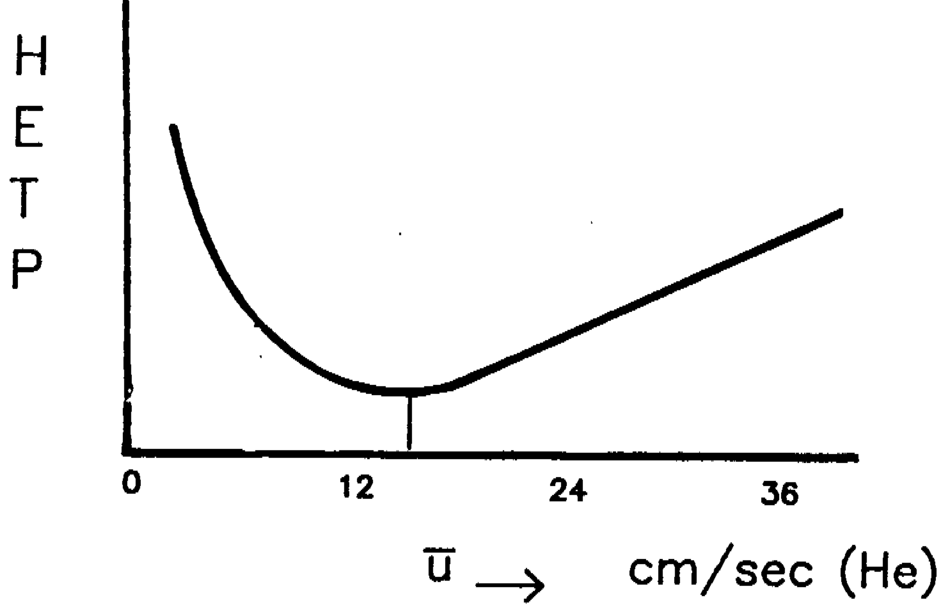


SETTING SPLIT FLOWS

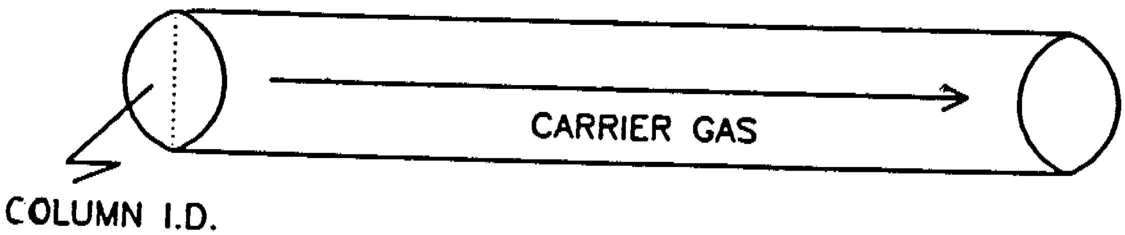


OP58802

LINEAR VELOCITY
COLUMN FLOW
SPLIT RATIO



Linear Velocity = $\bar{u}$ = Length/Gas Holdup Time



Column Flow $= (\pi r^2)(\bar{u})(60 \text{ sec/min})$

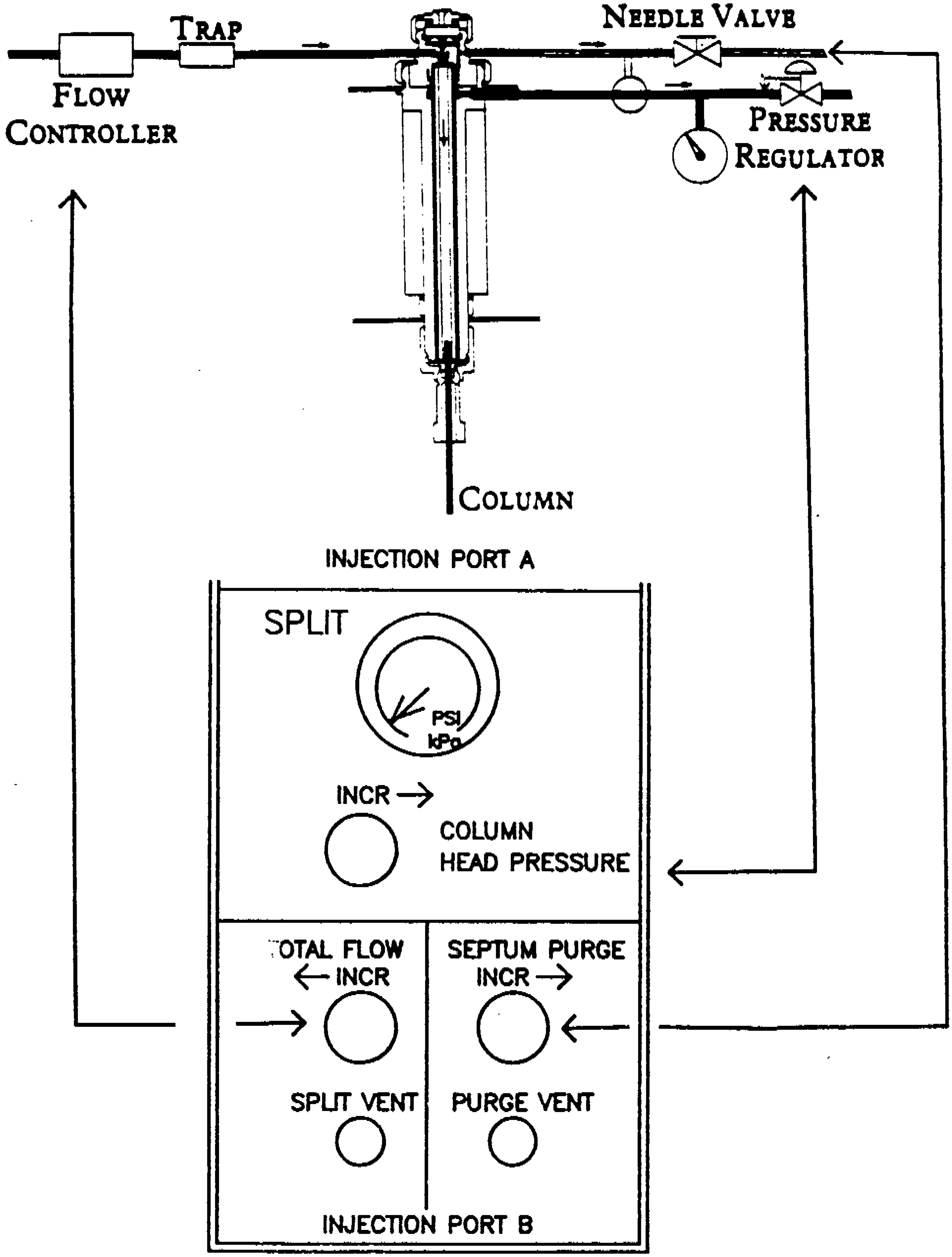
$= \text{cm}^3/\text{min} = \text{mL/min}$

Total Flow = Split Vent Flow + Column Flow

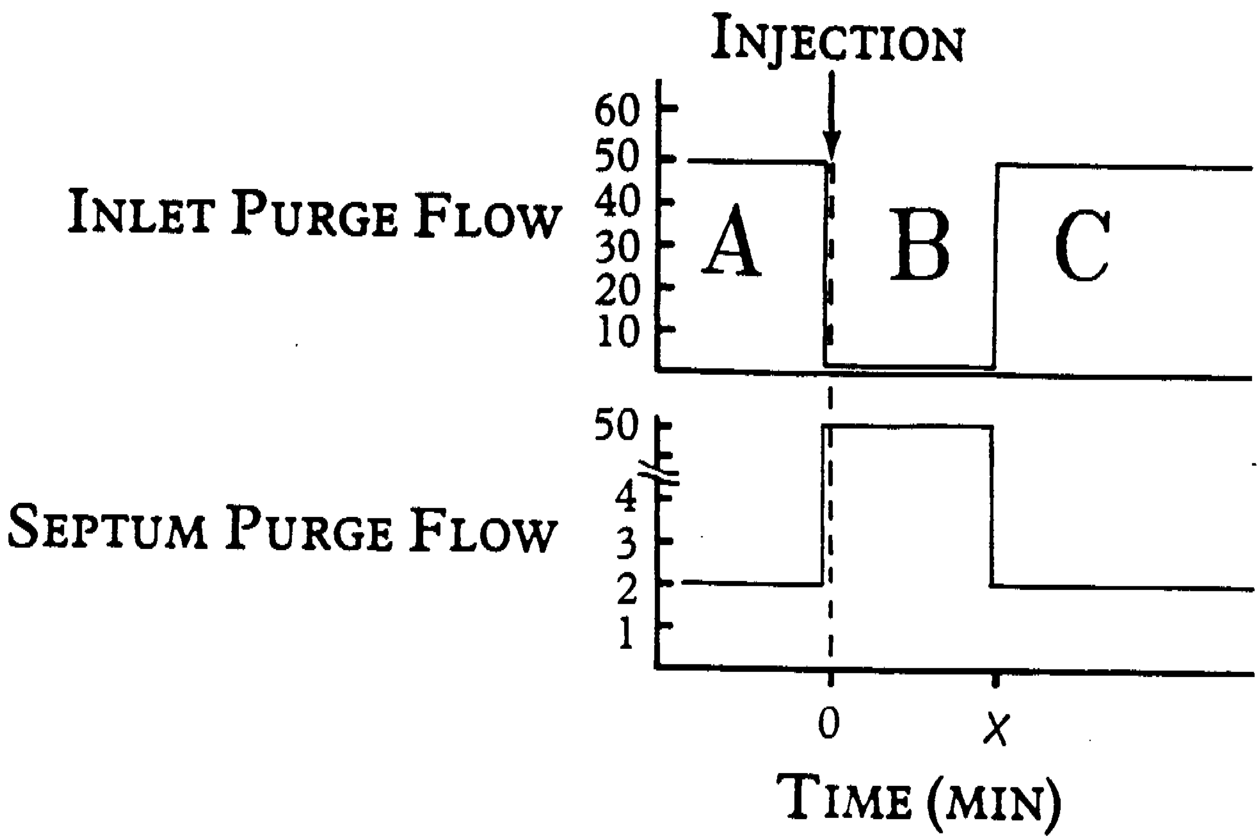
SPLIT RATIO:

Total Flow/Column Flow = Split Ratio

SETTING SPLITLESS FLOWS



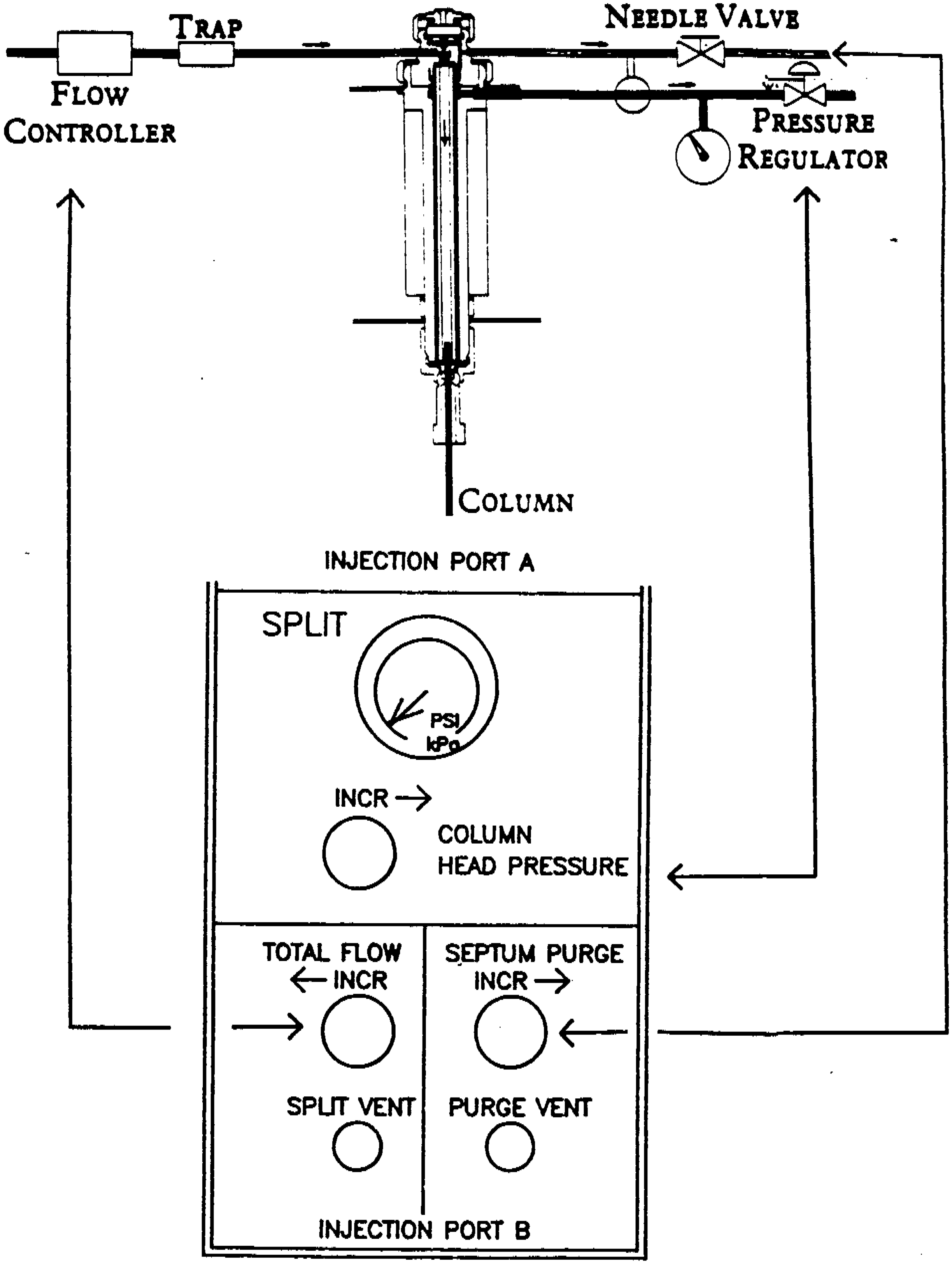
SPLITLESS PURGE TIMING



A	PURGE ON
B	PURGE OFF TIME 0.0
C	PURGE ON TIME XX

PURGE A/B TIME ON/OFF XX ENTER

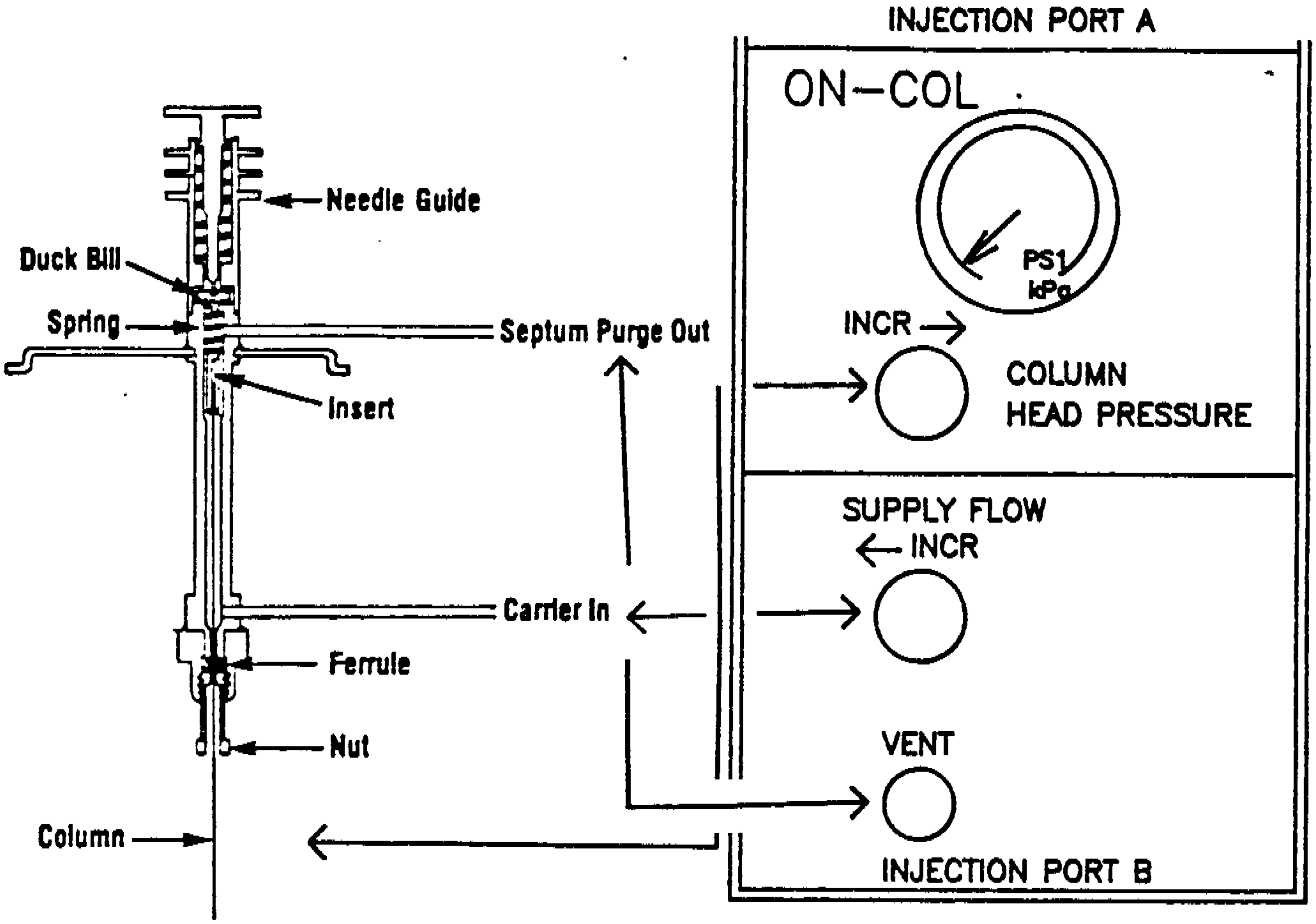
SETTING DIRECT FLOWS



OP58802

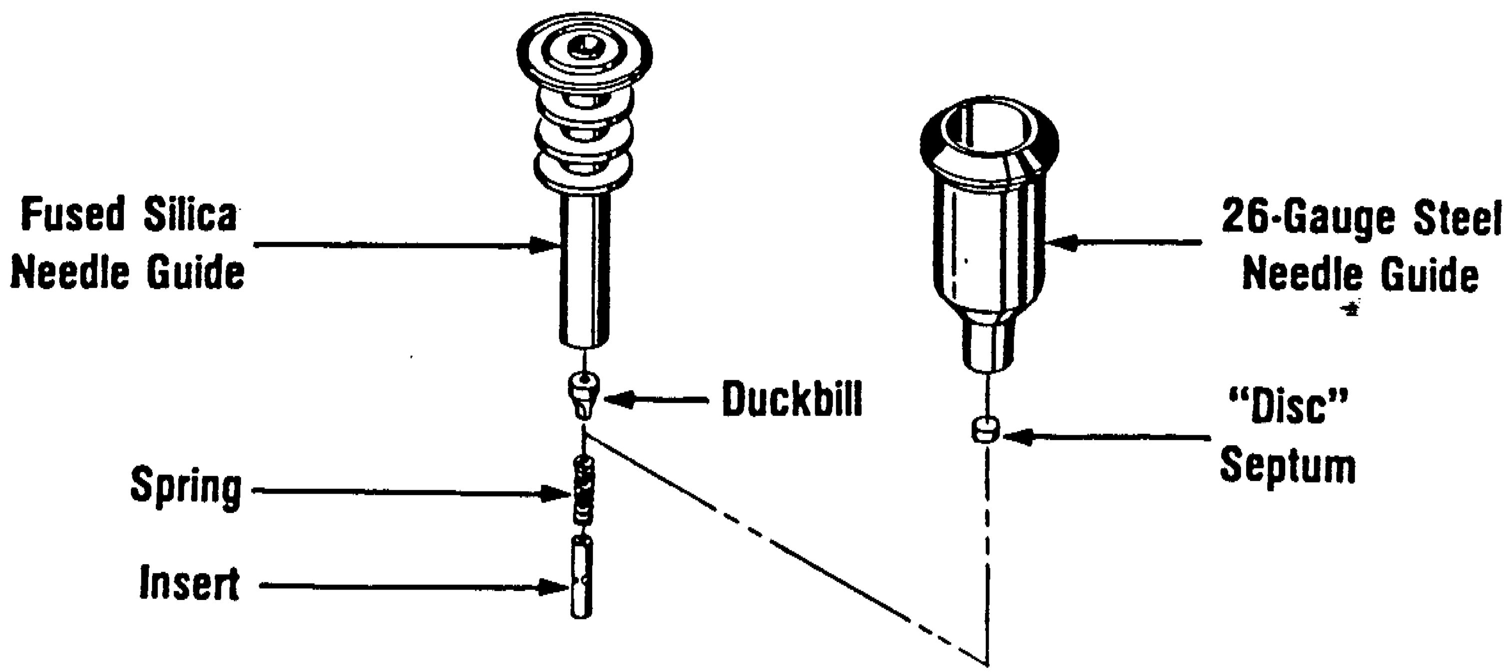
SETTING ON-COLUMN FLOWS

Done Below B.P. of Solvent.



F5890

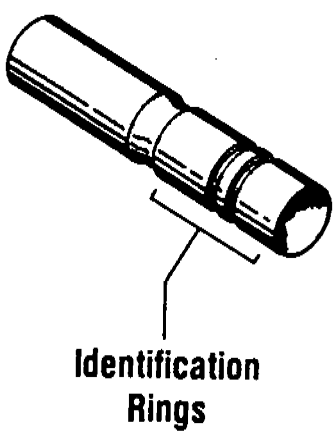
ON–COLUMN ACCESSORIES AND INSERTS



TYPICAL INSERT
DEDICATED ON-COLUMN CAPILLARY INLET

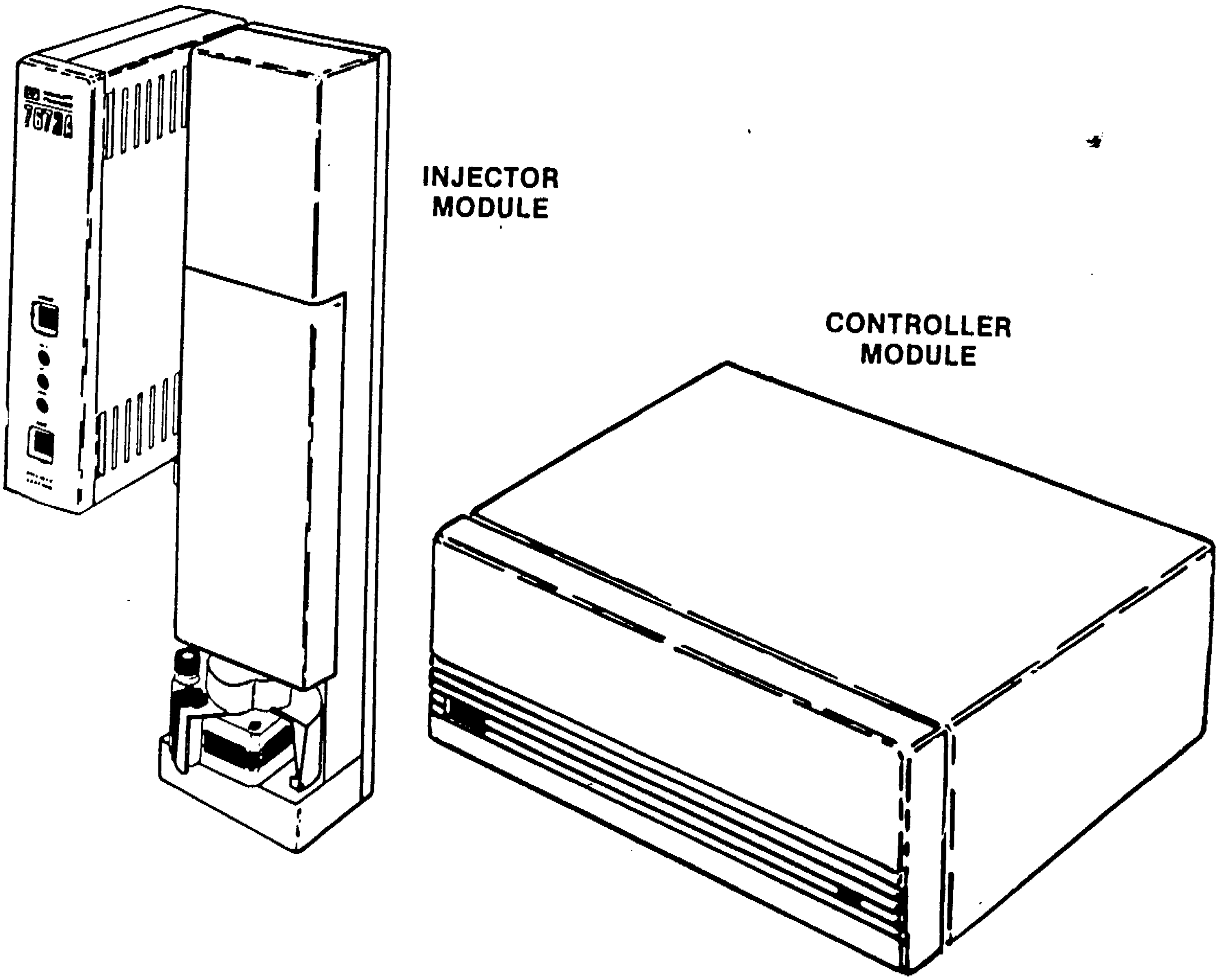
4 SIZES OF COLUMN INSERT

# OF RINGS	COLUMN ID
1	0.20 mm
2	0.32 mm
3	GLASS
NONE	0.53 mm

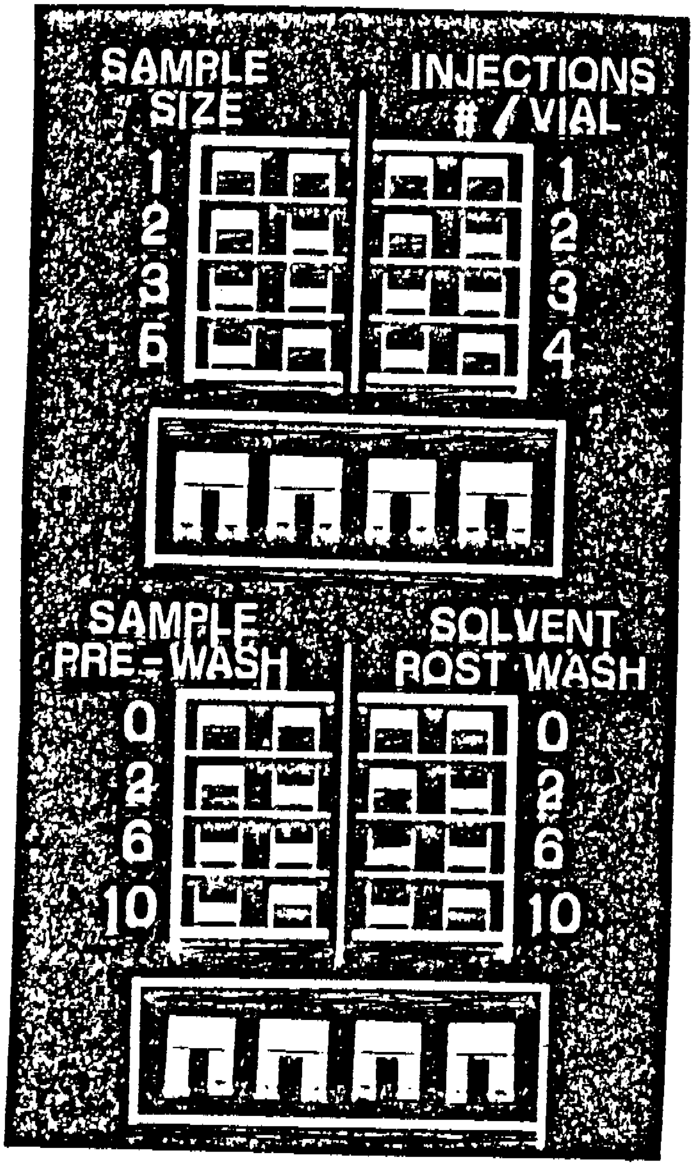


ALS

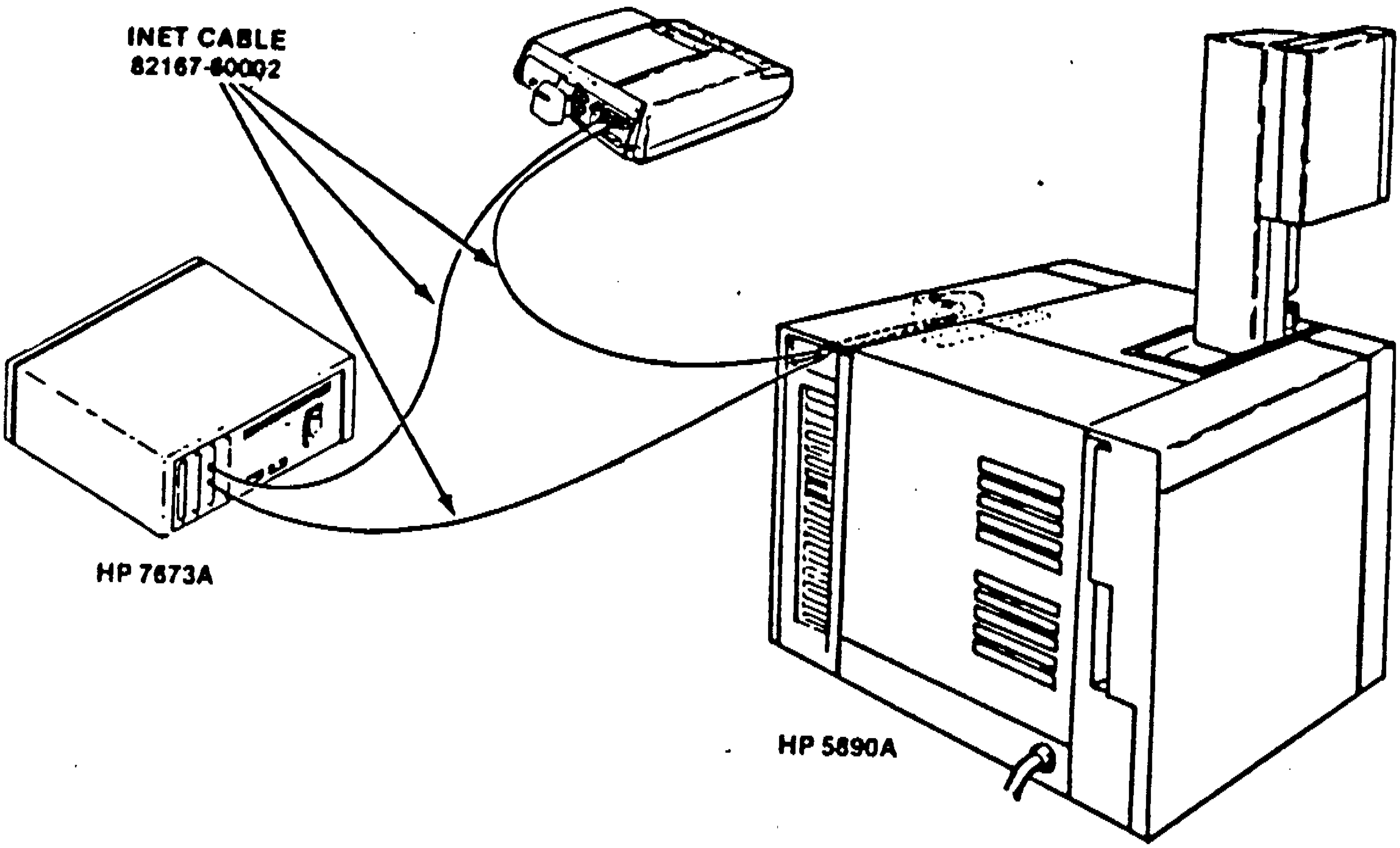
7673 SAMPLER SYSTEM
STAND ALONE CONTROL



INJECTION SEQUENCE CONTROL SWITHCES



7673 WITH HP5890 IN INET LOOP
3392 CONTROL



SAMPLER PARAMETER LISTING

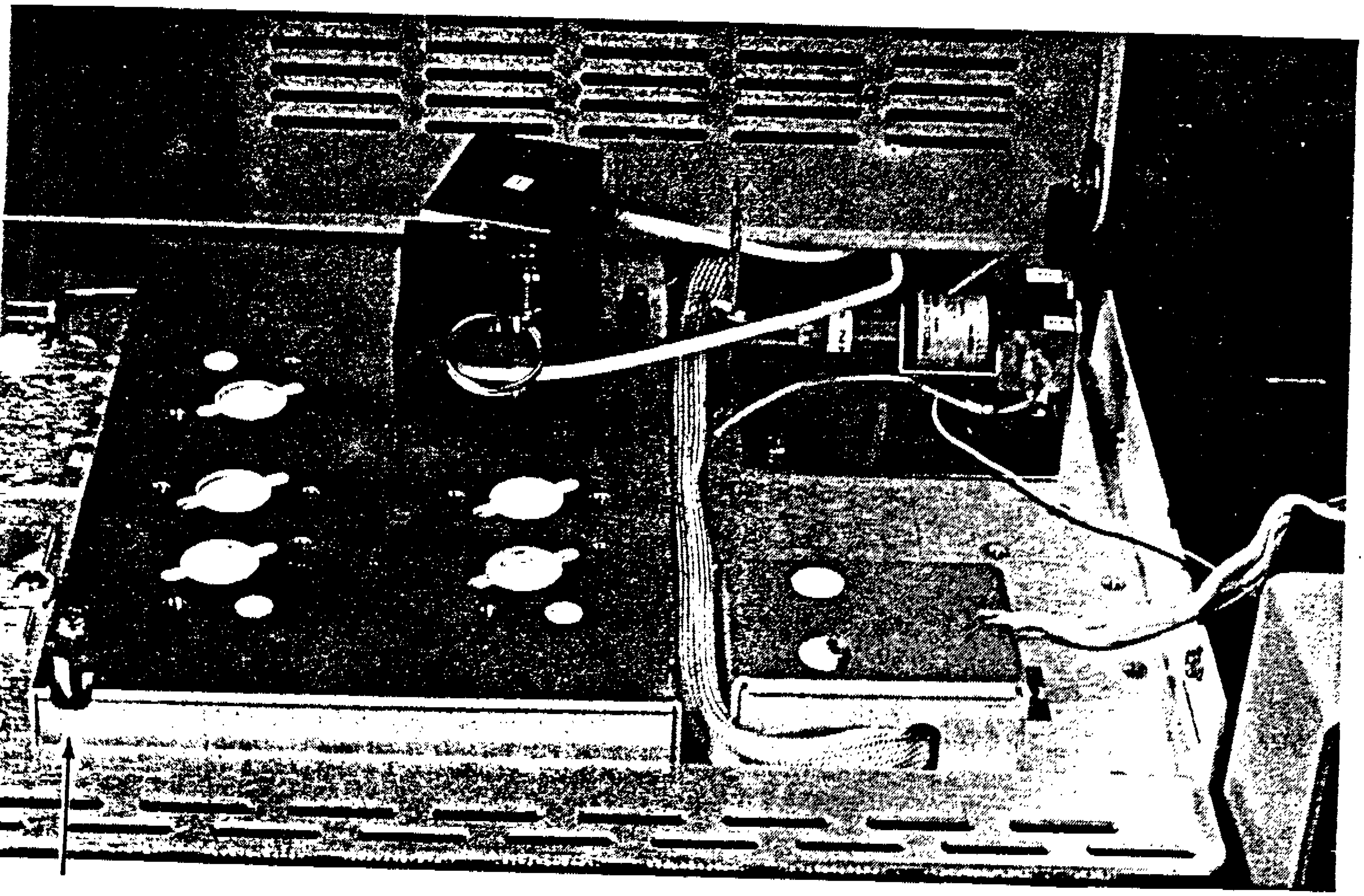
LIST: OP # 1 1 @

7673A SAMPLER:
LOOP ADDRESS: 1

REAR INJECTOR
INJ/BOTTLE
FIRST BOTTLE
LAST BOTTLE
OF SAMPLE WASHES
OF PUMPS
VISCOSITY
VOLUME
OF SOLVENT A WASHES
OF SOLVENT B WASHES
PRIORITY SAMPLE (1=YES)
ON-COLUMN (1=YES)

VALVING

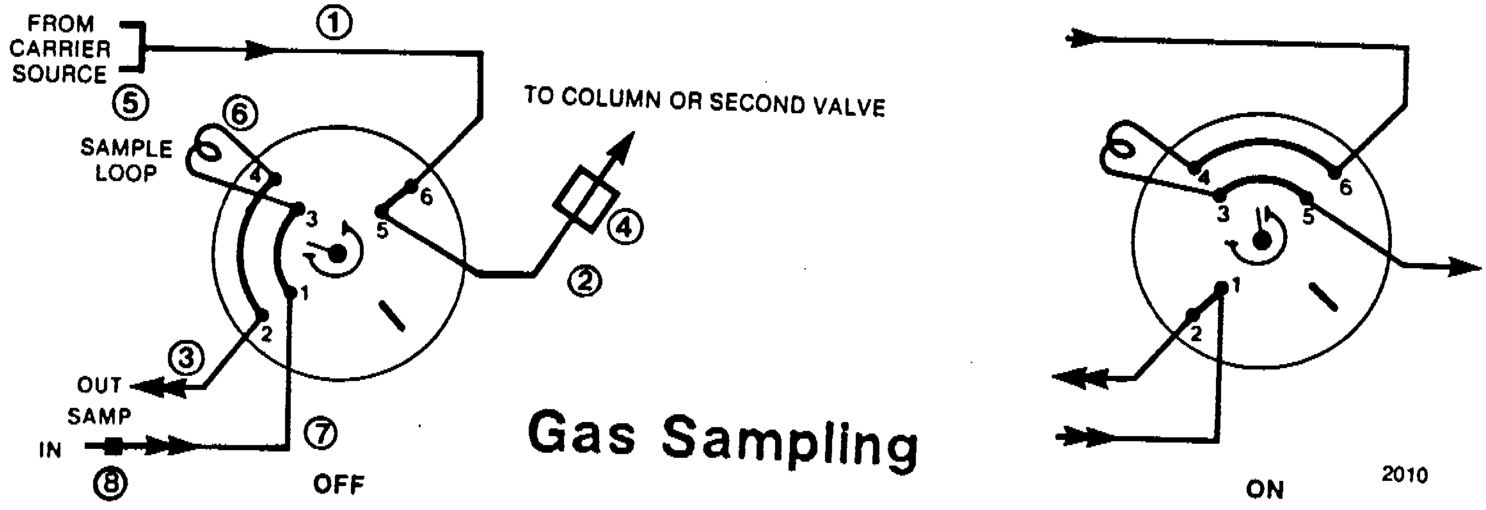
Accessory 19238A: Valve Box



HEX SCREW

1516

VALVE CONTROL



INTERNAL VALVE CONTROL

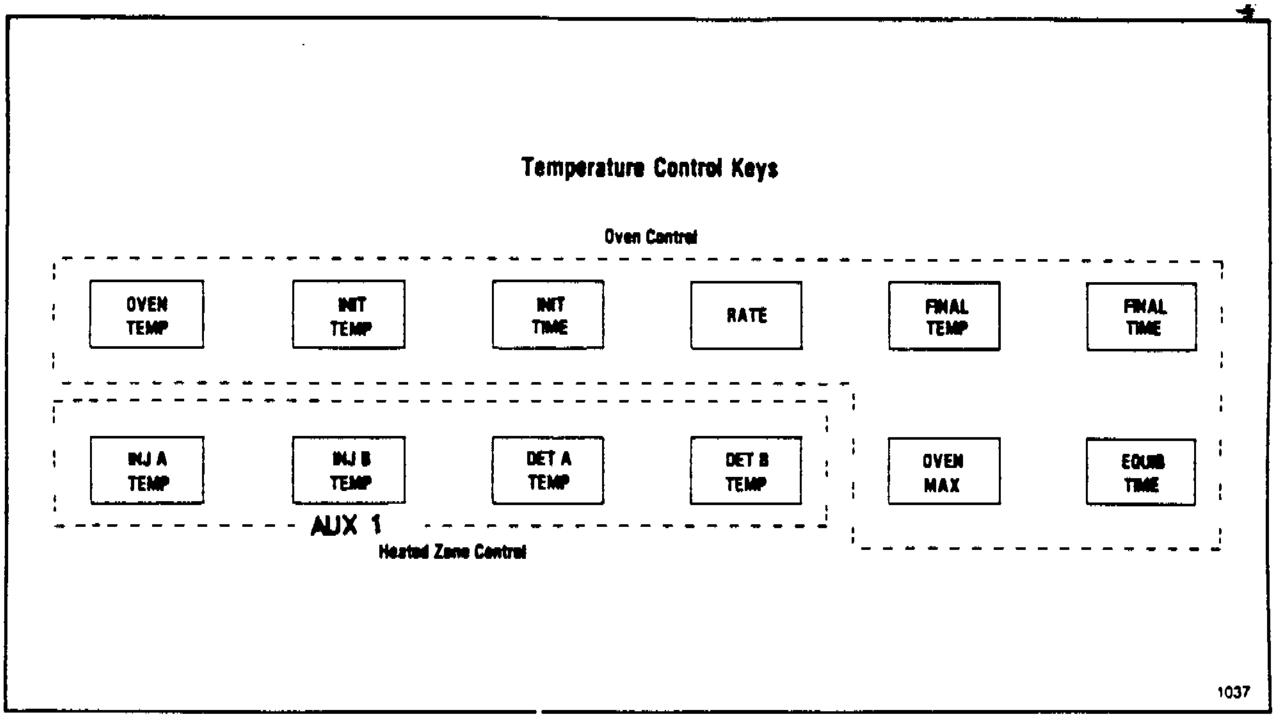
PURGE | A | ON

PURGE | A | TIME

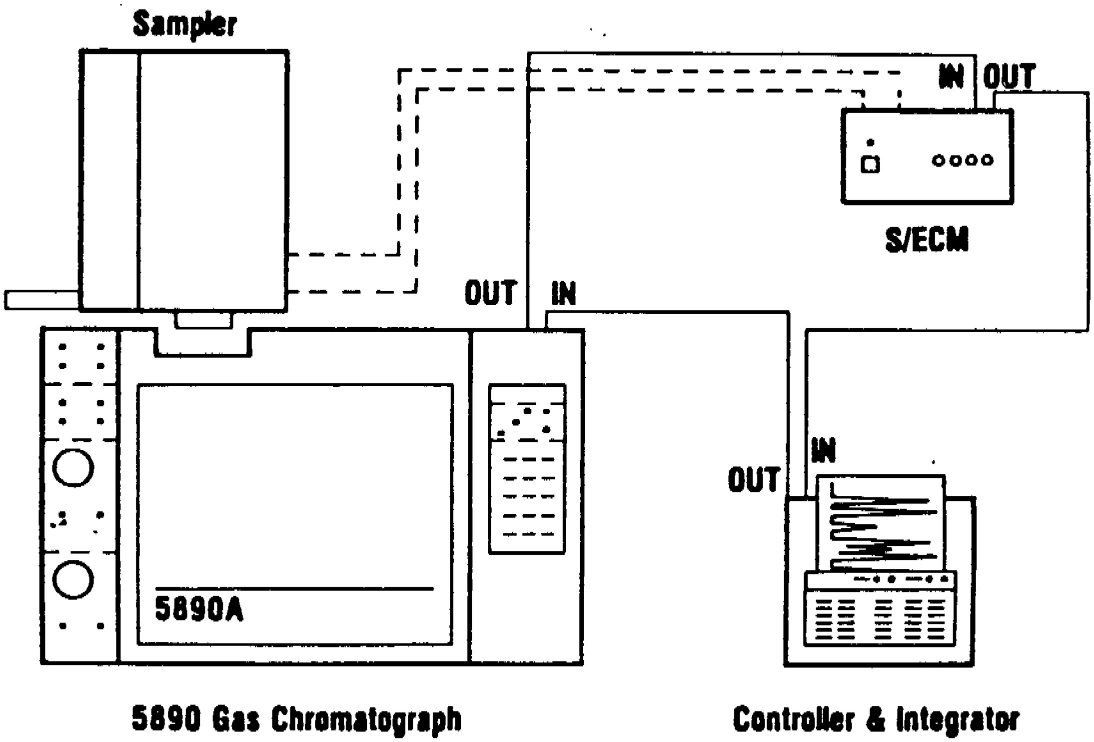
OFF | 0.0

ON | 5.0

Temperature Control



A Simple INET Loop



LABORATORY

EXERCISES

LAB 1: INSTRUMENT SET-UP (1 hour)

OBJECTIVE: *To become familiar with the set-up and turn on procedures for the 5890A Gas Chromatograph.*

1. *Connect the INET cables between the GC and the 3392.*

2. *Turn on the 5890 and observe the self test on the 5890 keyboard display.*

3. a). *Set the detector temperature for the FID to 300 C.*

   b). *Set the injector temperature for the packed or packed purged injection port to 200 C.*

   c). *Set the oven temperature to 150 C.*

4. *Set the column flow rate to 30 $\pm$ 2 mL/min.*

LAB 2A: PERFORMANCE CHECKOUT (0.5 hour)

1. a). Set H_2 flow to 30 $\pm$ 3 mL/min.

   b). Set air flow to 400 $\pm$ 20 mL/min.

2. a). Turn the FID detector ON

   b). Assign the FID to SIGNAL 1.

   c). While monitoring signal 1 from the keyboard display, ignite the flame.

   d). How do you know the flame is lit?

3. a). Turn the 339~~2~~3 integrator ON.

   b). What does "Loop-Up" indicate?

   c). Where do you need to press START to have the 3392 and 5890 both run at the same time? Try it.

4. a). Set Range to ? 8 on the 5890 and ATTN to ~~3~~ 6 on the 339~~2~~3

   b). What should ATTN be on the 5890? ?

   c). Make certain all heated zones are at Lab 1 setpoints.

5. Press the CLEAR button on the 5890. When the 5890 is ready, INJECT 1 microliter of sample #1 and press START.

   After the solvent plus 3 major peaks have eluted, press STOP.

   If the area counts of C14, C15 and C16 are each greater than 150,000, the system meets performance specifications.

LAB 2B: TEMPERATURE PROGRAMMING (1.5 hours)

1. *Set in the following temperature program:*

INIT. TEMP	*120*
INIT. TIME	*0.0*
RATE	*20*
FINAL TEMP	*160*
FINAL TIME	*1.0*

How long will this run take?

Did you have to calculate the time?

How could the instrument do this calculation for you?

2. *Inject 1 microliter of sample #1 and press START.*

Do you need to press STOP to end the run?

Do you have better separation between solvent and C14 than you did with the 150 C isothermal run?

3. *Set in the following temperature program:*

INIT. TIME	*120*
INIT. TIME	*0.0*
RATE	*30*
FINAL TEMP	*150*
FINAL TIME	*0.5*
RATE A	*10*
FINAL TEMP A	*170*
FINAL TIME A	*1.0*
RATE B	*20*
FINAL TEMP B	*190*
FINAL TIME B	*1.0*

4. *Set RANGE to 10* 0

   Set SIGNAL 1 to OVEN TEMP

   Set ZERO to 20480

   Set 339~~2~~ 3 ATTN to ~~3~~ 8

   Why is it necessary to set zero so high?

   What is signal 1 background with the oven at 120 C?

   Would that exceed 1 volt?

5. *Press START and observe the temperature ramp on the integrator.*

6. *Set RANGE to 8*

Set SIGNAL 1 to FID signal

Set ZERO to 0.0

Press COL COMP 1

Press A or B (appropriate FID Detector)

What occurs when you press enter?

7. *After the run is complete, assign COL COMP 1 to be subtracted from the FID signal and press START.*

Does this give you a better baseline for integration and presentation?

8. *DELETE Program A and B with one entry.*

Is RATE B still in memory?

Can you run RATE B without A?

9. *Reset the 5890 to default conditions.*

LAB 3: CAPILLARY (1.5 hour)

1. *Install the 12 meter, 0.20 mm column between the split/ splitless inlet and the FID detector.*

   28 mm dimension for inlet end

   92 mm dimension for detector end

2. *Install the split liner in the capillary inlet.*

3. *Heat the inlet to 200 C, the detector to 300 C and the column to 150 C.*

4. *Set column head pressure at 10psi and turn the FID detector on.*

5. *Adjust the column head pressure for a linear velocity of 30-35 cm/sec, using 1 microliter of methanol as a non-retained peak.*

6. *Add AUX. GAS to bring total flow up to 30 mL/min.*

7. *Set split vent flow to give a 30 to 1 split ratio.*

8. *Inject 1 microliter of sample #1.*

9. *Now set up as required to inject 1 microliter of sample #1 in the splitless mode.*

   What is the best purge on time?

3393

INTEGRATOR

HP3393A KEYBOARD

TWO MAJOR GROUPS

FUNCTION KEYS

ALPHANUMERIC KEYS

OPERATING MODES and PROMPTS

System Command Mode *

BASIC >

Plot [PLOT]

Integrate [START] or [ANALYZE]

Host Computer #

KEY SEQUENCE CONVENTIONS

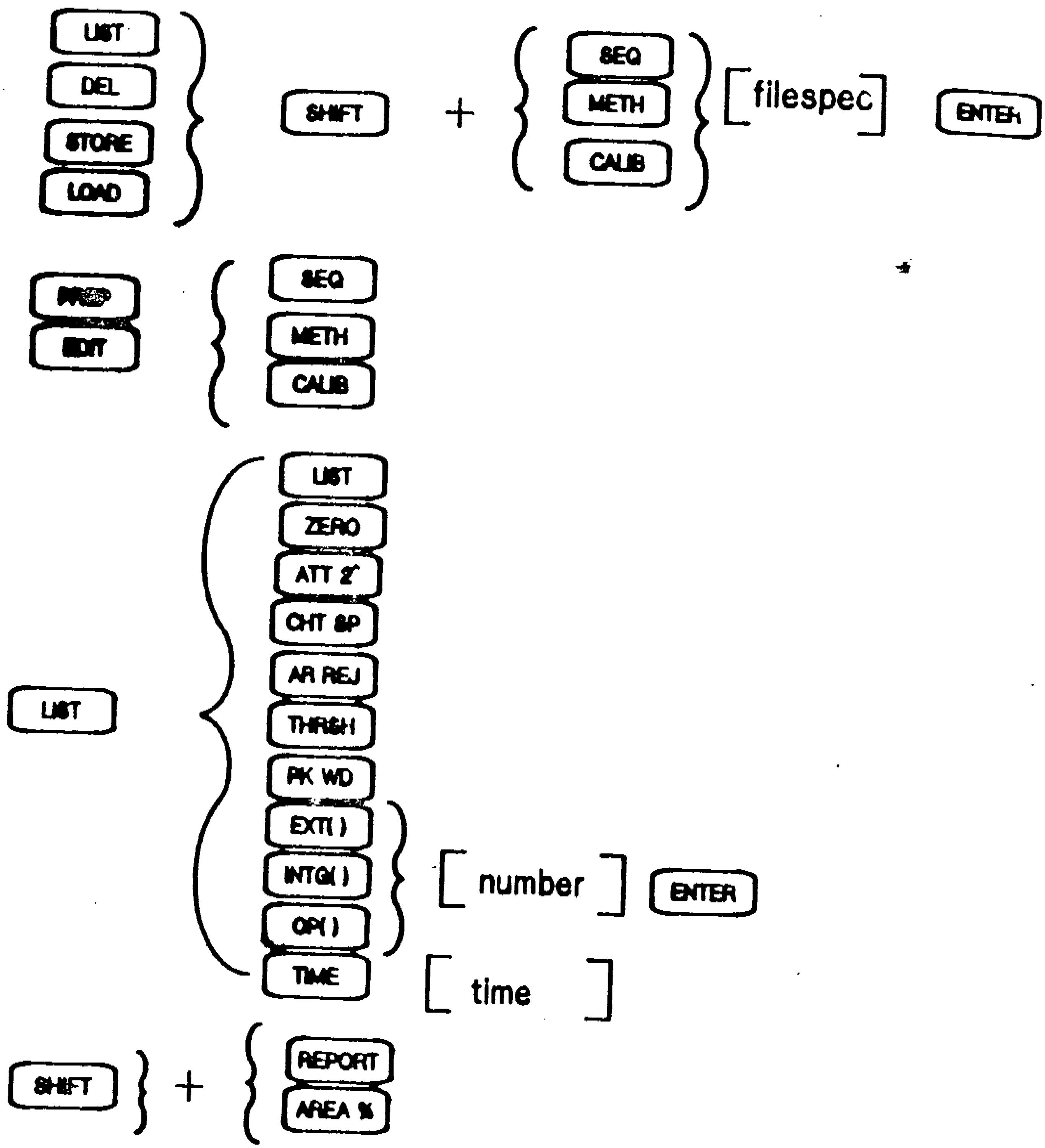
[ACTION] [FUNCTION] [VALUE] [ENTER]

[LIST] [OP()] [4] [ENTER]

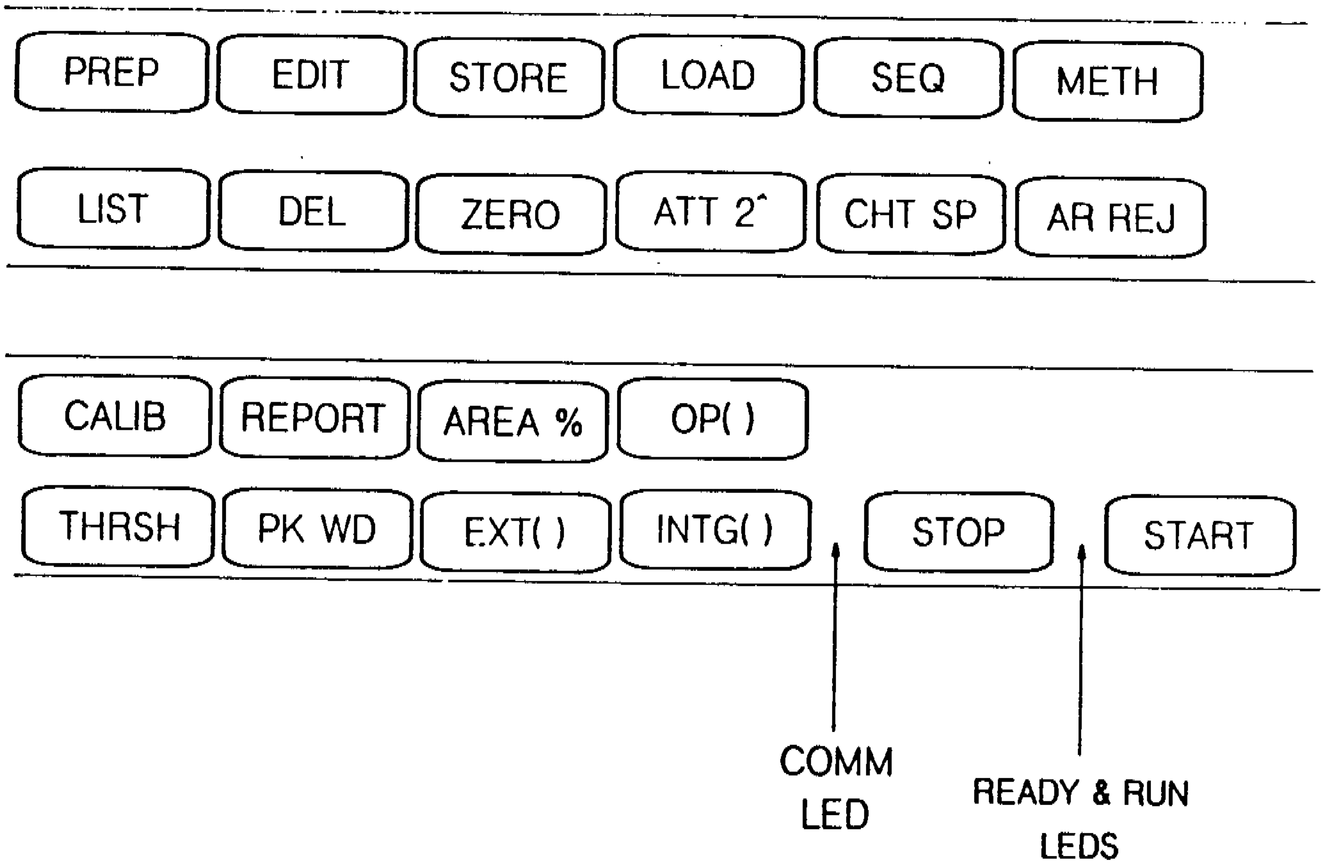
[SHIFT] TO FUNCTIONS APPEARING IN GOLD

HP 3393A SPECIAL KEYS

VALID SEQUENCES



HP 3393A SPECIAL KEYS



THE OPTION FUNCTIONS

To list an OP() function:

LIST   OP()   option#   ENTER

To activate an option:

OP()   option#   ENTER

To turn off an option:

OP()   –   option#   ENTER

LIST OF OPTIONS:

* LIST: OP #

(1) INTEGRATION PLOT TYPE

(2) RUN DATA STORAGE

(3) CALIBRATION OPTIONS

(4) REPORT OPTIONS

(5) POST–RUN LIST OPTIONS

(6) REMOTE DEVICE ACCESS

(7) DEFAULT SAMPLE INFORMATION

*

List of Integration functions:

most work with [Time]

LIST: INTG # @

0. SET BASELINE NOW
1. SET BASELINE NEXT VALLEY
2. SET BASELINE ALL VALLEYS
3. SKIM FROM NEXT PEAK
4. DISABLE AUTO TANGENT SKIM
5. EXTEND BASELINE HORIZONTALLY
6. MEASURE AND UPDATE THRESHOLD
7. TURN OFF RETENTION TIME LABEL
8. TURN OFF START/STOP MARKS
9. TURN OFF INTEGRATION
10. INCREMENT THRESHOLD

ALPHANUMERIC KEYBOARD

Typewriter keyboard except:

* $\boxed{\text{ENTER}}$ Replaces Carriage Return
* Includes $\boxed{\text{CTRL}}$
  $\boxed{\text{ESC}}$
  $\boxed{\text{BREAK}}$
* $\boxed{\text{SHIFT}}$ Prints Lowercase or Selects Superscript
* $\boxed{\text{CNTRL}}$ C $\equiv$ $\wedge$ $\boxed{\text{C}}$

  Reverse the shift key

ALPHANUMERIC KEYS

USED TO COMMUNICATE:

* numeric entries within key sequences
* system commands
* dialog responses for options
* analysis related information such as sample and component names, laboratory memo information, and report information
* BASIC program statements and commands
* text, when appropriate

SYSTEM COMMANDS

* ADMINISTRATIVE
* FILE MANAGEMENT
* REINTEGRATION
* BASIC
* HP-IL

LISTING ALL SYSTEM COMMANDS

```
*HELP
SYSTEM COMMANDS:
ANALYZE ASSIGN BASIC BX COPY CREATE DATE
DIRECTORY FORMAT HELP IDENTIFIER
INET_CONFIGURATION LOCK NOTEPAD PACK
PURGE READY RECONFIGURE RENAME SET SYSTEM
TIME UNLOCK XADDRESS
```

Command	Description
ANALYZE	Starts the reintegration of stored run data.
ASSIGN	Assigns a BASIC program to a numeric key for automatic execution when the key is pressed.
BASIC	Enters HP 3393A BASIC Mode
BX	Allows both input and output to BASIC mode from a terminal connected to the RS-232-C port
COPY	Makes a copy of a file.
CREATE	Creates a new file.
DATE	Lists and sets the date.
DIRECTORY	Provides a list of all files and space on the specified mass storage medium

Command	Description
FORMAT	Initializes a disc to the recording format used by the HP 3393A.
HELP	Lists all system commands.
IDENTIFIER	Sets the 12-character alphanumeric identifier.
INET_CONFIG	Lists and permits changes to the INET Configuration
LOCK	Prevents a host computer from communicating with the HP 3393A.
NOTEPAD	Allows you to make notations on the printer/plotter chart from command mode
PACK	Recovers lost disc space on specified disc
PURGE	Deletes a file.

Command	Description
READY	Reports on INET system readiness
RECONFIGURE	Reconfigures the HP-IL
RENAME	Changes the name of a file.
SET	Sets run parameters and the rur number listed on reports.
SYSTEM	Lists HP-IL and RS-232-C configura- tion: device numbers, instruments or devices, INET objects and some RS-232-C settings
TIME	Lists and sets the time of day.
UNLOCK	Allows a host computer to communi- cate with the HP 3393A.
XADDRESS	Sets the HP-IL printer address for

SPECIAL COMMANDS

$\boxed{\text{SHIFT}}$ $\boxed{\text{ENTER}}$

$\boxed{\text{BKSP}}$

$\boxed{\text{CNTRL}}$ $\boxed{\text{R}}$

$\boxed{\text{ESC}}$

$\boxed{\text{BREAK}}$

$\boxed{\text{BKSP}}$

```
*DATR※E
JAN  1, 1901  00:01:20
```

$\boxed{\text{CNTRL}}$ $\boxed{\text{R}}$

```
*DATR※E  =
DATE
JAN  1, 1901  00:01:35
```

WARNING SIGNALS

BEEP! (AUDIBLE)

* Key invalid in sequence
* System command during PLOT, INTEGRATION, or REINTEGRATION

? (Printed)

* Numeric value outside limits

HP 3393A — LED STATUS

- LEDs:
  * Keybd
  * Comm
  * Ready
  * Run
- Three states:
  * On solid
  * Off solid
  * Blink

HP 3393A LED STATUS

Meaning	Keybd	Comm	Ready	Run
Keyboard Entry Allowed	ON			
BX active or OUT OF PAPER	B			
HOST DRIVEN		ON		
COMMUNICATION in progress (node-driven)		B		
Processing data or listing	OFF			
No Communication Activity		OFF		
INET is ready			ON	
Auto-threshold in progress			B	
INET not ready for start			OFF	
Run in progress				ON
Post run				B

ON = ON SOLIDLY
OFF = OFF SOLIDLY
B = BLINKING

```
*  LIST: METH  @

RUN PARAMETERS
ZERO    =  0
ATT 2^ =  0
CHT SP =  1.0
AR REJ =  0
THRSH  =  0
PK WD  =  0.04

TIMETABLE EVENTS
EMPTY

CALIBRATION
NO CALIB TBL

INTEGRATION PLOT TYPE ....... FILTERED

RUN DATA STORAGE
Store signal data ........... NO
Store processed peaks ....... NO

REPORT OPTIONS
Suppress local report ....... NO
Peak height mode ............ NO
Report uncalibrated peaks ... NO
Extended report ............. NO

POST-RUN LIST OPTIONS
Store post-run report ....... NO
External post-run report .... NO
List run parameters ......... NO
List timetable .............. NO
List calibration table ...... NO
List remote method .......... NO

*
```