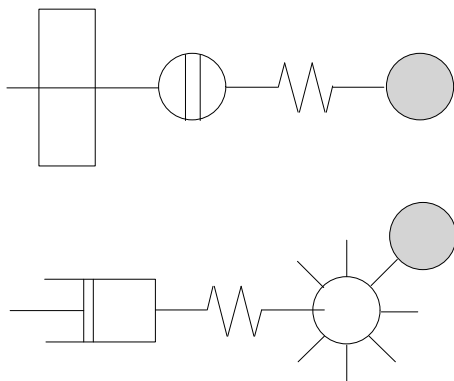


# Reference Manual



**FlowTEK**

Ver 1.5



**FlowTEK™**

**Reference**

**Manual**

**Device Control and Data Acquisition  
software  
Ver 1.5**



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# Welcome to FlowTEK

There is an increased demand for automation in analytical laboratories. In fact, this demand for quicker turn around time has spurred the development of process analysers for rapid analysis in the plant environment. Although this approach to quality assurance, environmental monitoring, and process control is the logical approach for the future, the need for a competent and efficient laboratory will remain.

The advent and wide use of the microprocessor will, in the coming years, continue to have a dramatic impact on the design of analytical instrumentation. Performance and capabilities of present instrumentation have benefited dramatically through the use of dedicated micro processors. Instrument engineers will continue to develop instrumentation along these lines.

Although the ultimate goal for the instrument engineer is to develop a specific interference-free sensor that requires no sample preparation, the commercial application of such devices still falls into the realm of science fiction. In many cases, some form of sample manipulation is still required. The techniques of Flow-Injection Analysis (FIA), and Sequential-Injection Analysis (SIA) together with several other flow-based procedures provide a convenient and easily automated option to the analyst to achieve the required sample manipulation.

FlowTEK, analyser control software produced by MINTEK, is capable of providing the flow-based analyser with micro processor control and data acquisition capabilities. This package, which is designed with numerous user-defined set-up options, will meet the requirements of research scientist, laboratory analyst, method developer, and process analyst. It is designed to improve productivity, simplify analyser use, and improve data quality through automated analyser control.

With FlowTEK, you will have the advantage of microprocessor control for your flow-based analyser at your fingertips.

Please complete and post your registration form as soon as possible to:

Mintek

Private bag x3015

Randburg, 2125

South Africa

If you do this, we will keep you informed about updates and enhancements. MINTEK is also in a position to discuss your particular application of this software with you so that you can maximise the use of this package.

# Manual conventions

When describing the procedure to follow to achieve a certain menu option, the keys to type are printed in **Bold** print. Often the rest of the word is given in normal print as in **Bold**; only the **B** must be typed.

The manual contains warnings and precautionary statements that can prevent personal injury, equipment damage, and the loss of data. All statements of this nature are called to your attention through the use of one of the following icons.



**WARNING!** Warnings alert you to situations that could result in personal injury or damage to the instrument or loss of data. They also tell you how to avoid them. This icon is also used for chemical hazards. Warnings always appear in bold type.



**WARNING - HIGH VOLTAGE!** This warning alerts you to the presence of high voltages and to the potential injury that could occur from electrical shocks. It also tells you how to avoid high voltage areas in the instrument. All high voltage warnings appear in bold type.



**CAUTION!** Cautions alert you to the correct operating or maintenance procedures to avoid damage of the instrument. All cautions appear in bold type.



**HINT:** *Hints draw your attention to general rules or short cuts. They specify ways to obtain the best performance or results from the equipment. All hints appear in italics.*



**NOTE:** *Notes alert you of important exceptions, side effects, or unexpected occurrences that might result from certain actions. All notes appear in italics*

# 1. Getting Started

## 1.1 YOU MUST READ THIS

If you are familiar with FlowTEK and do not want to read through the entire manual, then you can install the software as follows:

- Read the Licence agreement before you open the envelope with the disk in it to ensure that you are complying with the terms of the licence agreement.
- Place the FlowTEK diskette in drive A and type:
- **A:<Enter>**
- Type **INSTALL<Enter>**
- Enter the designation of the source and destination drives.
- Select the letter corresponding to the interface board that you are using:  
**E**-Eagle Electric PC-30B, **R**-RTD ADA2200, **P**-PC104 DM5406
- Register your system by returning the enclosed registration form.

The install program will create a directory called \FlowTEK, on your hard disk. All the program files will be copied to this directory. It will also create a sub-directory \FlowTEK\data, for your data files. Several sample data files will be copied to this sub-directory. The Quattro Pro work sheet for installing reduced data into Quattro Pro will be copied to the data sub-directory. You may want to copy this to your Quattro Pro data sub-directory. A batch file, ft.bat will be created as well. This batch file will specify the interface board that you use. You can copy this batch file into a sub directory in your path.



**NOTE :** *If you want to run FlowTEK by calling the executable you must add a command line switch to specify the board. The options are as follows  
/E for the Eagle Electric PC-30B,  
/R for the RTD ADA2200, or  
/P for PC104 equipped with RTD DM5406 multi IO card  
The format to use is FlowTEK /n where n is one of the above options.*



**HINT :** *You may run the install program at any time to repair a corrupted program or configuration file.*

## 1.2 System requirements

FlowTEK requires an IBM PC, XT, AT, PS/2 or 100% compatible computer equipped with at least:

- o a 360K floppy diskette (a fixed disk is highly recommended)
- o a minimum of 512KB of RAM
- o an EGA or VGA screen and screen adapter
- o a slot for the interface card. For the PC-30B this will be a fully busbed full length card. The ADA 2200 requires a half length fully busbed slot. Some devices require serial port connections.
- o an 80-column dot matrix printer (optional) (Epson or HPGL compatibility is required if you want to make use of the Graphic screen dump facility)
- o DOS 3.0 or later (DOS 5.0 is required if you want to use the Graphic screen dump with a Hewlett Packard compatible laser printer).
- o an interface board. At present the following interface boards are supported Eagle Electric's PC-30B, and RTD's ADA2200, and DM5406
- o a distribution board (This box is only available from MINTEK and is necessary for the software to allow data acquisition)

Although many of the functions of FlowTEK will function in the absence of the interface board and distribution board, data acquisition and device control is only possible when these components are installed.

FlowTEK will run on most IBM compatible equipment. If you have any doubt about whether a particular machine can be used, consult MINTEK.

We acknowledge the following copyrights and trademarks:

IBM	International Business Machines, Inc
ADA2200	Real Time Devices, Inc
DM5406	Real Time Devices, Inc
PC-30B	Eagle Electric, (Pty) Ltd
Quattro Pro	Borland Corp.
PC/104	PC/104 Consortium

### 1.3 Who will use FlowTEK?

FlowTEK is a flexible device control and data acquisition package for flow-based methods of analysis. This includes flow-injection analysis (FIA), sequential-injection analysis (SIA), segmented flow analysers, and single peak chromatographic techniques. Variations on these techniques may also be accommodated. FlowTEK may also be used as a simple and versatile device-control package with no detectors connected.

Because the package has been written with numerous user-selectable set-up options, this package is suitable for use

- o in the research environment for technique study and optimization,
- o by the method developer in the development of methods for the above mentioned techniques,
- o in the service laboratory for routine automated analysis, and
- o in the plant environment for unattended process analysis.

Limited data manipulation capabilities have been included in the package. Data files however, are in simple delimited ASCII format to facilitate capture by most editing, statistical, or spreadsheet packages. A macro driven spreadsheet for Quattro Pro is included.

## 1.4 Installing the software

This is what you need to install the FlowTEK software:

- o The FlowTEK diskette.
- o A spare formatted diskette on which to copy the original.
- o The drive name of your diskette drive (e.g. a:, b:).
- o The drive name of the drive where the program is to be installed (e.g. c:).
- o The interface card and distribution board.

We will assume the most common configuration, which is diskette drive a: and fixed disk c:.

- o Read the Licence agreement before you open the envelope with the disk in it to ensure that you are complying with the terms of the licence agreement.
- o Place the FlowTEK diskette in the diskette drive, and close the drive door.
- o Type
- o **A:<Enter>**
- o The DOS prompt should show A> or something similar.
- o Type
- o **INSTALL<Enter>**
- o This will install the software from the diskette in drive a: to a subdirectory called FlowTEK in drive c:. If you have selected other drives, follow the instructions on the screen.
- o Once you have accepted the disk drives, you will be asked to enter the letter corresponding to your interface card.
- o Select the letter corresponding to the interface board that you are using:  
E-Eagle Electric PC-30B, R-RTD ADA2200, P-PC104 equipped with RTD DM5406 multi IO board

If the computer responds with Bad command or file name, you have not followed the instructions correctly. Start again from the beginning.

If the installation is successful, a message to that effect will be displayed.

- o To make a backup copy of the original FlowTEK diskette, type **DISKCOPY A: A: <Enter>** (use your drive letters)  
Follow the instructions to copy the diskette. Keep the original diskette in a safe place.

Register your package with MINTEK in order to ensure that you are informed of future developments and enhancements.



The installation procedure for the software is now complete. The next section will tell you how to install the hardware.

## 1.5 Updating Software

To update software on normal computers, simply re-install the software using the installation disks.

To update software on a PC/104 system that does not include a floppy drive, the following procedure must be adopted.

- o Install the software on a normal desktop PC
- o Link the desktop PC with the PC/104 system by making use of either the serial or parallel ports.
- o Use software such as Laplink, or Interlnk (found in DOS 6.0 or later) to transfer files from the desktop computer to the PC/104. Refer to the relevant manuals for the steps to follow to transfer files from one computer to the next.

Another option is to connect a floppy drive to the FDD connector on the PC/104 CPU board. Then install the software directly from the installation disks in the normal way.

## 1.6 Installing the hardware

First, you must install the I/O card into your computer.



**WARNING! You must switch the computer off before you open it. Plugging in an interface card or even making a serial connection while the computer is powered up could damage the card or the computer.**

To install the PC-30B or ADA2200 interface cards carry out the following steps:

- o Discharge any static build-up by touching the chassis of the computer or any other suitable earth.
- o Before you plug the card in, ensure that the dip switch settings and jumpers on the board correspond to the settings given in chapter 13. The interface card manual, which has been included, gives further details on how this should be done.



**NOTE :** *Do not use the factory default jumper and dip switch settings for the interface card. Rather use the settings given in chapter 13.*

- o Insert the card into a vacant slot and fasten it to the computer chassis with the small locking screws.
- o Connect the supplied cable between the distribution board and the computer.
- o Ensure that the installation has been successful by running FlowTEK.
- o If the "Interface board not present" message shows, ensure that you specified the correct interface board during installation, ensure that the board is properly seated in the slot, ensure that the cable to the distribution board is properly connected.
- o If the computer hangs, and you have a network card present, you should remove the network card or move it to another address space. The I/O cards are configured to use the address space at 0x700 for the PC30-B and 0x300 (768) for the other boards.
- o If none of these options remedy the problem then the board is defective and must be returned for replacement.



**NOTE :** *Although FlowTEK comes with certain default settings and sample data files, you will have to configure the package to meet your requirements. See chapter 4, Configuring the system, for more details*

The PC/104 system will be fully configured when you receive it. The dip switch and jumper settings for the PC/104 DM5406 are also given for information in chapter 13.

## 1.7 Connecting devices

A Device is defined as an analytical instrument or component which you would like to control using the software package. These devices must be compatible with TTL or switch control signals. Provision is made for the control of Cavaro pumps which are controlled via a serial link.

The distribution board supplied with the various I/O boards is equipped with 12 digital I/O points. These points are divided into 8 digital output points and 4 digital input points. The digital output points are numbered 1 to 8. The digital input points are numbered 1 to 4. Chapter 14 gives a full description of the distribution board and explains how to configure the digital outputs for different types of logic (e.g. TTL or switch logic).

- Establish the logic that controls the device you want to connect to the package. For example, 10 binary (High Low) activates the pump in a forward direction, 11 binary (High High) activates it in the reverse direction, and 00 binary (Low Low) switches the pump off.
- Decide which output points on the distribution board you are going to use for each device, (e.g. 1 and 2 for the pump.)
- Configure the digital output points on the distribution board for the required logic as described in chapter 14.
- Enter the details of the device, the port you intend to connect it to, and the required logic on a copy of the data sheet given in Appendix 17.1.
- Connect each device to the distribution board while the computer and devices are switched off. Make use of any of the numerous digital ground points. All of these points are common. Quick connectors are supplied with your package to facilitate easy connection of the device to the distribution board.

Refer to the Cavaro pump manuals to establish how they are to be configured for control through a RS-232 or RS-485 interface.

- Install the required termination jumpers as indicated in the pump manual.
- Select a baud rate of 9600 by using the described jumper.
- Ensure that the pump is configured with a device address of 1 (ASCII).



**WARNING! Do not make changes to the pumps jumpers while the pump is powered up.**

## 1.8 Connecting detectors

The Detectors are those devices from which you want to acquire data. This may be the analytical measurement device you are using (e.g. a spectrophotometer) or any physical sensor (e.g. temperature sensor). FlowTEK allows up to four Analog inputs. The Analog input points are numbered 1 to 4 on the distribution board. The voltage range accepted by the interface board is 0-10V.



**WARNING! : Connecting sources with a higher voltage could damage the interface board and will cause other channels to become inaccurate or noisy.**

An amplifier circuit is included on the distribution board for Analog input port 1 and 2. This allows detectors with outputs < 1 V to be connected to the distribution board. (Settings for the amplifier for such devices are described in chapter 14.)

To link detectors to the distribution board:

- o Establish the output range of each detector (in most cases only one detector will be used).



**NOTE :** *The source impedance of all detectors should not exceed 1kW to maintain accuracy.*

- o Determine what, if any, mathematical function should be applied to the signal obtained from the detector (e.g.  $\log \frac{I_0}{I_t}$ )<sup>1</sup>
- o Choose the auto zero strategy that you intend to use (see Configuring the system for further details)
- o Choose an Analog input port on the distribution board for each detector. Only input port 1 and 2 allow amplification of the signal.
- o Enter the details of each detector, the Analog port you intend to connect it to, the output range, response range, and the required auto zero strategy on a copy of the data sheet given in Appendix 17.2.
- o Connect each detector to the distribution board while the computer and detectors are switched off. Quick connectors are supplied with your package to facilitate easy connection of the detector to the distribution board. The Analog signals are input as single ended inputs. Single ended inputs are depicted in figure 1.

- Wherever possible, leads should be shielded. The shield should be tied to Analog ground at the instrument end of the connection only. Make use of any of the Analog ground points. All of these points are common.

**CAUTION!** It is important to keep digital and Analog ground separate. Failure to do so could result in ground loops with unexpected results.

- Switch on the computer first and then the detectors. (When powering down, the detectors must be switched off before the computer.)

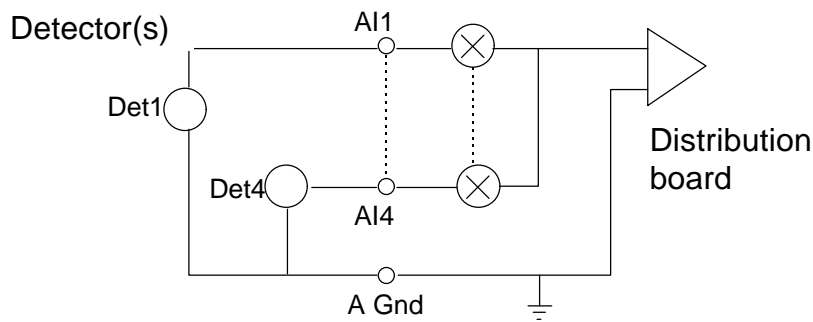


Figure 1 : Single-ended Analog inputs

You are now ready to enter FlowTEK to describe the devices and detectors to the package (see *Configuring the system*).

## 1.9 How to use this manual

This manual is divided into logical chapters which describe the various facets of the FlowTEK software package. Chapter 2, *Software overview*, describes the capabilities of the various menu options in the package. *The Keyboard conventions* used in FlowTEK are described in chapter 3.

Chapters 4 through 10 describe how each of FlowTEK features are used. When you are ready to *Configure the system*, refer to chapter 4. Subsequent chapters give a detailed description of each menu option. Particular attention should be given to the chapters, *Designing a method* and *Analysis by procedures*. You will have to be familiar with the *Designing a method* module before you move onto *Analysis by procedures*. The *Directives* available for inclusion in methods or procedures are described in detail in chapter 11. All of the *Data files* are described in chapter 12 together with a print out of the default data files.

Chapter 13 and 14 gives a description of the *RTD* and *Eagle* interface boards and the Distribution board. Also refer to the supplied interface manual for more technical information on the specific board you are using.

Details on *Customer support* including the capabilities of MINTEK in the development of methodologies, and design of Process Analysis systems, as well as the supply of instrumentation, is given in chapter 15.

A list of *Error messages* is provided in chapter 16.

## 2. Software overview

FlowTEK comprises eight menus. These menus are accessed from the main page. Several of the menus have pages of their own. A dendrogram portraying the program structure is given in figure 2. Menus are chosen by typing the first letter of the desired menu choice. Each page is portrayed on a graphics screen (as opposed to a text screen). Exiting from a menu is achieved by using the **ESC** key or **Q** for quit.

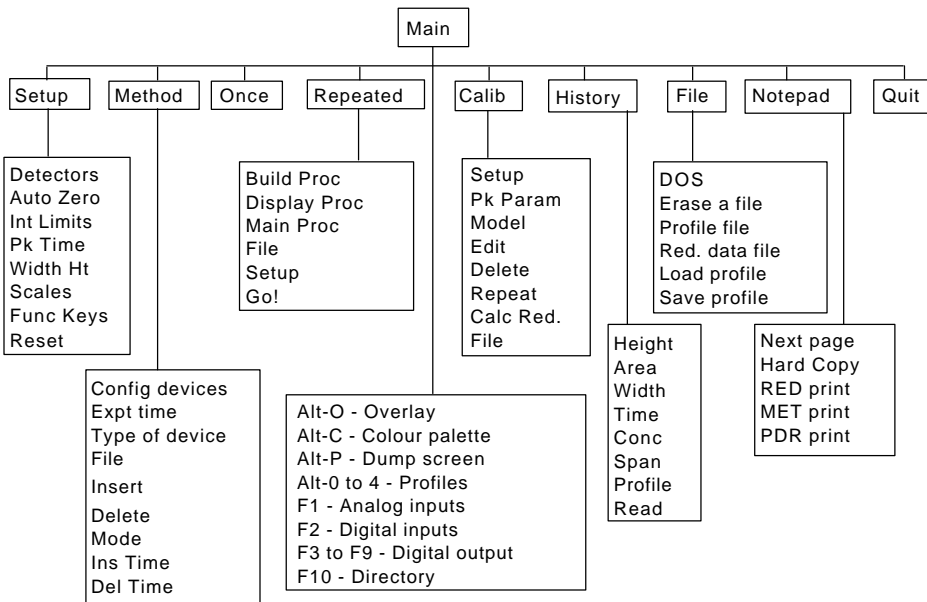


Figure 2 : Dendrogram of FlowTEK program structure

The software was written using Turbo C++ by Borland. More than 20 source files were compiled using the Borland compiler and linked together with various library routines to form the executable program file. Every attempt has been made to keep the program modular thereby allowing for easy future updates. Suggestions for future improvements to the software should be directed to Mintek.

Data compatibility with other products under development by Mintek will ensure an integrated and versatile approach to all your Flow-based Analysis control requirements.



## 2.1 The Main menu

This menu is displayed together with the Main screen as depicted in figure 3. The Main menu options are listed as follows:

- 0 *Set-up* allows you to configure the package to meet your particular requirements.
- 0 *Method* provides for the definition of devices and the description of device control methods.
- 0 *Once* initiates a single measurement sequence (called an experiment).
- 0 *Repeated* allows you to define and choose a procedure to accomplish multiple measurements.
- 0 *Calib* provides various option in performing a regression on acquired data or entered data. This option allows you to calibrate the system so that results are also reported in concentration units.
- 0 *History* provides a means of perusing data acquired by the package.
- 0 *File* gives certain file choice and manipulation utilities including a means of switching to DOS.
- 0 *Note pad* displays on the screen (or printer) a list of the current user-selected settings. Various reports depicting the data in data files are also selected and printed from this menu option.
- 0 *Quit* exits the program after checking that all updated files and the current configuration have been saved.

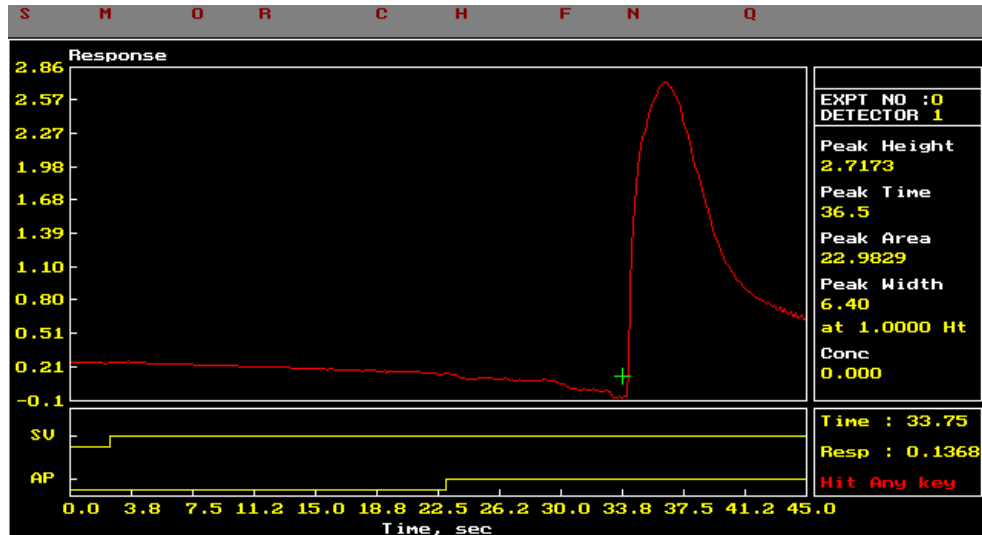


Figure 3 : Schematic representation of the FlowTEK Main screen

In addition to the Main menu options, function key **F1** gives an instantaneous reading of the current value of each of the Analog input channels. Function key **F2** shows the

present input to the four digital input points. The function keys **F3** to **F9** can be configured to force user-selected outputs to the digital ports. A directory of files in any subdirectory can be displayed by typing **F10**. Wild cards are supported.

At the main screen, it is also possible to call up four response profiles of previously collected data into pop-up windows for visual comparison. These pop-up windows are accessed by pressing **Alt-1** through **Alt-4**. They are cleared by using **Alt-0**.

The **Cursor Keys** activate a cross-hair cursor which can be used to read the scaled co-ordinates of any point on the profile.

A hard copy of any screen may be obtained on an Epson or Hewlett Packard printer by performing a screen dump. This is achieved by **pressing Shift-Print Screen** or **Alt-P**. For this feature to work, either PRTSC.COM or GRAPHICS.COM must be loaded before you enter FlowTEK. See section 3.6 for further information on the procedure to follow.

The colour palette used in the package can be changed by pressing **Alt-C**. When you reload the package, the original colour palette will be used.

An extremely powerful option in FlowTEK is obtained using **Alt-O**. This options allows subsequent profiles to be overlaid on the screen. In this way, peak profiles can be compared. This feature is particularly useful during method development and trouble shooting. **Alt-O** toggles this option on and off.



**HINT** : The border of the profile box changes colour to light green when this option is selected and white when it is not selected.

## 2.2 The Set-up menu

This menu allows you to configure the package to suit your particular requirements. This menu is also depicted together with the Main screen (see figure 3).

The following options are presented in the Set-up menu:

- o *Detectors* allows you to define the detectors from which data are to be acquired. The completed Detector Description Data Sheet (Appendix 17.2) should be used as source of information (see section 4.1).
- o *Auto Zero* lets you choose the mode of auto zero operation (see section 4.2). You can choose to zero the detectors response for every experiment, at the start of a series of experiments only, or not at all. You also can choose at what stage during an experiment the response is zeroed. Directive 41 is also useful to change the time at which the baseline is measured.
- o *Int Limits* defines the integration limits for peak area calculations (see section 4.3).
- o *Pk Time* specifies the time at which the peak height will be measured<sup>1</sup>. If you select 0, then the peak height at peak maximum is determined (see section 4.4).
- o *Width Ht* sets the height at which the peak width is determined<sup>2</sup> (see section 4.5).
- o *Scales* lets you choose between automatic re-scaling of the y-axis or a user-defined y-axis for scaling response profiles (see section 4.6).
- o *Func key* allows you to define digital output which is sent to the digital output points by each of the function keys (see section 3.3 and 4.7).
- o *Reset* sets all the set up parameters to their factory defaults (see Table 1 in section 4.8).
- o The function key settings (F3 to F9) can be tested while this menu is active.

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<sup>1</sup> This facility can be used to perform electronic dilution (see Ruzicka and Hansen, Flow Injection Analysis, 2nd ed., Wiley-Interscience).

<sup>2</sup> Given certain manifold requirements, this peak parameter allows FIA titrations to be carried out (See Ruzicka and Hansen, Flow Injection Analysis, 2nd ed., Wiley Interscience).

## 2.3 The Method menu

Method provides for the definition of devices (e.g. AP = Alitea pump, IV = Injection valve) and the description of device control Methods (e.g. at time 5 seconds set pump forward, etc.). The Method screen as depicted in figure 4 is displayed with this menu.

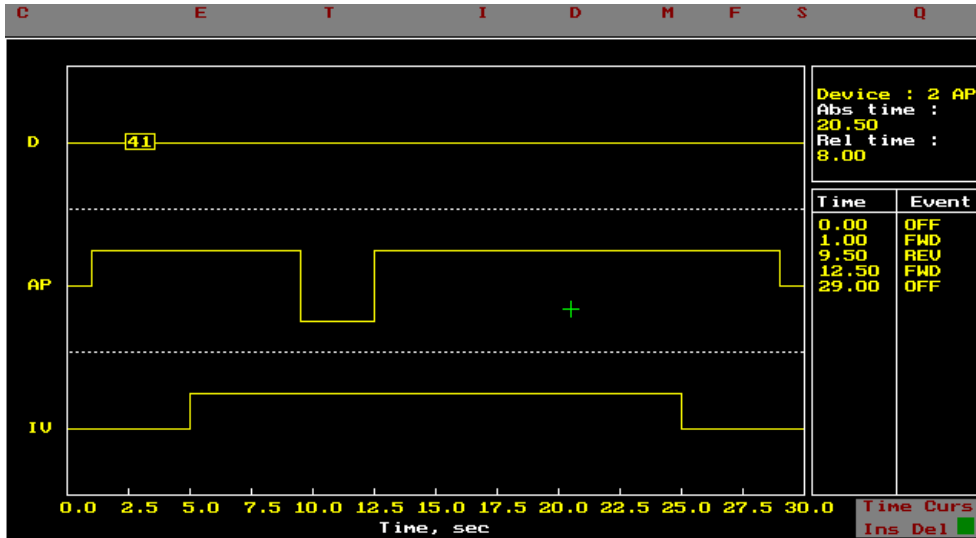


Figure 4 : Schematic representation of the FlowTEK method screen

The following options are presented in this menu:

- o *Config device* is where you enter device configuration data. Nine device descriptions are provided with the package and the facility to describe a further three is provided (see section 5.1).
- o *Expt time* allows you to select the duration of an experiment. An experiment is defined as a single measurement sequence. This option also allows you to set the time after the start of an experiment, that data collection should begin. This allows for lengthy sample manipulation, before the actual detection phase begins, without requiring redundant data acquisition (see section 5.2).
- o *Type Devices* is where the number and Type of devices is selected. The maximum number of devices that may be connected at any one time is eight. Choosing this options divides the Method screen into the chosen number of panels to allow programming of each device. All other device information is cleared when the No of Devices option is entered. The completed Device Description Data Sheet (Appendix 17.1) should be used as source of information (see section 5.3).

A Directive device can also be specified. This pseudo device allows a means

for executing directives during a method. Directives are discussed in more detail in chapter 11).

- O *Insert* is the menu choice which allows you to insert a device event for a chosen device. The device and time at which the event is to be inserted may either be typed in from the keyboard or else an arrow-key controlled cross-hair cursor determines the device and event time. The mode of operation depends on whether the Num Lock key switch is selected or not. Num Lock de-selected allows the cross-hair cursor mode of operation.



**HINT** : *This mode is depicted by a small green box in the bottom right hand corner of the screen. A red box indicates keyboard input of the event.*

Newer events replace previously defined events if they are inserted at the same time as a previously defined event. The time and event description for the current device is displayed in a Time and Event box. The chosen device and present time co-ordinates of the cross-hair cursor as well as the relative time from the previous event are also depicted in a Device box. Each device can have a maximum of three states. These states are depicted by means of a line chart in each devices panel. If the device is a syringe pump (SP), the time for events above the middle line less those below the middle line is given as the Net time. This figure is useful in sequential-injection analysis (see section 5.4).

Directive events are entered in an analogous fashion except the directive number and parameter are entered instead of the device action.

- O *Delete* allows an event to be deleted. The selection of the event to be deleted is accomplished in the same way as inserting an event. The delete event options takes into account whether the device is one which has pulsed or continuous events. It is therefore not necessary to delete the end of the pulse action. This is automatically deleted when the start of the pulse is deleted (see section 5.5).
- O *Ins Time* allows you to insert time between two events. When time is inserted, the experiment time is lengthened. Time inserted at a certain point in the method shifts the subsequent events of each of the defined devices. Other relevant times such as integration limits are also adjusted if applicable (see section 5.6).
- O *Del Time* provides a way of deleting time in the middle of a method. When time is deleted the overall experiment time is reduced. Deleting time shifts subsequent events for each of the defined devices. If the period to be deleted contains events, these events will also be deleted. Certain exceptions exist where one part of a pulse falls outside of the deleted period (see section 5.7).
- O *Mode* allows you to select between automatic or manual sample injection. If manual sample injection is selected, a micro switch connected to the manual

injection valve signals the start of the data acquisition when the sample is injected. In automatic mode, data acquisition commences at the time selected under Expt time (see section 5.8).

- o *Startup* allows you to start the program with the digital output points in a Startup configuration( see section 5.9).
- o *File* allows you to Save, Retrieve, or Erase a method file. A method file contains all the data necessary to define a method. The parameters which are saved are listed in chapter 11 (see section 5.10).

## 2.4 The Repeated menu

Repeated allows you to define and choose a procedure to carry out multiple experiments. Aside from normal laboratory operation, this facility allows for the automated operation of the analyser. In fact when the program starts, it automatically runs the Main Procedure defined in the Repeated menu option. By calling FlowTEK in the autoexec.bat batch file, automatic startup of the software after a power failure or when using directive 254, is assured.

Once you have defined one or more methods, you are ready to define a procedure. A Procedure can consist of

- o Methods,
- o other Procedures, and
- o Directives

Each element in the main procedure is carried out in sequence. The procedure definition screen is depicted in figure 5. Once Go! has been selected, the display returns to the main screen to display the profiles as they are acquired.

On the Repeated page, the present selections for file output (reduced files .RED, and profile files) are given. Also the present settings for critical automated operation settings are listed.

DEFAULT .PDR			
File	No.	Main Procedure File	: DEFAULT .PDR
\$IACN1.PDR	3	Reduced Data File	: DEFAULT .RED
\$IACN2.PDR	3	Profile File	: DEFAULT
\$IACN3.PDR	3	Warning Range	: ±10%
		Alarm Range	: ±15%
		Check Standard Range	: ±5%
		Permissible Noise	: ±50mV
		Maximum D to A change	: ±100.00 mV
		Setpoint	: 0.00 MG/L
		Check Standard	: 0.00 MG/L
		Recalib Pdr	: RECALIB .PDR

Figure 5 : Schematic representation of the FlowTEK Procedure definition screen

- o *Build Proc* - a procedure is built, by grouping a sequence of methods and / or procedures together with the number of repetitions for each method or procedure. In addition, a whole series of instrument and control directives can be invoked by including them in a procedure (see chapter 11). **ESC** terminates

the procedure definition (see section 7.2). Procedures and methods are specified by giving their file name, (e.g. CALIB.PDR or SAMPLE.MET). Directives are specified by entering the directive number together with an extension of .DIR, (e.g. 55.DIR will introduce a pause of x seconds). For Directives, instead of the number of repeats, the parameter is entered.

- *Display Proc* displays previously defined procedures for visual perusal (see section 7.3).
- *Main Proc* selects the defined procedure that will be executed when Go! is selected. This menu option identifies the main procedure (see section 7.4). This procedure will also be executed on startup of the program. **Esc** aborts a procedure.
- *File* selects a reduced data file for saving each experiments peak parameter data and relevant experiment identification information. The experiment number counter is reset to 1 when the reduced data file name is changed. The reduced data file format is given in chapter 12. *File* also selects the file root name for storing the profile data. (See chapter 12 for the profile file data format.) The file name extension gives the experiment number. This file name is stored in the reduced data file to enable linking the reduced data with the relevant profile data. If no profile root is chosen, profile files are not saved (see section 7.5).
- *Set-up* provides a means to enter certain critical data values used particularly in the process environment. (see section 7.6)
- *Go!* executes the Main procedure. The main screen is displayed on the computer screen. A procedure can be aborted by pressing **ESC**. The current method being executed is depicted at the top of the peak parameter box. In the event of a WAIT operation, the remaining time till the next experiment is displayed at the top of the peak parameter box. Pressing **ESC** during WAIT operation aborts the WAIT operation but not the procedure (see section 7.7).



**HINT** : A list of method and procedure files can be obtained by typing **F10**.



## 2.5 The Calibrate menu

Calibrate provides the facility to perform a regression analysis on acquired data or entered data in order to calibrate the system and allow for results to be presented in concentration units. The calibration screen is depicted in figure 6. Several user-selected options are available. Data points together with error bars (when more than one datum is used for a particular concentration level), and the calculated calibration curve are depicted graphically. The data used for the calibration are displayed in a table alongside the calibration curve. When the regression has been completed, the regression coefficient and correlation coefficient are depicted in an information box together with other relevant data.

The data for up to sixteen calibration standards can be entered into the calibration table. Alternatively, calibration data acquired through the measurement of standards can be allocated to the calibration table as described in the description of the directives. Other directives provide the capability to store and calculate the calibration curves of several calibration sets based on the data stored in the calibration table. See Chapter 11 for details.

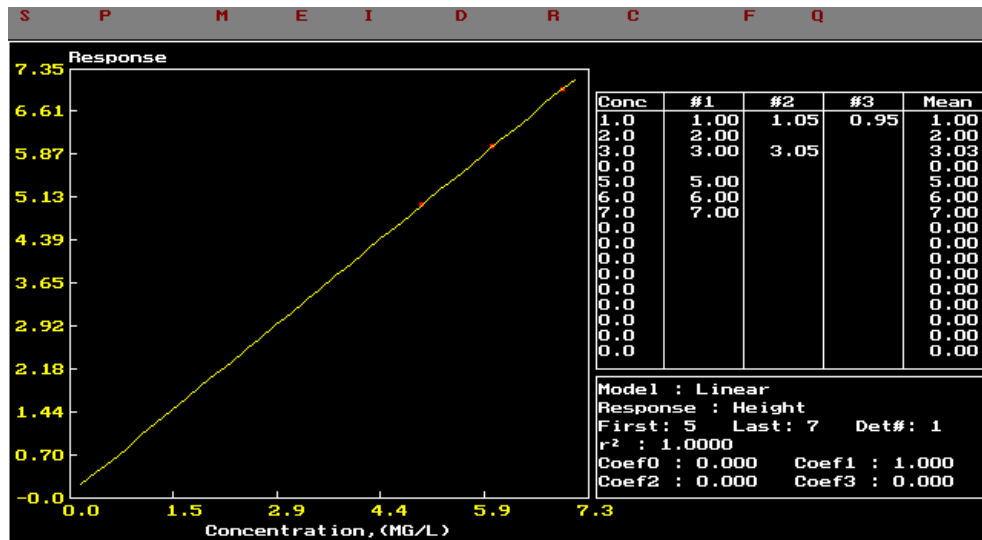


Figure 6 : Schematic representation of the FlowTEK Calibration screen

- 0 *Set-up* chooses the data to be used in the calibration. The number in the calibration table of the first standard and of the last standard in a calibration set is chosen. Also the concentration units for the standards are entered. (see section 8.1).
- 0 *Pk param* selects the peak parameter (Height, Area, Width, or Time) that will be used together with the concentration data to generate the calibration graph. This option allows you to investigate which parameter gives the best results (see section 8.2).

- *Model* chooses the regression model that will be fitted to the data. A choice of exponential, rectangular hyperbola, or first, second, or third order polynomial is possible. A further option allows each model to be tried. The one giving the best correlation coefficient is selected (see section 8.3).
- *Edit* provides a way of editing all or individual data points. This option is used changes are to be made to existing data (see section 8.4).
- *Insert* provides a way of adding calibration data and new standards to the calibration table. (See section 8.5).
- *Delete* allows outlier data to be deleted. The regression is repeated with the remaining data provided that there are still enough data for the chosen model (see section 8.6).
- *Repeat* enables the repeat measurement of any of the standards to be repeated as single experiments using the current method. The regression is then performed on the new data (see section 8.7).
- *Calc RED* recalculates the concentration field of a reduced data file using the current calibration data. The new concentration values are saved in the reduced data file (see section 8.8).
- *File* - The data pertaining to a calibration are saved, retrieved, or erased using this option (see section 8.9). The format of the data in the calibration file is given in chapter 12.

## 2.6 The History menu

History provides a means of perusing data acquired by the package. Data from a reduced data file are depicted for up to a hundred experiments at a time as either dots or bars on the history screen (see figure 7). Any of the peak parameters (height, area, width at a fixed height, time, or concentration) can be displayed. A pop-up window, activated by selecting Profile, displays the profile of a chosen experiment provided that the profile data were saved. All the peak parameters of the chosen experiment are displayed in the peak parameter box. An arrow-key driven cross-hair cursor facilitates the choice of experiments for closer scrutiny.

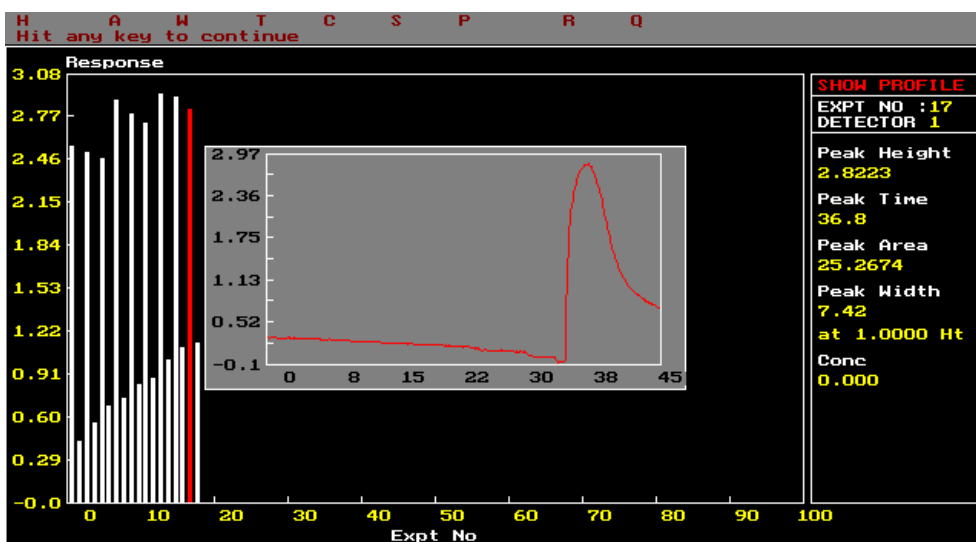


Figure 7 : Schematic representation of the FlowTEK history screen

- 0 *Peak Param* - The data from a reduced data file for one of the five peak parameters, peak height, area, width at a fixed height, time, and concentration, are chosen for display as dots or bars on the history screen (see section 9.1).
- 0 *Span* selects the experiment numbers that are displayed. The data of up to one hundred experiments can be displayed on the history screen (see section 9.2).
- 0 *Profile* displays the peak profile of a chosen experiment in a pop-up window. The profile data file must be stored. The peak parameter data for the chosen experiment are displayed in the peak parameter box (see section 9.3).
- 0 *Read* selects an experiment by requesting the experiment number (see section 9.3).
- 0 The arrow-key driven cross-hair cursor can also be used to select an experiment for closer examination (see section 9.3).
- 0 The data are represented by dots if . (period) is pressed. To depict the data as bars, press either / or \.

## 2.7 The File menu

File gives certain file choice and manipulation utilities including a means of shelling to DOS.

- o *DOS* exits the FlowTEK program and shells to DOS. The prompt is altered to remind you that a DOS shell is being used. Typing **EXIT** returns you to the FlowTEK program.
- o *Erase file* is a general purpose erase facility which allows wild cards.
- o *Red Data file* selects a reduced data file for saving peak parameter data and relevant experiment identification information. The experiment number counter is reset to 1 when the reduced data file name is changed. (See chapter 12 for the reduced data file format.)
- o *Profile file* selects the file root name for storing the profile data. (See chapter 12 for the profile file data format.) The file name extension gives the experiment number. This file name is stored in the reduced data file to enable linking the reduced data with the relevant profile data.
- o *Load profile* loads an experiment profile onto the main screen for closer examination.
- o *Save profile* toggles the switch which determines whether experiment peak profiles are saved or not. An option to create an abridged profile file containing just the response data is also provided. The format for the abridged profile file names is a<profile root>.<expt no>.

## 2.8 The Note pad

Note pad displays on the screen (or printer) a list of the current user-selected settings. It also allows the printing of several reports. This option is invaluable during system configuration.



**HINT** : *If the system ever behaves in an unexpected fashion, first examine the Note pad information page. Ensure that all parameters are set to the desired value.*

- o *Next Page* gives details on the other devices. Selecting it again returns the display to the original set of devices.
- o *Hard Copy* prints a list of the set-up parameters, as listed on the screen, on the printer. Space is provided on the printout for additional hand written notes pertaining to an experimental set-up.
- o *RED Print* prints out a report of the data in a chosen reduced data file.
- o *MET Print* prints out a table giving the data in a chosen method file.
- o *PDR Print* prints a report giving a chosen procedure file. The procedure file is also expanded to list all the methods that are executed when the procedure is executed in the Repeated mode of operation. This could be a long list.

## 3. Keyboard conventions

### 3.1 The menu system

The FlowTEK menu is displayed along the top line of each screen (see figure 8). The first letter of each menu choice is a different colour and highlights the letter that must be typed to access the chosen menu option. Exiting from a menu is achieved using the **ESC** key or typing **Q** for Quit. An invalid menu choice will result in a Beep from the computer. A single word describing the chosen menu option is displayed on the top line of the peak parameter (or equivalent) box. This serves as a confirmation of the menu choice selected.

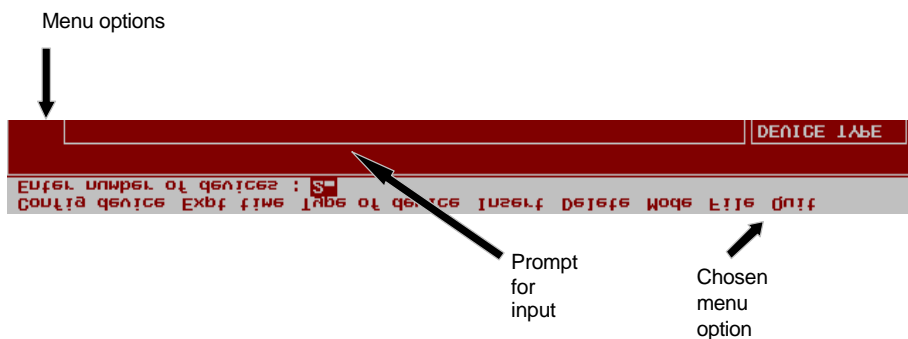


Figure 8 : The FlowTEK menu system

## 3.2 The ESC key

The **ESC** key is used as a general purpose escape, cancel, and quit key. It can be used in several situations as listed below:

- o to Quit the program,
- o to exit from a submenu to the main menu,
- o to cancel input; the original value is maintained,
- o to abort a wait period in a procedure; the procedure is not terminated,
- o to over ride the wait for digital input in a procedure,
- o to abort a procedure, or
- o to signal 'no more input' for a procedure definition.

### 3.3 The function keys

The function keys provide certain short cut options:

- **F1**, when used on the main page, displays the input voltage at each of the four Analog input channels, in a pop up window.
- **F2**, when used on the main screen, displays the status of the four digital inputs. A 1 signifies that the input is High while a 0 means that it is Low, relative to the digital ground.
- **F3 to F9** can be configured by the user to send output to any of the digital output ports. This option provides a way of checking digital ports without defining a method or manually controlling each device. This option is active from the Main Menu and the Set-up Menu.
- **F10** is used to display the directory. This function key is active for the Main menu, the Repeated menu (where only procedure and method files are displayed), the Method menu, the Calibration menu, and the File menu. Wild cards are permitted. The directory to be displayed, if other than the default path, must be entered.



### 3.4 Program input

There are five types of keyboard input to the program:

- o Integer,
- o Floating point,
- o Character strings,
- o File names, and
- o Single letter replies.

The second line of each screen is used for this kind of input. A prompt string is displayed together with a box for input. The size of the box determines the permissible length of input. The current value is displayed in the box. Pressing any key except **Ins** or one of the arrow keys as the first key will clear the box for new input.

The **Ins** key or one of the arrow keys will put the input in edit mode and allow the existing value to be edited. **Ins** also toggles between overwrite and insert input. The **Del** key deletes the character to the right of the cursor. **The Bk Spc** key (Back Space) deletes characters to the left of the cursor. **Home** and **End** moves the cursor to the beginning and end of the input string respectively. For some inputs, there are specific valid options which must be used. For some numeric input, the input must fall between certain limits. If one of the valid inputs are not used or numeric input falls outside of the valid range, the computer beeps, display the valid input, and waits for a valid reply. At any time **ESC** may be used to terminate the input. In such a case, the original value is restored.

For file names, the input routine also determines whether the file entered exists. If it does and the operation is *Save*, a warning that the file will be overwritten or appended is displayed. If it doesn't and the operation is *Retrieve*, a warning that the file does not exist is displayed. The default path used in the program is also updated by any path used in a file name. This ensures that all files pertaining to a particular application are in the same subdirectory, unless otherwise specified. If no path is given, the last used path is assumed.

Input that requires a single letter response does not need the **Enter** key. The typed key is not echoed to the screen.

### 3.5 The arrow keys for the cross-hair cursor

The cross-hair cursor can be activated:

- o on the main screen to give a scaled value of the x and y co-ordinates in the peak profile box,
- o on the method screen to select the device and choose a time for an event,
- o on the history screen to choose an experiment for closer perusal.

On the method screen, the cross-hair cursor is made active by de-selecting **Num Lock**. On the other two screens, using the arrow keys activates the cursor.

The step size is increased using the **+** and decreased using the **-** keys. Each time the **+** key is pressed, the step size is doubled. Each time the **-** key is pressed, the step size is halved, until it is one. The **\*** returns the step size to its default. The **Home** key sends the cursor to the middle of the screen. The **End** key together with any of the arrow keys moves the cursor to the respective box boundaries.

On the main screen, pressing **Enter** opens the cursor data window and gives the current scaled co-ordinates of the cursor. **Enter** on the History screen selects the experiment closest to the cursor. On the method screen **Enter** is used to mark a time position in the Insert and Delete time options.

## 3.6 Graphics screen dumps

**Alt-P** can be used on any screen to send a screen dump to a printer of the graphics screen presently displayed. Epson and IBM printers are supported if DOS versions earlier than 5.0 are used. If you want to dump graphics screens to a Hewlett Packard compatible laser printer you must use DOS 5.0. The following instructions tell you how to load graphics drivers to allow screen dumps of the graphics screens used by FlowTEK. **Alt-P** has the same effect as **Shift-Print screen**.

For computers equipped with versions of DOS before 5.0, the file **PRTSC.COM** supplied on the installation disk must be copied to your FlowTEK directory and then run before you run FlowTEK. **PRTSC.COM** replaces the now obsolete *GRAPHICS.COM* shipped with DOS ver 4.0 or lower. Copy and run **PRTSC.COM** as follows:

- o At the DOS prompt type **cd \FlowTEK**
- o Place the FlowTEK installation disk in drive a: or b:
- o Type **copy a:prtsc.com <enter>** (substitute your source drive letter)
- o The file will be copied to your FlowTEK sub directory.
- o This memory resident program must be run before you run FlowTEK simply by typing **prtsc** at the DOS prompt before you type **ft** to run FlowTEK. A better option is to add prtsc to your AUTOEXEC.BAT file. Refer to your DOS manual to see how this is done.

If your computer is running DOS 5.0 or later versions of DOS, you can dump graphics screens to a Hewlett Packard compatible printer. To do this, you must first load **GRAPHICS.COM**. This program is best run from your AUTOEXEC.BAT. Refer to your DOS manual to see how this is done. The syntax of the command to be included in your AUTOEXEC.BAT depends on the printer you will be using. Refer to your DOS manual for further information on this memory resident program. For a Hewlett Packard Laserjet II, for example use the following syntax:

- o `c:\dos\graphics.com laserjetii c:\dos\graphics.pro /r`



**HINT** : *For best results in converting colours to black and white images always use the /r command line switch as depicted in the above example.*

- o substitute the printer designation which best suits your printer in the above example. The options are listed in the DOS manual

If FlowTEK is being run from within the Windows environment, **prtsc** will save a copy of the present screen to the clip board where it can be pasted into any document capable of manipulating graphic files.

### 3.7 Overlaying profiles

The chart recorder was, and in many cases still is, the most popular way to display response data. Comparison of peak shape, reproducibility, stability, drift, and other parameters is achievable using a chart recorder. The advent of the microprocessor and software such as FlowTEK has given the user even greater power with regard to data analysis and interpretation. Future users of flow-based procedures will replace their chart recorders with computer-aided data acquisition and analysis.

FlowTEK is designed as a versatile data acquisition package. Basic data manipulation and inspection routines are provided. Arguably the most valuable of these is the facility to overlay peak profiles. Profiles can be overlaid during acquisition or, after acquisition, selected profiles can be loaded. Each peak is displayed in a different colour.

The start up setting for this option is single profile display with a screen clear between acquisitions. **Alt-O** (not Alt-0(zero)) toggles between profile overlay and clear screen between acquisitions. When overlay is selected, the border of the profile box is coloured light green. When clear screen is selected, the border of the profile box is white. This toggle can be used in the middle of a procedure or before a single experiment. All subsequent profiles will be overlaid until the switch is toggled again. To clear the screen before another batch of overlaid profiles toggle to clear screen and then back to profile overlay.

### 3.8 Changing the colour palette

For users with screen types that require alteration of the colour palette to improve contrast and visibility, the **Alt-C** keystroke combination allows the various colours to be changed.



**NOTE** : *The palette reverts to the default settings when FlowTEK is exited. Also changing the palette in this way does not affect the shading used by the printer to depict colours when you do a screen dump.*

## 4. Configuring the system

This menu allows you to configure the program to meet your particular requirements. As some of the settings have a direct effect on the method, you will be asked, after having made certain changes, whether you want to save the method before you exit this menu option.



**NOTE** : *Some of the parameters which are set in this menu are saved (and retrieved) with method files (see Chapter 12 for details). This means that if you change the conditions and then load in a method with different conditions your changes will be overwritten. By printing the method (from the Notepad menu), the values for each of the relevant parameters can be seen.*

## 4.1 Detector choices

Up to four detectors can be monitored by the software. This option defines the detectors for the program. To select this option type:

- o Detectors
- o Enter the number of detectors you have connected.
- o If there is more than one detector, you can choose whether to **O**verlay the peak profiles of each detector or **P**age through each one.



**NOTE** : *You can not select overlay of detector profiles and overlay of experimental profiles. This means that if you want to overlay experiments you must page from one detector to the next.*

- o Enter the mathematical transformation to be applied to the signal obtained for each detector. The choices are **L**og of ratio of signal intensity to initial intensity (i.e.  $\text{Abs}=\log I_0/I_t$ ), negative log (as in pH measurements), **D**erivative (i.e.  $Dy/Dt$ ), **I**nverse (resp(in mV)=10-resp) and **N**o transformation.
- o Enter the Analog channel that each detector is connected to on the distribution board. This number should be in the range 1 to 4.



**WARNING!** : **The output from the detector should be in the range 0 to 10 volts. Negative voltages will be treated as zero. Voltages greater than 10 could damage the system and will at best cause inaccuracies in other input channels. These voltages will be treated as 10 volts by the system.**

## 4.2 Auto zero options

Three approaches to zeroing the instrument are provided.

- **No** zeroing is implemented and the response obtained from the detector, modified by the chosen mathematical transformation, is displayed in the peak profile box.
- A zero offset is read **Once** during the first experiment in a range of experiments and this offset is subtracted from all subsequent response data.
- A zero offset is read at some time during **Every** experiment and subtracted from all the response data for that experiment.

Where one of the latter two modes of operation are chosen, the offset is usually measured at time zero, i.e. at the start of the experiment. This is not a pre-requisite. A time should be selected which gives the response of the detector, to the sample matrix, with no analyte present. The chosen mode of operation is selected for each detector type. Different modes may be applied to each detector.

To set the mode of operation type:

- **Auto Zero**
- Select for each detector either **Once** (during the first experiment), **Every** (during each experiment), or **Never**.
- If either of the first two are chosen, set the time, during the experiment, at which the zero offset is to be read.



**NOTE** : *Auto zero times will be altered where applicable when time is inserted or deleted in the Method menu.*

Directive 41 (see chapter 11) is provided so that at any time during a procedure or a method, it is possible to change the time at which the zero off set is to be read. This means that for different methods, different auto zero times may be selected. The approach to auto zeroing that is used must be selected in this menu option and then remains in place until changed using this menu option.



### 4.3 Integration limits

The integration limits set the time constraints between which the area under a peak will be determined. These limits must fall within the limits of an experiment. The area is calculated after the autozero offset has been subtracted (if applicable). Different integration limits may be set for each detector. The area is determined using Simpson's rule<sup>3</sup>. To set the limits type:

- o Int Lim
- o Enter the minimum and maximum integration limit for each detector as prompted.



**NOTE** : *Integration limits will be altered where applicable when time is inserted or deleted in the Method menu.*

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<sup>3</sup> Reviewed in M.A. Sharaf, D.L. Illman, and B.R. Kowalski, "Chemometrics", Wiley Interscience, New York, 1986

## 4.4 Peak time and electronic dilution

Peak time allows the choice of two different modes of determining the peak height response. Either the peak height is determined at the peak maximum, or the response is determined at a user-specified time during the experiment. This value is displayed as peak height in the peak parameter box. The peak time parameter in the peak parameter box gives the time at which the peak height is measured.

Measuring the peak height at peak maximum gives the most sensitive response for the experiment. It is, however, sometimes desirable to use the so-called electronic dilution method. In this approach, the peak height at a time either before (but usually after the peak maximum) is determined. This parameter represents the peak height at greater dispersion of the sample than that at the peak maximum. Different times can be selected for each detector. For this approach to work, it is imperative that reproducible flow conditions prevail.

To select peak height measurement mode and time, type:

- **Pk Time**
- **0** as the time at which the peak height is determined for peak height measurement at peak maximum.
- To select the peak height measurement at any time other than the peak maximum, enter the desired time (in seconds).
- Repeat for each detector.



**NOTE** : *The time at which the peak height will be determined, if other than the peak maximum will be altered where applicable when time is inserted or deleted in the Method menu.*

## 4.5 Peak width at a specified response

Peak width under certain manifold design conditions<sup>4</sup> has been used to do flow-injection titrations. Also, peak width is proportional to the  $\log_{10}$  of the concentration. Usually the peak width is determined at a specific peak height. The chosen peak height should ensure that a width for all peaks is obtained (i.e. less than the height of the shortest peak). Choosing a height of 0 could result in spurious results as noise in the detector signal may appear as the start of the peak. The chosen height must therefore be greater than the background noise. To select the desired peak height for width measurement type:

- o **Width Ht**
- o Enter the desired peak height at which width is measured for each detector.

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<sup>4</sup> J. Ruzicka and E.H. Hansen, "Flow Injection Analysis" 2nd ed., Wiley-Interscience, New York, 1988

## 4.6 Re-scaling the x- and y-axis

By default, the time axis extends from zero to the experiment time. The response axis is re-scaled after each experiment so that the peak profile fits comfortably within the peak profile box. It is sometimes desirable to view only a portion of the time axis, or to keep the response axis scale constant. This option in the set-up menu allows this choice. Note, if more than one detector is used and the fixed mode of y-axis display is chosen, the same y-axis will be used for each detector. If the default mode of operation is used and detector response is not overlaid, each peak profile will be re-scaled when it is displayed. To ensure re-scaling after every experiment, type:

- o **Scales**
- o Enter **0** as the minimum and maximum response values.

If a particular fixed scale is required, type:

- o **Scales**
- o Enter the desired minimum and maximum limits of the response axis. Negative values are permitted.

To view only a selected portion of the time axis, type:

- o **Scales**
- o Accept the current settings for the y-axis
- o Enter the minimum and maximum settings for the time axis.



**NOTE** : *The minimum and maximum time axes will be altered where applicable when time is inserted or deleted in the Method menu.*

## 4.7 Programming the function keys

The function keys, **F3** to **F9**, can be programmed to send user-specified values to the digital output points. This feature is useful in that it allows you to control the devices remotely from the computers key board.

In order to set up the function keys, the Device Description Data Sheet (Appendix 17.1) for your device set up will be helpful. From this sheet you will be able to establish which digital output points are connected to each device and what signals achieve the desired action. Bear in mind when you program the function keys that the value you send will affect all the devices connected to the digital output as a single binary number is sent to the digital output ports. This number is interpreted over all the output points. This means that if you want to maintain the status of one device as you change another, the relevant bits for the first device must be set accordingly.

To program the function keys, type:

- o **Func** key
- o Choose a function key to program (between F3 and F9). You can either press the desired function key or a number between 3 and 9. Then enter the number you want to send to the digital ports. You can enter the number in binary format (i.e. using 1s and 0s). To do this you must prefix the series of 1s and 0s with a B. The most significant bit will be sent to digital point 1 and the least significant bit will be sent to point 12. Only points 1 to 8 are available on the distribution board. When determining a number to send use the following formula:

$$N = \sum_{i=0}^{11} d * 2^{(p-1)}$$

N is the number to send

d is a 1 or 0 depending on whether you want to set the relevant bit or not

p is the digital output point (1 to 12)

- o Repeat this for each function key you want to program. Press ESC to return to the menu.
- o You can test the function key assignments from the Set-up menu by pressing the desired function key and watching the effect on the device.



**HINT** : *This facility is useful when testing the connection of devices and their definitions.*

## 4.8 The default settings

To return all settings to their default values as set out below, type:

0 **Reset.**

Table I : Default set up conditions

Parameter	Default Setting
Number of detectors	1
Analog channel for detector	1
Detector display mode	Paged
Function applied to data	None
Auto zero mode	Every experiment
Time to determine auto zero offset	0 sec
Minimum integration limit	0 sec
Maximum integration limit	Experiment time
Height at which peak width is measured	0
Time at which peak height is measured	At peak maximum
Time axis minimum	0 sec
Time axis maximum	Experiment time
Re-scale y-axis	Yes

## 5. Designing a method

A Method is defined as the sequence of events that must be carried out to obtain a single measurement experiment. Therefore, methods control the output sent to the devices connected to the digital outputs on the distribution board and the timing of these outputs. By allowing Directives to be included in Methods, it is possible to also effect certain changes to the analyser configuration. In the Method menu option, the devices are defined and the events, and timing of events are programmed. Methods may be retrieved, saved, or erased. Existing methods are also edited using this option. Methods form the basic building block of Procedures. Procedures will be discussed in detail in chapter 6.



**HINT:** *In preparation for defining a method, the Device Description Data Sheet given in Appendix 17.1 must be completed.*

## 5.1 Configuring Devices

The devices in an experimental set-up are defined as those components which require digital or serial control, (e.g. pumps, valves, auto sampler, etc.). Nine device definitions are supplied with the package. The first three are Cavaro devices which require serial control, the next six are digital devices which require TTL or switch control. The device definitions are given in Table II. An additional three user-specified device definitions may be added. These additional devices can be configured using the first option of the method menu. In preparation for the definition of a new device, the Device Description Data Sheet (Appendix 17.1) should be completed. A new definition at a specific definition number will overwrite the previous definition at that number.



**WARNING! Don't overwrite device definitions that are included in methods that you want to use again. If you do, unpredictable consequences will follow as the new device is unlikely to have the same logic.**

Table II : Default Device Definitions

Detail	1	2	3	4	5	6	7	8	9
Name	CP	CS	CV	AP	GP	IV	SV	AS	SW
Action	Pick	Pick	In	Fwd	Fwd	Inj	Adv	Next	True
	Disp	Disp	Out	Rev	Rev	Load	Home		False
	Stop	Stop		Off	Off				
Hotkey	P	P	I	F	F	I	A	N	T
	D	D	O	R	R	L	H		F
	T	T		O	O				
Output	01	01	1	01	10	01	10	1	1
	10	10	0	10	11	10	01	0	0
	00	00		00	00		11		
Pulse	0.0	0.0	0.0	0.0	0.0	0.0	0.3	5.0	0.0

To Configure a device, type:

- o **Config device.**
- o Enter the chosen device definition number. You can check the present status of the device definitions on the Notepad screen. User-specified device definitions are obtained by selecting **Next** page.
- o Enter the device name. This is a two letter identification label (e.g. SV for selection valve) for the device. This label must be unique.
- o Enter the number of actions the device must perform (e.g. 3 for forward, reverse, off). The maximum allowable number is three but fewer are



permissible. If you require more than three actions, then you must split them over more than one device definition.

- O Type in the action narration for action 1 (5 characters). Action 1 is the action to be depicted above the centre line in the Method time-line box. This narration will be used in the Time and Event box on the Method screen (see figure 4 section 2.3).
- O Enter the digital output for action 1. Note - if the device is to be connected to more than one digital output point, for example points 5 and 6, and you want to set point 6 high then you must output a 2 (10 binary). To set point 5 high, output a 1 (01 binary). To set both high, output a 3 (11 binary). To set both points low, output a 0 (00 binary).
- O Type in the hot key which will be used during method development to identify this action. This is usually the first letter of the narration. It must be unique within a device.
- O Repeat the last three steps for each of the actions. Bear in mind that the second action is the one that will be depicted below the centre line in the Method time-line box. The third action will be depicted on the centre line in the Method time-line box.
- O Finally enter the pulse length (in seconds) if the device is controlled by pulses rather than a continuous signals. A pulse length of zero will be interpreted as continuous output rather than pulsed output.
- O Once all the desired devices have been configured, pressing **ESC** will return you to the method menu.

## 5.2 Setting the experiment time

The experiment time is the time necessary to complete a single measurement, i.e. the time required to load the sample loop, inject and manipulate the sample, carry it into the flow cell of the detector, and flush it from the detectors flow cell.

This menu option also allows the setting of the time at which data collection should commence. Although in most cases, this will be zero, i.e. data collection should commence at the start of the experiment, it is conceivable that for a particularly lengthy sample manipulation experiment, data collection should be delayed to commence once the manipulation is completed. This will mean that profile data files will be smaller in size. Also, the maximum data collection time is 300 seconds (5 minutes) divided by the number of detectors. The data collection time is the experiment time minus the start data collection time. To set these two times type:

- o **E**xpt time.
- o Enter the time at which data collection is to commence in seconds.
- o Enter the experiment time in seconds.



**NOTE** : *Integration limits, autozero times, time for peak height measurement, and time axis times will be set to either 0 or the experiment time (which ever is relevant) when the experimental time is changed.*

### 5.3 Specifying the devices

This option specifies the devices connected to the distribution board which are to be controlled by FlowTEK. It also provides a means of specifying a Directive pseudo device. These devices will each be displayed in a panel on the Method screen and also in the device box at the bottom of the Main screen. Data from a completed Device Description Data Sheet (Appendix 17.1) should be used to specify the devices. First the number of devices is chosen and then each device is specified.

To set the number of devices, type:

- o Type of device.
- o Enter the number of devices (max. 8).

To specify each device:

- o Enter the two letter device description or **D** for Directive
- o Enter the digital output point to which the device is connected (1-8). In the event of a device being connected to two digital points, it must be connected to adjacent points and the number of the lower point must be specified.
- o For serial devices set this value to the device address as configured on the device. (See the Cavaro manuals for details on setting the device address). When you use a syringe pump with a built in selection valve, you must specify the same address for both devices.

Once the devices have been defined, the Method screen will be divided into separate panels for each device. The device descriptions will indicate the panel that depicts the events for each device. All existing events are deleted when the devices are specified.

## 5.4 Inserting an event

There are two ways of inserting an event. The first makes use of the cross-hair cursor and is activated by de-selecting the Num Lock key. The second allows you to specify the device and exact time of an event by typing in the relevant numbers. This option is chosen by setting Num Lock on. A little square in the bottom right hand corner indicates Green for cross-hair cursor input and Red for numeric key pad input.

Directive events are treated in a similar manner to the events for the other devices except that the directive number and parameter are entered.

To insert an event using the cross-hair cursor:

- Ensure that **Num Lock** is off. The little square in the bottom right hand corner of the screen should be green when you move the cursor.
- Type **Insert**
- Use the arrow keys to move the cross hair cursor to the panel of the device you want to program. As you move from one device to the next, the Time and Event box and Device info box will be updated.
- Remember, the **Home** key returns you to the centre of the screen, the **End** key and one of the arrow keys sends you to one of the four borders. The **+** key increases the step size, while the **-** key reduces the step size. The **\*** key return the step size to its default.
- Press the single hot key defined in the Configuring device menu option which represents the action you want to insert, e.g. **F** for forward.
- In the case of a Directive, enter the directive number (see Chapter 11 and Appendix 17.4). Then enter the desired parameter for the chosen directive.
- The *Abs time* figure in the device information box gives the absolute time from the start of the experiment represented by the current cursor position. The *Rel time* figure gives the time since the last event for the selected device. The *Device number* and label indicate which device is currently being programmed.

To insert an event by typing in the exact time and device number:

- Ensure that Num Lock is selected. The little square in the bottom right hand corner of the screen should be red.
- Type **Insert**.
- Enter the device number.
- The Time and Event box and device info boxes will be updated to indicate the current status of the method for the selected device.
- Enter the desired time of the event.
- Press the single letter key representing the action you want to insert, e.g. **F** for forward.

- o In the case of a Directive, enter the directive number (see Chapter 11 and Appendix 17.4). Then enter the desired parameter for the chosen directive.

## 5.5 Deleting an event

The two modes of inserting an event hold for deleting an event as well. Either (with **Num Lock** off) the cross-hair cursor may be used or (with **Num Lock** on) the device number and time of event may be entered from the key board.

To delete an event using the cross-hair cursor:

- Ensure that **Num Lock** is off. The little square in the bottom right hand corner of the screen should be green when you move the cursor.
- Use the arrow keys to move the cross hair cursor to the panel of the device for which you want to delete an event. As you move from one device to the next, the Time and Event box and Device info box will be updated. Rel time should read zero when you are aligned with an event.
- Remember, the **Home** key returns you to the centre of the screen, the **End** key and one of the arrow keys sends you to one of the four borders. The **+** key increases the step size, while the **-** key reduces the step size. The **\*** key return the step size to its default.
- Align the cross-hair cursor vertically with the event to be deleted in the relevant device panel.
- Type **Delete**.

To delete an event by typing the exact time and device number:

- Ensure that **Num Lock** is selected. The little square in the bottom right hand corner of the screen should be red.
- Type **Delete**.
- Enter the device number.
- The Time and Event box and device info boxes will be updated to indicate the current time and event list for the selected device.
- Enter the desired time of the event to be deleted.

A pulsed event is deleted by deleting the start of the pulse. Deleting an event that is actually a pulse will delete both actions required to give the pulse. Directives are deleted in exactly the same way.

## 5.6 Inserting time

During method development it is sometimes desirable to increase the duration of a particular stage in a method (e.g. the sample load cycle is not long enough.) This option provides a way of inserting time at any point in the method. When time is inserted, all subsequent events are shifted up by the time inserted and the experiment time is increased accordingly<sup>5</sup>.

There are two ways of inserting time. The first allows for key board specified time to be inserted. The second allows for a time selected using the cross hair cursor to be inserted.

To insert time using the cross hair cursor:

- Move the cursor to the position where time must be inserted. The Device information box will provide useful information about the current position of the cursor. It is not important in which panel the cursor is positioned as the time will be inserted at the cursor point into each of the device programs.
- Press the **Enter** key. This marks the present position of the cursor and sets *Rel time* to zero.
- Use the arrow keys to move the cursor (left or right) to give the desired amount of extra time. *Rel time* will indicate the amount of time inserted.
- When the desired amount of time has been reached, press the **Ins** key.
- Time is inserted to the right of the marked position.

To insert time using the key board:

- Move the cursor to the position where time must be inserted. The Device information box will provide useful information about the exact present position of the cursor. It is not important in which panel the cursor is positioned as the time will be inserted at the cursor point into each of the device event lists.
- Press the **Ins** key. Because no position has been 'Marked' as in step 2 above, FlowTEK will prompt you to enter the time to insert (in seconds).
- Enter the time to insert.
- Time is inserted to the right of the position of the cursor.



**NOTE** : *The program will not allow you to insert time so that the experimental time exceeds the allowable maximum (i.e. 300 seconds / number of detectors).*

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<sup>5</sup> This option is particularly useful in Sequential Injection Analysis. See J. Ruzicka and G.D. Marshall, *Anal. Chim. Acta*, **237**, (1990), 329-343.

## 5.7 Deleting time

During method development, it is frequently found that a method can be shortened by cutting down on the time for certain events. Deleting time is achieved in an analogous fashion to inserting time and the experiment time is reduced by the extend of the deleted time.



**Warning! : Events falling into the time period marked for deletion, will also be deleted. The only exception to this rule is if the second part of a pulsed event falls within the time to be deleted. In this case, the pulse is reconstructed after the time has been deleted.**

There are two ways of deleting time. The first allows for key board specified time to be deleted. The second allows for a time selected using the cross hair cursor to be deleted.

To delete time using the cross hair cursor:

- Move the cursor to the position where time must be deleted. The Device information box will provide useful information about the current position of the cursor. It is not important in which partition the cursor is positioned as the time will be deleted at the cursor point from each of the device event lists.
- Press the **Enter** key. This marks the present position of the cursor and sets *Rel time* to zero.
- Use the arrow keys to move the cursor (left or right) to define the time to be deleted. *Rel time* will indicate the amount of time to be deleted.
- When the desired amount of time has been reached, press the Del key.

The time will be deleted to the right of the marked position irrespective of the direction that the cursor was moved.

To delete time using the key board:

- Move the cursor to the position where time must be deleted. The Device information box will provide useful information about the exact present position of the cursor. It is not important in which panel the cursor is positioned as the time will be deleted at the cursor point from each of the device event lists.
- Press the **Del** key. Because no position has been `Marked' as in step 2 above, FlowTEK will prompt you to enter the time to be deleted (in seconds).
- Enter the time.
- Time is deleted to the right of the position of the cursor.





## 5.8 Mode of injection

FlowTEK provides for automatic sample injection or the use of a manual sample injection valve. When a manual sample injection valve is used, FlowTEK still provides the facility of data acquisition. The injection valve must be linked to a proximity switch that will signal an injection. This switch should be connected to any one of the digital input points. The experiment must be initiated as usual from FlowTEK either using Single measurements or Repeated measurements. Data acquisition and device control will wait until the switch on the injection valve is closed.

To specify Manual sample injection, type:

- Mode**
- Manual**
- Enter the digital input point to which the injection valve proximity switch is connected.

When automatic sample injection is selected, the injection valve must be specified as one of the devices and programmed as described above.

## 5.9 Choosing the start up value for the digital output points

It is often desirable to have the program start up with a specific output to the digital output points. For example, valves and other devices could be set to start up positions. The actual digital value or binary equivalent (entered with a B as prefix) can be stored in the configuration file FT.CFG.

To set the start up values for the digital points, type:

- 0 **Startup**
- 0 Enter the desired value either as an integer or a binary string with prefix B. Use the same rules as given in section 4.7 to determine the number to use.

If serial devices are used, the initialisation command is sent to all Cavaro devices.

## 5.10 Saving, retrieving, and erasing method files

Once a method has been defined, it can be saved to disk. Alternatively, a previously defined method may be retrieved from disk. This option also provides a way to delete obsolete methods. As in other menus, the **F10** function key allows for a directory of files to be displayed on the screen. The file format of the method file is given in Chapter 11. The rules for entering a file name as specified in section 3.4 hold for method files as well.

To Save, Retrieve, or Erase a file, type:

- 0 **File.**
- 0 Choose **S**ave, **R**etrieve, or **E**rase a file.
- 0 Enter the file name. Wild cards may be used for the erase option.
- 0 If the file exists and the operation is Save, you will be asked whether you want to overwrite the file. If the operation is Retrieve and the file does not exist, you will be notified and given a chance to re-enter the file name.

## 6. A single experiment

This option on the main menu allows you to perform a single experiment.

Before activating this option, it is necessary to configure the software and instrumental system as you want it to be. The last used software configuration as saved in the set up file (FT.CFG - see chapter 12) may be acceptable or require minor alteration.

The **S**et-up (chapter 4) and **M**ethod (chapter 5) menu options can be used to achieve the desired configuration. The **N**otepad option provides a means of examining the current settings. The function keys (**F3** to **F9**) can be programmed to manually ensure that the all devices are in their start up positions and the various lengths of tubing in the flow manifold are filled with the correct solution, e.g. the carrier stream has been flushed through the manifold.

Once the devices connected to the distribution board have been powered up and the user-selectable options have been correctly set, a single experiment can be performed by typing **O**nce.

The data from the experiment will be saved in the relevant data files (Reduced data and Peak profile data, if this has been selected). The experiment counter will be incremented and the system will be ready for the next experiment.

It is permissible to intermix Single and Repeated measurements. The experiment counter will be reset to 1 when the reduced data file name is changed. When the reduced data file is changed, the profile root file name should also be changed to prevent profile data files from being over written. Remember the profile data are stored in a file with the name comprising the root with the experiment number as extension e.g. profile.1, profile.2, .....

The experiment counter is set to one when you:

- use the **R**eset option on the Set-up menu. This will reset all parameters to their default values,
- change the name of the reduced data file,
- enter the program.

## 7. Analysis by procedures

The concept of Analysis by Procedures is one of the most powerful options of this package, particularly when this is combined with the option of including instrument directives in the procedure. It enables you to select any combination of directives, methods, and procedures for execution. It is also possible to program the number of repetitions of each method or procedure. Because a procedure can comprise other procedures, it is possible to define common procedures such as Calibrate, Wash, InstrOff and use these as building blocks to suit the particular requirements of each application.

A procedure is made up of one or more methods, other procedures, and directives, or any combination of these. For methods and procedures, the number of repetitions must be specified. When directives are used, the appropriate parameter must be specified (see chapter 11).

A procedure may be aborted at any time by pressing the **ESC** key. If the **ESC** key is pressed during a wait period, the wait period will be aborted but the procedure will remain active. To abort the procedure in such an instance, just press **ESC** again. If a particular method or procedure is included in a procedure definition and subsequently erased, this will cause the procedure to abort when the program tries to access that erased file. A message is displayed on the second line of the screen indicating the status at exit from the procedure.

The Analyser can be programmed to run *ad infinitum* by using the Reset Directive (Directive number 254). This directive reboots the computer after saving the relevant data. If FT.BAT has been included in the AUTOEXEC.BAT, then FlowTEK will start up again and repeat the Main procedure from the beginning.



**WARNING! Although reduced data will be appended under these conditions on the Reduced Data file, profile data will be overwritten because the experiment counter is reset to 1 when the program starts again.**

## 7.1 Listing a directory of procedure and method files

The **F10** function key displays a list of procedure (.PDR) and method (.MET) files in a window on the Repeated set-up screen. The list is split into .PDR and .MET files. There is no sorting of these files within the group. This list remains visible during procedure development. It is not updated as new procedures are added. To refresh the list, you must press **F10** again.

## 7.2 Building a procedure

Before building a procedure, you must ensure that all the files (both .PDR and .MET) that you plan to use exist and are in the same subdirectory. You will not be allowed to use a file that does not exist. You can use the Procedure Description Data Sheet given in Appendix 17.3 to prepare your procedure. You can get a permanent record of your procedure on the Notepad menu.

Although directives are entered with the same format as files, they are not stored as files but are represented internally in the software. The same file format is used to keep the interface consistent.

To build a procedure, type:

- **Build Proc.**
- Enter the desired method or procedure file name. You must enter the extension.
- You can enter a directive by typing the directive number with the extension .DIR. (e.g. to set the baud rate of the serial port use directive 34 and enter it as follows, 34.DIR.)
- Enter the number of repeats for the method or procedure, or in the case of a directive enter the appropriate parameter, (e.g. to select a baud rate of 9 600, enter a parameter of 96).
- Repeat the last two steps until there are no more entries, then press **ESC** to signal the end of the procedure. A maximum of 20 steps are allowed.



### 7.3 Displaying a procedure

You may wish to view a previously defined procedure. This menu option allows you to do that. The procedure is displayed in the box where the procedure is usually defined.

To display a procedure, type:

- o **D**isplay Proc.
- o Enter the procedure file name to display. If the file does not exist, a message to that effect will be displayed.
- o The procedure is cleared to display the next procedure or to build a new one.

## 7.4 Declaring the Main procedure

Once all the desired procedures have been built, you must indicate to the program which is the Main procedure. This is the procedure that the program must begin with when you start the Repeated analysis. This is also the procedure which will automatically commence when the program is started. The program is set up so that the Main procedure is executed on start-up to accommodate a power failure when the software is used in an automated mode. To achieve this, FT.BAT must be included as the last command in the AUTOEXEC.BAT file. To declare the Main procedure, type:

- o **Main.**
- o Enter the file name of the main procedure.
- o The entered file will be displayed in the set-up conditions box.

## 7.5 Specifying the reduced data file and profile file root

The reduced data file specified on this page will be the file where all reduced data for the procedure are saved. If the file exists already, the reduced data will be appended. See chapter 12 for the format of the reduced data file.

The profile file name will be the profile root with the extension giving the experiment number, e.g. test.1, test.2, test.3, .... If the procedure is to be repeated indefinitely, it is important to determine whether more than 999 experiments will be carried out. If so, profile saving should be disabled as all the profiles from 1000 on will overwrite existing profile files, and could lead to confusion.

In addition to profile files, it is also possible to store abridged profile files which only contain the response data. These files are useful for importing the response data into a third party package without all the other supporting data usually stored in a profile file. The format of the file name is a<profile root>.<expt no>, e.g. atest.1, atest.2, atest.3, .... If no profile root is entered, no profile files will be saved. To select abridged profile file saving use the Save profile option on the file menu (see section 12.6).

To choose a reduced data file and profile root, type:

- File**
- Enter the desired reduced data file name.
- The entered file will be displayed in the set-up conditions box.



**NOTE** : *When the reduced data file name is changed, the experiment counter is reset to 1. If the reduced data file already exists, reduced data will be appended but profile files could overwrite previous profile files.*

- Enter the desired file root.
- The entered file will be displayed in the set-up conditions box.
- If the profile filing was previously disabled, you will be prompted to enter whether you want profiles and / or abridged profile files to be saved.

## 7.6 Setting parameters required for certain directives

Certain directives require additional information to operate correctly. (e.g. directive 48 compares the concentration measured in the last method to a value set up under this menu option to establish whether the calibration is still valid.). These values must be entered before you begin the procedure and they remain constant for the duration of the experiment. These values are stored in ft.cfg (see chapter 12). The present values for each of the parameters are listed on the Repeated screen (see figure 5 section 2.4).

To enter new values for these parameters, type

- o **Set-up**
- o Enter a value for the percentage range variation permissible in concentrations measured around the setpoint before a Warning is generated (see Directive 46).
- o Enter a value for the percentage range variation permissible in concentrations measured around the setpoint before an Alarm is generated (see Directive 47).
- o Enter a value for the percentage range variation permissible in the measured concentration for the check standard around the pre-set value before recalibration is required (see Directive 48).
- o Enter the permissible variation in the baseline signal before a Warning is generated indicating excessive noise (see Directive 62)
- o Enter the maximum change in voltage sent to the D to A converter. This voltage is proportional to the concentration measured in the last sample and is useful for tracking a particular stream. The limit is set to avoid rapid changes in this response resultant from outliers.
- o Enter the desired Setpoint.
- o Enter the concentration of the check standard used to validate the calibration.
- o Enter the name of the procedure used to recalibrate the instrument if the calibration is found to be invalid.

## 7.7 Executing a procedure

Once the Main procedure and data file names have been selected, all is set to begin the procedure. Start the procedure by typing:

- o **Go!**
- o The Main screen will be displayed and the first method in the procedure will be loaded and executed. The name of the method being executed is displayed at the top of the peak parameter box.
- o At any time during the procedure, you can abort the procedure by pressing **ESC**. If you press **ESC** during a WAIT period, the WAIT will be aborted and the procedure will continue with the next method.

## 8. Using the calibration option

A calibration option has been included for simple conversion of the response from the detectors to concentration units. This calibration option provides for manual input of calibration data. Alternatively, the data can be acquired by executing a procedure. Directive 49 is used to allocate a response to the calibration table. Basic data editing options are provided to allow a data point to be added, deleted, changed, or the experiment to be repeated to obtain a new value.

Several regression models are offered and the regression can be carried out on any of the peak parameters. The calibration data and regression results are depicted graphically on the calibration screen.

Where more than one response is obtained for a particular concentration level, the data are represented as a span in the y direction showing the highest and lowest points. The regression is carried out on the mean of the points. If one or more data points are deleted, the simple mean of the remaining points is used in the regression calculation.

You can try various combinations of peak parameters and models until you find a combination which gives you a satisfactory calibration curve. All subsequent concentration calculations will be based on the last curve calculated.

The data for more than one calibration set can be stored in the calibration table. The *First Standard* and *Last Standard* parameters indicate which data to use in the calibration calculation.

An option is also included to recalculate the concentration for the data in any reduced data file using the current calibration curve.

The calibration data and curve can be saved or retrieved from a file.

## 8.1 Specifying the calibration data

There are two methods of data entry. The response data can be acquired and stored in the calibration table using Directive 49. Or, the data can be entered manually from the keyboard.

If manual data is used, choose the peak parameter for which data are to be entered. It is not necessary to enter data for each peak parameter. To evaluate other peak parameters, re-enter the response data and specify the desired parameter. In this case, the peak parameter is simply for information purposes.

To specify the calibration data to use in a regression calculation, type:

- 0 **S**et-up.
- 0 Enter the concentration units of the standards. These units will be used for samples as well.
- 0 Enter the First standard number in the calibration table.
- 0 Enter the Last standard number in the calibration table.

## 8.2 Changing the peak parameter

If the calculated correlation coefficient is not satisfactory, you can try performing the regression on a different peak parameter. To change the parameter, type:

- 0 **P**k param.
- 0 Choose **H**eight, **A**rea, **W**idth, or **T**ime.
- 0 Use **E**dit, **I**nsert, or **D**elete to update the data.



### 8.3 Changing the model

One of five different regression models can be selected. There is a choice of linear, quadratic, trinomial, rectangular hyperbola, or exponential models. The regression can be carried out on any of the peak parameters (i.e. peak height, area, width at a fixed height, or time). In the regression calculation, a least squares regression on concentration as a function of response is calculated. In the case of the exponential and rectangular hyperbola models, the data are first transformed and a linear regression is carried out on the resultant transformed data.

The regression coefficients are displayed and can, given the response, be used directly to determine the concentration of an unknown. The concentration is calculated as follows for C=concentration, r=regression coefficient, and R=response:

o for the polynomial models 
$$C = \sum_{i=0}^n r_i R^i \quad n = 1, 2, 3$$

3

o for the exponential model 
$$C = r'_0 e^{r_1 R} \quad r'_0 = e^{r_0} \quad 4$$

o for the rectangular hyperbola model 
$$C = \frac{r'_0 R}{r'_1 - R} \quad r'_0 = \frac{1}{r_0} \quad r'_1 = \frac{r_1}{r_0} \quad 5$$

The correlation coefficient ( $r^2$ ) is calculated in each case and serves as an indication of the goodness of fit. An additional option is offered in the choice of the model. If All is chosen, the regression is carried out using each of the five models and the correlation coefficient is calculated for each case. The model which gives the highest correlation coefficient is automatically selected. (If there is not a unique solution to a particular model, the correlation coefficient is set to zero.)

A different calibration model can be tried by typing:

- o **Model.**
- o Choose **L**inear, **Q**uadratic, **T**rinomial, **E**xponential, **H**yperbola, or **A**ll.

The regression will be repeated and the regression information box will display the relevant regression and correlation data.

## 8.4 Editing the calibration data

A facility is also provided to edit any or all of the response data. This option is also used for keyboard data correction.

To use this facility, type:

- o **Edit.**
- o Enter the number of the standard you want to edit, **0** for all, i.e. keyboard data entry.
- o Enter the replicate number to edit, **0** to change the concentration value.
- o Once you have made all the changes you want to, press **ESC** to go onto choosing the model.

This option is only used for editing existing data. To add new data use the **Insert** menu option.

When you select the calibration model, the regression will be performed on the data and the calibration curve and regression information will be displayed on the screen.

## 8.5 Inserting data points

To enter new data points or assign a concentration value to a particular standard in the calibration table, use this menu option. When inserting data for a standard, the program will first establish whether the standard has been assigned a non-zero concentration value. If the concentration for the chosen standard is zero, the program will ask for a concentration value. In this way, new standards are inserted into the calibration table.

If the chosen standard has a value already, this menu option will establish how many replicates are already stored. New inserted replicates will be added at the end of the existing replicate list. If there are already three replicates, the first replicate will be deleted, and the other replicates will be shifted up to make a place at replicate number three for the new value. In this way, the oldest data is always deleted. This approach is also used when using a directive to add response values to the calibration table.

To insert a new standard:

- o Select a standard in the calibration table that has a concentration of zero and forms a contiguous series with the other calibration data you will use for this specific calibration.
- o Type **Insert**.
- o Enter the standard number.
- o Enter the concentration using the units that will be used to report the sample concentration as selected using the **Set-up** menu option.
- o Enter the response for the first replicate of the new standard. If the response is not yet available, simply press **ESC**.

To insert a new response for an existing standard:

- o Type **Insert**.
- o Enter the standard number.
- o Enter the response for the new replicate.

## 8.6 Deleting data points

If a particular data point is an outlier, you can delete it. The regression is then carried out using the mean of the remaining data. You may even delete all the replicates of a particular concentration level. Provided that there are still sufficient points to yield a unique solution, the regression will be carried out on the remaining points.

To delete a data point, type:

- Delete**
- Enter the standard number
- Enter the replicate number

Deleted replicates will be blanked out. Remaining replicates will be shifted left to fill up the response table for a particular standard from the left.

## 8.7 Reset Calibration table entries

The entire calibration table can be cleared using the Reset menu option. This option sets all concentration values to zero. All Responses are erased, and the replicate counters for each standard are set to zero. Also the regression coefficients for each detector are also set to zero.

To Reset all variables, type

0     **Reset**

## 8.8 Recalculating data in a reduced data file

If a series of experiments were carried out without a calibration curve being active or using an invalid calibration data set, it is possible to return afterwards and re-calculate the concentration values by referring to a new or different calibration set. The calibration data used may originate from acquired data or from manually entered data. The option which allows this is called Calc Red.

To recalculate the concentration field in a reduced data file, type:

- o **C**alc Red.
- o Enter the name of the reduced data file to recalculate.
- o You can print out the recalculated data file using the **P**rint RED option on the **N**otepad menu.

The new values will be stored in a new reduced data file with the same name as the old.

## 8.9 Saving, retrieving, and erasing calibration files

Once a regression calculation has been concluded, it can be saved to disk. Alternatively, a previously defined calibration may be retrieved from disk. This option also provides a way to delete obsolete calibration files. The file format of the method file is given in Chapter 12. The rules for entering a file name as specified in section 3.4 hold for calibration files.

To Save, Retrieve, or Erase a file, type:

- o **F**ile.
- o Choose **S**ave, **R**etrieve, or **E**rase a file.
- o Enter the file name. Wild cards may be used for the erase option.
- o If the file exists and the operation is Save, you will be asked whether you want to overwrite the file. If the operation is Retrieve and the file does not exist, you will be notified and given a chance to re-enter the file name.

## 9. Data perusal with the history module

Data manipulation, in this program, has been limited to a simple graphical data viewing module. However, all data files are stored in ASCII format with the structure of the files being outlined in chapter 12. In addition, a Quattro Pro macro is provided which reads a reduced data file and loads it into a Quattro Pro work sheet.

The data perusal module, obtained by selecting the History menu option from the Main screen, displays by means of bars or points the peak parameter response of up to a hundred experiments. Where more than one detector has been used, the data for each detector can be accessed using the **Pg Up** and **Pg Dn** keys.

It is also possible, using the History menu option, to recall and display in a pop-up window, any of the profiles of the data on the screen, if the respective profile files were saved. All the peak parameters of a chosen experiment are also displayed in a peak parameter box on the History screen.



## 9.1 Choosing a peak parameter

The peak parameter is selected by pressing the respective Menu key. The relevant parameter will be displayed as dots or bars for the given span of experiments (default 1 to 100). The peak parameter to display and mode of display is chosen as follows:

- o Select the peak parameter, Peak **H**eight, **A**rea, **W**idth at a fixed height, **T**ime, or **C**oncentration .
- o for points, type . (period).
- o for bars, type / (forward slash) or \ (back slash).

## 9.2 Choosing the experiments to display

A maximum of a hundred experiments may be displayed on the History screen at any one time. This enables the perusal of data from extended procedures. To choose the span of experiments to display, type:

- o **Span.**
- o Enter the first and last experiment numbers to display.

### 9.3 Displaying a profile

The peak parameters of a single experiment, (the one highlighted in red), are given in the peak parameter box. The profile of this experiment can also be recalled by using the Profile option. This experiment is chosen in one of two ways:

- o Using the cross-hair cursor: Use the arrow keys to vertically align the cursor with the desired experiment. Press the Enter key.
- o Typing an experiment number: Type Read and enter the number of the desired experiment. The peak parameter box will be updated to display the chosen experiments data and the corresponding experiment on the history screen will be highlighted in red.

Once an experiment has been selected, and providing that the profile data were saved, an experiment profile can be recalled. The profile is displayed in a small pop-up window. The profile is recalled by:

- o selecting the desired experiment.
- o type **P**rofile.
- o The profile window is closed by pressing any key.

## 10. Directives

A Directive alerts the software to user-selectable configuration options. Appendix 17.4 gives a complete list of available Directives. Each directive is discussed in more detail. Directives can be incorporated in both Procedures and Methods. FlowTEK has been structured to permit the convenient addition of further Directives in the future. Suggestions from users are therefore encouraged.

Directives are labelled from 32 to 255. The reasons behind this numbering system is historical and has been maintained to ensure compatibility with other systems. Appendix 17.4 lists the valid parameters for each Directive and the default settings when FlowTEK is run.

## 10.1 Definition of Directives

### ***Send diagnostic data to the Printer (Directive 32)***

When this Directive is selected with the parameter set to 1, FlowTEK will send additional diagnostic data to the printer port. This is useful during Method and Procedure testing. This data key peak data and a narrative detailing all analyser events. Usually, this directive is used as one of the first directives in the Startup Procedure. Alternatively, if detailed information is only required for a particular method, this option can be switched on (parameter=1) and off (parameter=0).

### ***Select output to the printer (Directive 33)***

FlowTEK can output response and calibration data to a printer port. In order for this option to work, the printer or suitable logging device must be connected to the parallel port and be on-line. Failure to do so will result in an error. By default, output to the printer is disabled (parameter=0). This directive must be used with a parameter of 1 to ensure that output is sent to the printer.

### ***Set the baud rate and COM Port (Directive 34)***

This directive is used to set the desired COM port and baud rate. The protocol used for serial communications is fixed as No parity, 8 bits, and 1 stop bit. The default COM port is 1 at a baud rate of 9 600.

The parameter used with this directive allows the selection of a different COM port and baud rate. The last saved baud rate and COM port are stored in the ft.cfg file (see chapter 12) so that if power is interrupted, the last saved baud rate will be used when FlowTEK is powered up again. If no serial device is connected to the chosen serial port, undue delays while the serial coms modules “time-out” will be introduced if the COM port is not set to 0. A COM port setting of 0 indicates that serial output is disabled.

Parameters for this directive are as follows:

Parameter	Result
0	Disable
1	COM 1
2	COM 2
3	COM 3
4	COM 4
12	1200 baud
24	2400 baud
96	9600 baud

### ***Check the correlation coefficient (Directive 35)***

This Directive should be used before a regression is carried out. Then, once the regression has been performed, if the calculated correlation coefficient ( $r^2$ ) is less than 0.98 (parameter 1), 0.99 (parameter 2), or 0.999 (parameter 3), the old regression coefficients are retained and the regression just performed is ignored. If the parameter is set to 0 (default), this function is deactivated.

### ***Select detector channel (Directive 36)***

FlowTEK has four Analog input channels. This Directive specifies the desired input channel. While data are acquired simultaneously from all channels, the calculation of concentration is based on the regression coefficients of the chosen detector channel, using the chosen autozero scheme for the selected channel. To calculate and print the concentration values of other channels, select the channel number and perform the relevant calibration calculation. Of course, the response of one channel should be calculated and displayed (Directive 42) before the next is chosen.

### ***Peak or Valley (Directive 37)***

The signal from most detectors starts low and increases when the sample passes through the detector and then returns to the baseline. The response is a typical peak. In some cases, the baseline response is high and when the sample passes through the detector, the response decreases to give a typical valley shaped signal. This Directive instructs FlowTEK whether to follow a peak or a valley. For Peak tracking the analytical signal is equal to the maximum response registered minus the auto zero response. For Valley tracking the analytical signal is equal to the auto zero response minus the minimum response registered.

### ***Number of decimal places reported (Directive 38)***

Calculation of the concentration data is carried out using the maximum number of available decimal places. Once the result is determined, it is printed and displayed with the number of decimal places selected with this directive. The default is 2 and the maximum number is 4. Of course by choosing the concentration units sensibly, the most appropriate output will be obtained.

### ***Time to start measurement (Directive 39 and 40)***

Some Flow-injection methods require a lengthy sample manipulation period before the sample is presented to the detector. A typical example is an incubation period in the case of certain enzymatic reactions. The detector response during this period is of

little consequence. Directive 39 and 40 instruct FlowTEK to start data acquisition at a specified time during the Method. This option provides a way of ignoring spurious peaks before the analytical signal is generated. This value is the same as is set in the **Method** menu option under **Expt Time**

Directive 39 interprets the parameter as minutes while Directive 40 interprets the parameter as seconds.

#### ***Auto zero (Directive 41)***

Directive 41 changes the time at which the reading for the selected channel is taken that is subtracted from response values as an auto zero. It also sets Auto zero frequency to Every experiment. Ensure that the same channel is used for determining the zero response and the analytical signal by using directive 36 before this directive. This Directive is usually placed before the Method, if it is necessary to change the time at which the baseline is measured. This value should preferably be set up using the **AutoZero** option under the **Set-up** menu option. Although it is recommended that the base line response be determined for each measurement, this is not a requirement specially where there is a stable or negligible baseline response from the detector.

#### ***Analog output corresponding to Set Point concentration (Directive 42)***

An output is sent to the Analog output channel specified by the parameter. The value sent is determined as follows:

$$Val = \frac{R_{SA} - R_{SF}}{R_{SL} - R_{SF}} * 10$$

*Val* - value sent to the Analog output

*R<sub>SA</sub>* - Concentration of Set Point

*R<sub>SF</sub>* - Concentration of first calibration standard

*R<sub>SL</sub>* - Concentration of last calibration standard

If 4-20 mA circuitry is included, this option can be used to generate a 4-20 mA signal for process control applications. This directive is useful when the 4-20mA signal is being used to control a component at a particular set point. Typically it would be used at startup to establish a value for the 4-20 mA output to be maintained until the first reading from the plant was received.

The value of the Set Point is specified in the **Repeated** menu **Set-up** option.

#### ***Analog output corresponding to the parameter (Directive 43 and 44)***

An output voltage is sent to Analog output channel 1 (Directive 43) or channel 2 (Directive 44) corresponding to the value specified as the parameter divided by 10, i.e. if parameter = 55, then Analog output = 5.5V. (This output is also available as a 4-20 mA signal if this circuitry has been coupled to one of the Analog output channels.)

Where a manifold device requires an analog input to effect one or other change, this Directive can be used, (e.g. the pump speed of the Alitea pump can be altered using a 0 to 5 V signal.)

#### ***Analog output corresponding to response (Directive 45)***

An output is sent to the Analog output channel specified by the parameter. The value sent is determined as follows:

$$Val = \frac{R_{SA} - R_{SF}}{R_{SL} - R_{SF}} * 10$$

*Val* - value sent to the Analog output

$R_{SA}$  - Concentration of sample

$R_{SF}$  - Concentration of first calibration standard

$R_{SL}$  - Concentration of last calibration standard

If 4-20 mA circuitry is included, this option can be used to generate a 4-20 mA signal for process control applications. This output can also be modulated to minimise the effect of outliers. The maximum permissible change from one measurement to the next is set using Directive 63. If the deviation persists then the Analog output will be altered by an amount equal to the value specified in Directive 63 in successive measurements.

#### ***Compare to Setpoint - Warn (Directive 46)***

The last obtained concentration response is compared to the set point. The set point is specified in the **Repeated** menu, option **Set-up**. If it deviates from the set point by a percentage greater than that set up in the same **Set-up** option, a warning message is displayed and Warning Output Point 1 is set high. This output can be used to trigger a visual or audible alert.

#### ***Compare to Setpoint - Alarm (Directive 47)***

The last obtained concentration response is compared to the set point. The set point is specified in the **Repeated** menu, option **Set-up**. If it deviates from the set point by a



percentage greater than that set up in the same **Set-up** option, an alarm message is displayed and Warning Output Point 1 is set high. FlowTEK waits until the alarm status has been cleared. The alarm status is cleared by setting digital input point 1 high. A Reset button should therefore be connected to this Input point.

#### ***Compare to Check standard (Directive 48)***

When the calibration for a particular Method is fairly stable, this Directive provides a means of only recalibrating when it is absolutely necessary. Periodically (say after 50 measurements), a check standard is measured. If the calculated response falls outside the limits set in the **Repeated** menu, option **Set-up**, the calibration Procedure entitled RECALIB.PDR is executed. This file name is stored in the FT.CFG file (see chapter 12). If it falls within the range the calibration is omitted and the Procedure continues.

#### ***Allocate standard response (Directive 49)***

This directive indicates that the response from the next measurement is to be stored in the calibration response table. Having executed a Method to analyse a calibration standard, this Directive ensures that the response is stored in the calibration response table. This Directive must precede the end of the method or the Peak Calc Directive (Directive 42) and will usually precede the Method. Because this step is independent of the Method, the same Method can be used to analyse samples and standards. Also standards can be analysed in any order and at any time during a Procedure.

The Regression Directive (Directive 53) uses data stored in the calibration response table. This table can store up to three replicates for each standard. Where more than three replicates are measured, the oldest response is overwritten. Before the regression calculation is executed, the responses for each standard are averaged. Directive 51 and 52 inform the Regression algorithm which data in the calibration response table to use.

Directive 50 clears the regression response table and sets replicate counters back to zero. Calculation of new regression coefficients is only carried out when Directive 53 is selected.

#### ***Clear the calibration response table (Directive 50)***

This Directive clears the calibration response table and sets the replicate counters back to zero. See Directive 49.

#### ***Select the first standard (Directive 51)***

The data for several calibrations can be stored in the calibration response table. These data are stored using Directive 49. Two directives are then required to inform the regression algorithm which data to use. This directive marks the first standard in the calibration response table. Directive 52 specifies the last standard in the calibration set. These two pointers also determine the values used in Directives 42 and 45.

**Select the last standard (Directive 52)**

In order to allow more than one calibration set to be stored in the calibration response table, it is necessary to be able to determine the first and last standard in a particular calibration set. This Directive points to the last standard in a particular calibration set. Refer to Directive 51 for an important application of this directive.

**Regression model (Directive 53)**

One of five regression models can be applied to calibration data collected using FlowTEK. The data to use are specified by the first standard number (Directive 51) and the last standard number (Directive 52). Responses collected for a particular standard are averaged before the regression is carried out. A maximum of three replicates can be stored. A linear least squares regression function is used to determine the regression coefficients. Note, this function uses the peak response data selected by the calibration mode which is set using Directive 65 or set in the Calib menu, Peak response option. The regression module also calculates and prints (where applicable) the regression coefficient,  $r^2$ .

The five regression functions supported are:

$C = a_1R + a_0$	<i>Linear</i>
$C = a_2R^2 + a_1R + a_0$	<i>Quadratic</i>
$C = a_3R^3 + a_2R^2 + a_1R + a_0$	<i>Trinomial</i>
$C = r'_0 e^{r_1R}$ $r'_0 = e^{r_0}$	<i>Exponential</i>
$C = \frac{r'_0 R}{r'_1 - R}$ $r'_0 = \frac{1}{r_0}$ $r'_1 = \frac{r_1}{r_0}$	<i>Rectangular hyperbola</i>

In each case

- C refers to concentration
- R refers to response, and
- $a_0$  to  $a_3$  are the regression coefficients.

### ***Wait for a period (Directive 54 and 55)***

A pause between instructions in a Procedure can be achieved by using these Directives. Directive 54 interprets the parameter as minutes while Directive 55 interprets the parameter as seconds.

### ***Wait for digital input high (Directive 56)***

Execution of a Procedure or Method can be suspended until a digital input is set high. The parameter specifies the digital input point.

### ***Wait for digital input low (Directive 57)***

Execution of a Procedure or Method can be suspended until a digital input is set low. The parameter specifies the digital input point.

### ***Wait for keypad input (Directive 58)***

Execution of a Procedure or Method is suspended until the number given by the parameter is pressed on the keypad. Note only single digit numbers are valid, i.e. 0 to 9

### ***Digital output to port 1 (Directive 59)***

This Directive sets the output at port 1 (i.e. digital outputs 1 to 8) to the value given by the parameter. Calculate the value to send to the port using the following equation:

$$Val = \sum_{p=1}^8 d * 2^{p-1}$$

*Val* - is the value to use as the parameter,

*p* - is the digital output point, and

*d* - is 1 or 0 depending on whether to set the relevant point high or not

### ***Digital output to port 2 (Directive 60)***

This Directive sets the output at port 2 (i.e. digital outputs 9 to 12) to the value given by the parameter. Use the same equation as for Directive 59 but divide the value by 256 before you use it. i.e. suppose that you wanted 9 and 10 set high:

Apply the equation  $Val = 1 * 2^{(9-1)} + 1 * 2^{(10-1)} = 768$

Divide by 256  $Val = 768 / 256 = 3$

Use the value 3 as the parameter. These digital output points are not wired to terminals in the Distribution box.

### ***Serial output (Directive 61)***

This directive can be used to send characters down the serial line. The ASCII value of the desired character must be used as the parameter. The COM port and baud rate selected with Directive 34 is used.

### ***Select acceptable baseline noise levels (Directive 62)***

This directive selects the maximum permissible baseline noise levels. The baseline noise is determined by taking 10 measurements of the detector response when Directive 71 is executed. The spread of these values is obtained. If the spread exceeds the value set by this Directive or in the **Repeated** menu, **Set-up** option, then a warning is generated. The parameter is interpreted as mV, i.e. 1 to 10 000 mV. If the parameter is set to 0 or Instrument Diagnostics are disabled (Directive 70), then this option is disabled.

### ***Select limits for Outlier damping (Directive 63)***

When the 4-20mA signal is used for control purposes, it is sometimes undesirable to have rapid changes in the 4-20 mA signal. In some cases, such increases are simply the result of a spurious signal or outlier. This directive provides a means of damping the change in 4-20 mA signal.

The concentration is determined in the normal way and is reported (to the screen and printer port). Then the voltage necessary to generate the desired output signal (Directive 45) is calculated. If Outlier damping has been selected, the newly calculated voltage is compared to the last voltage sent to the specified channel. If it differs by more than the value specified in the parameter, then the voltage that is sent to the D to A converter is equal to the last voltage sent plus the limit specified by the parameter. This directive interprets the parameter as mV, i.e. if you use a parameter of 10, the maximum allowable change in voltage will be 10 mV. This means that permissible levels of damping between 1 and 10 000 mV can be used.

If the parameter is set to 0, then outlier damping is disabled.

This directive should be used with caution and only once the outliers have been minimised by sound manifold and detector design.

### ***Calculate and print the last response (Directive 64)***

Although the response of the last chosen channel is automatically calculated and displayed at the end of a Method, it is sometimes necessary to carry this out during a Method. Specifically, this is necessary where more than one channel is being used and it is necessary to output the results of other channels in real time (as a 4-20mA signal) as opposed to simply storing them to the .RED file. It also allows for the use of different calibration curves. Where this option is required, it would be necessary to run the following directive sequence:

Directive	Parameter	Description
36	2	Select detector channel 2
51	1	Set First standard = 1
52	4	Set Last standard = 4
53	1	Calibrate using Linear model
64		Output the peak parameters, concentration, and Analog output for the last obtained peak

### ***Select peak parameter for calibration data (Directive 65)***

Most often, peak height is used as the parameter for quantifying the response from the detector. FlowTEK also permits the use of peak area, peak width at a specified height, peak time, or peak height at a specific time (other than at peak maximum). This directive selects the desired mode. Parameters such as the integration limits for area, the height at which the peak width is determined, and the time at which peak height is determined are selected in the **Set-up** menu.

### ***Enable Instrument Diagnostics (Directive 70)***

Several Instrument Diagnostics have been added to FlowTEK. These directives allow the checking of key diagnostic information. An example of this is the monitoring of the baseline noise. This Directive enables these instrumental diagnostics. The output from these diagnostic tests are sent to the printer port.

### ***Noise Check (Directive 71)***

It is useful to periodically monitor the noise from the detector circuitry. This directive checks the noise on the selected detector channel and raises a warning if it exceeds a pre-determined level. The permissible noise level is specified in the **Repeated** menu, **Set-up** option and is expressed in mV. Warning Output Point 6 is set high.

### ***Generate a Warning based on digital input (Directive 72 and 73)***

The digital inputs can be connected to monitoring devices that generate a digital signal in the event of an unacceptable condition. These directives can be used to monitor these digital inputs and raise a warning if the input goes High (Directive 72) or if it goes Low (Directive 73). When a Warning is generated, a warning message is displayed and Warning Output Point 3 is set high.

#### ***Generate an Alarm based on digital input (Directive 74 and 75)***

The digital inputs can be connected to monitoring devices that generate a digital signal in the event of an unacceptable condition. These directives can be used to monitor these digital inputs and raise a warning if the input goes High (Directive 74) or if it goes Low (Directive 75). When an Alarm is generated, an Alarm message is displayed, and Warning Output Point 3 is set high. FlowTEK waits until the alarm status has been cleared. The alarm status is cleared by setting digital input point 1 high.

#### ***Generate a Warning if the last Response drops below a specified value (Directive 76)***

It is useful to monitor the detector output to ensure that there is not a loss in sensitivity. This is particularly important where there is a possibility of the detector becoming fouled or reagents degrading. This directive is usually used after the top calibration standard or Check Standard. If the last response drops below a value specified by the parameter a Warning is generated. When a Warning is generated, a warning message is displayed and Warning Output Point 4 is set high.

#### ***Execute a Procedure based on a digital input (Directive 80)***

Where remote control of the analyser is required, it may be necessary to send the analyser a digital signal that can be used to initiate a Procedure. Such a procedure may select a different stream or initiate a wash routine or carry out a calibration. This Directive allows this kind of remote control. The parameter acts as a mask for the digital input and enables various digital inputs to specify different Procedures. It also means that digital inputs that are used for other purposes can be masked out. The procedure to be executed is determined by ANDing the Digital input (from the configured four digital inputs) with the parameter, e.g. if the parameter is 6 (0110) and the digital input is 1010, Procedure 2.PDR will be executed (0110 and 1010 = 0010 (2)). In this example digital input 1 and 4 are not brought into reckoning in determining the required procedure. Of course, procedure 2.PDR must be defined and resident in the default path.

#### ***Reset micro (Directive 254)***

This Directive performs the same action as toggling the reset switch on the computer. Its main function is to reset all set-up options (except those stored in files) to their default values and begin the startup Procedure again. In this way, a never ending repetition of the Main Procedure is achieved. Remember, that when the program starts up, the experiment counter is set to 1. This means that if profile saving is enabled, profiles will be over written when this Directive is used.

## 11. The File manipulation options

The File menu in the program provides some file manipulation and set up options. The facility of being able to exit the program temporarily and shell to DOS is a useful facility which provides access to all the usual DOS commands without losing the current operating conditions. The present status of file options such as file names, the default sub directory, and whether Peak profile saving is enabled, can be verified using the Note pad menu option. A facility is also provided to delete files. In this option wild cards are supported. As in other menu options pressing **F10** gives a list of files in any sub directory.



## 11.1 The DOS shell

It is often necessary to leave the program temporarily to execute a standard DOS command. The option is provided in the File menu option. To Shell to DOS, type:

- o **DOS**.

Once you have entered the DOS shell, the DOS prompt is altered to remind you to return to FlowTEK. To return to FlowTEK, type:

- o **exit** at the DOS prompt.

## 11.2 Erasing a file

The Erase file option on the file menu provides the option to erase a file or group of files from a valid disk drives. To erase file(s), type:

- 0 **E**rase.
- 0 Enter the files name to delete (wild cards are supported).

### 11.3 Choosing a reduced data file name

There are two places in the package where the Reduced data file name can be set. If the procedures approach to Repeated analysis is used, an option on the Repeated menu allows for the selection of a Reduced data file name. This option on the Files menu, allows the choice of a reduced data file name for all other situation, notably for single measurements. To select a reduced data file name, type:

- o **Reduced data file.**
- o Enter the desired file name. It is not necessary to enter the .RED extension. If a sub directory other than the current one is desired, it to can be entered with the file name. It is important to note though that all data files for the current session, including method files (.MET), must be saved or retrieved from the same data subdirectory.

When the Reduced data file name is altered, it is assumed that the profile file root should also be altered. The option to change this file name is automatically entered with the same filename root but different extensions (see Profile file in the next section for details).



**NOTE :** *When the reduced data file name is changed, the experiment counter is reset to 1. Remember, the experiment number is used as the extension for profile and abridged profile file names. If you select a reduced data file which already exists, the new data will be appended. However, existing profile files could be overwritten.*

## 11.4 Choosing a Profile file name

This option allows you to choose the root for future profile data and abridged profile data files. Remember, profile files have the same root file name with a numeric extension (between 1 and 999) which indicates the experiment number. Like the Reduced data file name, this file name can also be set on the Repeated menu. To set the profile file root on this menu, type:

- **Profile file.**
- Enter the file name with no extension. If a sub directory other than the current one is desired, it to can be entered with the file name. It is important to note though that all data files for the current session, including method files (.MET), must be saved or retrieved from the same data subdirectory.

## 11.5 Loading a profile file

Instead of using the pop up windows to peruse an experiment profile, it may be desirable to load a profile file onto the main peak profile screen. This option allows you to do that. To load a profile file, ensure that the desired profile root has been selected using the Profile file name option. Then type:

- 0 Load profile file.
- 0 Enter the experiment number of the profile to be loaded.

To overlay retrieved profiles, first enable profile overlaying by pressing **Alt-O**. The border of the profile box will change colour to light green. Then load the profile files as described above.



**WARNING! Where there are more than 999 experiments in a procedure, profile files will begin to overwrite earlier profile files for experiments >999. Also if Directive 254 is used, the experiment counter is reset to 1 when the programme re-starts. If profile saving is selected, earlier profile data will be overwritten. Under these situations, it is wise to disable profile or abridged profile saving.**

## 11.6 Save a profile

It is often desirable to only save the reduced data in the Reduced data file. This option allows you to toggle between Enabling profile saving and Disabling profile saving. You can also select whether you want to save abridged data files in this menu option. An abridged profile data file contains only the response data and is useful for importing response profiles into third party data manipulation packages. The file name of abridged profiles is given by **A<profile file root>.<expt no>**.

The present status of these selections is displayed on the Note pad page. When you enable profile saving, the current profile will be saved. This means that if you have had profile saving disabled and you obtain a profile which you want to keep, you can save it with this option. To switch this option, type:

- 0 **S**ave profile. A comment on the second line of the display will alert you to whether profile saving has been enabled or disabled.
- 0 Answer **Y**es or **N**o to the question of whether you want to save abridged profile data files
- 0 Answer **Y**es or **N**o to the question of whether you want to save profile data files

## 11.7 Data files

This section outlines the structure and composition of the data files used by FlowTEK. All data files are stored in ASCII format. The configuration file, FT.CFG, and the device configuration file, DEV.CFG, must be in the same subdirectory as the program. All other data files may be placed in any user-specified subdirectory.



**NOTE :** *You must keep all data files pertaining to one experiment in the same subdirectory.*

The Install program will place the program and configuration files and a few other necessary files in a subdirectory called \FlowTEK. All the data files will be placed in a subdirectory of \FlowTEK called \FlowTEK\DATA.

The format of each of the data files is given below, together with the default data for each file.

Table III : The configuration file (FT.CFG)

Data	Description
C:\FlowTEK\DATA\	Path
DEFAULT.RED	Reduced data file name
DEFAULT.MET	Method file name
DEFAULT.CAL	Calibration file name
DEFAULT 1 1	Profile root, save profile, save abridged profile
DEFAULT.PDR	Main procedure file name
80 1 -0.005 1.0	Detector display mode, re-scale y-axis, ymin, ymax
1	No. of detectors
0 60.0 0.0 0.0 0.0 69 0.0 78 0	min integration limit, max. integration limit, width height, peak time, time auto zero, mode auto zero, auto zero offset
0 0	value for startup of the digital output points
0 0 0 0 0 0 0 0 0	Values for each of the function keys
10	Allowable % deviation from setpoint before Warning
15	Allowable % deviation from setpoint before Alarm
5	Allowable % deviation of check standard
50	Permissible noise in detector channel (mV)
100.0	Max. permissible change in Analog output voltage (mV)
0.0	Setpoint concentration
0.0	Check Standard concentration
RECALIB.PDR	Recalibration procedure file name
0	Printer Port (0 - disabled)
0XE3	Serial Port configuration string

Table IV : The reduced data file (DEFAULT.RED)

Description
Date, Time, Peak height, Peak time, Peak area, Peak width, Width height, Concentration, Profile name, Method name
.....Repeated for each detector
.....Repeated for each experiment



Table V : The device configuration file (DEV.CFG)

Data	Description
AP	Device label
3	no of actions
FWD 1	action narration, digital output
REV 2	action narration, digital output
OFF 0	action narration, digital output
FRO	hot keys
3	bit mask
0.0	pulse length
.....REPEATED for each device	

Table VI : The method file (DEFAULT.MET)

Data	Description
0.0 60.0	time data-start, experiment time
0.0 60.0	time axis min, time axis max.
2	no of devices
6 0	ptr to dev defn 1, digital pt
3 2	ptr to dev defn 2, digital pt
2	no of events
0.0 0 79 0	time, ptr to device table, event, output
0.0 1 76 8	time, ptr to device table, event, output
1	no of detectors
0.0 60.0 1.0 0.0 0.0 69 0.0 78 0	min integration limit, max. integration limit, width height, peak time, time auto zero, mode auto zero, auto zero offset, signal function, detector channel
65 0	inject mode, inject switch

Table VII : The procedure file (DEFAULT.PDR)

Data	Description
c:\flowtek\data\default.met 10 .... Repeated for each file in the procedure	Method/Procedure/Wait file, no of repeats

Table VIII : The profile file (DEFAULT.001)

Data	Description
Method file data as per .MET -0.01 0.005 136 The raw profile data	Resp min, Resp max. No of data pts

Table IX : The calibration file (DEFAULT.CAL)

Data	Description
1	Detector Number
1 4	First and Last standard numbers
72 76	Regression mode, Regression model
0.0 1.0 0.0 0.0	Regression coef
1.0	Correlation coef
MG/L	Units
1.0 3 1.0 1.0	Std conc, no of replicates, replicate response, mean response
.... Repeated for each standard	

## 12. The interface board

The PC-30B, ADA2200 (or ADA2210), and DM5406 (or DM406) boards are high accuracy Analog and digital input/output (I/O) boards. These boards can be plugged into any of the fully bussed slots in an IBM or compatible computer or in the case of the DM5406 onto a PC/104 format stack. The boards have been designed as versatile general purpose boards with several user-configurable options. The board supplied with your package, has been configured to operate with FlowTEK. For this reason, dip switches and jumpers on the board should not be changed. Table X to Table XV give a list of the settings for the dip switches and jumpers on the board. In the case of the PC30B, the address of the board is 0x700 for the other two boards, the address is 0x300. This may clash with other cards installed in your computer. If this occurs, you must remove the other card or reconfigure it at another address.

Table X : PC-30B Dip Switch settings

Switch	1	2	3	4	5	6	7	8
Setting	on	on	off	off	off	on	on	on

Table XI : PC-30B Jumper settings

Jumpers in	W2	W3	W6	W7	W10	W11	W13	W17
	W18	W21	W23	W24	W27	W29	W30	W52
Jumpers out	W1	W4	W5	W8	W9	W12	W14	W15
	W16	W19	W20	W22	W25	W26	W28	W31
	W32	W33	W53	W54				

Table XII : ADA2200 Dip Switch settings

Switch	1	2	3	4	5
Setting	off	off	off	off	on

Table XIII : ADA2200 Jumper settings

P3	10 volts
P4	+ (unipolar)
P5	DRQ1
P6	DACK1
P7	XTAL, OT1, and PCK
P8	OT2, IRQ6 -> IRQ7, G
P9	5 and x2
P10	5 and x2
P11	Single ended
P12	EOC
P13	+

Table XIV : DM5406 Dip Switch settings

Switch	1	2	3	4	5
Setting	off	off	off	off	on

Table XV : DM5406 Jumper settings

P3	OSC, OT1, PCK, TRIG, 5V
P4	IRQ2->IRQ3, G
P5	DACK1, DRQ1
P6	UNI, 10V
P7	S, S, S
P8	10
P9	10



**WARNING!** Changing the address on the dip switches of the interface card will render the software useless.

## 13. The Distribution board

The distribution board enables convenient connection of the devices to the interface board and ensures that the correct connections on the interface board are used. It also provides certain optional features such as an amplifier and filter for the detectors connected to Analog input point 1 and 2. The gain (amplification), offset (baseline), and level of filtering can be independently set for each amplifier. The digital input signals accept both TTL and switch logic. In addition, the 8 digital outputs (1 to 8) can be configured for either TTL or switch logic to match the control logic of the devices connected to them. (Digital outputs 9 to 12 are configured for TTL output but are not routed through to terminal points).

Refer to sections 1.6 and 1.7 when connecting devices and detectors to the distribution board. Figure 9 gives the lay out of the various I/O points and amplifier and filter switches on the board.

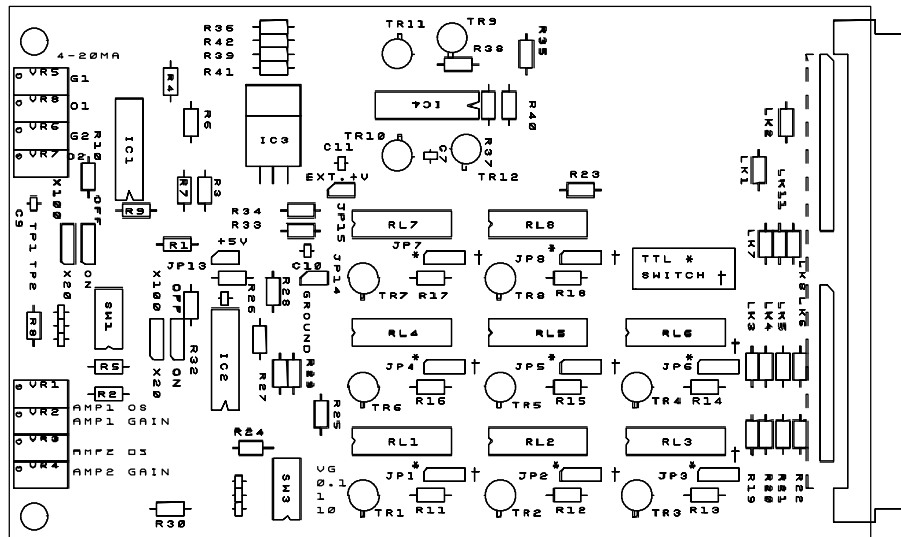


Figure 9

Figure 9 : Schematic diagram of the Mintek Distribution board



**WARNING! : The supplied  $\pm 12$  volt power point can draw a maximum current of 100 mA. A larger current could damage the interface board.**

## 13.1 Using the amplifier

The Mintek Distribution board offers the option of amplifying the incoming signal from Analog input channel 1 and 2. This signal can also be subjected to various degrees of electronic filtering. The default settings are unity amplification with no electronic filtering. The following description explains how to set up the amplifier or enable signal filtering.

To set up the two amplifier circuits:

- o Establish whether you need amplification by determining the output range of the detector. The default input range for FlowTEK is 0 to 10V
- o To enable the amplifier insert the jumper at the relevant channel jumper selection to the ON side. Note there is a jumper for channel 1 and another one for channel 2. The channels are labelled on the board. In the OFF position all the features of the amplifier are disabled.
- o Use OS1 and OS2 to adjust the Offset. To do this connect a power source or even the detector to the relevant input channel and provide 0V or set the baseline of the detector. Measure the obtained input at test point TP1 or TP2 depending on the input channel. Offsets of  $\pm 10$  V can be trimmed out. The factory setting is 0V corresponding to no offset.
- o Decide on the required gain. A pre-set gain of 20 is selected (i.e. for detectors with an output range of 0 to 500 mV), with the x20 jumper. If the x100 jumper is inserted, a gain of 100 is selected (i.e. for detectors with an output range of 0 to 100 mV). For fixed gain settings VG for the relevant channel must be off!
- o For detectors with an output range requiring amplification other than these two fixed levels, the variable amplifier option can be used. The variable option allows the adjustment of the gain between 1 and 20 or between 20 and 100 times. To use the variable gain amplifier set VG to the ON position and select the desired maximum using the x20 / x100 jumper settings.
- o Use G1 or G2 to adjusted the Gain for channel 1 or 2 respectively. Note if VG is off adjusting the gain will not affect the achievable gain. When variable gain is ON set up the gain by supplying another voltage (usually near full scale) and then adjust the G1 or G2 pot to give the correct voltage at the relevant test point TP1 or TP2.

Switches 10, 1, 0.1 determine the level of electronic filtering applied to the incoming signal. The labels indicate the time constant (in seconds) applied. Switching on more than one of these filter selection switches will provide a time constant corresponding to the value of the highest switch.

## 13.2 Configuring the digital output points

The first eight digital outputs (1 to 8) can be configured to interface TTL (5V) or switch controlled devices. The remaining digital output points (i.e. from 9 to 12) are configured for TTL output but are not wired through to the terminals.

The lower eight digital outputs (1 to 8) can be configured to provide either TTL (0 to 5V) or switch (On or Off) signals. The mode of operation is selected using the jumper at each circuit board relay. Placing the jumper in the † position configures the output for switch control. Placing it in the \* position configures it for TTL control. Output points are numbered on the board.

## 14. Customer support

Mintek or its appointed dealers will gladly assist you to install your system and get it operating to pre-defined specifications. The fee for this service depends on the circumstances surrounding the installation. Advice on components and suitable devices is also provided.

Mintek began its research effort into flow-injection analysis soon after Ruzicka and Hansen defined the concept in 1975. Staff from Mintek's Process Analytical Science Group were involved in the original work which led to the emergence of Sequential-injection Analysis. For more than a decade, Mintek staff have been involved in

- o extensive method development (many of these methods are in routine use in this and other laboratories),
- o evaluation studies of components, devices, and accessories used in flow-based techniques,
- o the development of certain components, devices, and accessories, and
- o training in the fundamentals and implementation of flow-injection analysis and other flow based methodologies.

Mintek therefore has at its disposal extensive applications development facilities. Staff are also willing to discuss your application and provide consulting services or full method development facilities to you.

A hot-line to the software author and several flow-injection authorities is provided and may be used by registered owners of the FlowTEK package. Details are provided on your registration card. The telephone number to call in South Africa is (011)-709-4111 or (27-11-709-4111 for international calls). This advice is a free service, but assumes that no development work is required. Development work can be undertaken at a negotiated fee.

Like all software packages, it is expected that FlowTEK will continue to develop. The features included in future versions will reflect, to a large extent, the feed-back we get from users of the software. You are therefore encouraged to make your ideas for enhancements to the package known to us.



## 15. Warning and Error messages

### 15.1 Warnings

#### ***BEEPS***

As a rule, a BEEP by the computer indicates some undesirable condition and needs your attention. The only exception to this is the end of a procedure which is signalled by a BEEP as well.

#### ***Data entry warnings***

The data input routines used in FlowTEK have been specifically designed to minimise errors through invalid data entry. For this reason, you may obtain a message giving the valid range of data values that you may enter. These warnings are cleared from the screen by pressing **Enter** and then entering a valid response. If you want to back out of a data entry routine just press **ESC**.

#### ***File name entry warnings***

When a file name is entered, the context of the request for a file name is determined by FlowTEK and a logical deduction of whether you intend to SAVE data to the file or RETRIEVE data from it. FlowTEK then examines the storage media and determines whether the file already exists. If the file exists and you want to Save data to the file, FlowTEK warns you that you may *overwrite* (or in the case of a reduced data file, *append* to) existing data. You then have the opportunity to continue or enter a new file name. If the file does not exist and you want to Retrieve data from it, FlowTEK warns you that the *File does not exist*. You must then retype a valid file name. In this case, ensure that you are specifying the correct path. Remember, once you use a path, it becomes the default for future files.

#### ***No unique solution in a calibration***

If there are insufficient data for the chosen regression model, or the supplied data do not provide a unique solution to the regression analysis, a *No unique solution* warning will be displayed and concentration data will not be calculated.

#### ***Interface board not present***

Many of the functions in the FlowTEK package are not dependent on the interface board or distribution board. For example, calibration, method development, and the design of procedures may be carried out without the board being present. All data acquisition or device control functions obviously need the board. In the event of poor contact between the board and the computer or the link between the interface board and the distribution board, a warning that the *Board is not present* will be displayed in the lower left corner of the main screen. If this happens, ensure that all connections are secure.

### ***MET or CAL file not present***

FlowTEK remembers the method and calibration file that you were using in your last session. When the program is entered, the last conditions used are loaded as a starting point for the new session. (Of course calibration data are deleted as it is unusual to use the same calibration data from one session to the next. If you do want to use them, you can load the calibration data using the Calibration, File, Retrieve option). If these files have been deleted in the interim, a warning to that effect will be displayed on the screen.

### ***Time out on Serial Port***

You have selected devices that make use of serial coms for control however, no serial devices are connected or there is trouble with the serial port. Alternatively, you have saved a serial output port >0 and you do not have any serial devices. To remedy this problem check your serial port. Ensure that the device is plugged into the correct serial port. If you are not using a serial controlled device use directive 32 to select a serial port with parameter 0.

### ***Printer not connected or off-line***

You have selected output to the printer but no printer is connected to the parallel port or the connected printer is not on-line. Rectify this problem by connecting a printer, putting it on-line or disabling printing with directive 33.

## 15.2 Errors

### ***Incompatible screen driver***

FlowTEK was written to operate with an EGA or VGA screen. This means that high resolution colour coded plots of the data acquisitions can be displayed on the screen. FlowTEK will not work with any other screen formats.

### ***DMA error***

This error is generated by the interface board and means that there has been some trouble with the acquisition of data. If this occurs, ensure that the output of detectors linked to the system are within the required ranges. Remember the interface board is configured for 0 to 10 V input. If you use channel 0 or 1, you can input a smaller voltage and amplify your signal with the amplifier on the distribution board. If you use this option, take into account the amplification factor that you are applying. If the error persists, there could be a fault on the interface board. This would necessitate replacement and repair of the interface board.

### ***Parity error***

A parity error is another error condition generated by the interface board. Check all connections to the board. Pay particular attention to the ground connections. If the problem persists, report it to Mintek.

### ***Trouble with device definition file***

If the device definition file (**DEV.CFG**) becomes corrupted or is inadvertently deleted, FlowTEK will not load. To rectify this problem, re-install the package from the floppy disk.

# 16. Appendices

## 16.1 Device Description Data Sheet

Device No.				
Device Name				
No. of actions				
Action narration 1				
Digital output 1				
Hotkey 1				
Action narration 2				
Digital output 2				
Hotkey 2				
Action narration 3				
Digital output 3				
Hotkey 3				
Pulse length				

Method file name :

Date :

No of devices  Time start data  Expt. time

Device No	1		2		3		4	
	Typ	Pt	Typ	Pt	Typ	Pt	Typ	Pt

Device No	5		6		7		8	
	Typ	Pt	Typ	Pt	Typ	Pt	Typ	Pt

## 16.2 Detector Description Data Sheet

Date :

No of detectors  Page or Overlay plots

Output point	1	2	3	4
Description				
Output range				
Response range				
Signal Transform				
AutoZero mode				
AutoZero time				
Int limit min				
Int limit max.				
Peak time				
Width Height				



## 16.4 Directives

Dir	Description	Parm	Description	Def
32	Additional diagnostic data	0	No	0
		1	Yes	
33	Select printer on	0	Printer Off	0
		1	Printer On	
34	Select COM port and baud rate	0	Disable Serial COMS	1 & 96
		1	COM1	
		2	COM2	
		3	COM3	
		4	COM4	
		12	1200 baud	
		24	2400 baud	
		96	9600 baud	
35	Check Correlation coef	0	Accept if $r^2 > 0.0$	0
		1	Accept if $r^2 > 0.98$	
		2	Accept if $r^2 > 0.99$	
		3	Accept if $r^2 > 0.999$	
36	Select detector channel	1-4	Channel Number	1
37	Follow peak or valley	0	Valley	1
		1	Peak	
38	Concentration data format	0-4	No. of decimal places	3
39	Time to start measurement	0-999	Time in minutes	0
40	Time to start measurement	0-999	Time in seconds	0
41	Time for AutoZero	0-999	Time in seconds	0
42	Analog output based on the Set Point concentration	1-2	Analog output channel	1
43	Analog output to channel 1	0-100	Voltage output * 10	0
44	Analog output to channel 2	0-100	Voltage output * 10	0
45	Analog output based on response	1-2	Analog output channel	1
46	Compare to Setpoint - Warn			
47	Compare to setpoint - Alarm			

Dir	Description	Parm	Description	Def
48	Compare to Check std			
49	Allocate standard response	1-16	Std number	1
50	Clear calibration response table			
51	Select First Standard	1-16	First Std number	1
52	Select Last Standard	1-16	Last Std number	3
53	Regression Model	1	Linear	1
		2	Quadratic	
		3	Trinomial	
		4	Rectangular hyperbola	
		5	Exponential	
54	Wait for x min	0-999	No of minutes	0
55	Wait for x seconds	0-999	No of seconds	0
56	Wait for digital input High	1-4	Input point	1
57	Wait for digital input Low	1-4	Input point	1
58	Wait for keypad input	0-9	Key on keyboard	0
59	Digital output to port 1	0-255	Value to output	0
60	Digital output to port 2	0-255	Value to output	0
61	Digital output to port 3	0-255	Value to output	0
62	Select acceptable baseline noise level	0-1000	Noise (in mV) 0 disable checking	0
63	Select amount of output damping	0-10000	Maximum change in voltage Damping (mV) 0 disables damping	0
64	Calculate and print the last response			
70	Select Instrument Diagnostics	0	Disable diagnostics	0
		1	Enable diagnostics	
72	Generate a Warning if Digital Input High	0-4	Digital Input Point	0
73	Generate a Warning if Digital Input Low	0-4	Digital Input Point	0



Dir	Description	Parm	Description	Def
74	Generate a Alarm if Digital Input High	0-4	Digital Input Point	0
75	Generate a Alarm if Digital Input Low	0-4	Digital Input Point	0
76	Generate a Warning if last Peak height drops below specified limit	0-10000	Peak height in mV	0
80	Perform a Procedure or Method based on Digital Input	0-999	Procedure Number	0
254	Reset Micro			

