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# Lambda 2

## UV/VIS Spectrometer

### Operator's Manual

B 2131 – A 2.2 – M 1261 / 4.89 Printed in Federal Republic of Germany 6018-2308

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PERKIN ELMER

Bodenseewerk
Perkin-Elmer & Co GmbH

D-7770 Überlingen

# L A M B D A 2

A UV/VIS Spectrometer
For Routine Analysis Applications

## OPERATOR'S MANUAL

Valid for Software Version 3.x

Publication: B2131
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Part Number: B018-2308

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Technical Documentation,
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# ATTENTION

BEFORE USING THIS INSTRUMENT IT IS ESSENTIAL TO READ THE MANUAL CAREFULLY AND TO PAY PARTICULAR ATTENTION TO ANY ADVICE IT CONTAINS CONCERNING POTENTIAL HAZARDS THAT MAY ARISE FROM THE USE OF THE INSTRUMENT.

This advice is intended to supplement, not supersede, the normal safety code of behavior prevailing in the user's country.

## ELECTRICITY

If any part of the instrument is not installed by a Perkin-Elmer Service Representative, ensure that the line power plug is wired correctly:

| | Cable Lead Colors | |
|---|---|---|
| | International | USA |
| Live terminal | Brown | Black |
| Neutral terminal | Blue | White |
| Earth-grounded terminal | Green/Yellow | Green |

To ensure satisfactory and safe operation of the instrument it is essential that the green/yellow lead of the line power cable is connected to true electrical earth.

Even with the POWER switch off line power voltages can still be present within the instrument; the instrument must therefore be unplugged at the line power supply before any work is undertaken inside it.

Servicing should be carried out only by a Perkin-Elmer Service Representative or similarly authorized person.

WARNING: This instrument is not designed for operation in an explosive atmosphere.



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# SAFETY PRECAUTIONS

This instrument has been designed and tested in accordance with Perkin-Elmer Specifications and IEC 348 'Safety Requirements for Electronic Measuring Apparatus'. This instrument is protected in accordance with IEC Class 1 rating. This manual contains information and warnings that must be followed by the user to ensure safe operation and to maintain the instrument in a safe condition.

The instruments have been designed for indoor use and will operate correctly under the following conditions:

| | |
|---|---|
| Temperature | 15 °C to 35 °C |
| Relative Humidity | 75% maximum |

The instrument should be connected to a power supply line that includes a switch or other adequate means of disconnection from the electrical supply. The instrument power plug shall only be inserted into a socket outlet provided with a protective earth connection.

WARNING: Any interruption of the protective conductor inside or outside the instrument or disconnection of the protective ground terminal is likely to make the instrument dangerous. Intentional interruption is prohibited.

When the instrument is connected to its supply, terminals may be live, and the opening of covers or removal of parts (except those to which access can be gained by hand) is likely to expose live parts.

The instrument shall be disconnected from all voltage sources before it is opened for any adjustment, replacement, maintenance or repair.

Capacitors inside the instrument may still be charged even if the instrument has been disconnected from all voltage sources.

Any adjustment, maintenance or repair of the opened operating instrument shall be avoided as far as possible, and, if inevitable, shall be carried out only by a skilled person who is aware of the hazard involved.

Only fuses with the required current rating and of the specified type are to be used for replacement. The use of makeshift fuses and the short-circuiting of fuse holders is prohibited.

Whenever it is likely that the protection has been impaired, the instrument shall be made inoperative and secured against any unauthorized operation.

The protection is likely to be impaired if, for example, the instrument:

a) shows visible damage;
b) fails to perform the intended measurements;
c) has been subjected to prolonged storage under unfavorable conditions;
d) has been subjected to severe transport stresses.

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# 1. INTRODUCTION

## 1.1 BRIEF APPARATUS DESCRIPTION

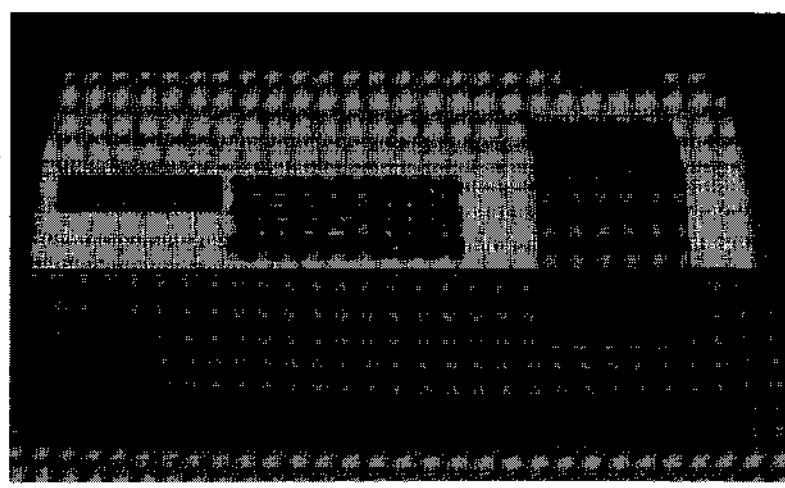


**Figure 1-1. Lambda 2 UV/VIS Spectrometer**

Lambda 2 is a versatile instrument for routine and automated analysis.
Design features making Lambda 2 ideal for routine analysis applications are:

* Double beam optics for excellent long term stability, reference compensation and baseline correction.

* Basic methods for timedrive, scan, wavelength program and concentration. Up to 20 ready-to-run methods can be stored.

* Cassette storage for preprogrammed methods, allowing transfer from instrument to instrument, lab to lab.

* A wide wavelength range: 190 nm to 1100 nm.

* Scan speeds from 7.5 nm/min to 2880 nm/min.

* Simple keyboard layout for ease of use. HELP prompt.

* Security of operation, allowing some instrument functions to be disabled to prevent misuse.

* Connection to PC operating with PECSS software to give total system flexibility, and allowing for future expansion.

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## 1.2 SCOPE OF THIS MANUAL

This manual is intended to help the user in getting to know the Lambda 2. After reading it he/she should be able to:

* Make full use of the analytical possibilities offered by the instrument.

* Solve quickly and surely any problems that may arise in the future.

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# 2. INSTALLATION

## 2.1 EQUIPMENT PROVIDED

| | | PART NUMBER |
|---|---|---|
| **LAMBDA 2** | Lambda 2 Spectrometer | B016-6351 |
| | **Either** | |
| | Standard EX-800 printer * | B017-0717 |
| | Cable: Lambda 2 to printer | B015-3907 |
| | **Or:** | |
| | EX-800 with RS-232-C interface * | B016-8281 |
| | Cable: Lambda 2 to printer | B016-6569 |
| **LAMBDA 2/PC** | Lambda 2 Spectrometer | B016-6351 |
| | Cable: Lambda 2 to PC | B018-0242 |
| | Cable: PC to EX-800 printer | B016-9358 |
| | Epson PCe * | B017-0885 |
| | PECSS software package * | B016-2859 |

* These items are not delivered with all instrument versions.

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## 2.2 UNPACKING AND INSPECTION

Unpack the components carefully. Keep the packing materials for possible future shipping or storage.
Examine the components for any signs of damage in shipment. In the event of damage, file an immediate claim with the authorized carrier, and inform your local Perkin-Elmer office or representative.

After the instrument has been unpacked check the exterior and interior for possible damage as follows:

* Check the entire outer cabinet for damage.
  Check that terminals, fuse holders etc. are not damaged.

* Open the sample compartment cover, checking that it moves freely without binding. The sample compartment must be free from dust and other foreign material.
  Close the sample compartment cover.

* Remove the top cover securing screws at each end of the spectrometer and open the top cover by lifting it at the front edge and swinging it fully back.

  Check the inside of the instrument to see that source lamps have not been damaged and that there are no loose components.

  Close the top cover and replace the two securing screws.

**INSTALLATION** The Lambda 2 is a very straightforward instrument to install and the user should be able to install it himself.
A suitable working area should be prepared for the instrument before proceeding with installation. Details concerning the working area and installation procedures are given in the following section.
Should the user have any questions concerning the installation of the instrument he should not hesitate to contact his local Perkin-Elmer office.

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## 2.3 LABORATORY ENVIRONMENT

The following recommendations should be adhered to as closely as possible to attain maximum instrument stability and minimum maintenance.
The laboratory atmosphere should be free from dust, smoke and corrosive fumes. The instrument should not be operated in an explosive atmosphere.

**TEMPERATURE** Ambient temperature should lie between 15 °C and 30 °C. Avoid significant temperature fluctuations.

**HUMIDITY** Ambient relative humidity should lie between 10% - 75% without condensation.

**LIGHTING** The instrument should not stand in direct sunlight. Good illumination with diffuse light is ideal.

**DIMENSIONS** 650 mm x 233 mm x 560 mm W x H x D .

Sufficient room should be left at the back of the instrument to allow sufficient air circulation.

**WEIGHT** 26 kg. The instrument should be placed on a sturdy bench.

**POWER** Any voltage between 100 and 240 V, 50/60 Hz; instrument automatically adjusts to prevailing voltage.

FUSES 200 - 240 V operation requires one 2.5 ampere fuse.
100 - 120 V operation requires one 4.0 ampere fuse.

**CONFIGURATION**



**Figure 2-1. Lambda 2 Spectrometer with Epson EX-800 Printer**

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## 2.4 PRINTER CONNECTION

**GENERAL**

For output of alphanumeric and graphical results the spectrometer has been designed to operate with an Epson EX-800 printer.
For faster and more efficient operation the printer can be fitted with an RS-232-C interface which has a memory buffer.

Procedures for setting up the printer, loading paper, etc. are presented in the User's Guide provided with the printer. Please refer to that guide when operating the printer.

**CONNECTING THE PRINTER**

The printer is connected to the spectrometer with the cable provided.

* Plug the 6 pin DIN plug into the socket on the rear panel of the printer.
* Plug the 25 pin plug into the socket on the right hand panel of the spectrometer.

**PRINTER + INTERFACE**

- If the printer is fitted with an optional RS-232-C interface, the 6 pin DIN socket is redundant.
- The spectrometer must be connected to the printer via the 25 pin cannon socket on the interface board. This is located at the rear of the printer.
- If the printer is ordered complete with the RS-232-C interface, the correct cable for connecting the Lambda 2 to the printer is supplied with the instrument.

**SETTING PRINTER DIP SWITCHES**

**WARNING:**

Electronic components can be damaged by static discharges. Before changing the DIP switch settings, make sure that you are not statically charged by grasping a water pipe or similar. Disconnect the printer from the electrical supply. Apart from the DIP switches do not touch any other electrical components in the printer.

The factory DIP switch settings in the printer correspond to the operating protocol of the spectrometer software. Hence, if the printer is delivered with the instrument, no changes to the settings should be necessary. However, if the printer has been used elsewhere beforehand, then the DIP switches settings may have been altered. DIP switches to control the printer are found at the back of the printer and are easily accessible. DIP switches controlling the optional RS-232-C serial interface are found on the interface circuit board, which is only accessible if the printer housing is removed. To locate the DIP switches and to remove the printer housing, follow the instructions in the printer user's guide.

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## 2.4.1. PRINTER DIP SWITCHES

The two banks of DIP switches are found on the back panel of the printer and are easily accessible.
The settings enabling communication between spectrometer and printer are as follows:

(These settings are valid only for the standard printer not fitted with the optional RS-232-C interface).

| SWITCH | SETTING | SIGNIFICANCE |
|---|---|---|
| SW1-1 | DOWN | Normal characters, 10 characters/inch |
| SW1-2 | DOWN | Zero not slashed. |
| SW1-3 | DOWN | Typestyle. |
| SW1-4 | DOWN | ESC/P control codes used. |
| SW1-5 | DOWN | Normal Typestyle. |
| SW1-6 | UP | |
| SW1-7 | UP | US English Character Set. |
| SW1-8 | UP | |
| SW2-1 | UP | 12 inch page length. |
| SW2-2 | DOWN | Cut sheet feeder disabled. |
| SW2-3 | DOWN | Formfeed sent by spectrometer. |
| SW2-4 | DOWN | Linefeed sent by spectrometer. |
| SW2-5 | UP | Built in serial interface, |
| SW2-6 | UP | no parity. |
| SW2-7 | DOWN | Baud rate 9600. |
| SW2-8 | DOWN | |

**NOTE:** DIP switch settings are only checked by the printer at power on. Hence, turn the instrument off when changing settings.

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**NOTE:** This section only applies to printers fitted with the optional RS-232-C interface.

## 2.4.2. PRINTER DIP SWITCHES

The DIP switch settings at the back of the printer must be set as follows:

| SWITCH | SETTING | SIGNIFICANCE |
|---|---|---|
| SW1-1 | DOWN | Normal characters, 10 characters/inch |
| SW1-2 | DOWN | Zero not slashed. |
| SW1-3 | DOWN | Typestyle. |
| SW1-4 | DOWN | ESC/P control codes used. |
| SW1-5 | DOWN | Normal Typestyle. |
| SW1-6 | UP | |
| SW1-7 | UP | US English Character Set. |
| SW1-8 | UP | |
| SW2-1 | UP | 12 inch page length. |
| SW2-2 | DOWN | Cut sheet feeder disabled. |
| SW2-3 | DOWN | Formfeed sent by spectrometer. |
| SW2-4 | DOWN | Linefeed sent by spectrometer. |
| SW2-5 | DOWN | Any option interface parity. |
| SW2-6 | DOWN | |
| SW2-7 | DOWN | Baud rate 9600. |
| SW2-8 | DOWN | |

**NOTE:** DIP switch settings are only checked by the printer at power on. Hence, turn the printer off when changing settings.

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**NOTE:** This section only applies to printers fitted with the optional RS-232-C interface.

## 2.4.2. INTERFACE DIP SWITCHES

The serial interface in the printer can be identified as follows:
The number 8148 is printed on the board.
Two banks of DIP switches are found on the circuit board:
SW1 with 8 switches, SW2 with 6 switches.

| SWITCH | SETTING | SIGNIFICANCE |
|---|---|---|
| SW1-1 | OFF | 8-bit word length. |
| SW1-2 | OFF | Parity check disabled. |
| SW1-3 | OFF | Parity selection. |
| SW1-4 | OFF | Flag selection. |
| SW1-5 | OFF | |
| SW1-6 | ON | Baud rate 9600. |
| SW1-7 | OFF | |
| SW1-8 | OFF | |
| SW2-1 | ON | I/F Board enable. |
| SW2-2 | ON | Buffer enable. |
| SW2-3 | OFF | Flag reset timing. |
| SW2-4 | OFF | |
| SW2-5 | OFF | Self test disable. |
| SW2-6 | OFF | Self test mode selection. |

**NOTE:** If the EX-800 printer has been used with the Perkin-Elmer Lambda 15 Spectrometer, then only one change on the interface DIP switch settings is necessary:

For use with Lambda 15 switch SW1-4 is ON. Switch this to OFF for operation with the Lambda 2.

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## 2.5 STANDARD CELL HOLDER

The standard cell holder accommodates a large selection of rectangular cells up to 12.4 mm x 12.4 mm square (outside dimensions). Spacers for short pathlength cells are available to extend the applications of the holder.
The holder is both vertically and horizontally adjustable to ensure perfect alignment of the cell sample area in the radiation beam.
An integral lifter can be used to raise short cells.
Two standard cell holders are provided with the spectrometer.

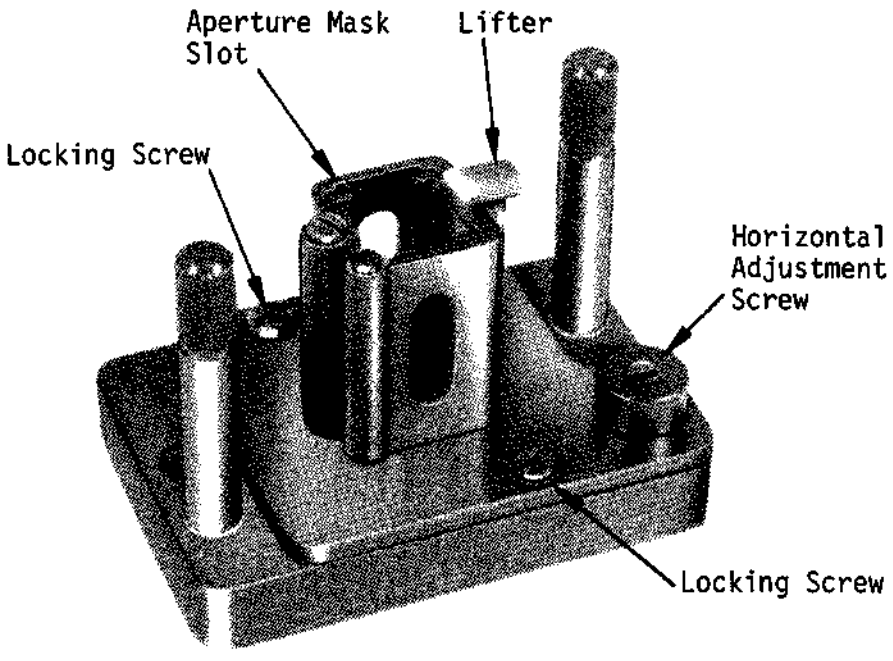



**Figure 2-2. Standard Cell Holder**

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## 2.5.1. CELL HOLDER INSTALLATION

The cell holder is installed as follows:

1) Orientate the holder so that the lifter is toward the rear of the sample compartment.

2) Lower the holder so that the two alignment holes slip over the two studs on the baseplate at the bottom of the sample compartment.

3) Move the milled posts a little to locate the threaded holes in the baseplate, and then tighten the milled posts.

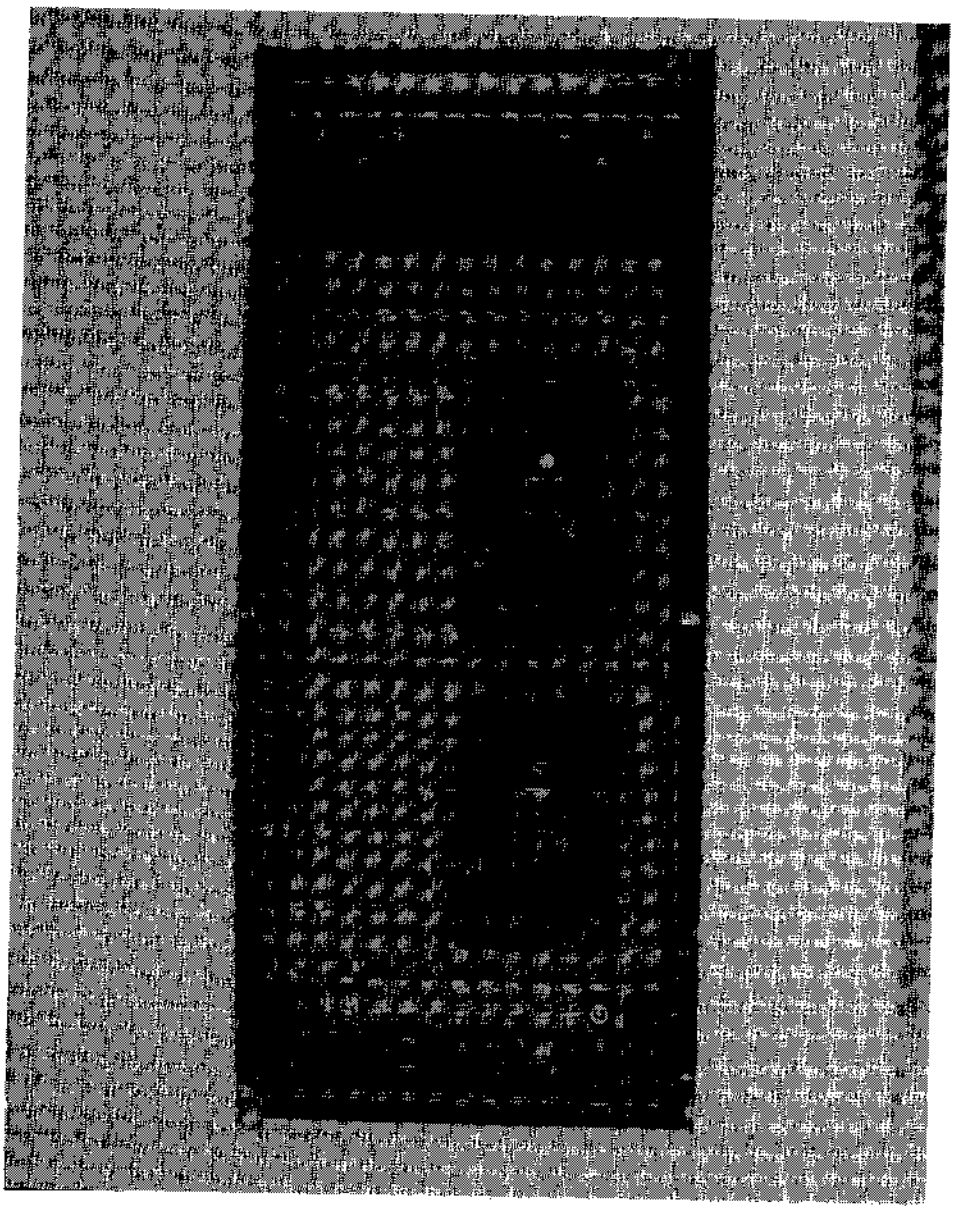


**Figure 2-3. Sample Compartment**

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## 2.5.2. STANDARD RECTANGULAR CELLS

The standard cell holder accommodates rectangular cells up to 12.4 mm x 12.4 mm square (10 mm optical pathlength). No horizontal alignment of the cell in the radiation beam is necessary.

The height of the liquid in the cell, from the cell base to the meniscus, should not be less than about 25 mm. If it is less than this, then the cell must be raised by means of the vertical lifter, or a microcell must be used.

Standard rectangular cells with pathlengths of less than 10 mm may be accommodated by using appropriate spacers.

Procedures for aligning microcells when using the standard cell holder are given in section 7.3

## STOPPERED CELLS

When using stoppered cells, a build up of pressure can cause the cells to split.
To avoid this:

* only fill the cell sufficiently for the radiation beam to pass through the solution. The air space above the liquid should be adequate to compensate for presssure changes due to evaporation and temperature changes.
* If the cell must be filled completely with solution, do not press the stopper right in. Set the stopper lightly in the cell opening.
  If high temperature measurements are to be made, the use of stoppers with drilled capillaries is recommended.

## REMOVAL OF THE SAMPLE COMPARTMENT COVER

Some of the accessories require removal of the sample compartment cover.
This must be done carefully as follows:

1) Open the cover only about 30 degrees to provide clearance between the hinged end of the cover and the top of the spectrometer.

2) While holding the cover at the 30 degrees angle with one hand, use the other to grasp the the cover at its long side, near the hinge, and pull straight up to release one hinge.

3) Repeat the above step on the other side to release the cover from the other hinge.

The sample compartment cover, or other accessory cover, is installed by following the above procedure in reverse.

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## 2.5.3. ACCESSORIES

For accessory operation an accessory control board must be fitted. This should only be carried out by a Perkin-Elmer service representative, or similarly qualified person.

If installation of accessories requires the removal of the sample compartment baseplate, proceed as follows:

* Unscrew the 4 allen screws and lift out the baseplate, complete with cell holders.

* The accessory may now be installed by following the instructions in the manual supplied with the accessory.

* Electrical connection of the accessory to the instrument is made at the right hand side of the instrument where all the communication ports are found.
  The accessory ports are marked for each accessory.
  See Figure 2.4.

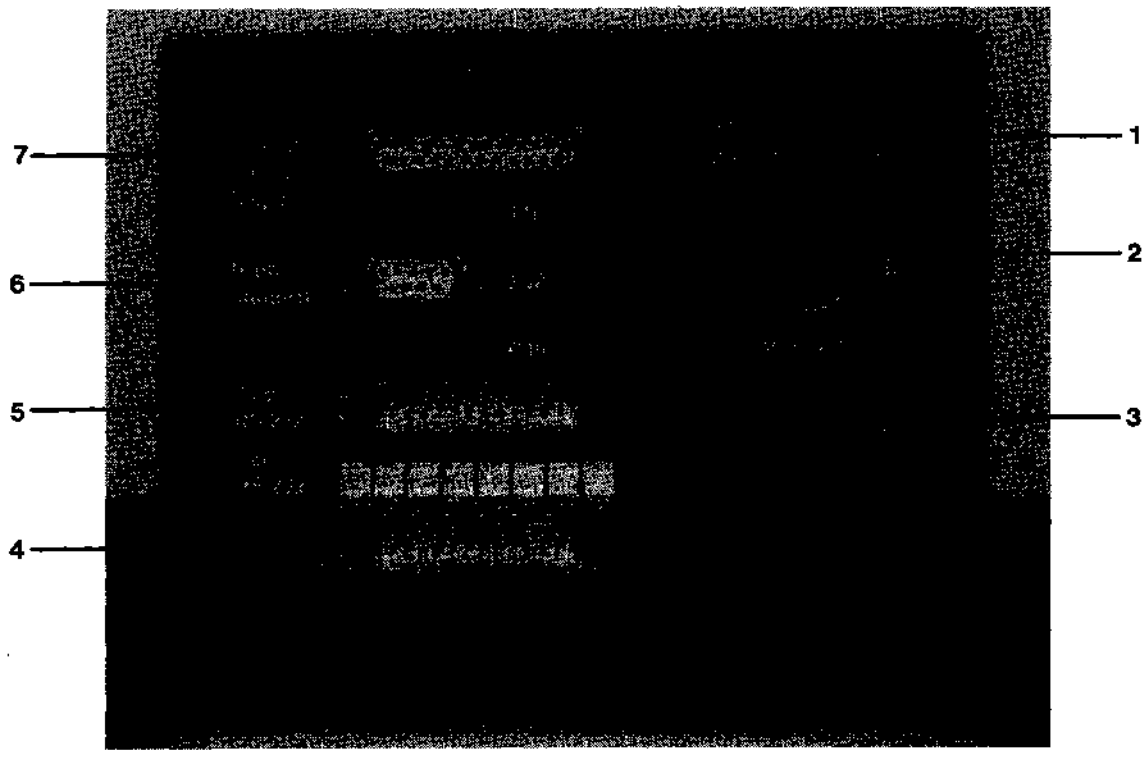
**NOTE:** Lambda 2, unlike older Lambda instruments has a 1 piece baseplate in the sample compartment. Because of this some accessories may have to be fitted slightly differently to the directions in the accessory manual.

**THERMOSTATTED SUPER SIPPER** (Part no. 126-830).
Remove the cell holder from the baseplate delivered with the accessory and mount it directly onto the Lambda 2 sample compartment baseplate.

**LC MICRO-FLOWCELL** (Part no. 110-662).
When using this accessory with the Lambda 2, the sample compartment baseplate small kit (180-210) is required.

**LIGHT PIPE ACCESSORY** When using this accessory with Lambda 2, the sample compartment baseplate small kit (180-210) is required.

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1. On/Off switch for overlay ouput with analog recorder
2. Connector for analog recorder control cable (J56)
3. Output for analog recorder
4. 1st RS-232 (standard)
5. 2nd RS-232 (P15)
6. Connector for multisampler (J48)
7. Connector for: (J14) cell changers, Single Sipper, Super Sipper, Econo Sipper

**Figure 2.4 Lambda 2 Terminal Panel for Connection of Accessories**

**NOTE:** In the standard version of the instrument with no accessory control board fitted, only one RS-232-C interface (here marked 1st RS-232-C) is found on the terminal panel. No accessory connectors are found on the standard instrument.

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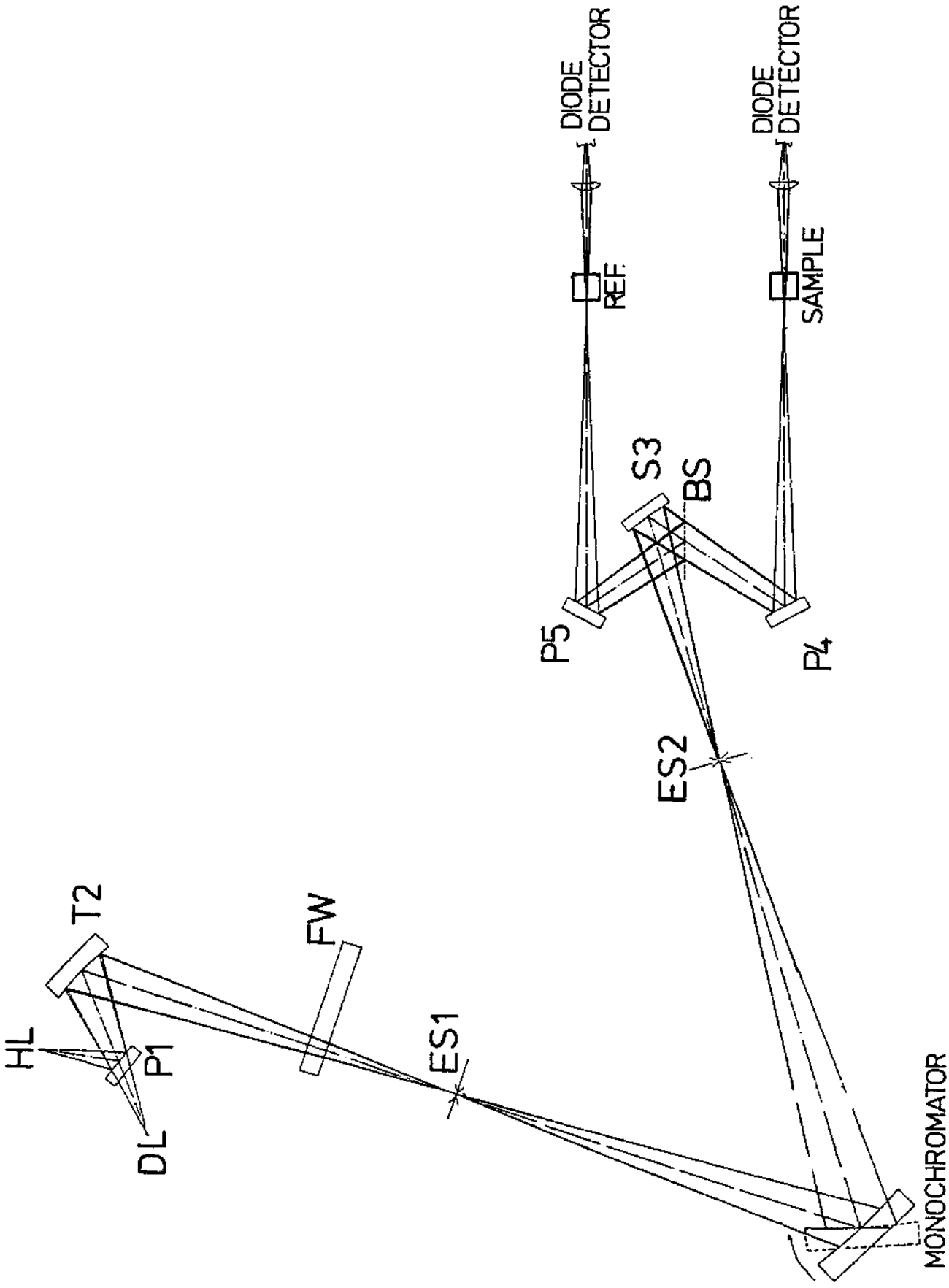
**WARNING:** **SUPER SIPPER CONNECTION:** if the super sipper has been used with other lambda instruments before, or has not been ordered together with Lambda 2, then an alteration must be made to the connecting cable, (part number B013-2227).
The connection to **PIN 14** of the 25 pin cannon socket must be interrupted, and the wire must be lead off to the cable shielding.
If this is not done, damage to the internal circuitry of the Lambda 2 can occur.
This work should be carried out by a suitably qualified person.

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# 3. PRINCIPLES OF OPERATION

This page has been left blank intentionally.
The instrument optical path and explanation are found on the following pages.

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**OPTICAL SYSTEM**

The optical path of the Lambda 2 is represented in Figure 3-1 on the facing page.

The monochromator is a concave holographic grating with 1053 lines/mm.

Planar mirror P1 is moved into position by a mechanical arm. When it is in position, light from the deuterium lamp DL, is blocked, and light from the halogen lamp HL, is reflected onto the toroidal mirror T2.
Source changes, due to the positioning of mirror P1, occur in synchronization with the monochromator, at a specified wavelength. The monochromator stops slewing until the source change is complete.

Radiation is focussed by T2, passing through the filter wheel (FW) onto the entry slit ES1.
The filter wheel rotates different optical filters into the radiation beam. It is synchronized with the monochromator. The filters serve to limit the wavelength range reaching the monochromator, and so reduce stray radiation.
Radiation passes through the entry slit to the monochromator. Radiation is spectrally dispersed by the monochromator and focussed on the exit slit ES2.

After passing through the exit slit, radiation passes to the spherical mirror S3. Radiation is reflected onto the beam splitter BS, which allows 50% of the radiation to pass through to planar mirror P4. 50% of the radiation is reflected onto planar mirror P5.
Mirror P4 focuses the radiation beam in the sample cuvette. The radiation is then focused onto the sample photo-diode detector by a simple convex lens.
Mirror P5 focuses the radiation beam in the reference cuvette. The radiation is then focused onto the reference photo-diode detector by a simple convex lens.

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# 4. SPECIFICATION

| | |
|---|---|
| **OPERATING PRINCIPLE** | Double beam spectrometer for the UV/VIS region, with microcomputer electronics, keyboard entry and vacuum fluorescence display. |
| **MONOCHROMATOR** | Holographic concave grating with 1053 lines/mm. |
| **RADIATION SOURCES** | Prealigned Deuterium and Tungsten-halogen lamps with automatic lamp switching. |
| **WAVELENGTH RANGE** | 190 nm through 1100 nm. |
| **STRAY RADIATION** | Measured with 2 sec response time:<br>< 0.03 %T at 220 nm,<br>< 0.02 %T at 340 nm, 370 nm.<br>Absorbance: > 2 A at 200 nm, measured with a 2% KCl solution against distilled water. |
| **WAVELENGTH:** | |
| **Accuracy** | ± 0.3 nm at Deuterium peak 656.1 nm. |
| **Reproducibility** | ± 0.1 nm |
| **SPECTRAL SLIT WIDTH** | 2 nm |
| **PHOTOMETRIC:** | |
| **Accuracy** | ± 0.005 A at 1 A (measured with NBS 930 filters). |
| **Reproducibility** | ± 0.002 A at 1A. |
| **Range** | -3 A to +3 A. |
| **Stability** | < 0.0003 A/h (at 500 nm, 1 sec response time, after warmup). |
| **BASELINE STABILITY** | ± 0.001 A (200 - 1100 nm at 0 A, 2 sec response time, 240 nm/min scan speed). |
| **NOISE** | < 0.0003 A (at 0 A, 500 nm, 2 sec response time). |

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## 4.1 DATA OUTPUT

| | |
|---|---|
| **DIGITAL PORT** | RS-232-C/V24 interface for connecting an Epson printer or an Epson PC; optional second RS-232-C/V24 |
| **ANALOG PORT** | For connecting an analog chart recorder (option) |
| **DISPLAY** | 40 character, alphanumeric vacuum fluorescence display for parameter, results and operating information |

## 4.2 ELECTRICAL SPECIFICATION

| | |
|---|---|
| **POWER REQUIREMENTS** | Any voltage between 100 and 240 V, 50/60 Hz; instrument automatically adjusts to the prevailing voltage. |
| **FUSES** | 200 - 240 V operation requires one 2.5 ampere SloBlo fuse.<br>100 - 110 V operation requires one 4.0 ampere SloBlo fuse. |
| **TECHNICAL STANDARD** | In compliance with the requirements for technical instruments stipulated by IEC 348, VDE 0411. |
| **RADIO SUPPRESSION** | Fulfils regulation VDE 0871/B of the F.R.G. |

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## 4.3 METHOD SPECIFIC PARAMETERS

### TIMEDRIVE METHODS

| | |
|---|---|
| **Ordinate Mode** | Choice of %T, ABS or conc |
| **Response Time** | 0.1; 0.2; 0.5; 1; 2; 5; 10 sec |
| **Cycles** | 0-99 cycles, 0.01 - 999.9 min cycle time |
| **Batches** | 0-99 samples, start sample ID 0-99 |
| **Graphics, Method and Data output** | Individually selectable |
| **Sample/User Identification** | Up to 8 characters on the numeric keypad |

### SCAN METHODS

| | |
|---|---|
| **Ordinate Mode** | Choice of %T, ABS, and first to fourth derivatives |
| **Scan Speed** | 7.5; 15; 30; 60; 120; 240; 480; 960; 1920; 2880 nm/min |
| **Smoothing** | 0, 2, 3, 4, 6, 8, 10 nm |
| **Cycles** | 0-99 cycles, 0 - 999.9 min cycle time |
| **Batches** | 0-99 samples, start sample ID 0-99 |
| **Graphics, Method and Data Output** | Individually selectable |
| **Sample/User Identification** | Up to 8 characters on the numeric keypad |

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**WAVPROG METHODS**

| | |
|---|---|
| **Ordinate Mode** | Choice of %T, ABS, Ratio, Difference. |
| **Response Time** | 0.1; 0.2; 0.5; 1; 2; 5; 10 sec |
| **Cycles** | 0-99 cycles, 0 - 999.9 min cycle time |
| **Batches** | 0-99 samples, start sample ID 0-99 |
| **Graphics, Method and Data Output** | Individually selectable |
| **Sample/User Identification** | Up to 8 characters on the numeric keypad |

**CONC METHODS**

| | |
|---|---|
| **Ordinate Mode** | Choice of ABS, 2 or 3 wavelength analysis, 2nd derivative and peak area. |
| **Response Time** | 0.1; 0.2; 0.5; 1; 2; 5; 10 sec<br>(For ABS and 2 or 3 wavelength analysis only) |
| **Smoothing** | 0, 2, 3, 4, 6, 8, 10 nm<br>(for 2nd derivative and peak area analysis only) |
| **Calibration** | Using up to 15 standards, with 4 possible curve fits |
| **Batches** | 0-99 samples, start sample ID 0-99 |
| **Graphics, Method and Data Output** | Individually selectable |
| **Sample/User Identification** | Up to 8 characters on the numeric keypad |

Perkin-Elmer reserve the right to make technical alterations without notice.

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# 5 OPERATING CONTROLS AND DISPLAY

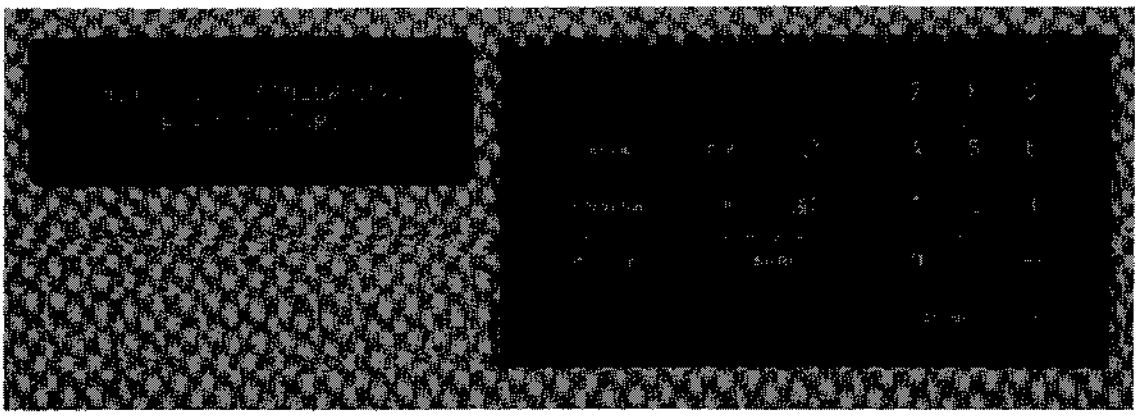


**Figure 5-1. Lambda 2 Display and Keyboard.**

Instrument operation is straightforward.
The display shows instrument status and parameters in the top line. The bottom line displays prompts for any input the user must make.
All user input is made from the instrument keyboard.

The following keys are found on the keyboard:

| Key | Function |
|---|---|
| **0 to 9** | Numerical keys for numerical input. |
| **CE** | Clears any unentered input from the display. |
| **METHOD** | Allows the user to call up methods from the method memory. That method may then be used for analysis, or reviewed, and if necessary, amended. |
| **PARAMETER** | If the active method is to be reviewed, this key allows the user to scroll method parameters onto the display. If necessary the parameter value may be amended. When PARAM appears as a prompt below an option, pressing **PARAMETER** will carry out the option on the display. |
| < > | When one or two arrows are shown on a display prompt, either of the arrow keys should be pressed. This may be because method parameters can only take instrument set values, or because a number of different options are available, these are accessed using the arrow keys. |

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| Key | Function |
|---|---|
| **ENTER** | If a parameter value is to be amended, after the new value has been typed into the prompt line, pressing **ENTER** will fix the new value in the method. |
| **GOTO LAMBDA** | Allows the user to change the wavelength shown on the status display. Press **GOTO**, type in the new wavelength and press **ENTER**. Or type in wavelength and press **GOTO**. |
| **BACK CORR** | Carries out an autozero at the wavelength shown on the status display. |
| **POINT** | Decimal point for numerical entry.<br>Pressed in conjunction with **PARAMETER**, access to TAGGED parameters can be gained (see Section 5.4.8).<br>Pressed in conjunction with **HELP** a directory of stored methods can be printed out. |
| **HELP** | Pressing HELP at any stage will display hints and advice concerning a particular parameter or instrument operation.<br>Pressing . **HELP** in a branch prints out a branch directory.<br>Pressing . **HELP** at a branch header prints out a complete instrument directory. |
| **STOP** | STOP will terminate an analysis run.<br>Also during method modification pressing STOP at any stage will return the display to the method header.<br>Pressing **STOP** while the method header is on the display will return the display to the status display.<br>Pressing **STOP** while on the status display will return the software to the branch header. |

**METHODS**

Instrument software allows a number of analytical methods to be carried out.

**TIMEDRIVE** methods measure sample absorption at one wavelength against time. This allows simple reaction kinetics to be studied, for example.

**SCAN** methods allow samples to be scanned between two wavelengths and their spectra obtained.

**WAVPROG** methods allow sample absorption to be measured at up to 20 different wavelengths.

**CONC 1** methods allow calibration of a concentration standard curve based on sample absorbance, and analysis of samples of unknown concentration.

**CONC 2** methods allow calibration of a concentration standard curve based on peak area or derivative magnitude, and analysis of samples of unknown concentration.

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5.2 **TURNING ON**

Flip the rocker switch to turn on the instrument.
During the startup routine the display will read:

```
"   VERSION 3.0
       BUSY   "
```

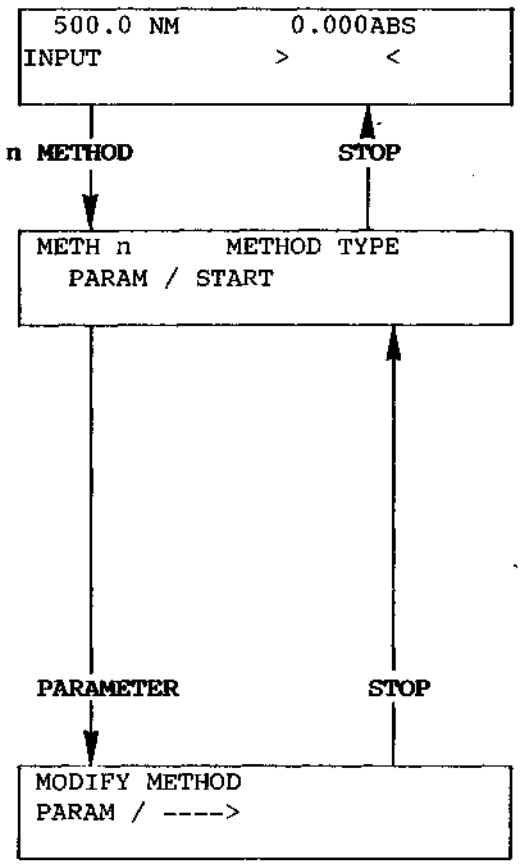
After completing the startup routine, the display will show the status display:

```
"500.0 NM        0.000ABS
 INPUT          >       <"
```

Pressing **START** will start the last method used before power off.
Alternatively, the user can call a method to the active memory. That method can then be used for analysis runs, or if required, the method parameters can be reviewed and amended. Type in the method number and press **METHOD**.

(The user is also able, using the status display, to measure sample absorbance manually. To do this, amend the displayed wavelength using the **GOTO LAM** key, and then place a reference and a sample in the sample compartment. The absorbance now shown on the display is that of the sample at that wavelength.)

**CALLING A METHOD**




Status Display

Method Header

There are three options to choose at this position:

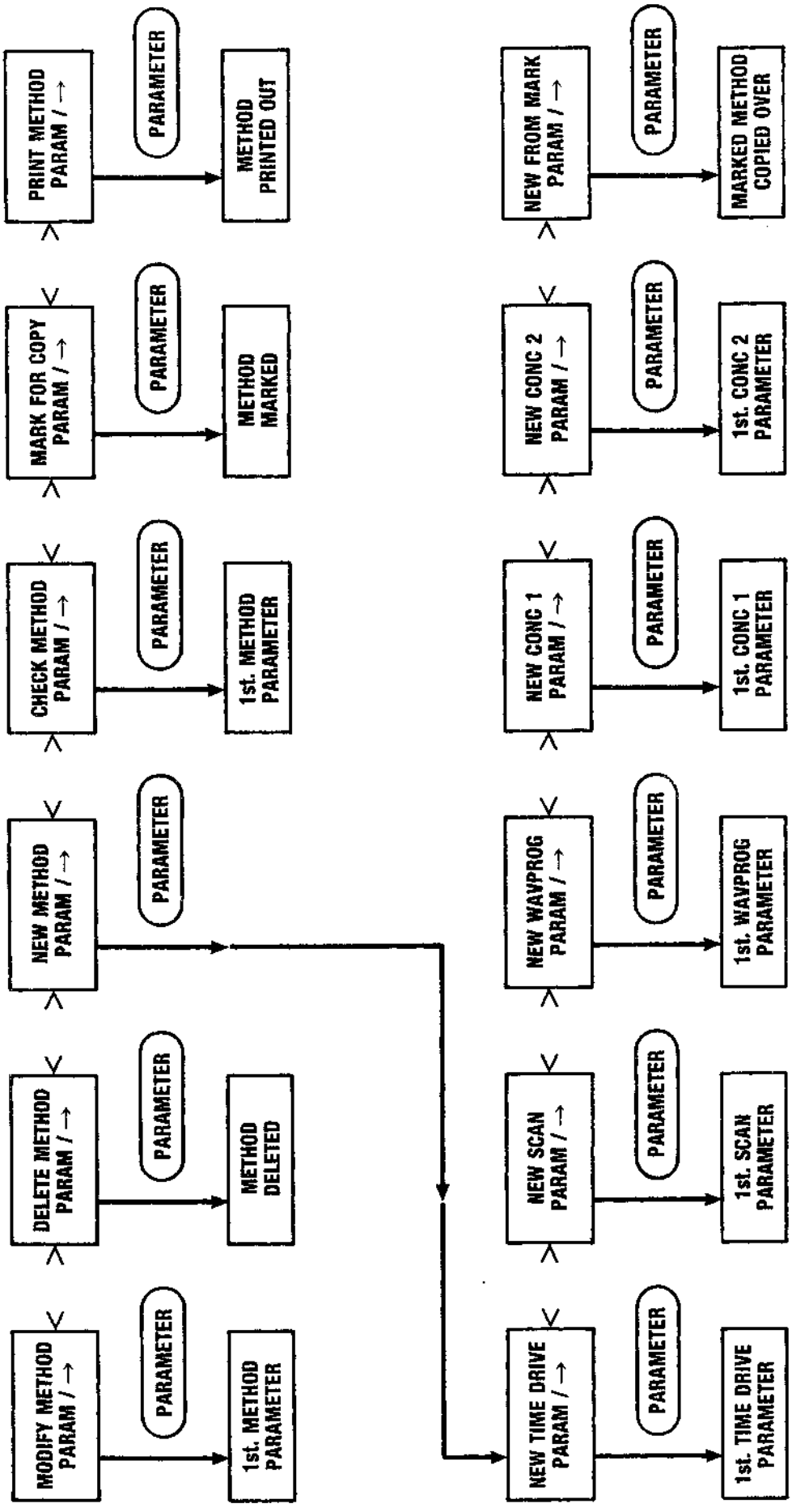
1 Press **START** to begin an analysis run.

2 If method n is not needed press **METHOD** and then select another method. Or access another method with the arrow keys.

3 Press **PARAMETER** to access the modification options.

Modification Options are detailed on the next pages.

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## 5.3 METHOD MODIFICATION OPTIONS

**MODIFY METHOD** Allows the user to review all the method parameters, and then, if necessary, amend them.
Repeatedly pressing **PARAMETER** allows the user to scroll through the method parameters. Whilst a parameter is on the display it can be amended.
If the user only wants to use existing methods and change parameters in those methods he only needs to press **PARAMETER** when at the method header and he will automatically come to the Modify Method option.
Pressing **PARAMETER** again will scroll the first method parameter onto the display.

**DELETE METHOD** Allows the user to delete the method.
Upon pressing **PARAMETER** the method file in the memory will be deleted, and simultaneously the next method will be called to the active memory.
(e.g. If method 1 is deleted here, then method 2 will automatically be called to the active memory.)

**NEW METHOD** Once **PARAMETER** has been pressed a number of NEW METHOD options can be accessed by pressing the arrow keys:

NEW TIMEDRIVE: If **PARAMETER** is pressed then a new timedrive method can be created in this method file.

NEW SCAN: If **PARAMETER** is pressed then a new scan method can be created in this method file.

NEW WAVPROG: If **PARAMETER** is pressed then a new wavprog method can be created in this method file.

NEW CONC 1: If **PARAMETER** is pressed then a new conc 1 method can be created in this method file.

NEW CONC 2: If **PARAMETER** is pressed then a new conc 2 method can be created in this method file.

($) NEW FROM MARK: If **PARAMETER** is pressed then a method that has been marked will be copied into this method file.
(See also MARK FOR COPY below).

**EMPTY METHOD FILES** If no method is stored at the number entered the new method options will immediately be displayed.
This allows the user to create completely new methods without altering existing ones.

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**CHECK METHOD** Allows the method parameters to be checked, but it is not possible to amend the parameter values. This option is useful when a method file has been write-protected and no MODIFY method option is available. CHECK still allows the method parameters to be checked.

**MARK FOR COPY ($)** Pressing **PARAMETER** will mark the method. This marked method can then be copied into another method file. To do this, call the second method file, go to NEW FROM MARK ($) in the new method options and press **PARAMETER**. The marked method will be copied over into this method file. Immediately after pressing **PARAMETER** to mark a method the display returns to the method header.

**PRINT METHOD** Pressing **PARAMETER** will print out the method parameters.

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## 5.4 ANALYSIS METHODS

The next section describes the various methods present in the instrument software. Flowcharts show the sequence of parameters in the methods, and on the facing pages each parameter is explained in detail.
Some parameters dealing with the output of analysis results are common to all methods. These common parameters are explained in a section of their own immediately after the method specific parameters.

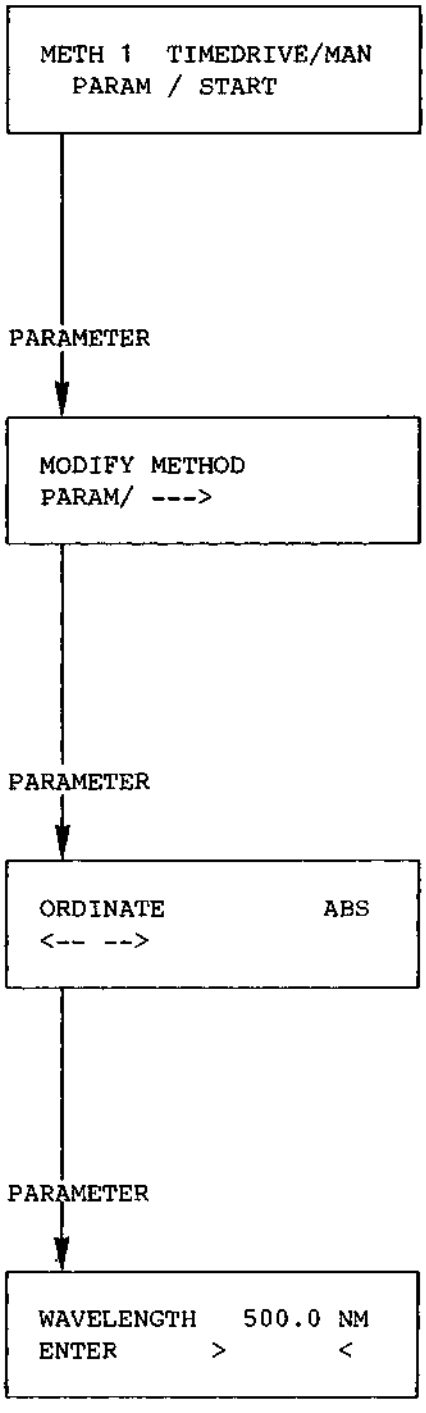
Up to 20 methods can be stored in the instrument.
When the instrument is delivered, methods 1 to 5 contain 1 each of the five different analytical methods.

Methods are "named" with numbers and can take any name between 1 to 999.
On delivery method 999 contains the DATE/TIME method, (8.3.3).

**CREATING NEW METHODS**

Enter a method number which is not occupied. The new method options will be displayed immediately. A new method can be written in this file.
If there are no free method files, decide which method can be overwritten. Select that method and go to the New Method options. A new method can be written.

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(cont'd)

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**METHOD HEADER** When the method header is on the display the method is in the active memory. Two options are available:
* Carry out an analysis run by pressing **START**.
* Review or amend the method parameters by pressing **PARAMETER**

**METHOD MODIFICATION** Using the arrow keys, select what type of task is to be carried out on the method. Select MODIFY method to review and amend the method.

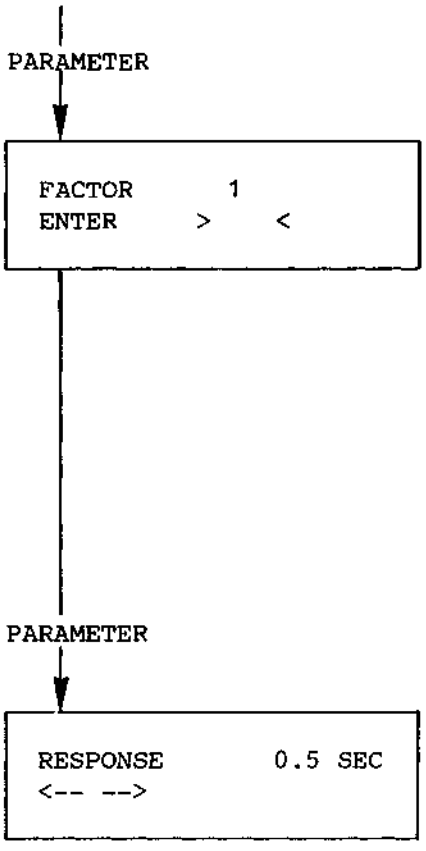
**ORDINATE** Sets the units in which readings are to be made.
The options are:
%T = Percent Transmittance units
ABS = Absorbance units
CONC = Concentration units (see the Factor parameter also)

The required ordinate is brought into the display by pressing the arrow keys.

**WAVELENGTH** The wavelength at which sample absorption is to be measured. Any wavelength between 190.0 nm and 1100.0 nm can be selected in 0.1 nm increments.
Type in the wavelength value and press **ENTER**.

(cont'd)

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(common parameters follow: 5.4.6)

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**FACTOR**

Allows a factor to be applied to the instrument reading. For instance, measurements can be made directly in concentration.
The absorbance value is multiplied by the factor to give a reading in concentration.
This is based on the Beer-Lambert law, the final form of which gives a linear relation between the absorbance and concentration of a sample:

$A = \epsilon Cl$ where A is the absorbance,
$\epsilon$ is the molar absorption coefficient,
C is the concentration of the sample,
and l is the pathlength of the cell.

Thus, if we know the molar absorption coefficient $\epsilon$, the cell pathlength l, and the absorbance A, we can determine the sample concentration using the relation:

$$C = A/\epsilon l.$$

Hence to get a concentration reading we must enter the factor $1/\epsilon l$ for the sample being dealt with.

If no concentration readout is required, then enter a factor of 1 (default value).

A factor can also be used to make corrections for dilutions of original solutions or to correct for weighing errors.

**RESPONSE**

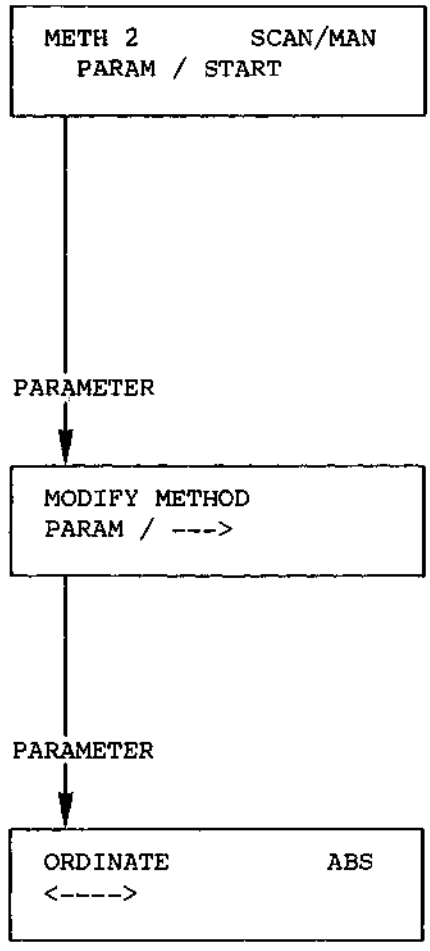
Sets the time interval between registration of readings by the instrument.
Long response times result in a good signal-to-noise ratio, but can lead to a smoothing of the signal with a rapidly changing sample. In general choose as short a response time as possible without noise becoming a problem.
The required response time is scrolled onto the display with the arrow keys.
Possible values are:

0.1, 0.2, 0.5, 1, 2, 5, 10 seconds.

(common parameters follow: 5.4.6)



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(cont'd)

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**METHOD HEADER** When the method header is in the display the method is in the active memory. Two options are available:
* Carry out an analysis run by pressing **START**.
* Review or amend the method parameters by pressing **PARAMETER**

**METHOD MODIFICATION** Using the arrow keys, select what type of task is to be carried out on the method. Select MODIFY method to review and amend the method.

**ORDINATE MODE** Sets the units in which readings are to be made.
The options are:
%T = Percent Transmittance units
ABS = Absorbance units
D1 to D4 = First to Fourth Derivatives of the spectrum.
Select the appropriate units with the arrow keys.

**CHOICE OF DERIVATIVE** Derivative operation resolves overlapping bands and discriminates in favor of steeper characteristics. Broad band interference is reduced and fine structure within absorption bands is enhanced. Derivative spectra provide more easily interpreted qualitative information from samples with overlapping absorption bands, and aid quantitative analysis when spectra contain unwanted background absorption.

**DERIVATIVE ORDER** Resolution and discrimination increase with the derivative order, but the signal-to-noise ratio decreases. The second derivative is usually more useful than the first. As well as greater resolution, the characteristic minimum at a primary signal peak is easy to identify. If second derivative resolution is insufficient, third or fourth derivatives may be necessary, if the noise can be tolerated.

Setting instrument parameters to optimize derivative spectra is often a matter of trial and error. However, the following notes provide some general information for parameter selection.

SCAN SPEED As a general rule:

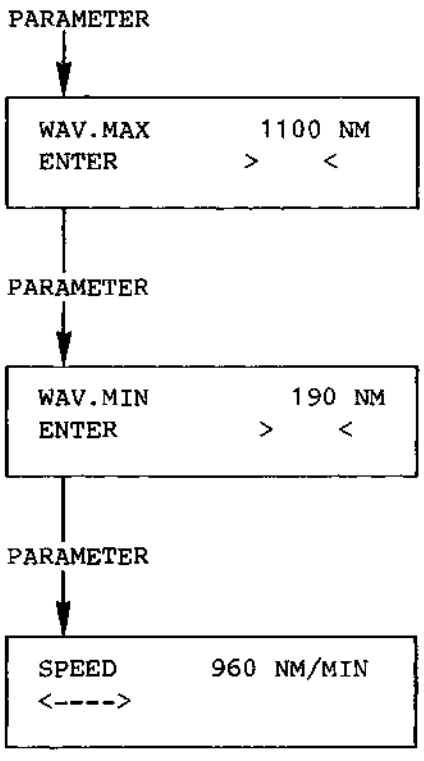
Scan speed (nm/min) = PW (nm) x 10

PW being the peak width of the component to be resolved.
Scan speeds that are too high lead to a loss of resolution.

SMOOTH The smooth setting of the instrument should be small compared to the features of interest. The influence of smooth setting is more pronounced in derivative operation than in absorbance operation.
Smooth value should have a value 1/2 of the peak width.

**NOTE:** (A smooth value of 0 gives no derivative, regardless of the ordinate chosen).

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**WAV MAX**
**WAV MIN**

These two parameters set the scan range within which the sample spectrum is to be scanned.
WAV MAX is the top limit of that range.
WAV MIN is the lower limit of that range.
Permitted values: WAV MAX 1100.0 - 190.0 nm
WAV MIN 190.0 - 1100.0 nm.
Type in the appropriate value and press **ENTER**.

**SPEED**

Scan speed selection depends upon analytical requirements and the nature of the sample.

| | |
|---|---|
| Survey spectra and spectra of broad band samples | 2880; 1920; 480; 240 nm/min |
| Spectra of solid and liquid samples | 120; 60; 30 nm/min |
| Gaseous samples, high resolution or expanded spectra | 30; 15; 7.5 nm/min |

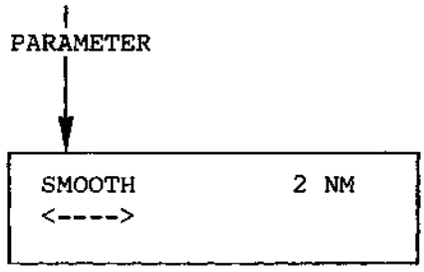
Tracing the natural form of an absorption band is influenced by the scan speed and smooth setting.
No given set of instrument parameters will be valid for all absorption bands.
These general recommendations should help the user to trace an absorption band as close to its natural form as possible:

1. For narrow absorption bands, slow scanning speeds and the smallest possible Smooth setting in keeping with the noise should be chosen.

2. Broad absorption bands allow the selection of faster scanning speeds.

(If a derivative ordinate mode has been chosen see the previous page for recommendations on speed in derivative mode.)

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**SMOOTH** The selectable smooth settings allow spectral resolution to be controlled and noise filtered out.
This is done by processing the signal using the Savitzky/Golay algorithm.
Signal-to-noise ratio is enhanced as noise is smoothed out.

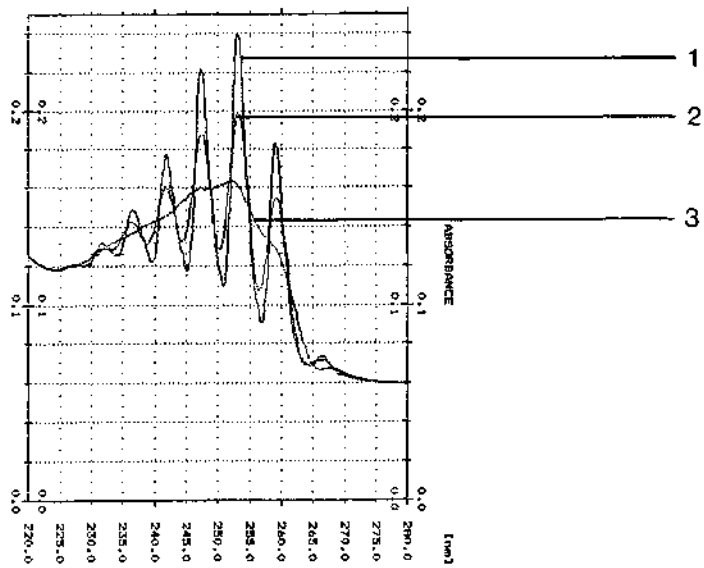



**Figure 5-2.** Benzene spectra recorded with different SMOOTH settings
1. Spectrum recorded with 2 nm smooth. This is a good setting 1/2 the peak width. Spectrum quality is not compromised.
2. Spectrum recorded with 6 nm smooth. Spectrum quality is poor.
3. Spectrum recorded with 10 nm smooth. It is no longer possible to recognise any spectrum.

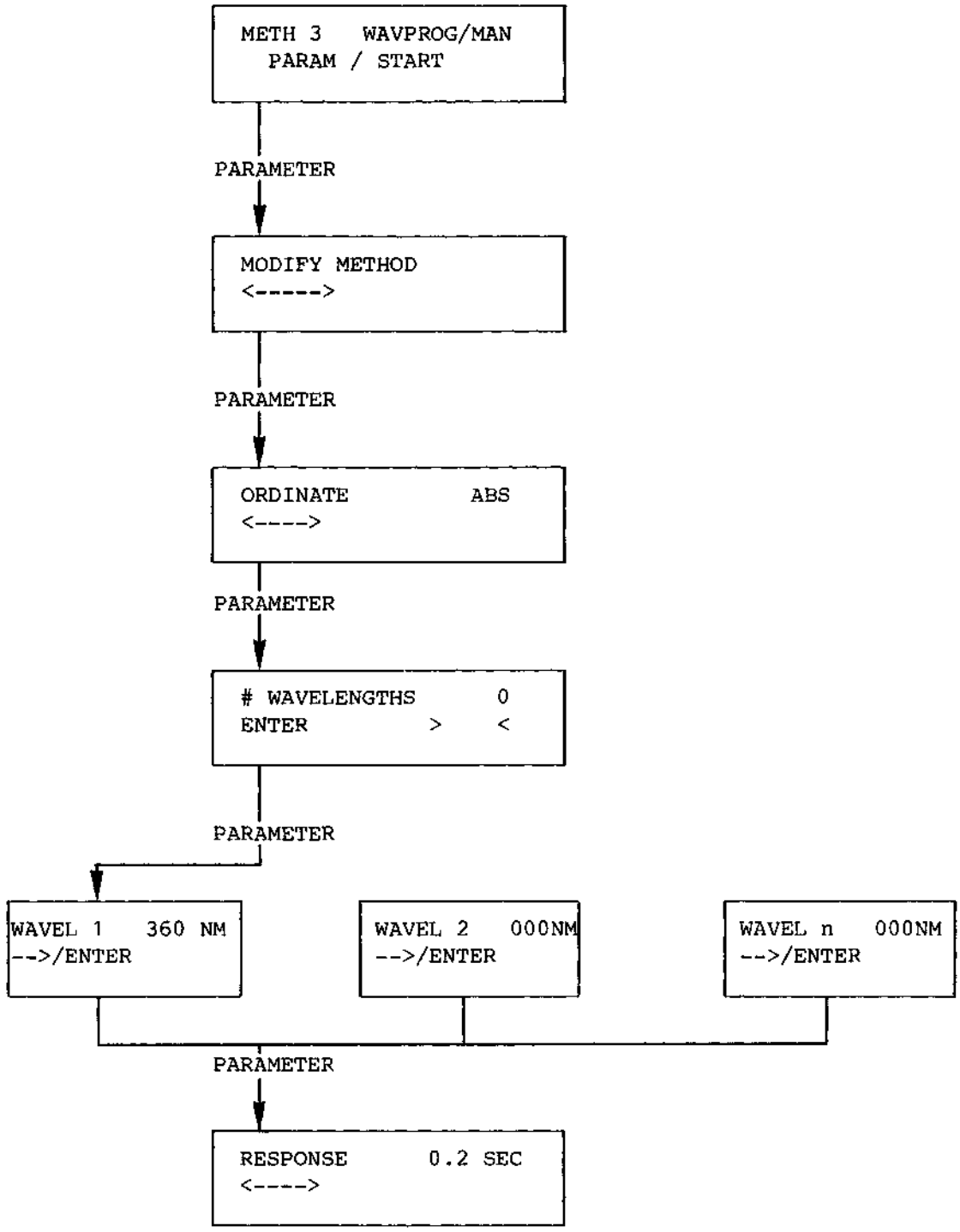
Too large a Smooth setting can result in too extreme averaging of the signal. Some spectral fine structure may be smoothed and lost.
On the other hand, if the smooth setting is too small, spectral features may be obscured by noise.
A compromise must be found.
In general, the Smooth should have a value of half the peakwidth of the narrowest peak in the spectrum.
Permitted smooth values vary with the scanning speed:

**NOTE:** (A smooth of 0 nm will result in no derivative spectra being obtained, even with a derivative ordinate).

| SCANNING SPEED | up to 960 nm/min | 1920 nm/min | 2880 nm/min |
|---|---|---|---|
| SMOOTH VALUES | 0, 2, 3, 4, 6, 8, 10 nm | 0, 4, 6, 8, 10 nm | 0, 6, 8, 10 nm |

Should an incorrect setting be chosen, the instrument will display a hint that the speed or smooth should be changed, otherwise no smoothing of the signal will occur.

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**METHOD HEADER** When the method header is in the display the method is in the active memory. Two options are available:
* Carry out the analysis run by pressing **START**
* Review or amend the method parameters by pressing **PARAMETER**

**METHOD MODIFICATION** Using the arrow keys, select what type of task is to be carried out on the method. Select MODIFY method to review and amend the method.

**ORDINATE MODE** Sets the units in which readings are to be made.
The options are:
%T = Percent Transmittance units.
ABS = Absorbance units.
RAT = Ratio = ABS 1/ABS 2
DIF = Difference = ABS1 - ABS 2
Select the required unit with the arrow keys.

**# WAVELENGTHS** This is the number of wavelengths at which sample absorption is to be measured.
Any number between 1 and 20 can be entered. Type the number and press **ENTER.**
For the Difference and Ratio ordinates the number of wavelengths entered must be a multiple of 2.
When differences and ratios are calculated it is always

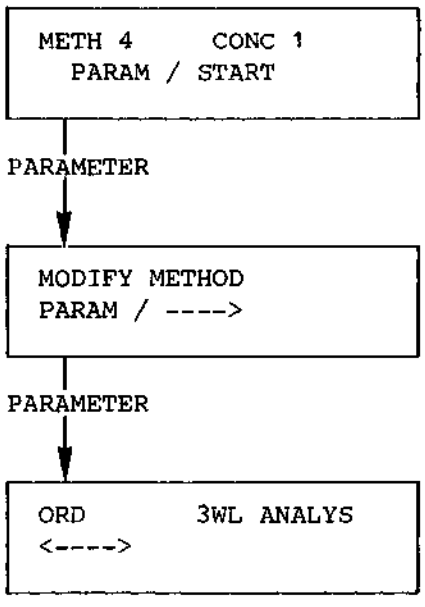
ABS 1 - ABS 2, ABS 3 - ABS4 and so on, or

ABS 1/ ABS 2, ABS 3/ ABS 4 and so on.

**WAVEL 1 – n** Allows the measurement wavelengths to be entered. After entering one wavelength press the arrows to progress to the next wavelength. Wavelengths will be scanned in the order in which they are entered. Values between 190.0 - 1100.0 nm in 0.1 nm increments are permitted.
Type in the wavelength and press **ENTER.**

**RESPONSE** Sets the time interval between registration of readings by the instrument.
Long response times lead to a good signal-to-noise ratio, but can result in a distorted signal. Hence choose as short a response time as possible without noise becoming a problem.
Possible values are: 0.1, 0.2, 0.5, 1, 2, 5, 10 sec.
Select the required response time with the arrow keys.

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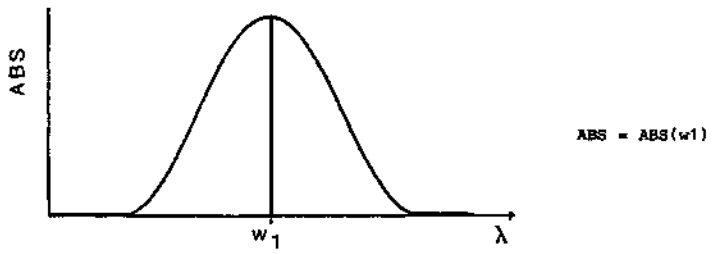

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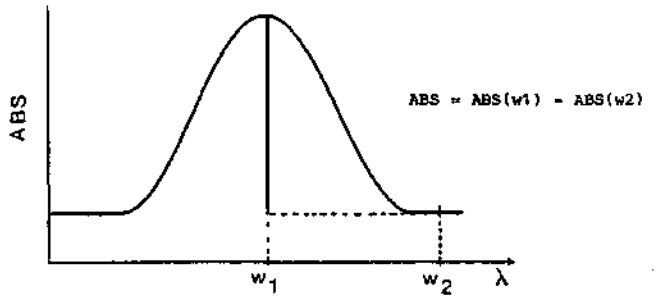
**METHOD HEADER** When the method header is in the display the method is in the active memory. Two options are available:
* Carry out an analysis run by pressing **START**
* Review or amend the method by pressing **PARAMETER**

**METHOD MODIFICATION** Using the arrow keys, select what type of task is to be carried out on the method. Select MODIFY method to review and amend the method.

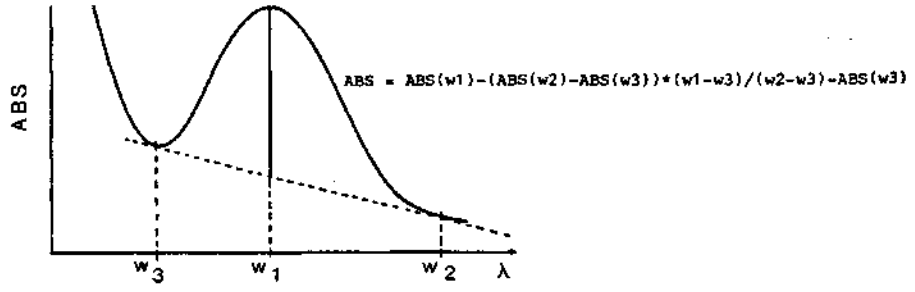
**ORDINATE** Sets the process by which the absorbance and hence the concentration is measured from the readings.
Three options are available:




ABS The absorbance at one wavelength is used to calculate the sample concentration.




DELTA ABS The absolute absorbance is calculated from two wavelengths to compensate for baseline offset.

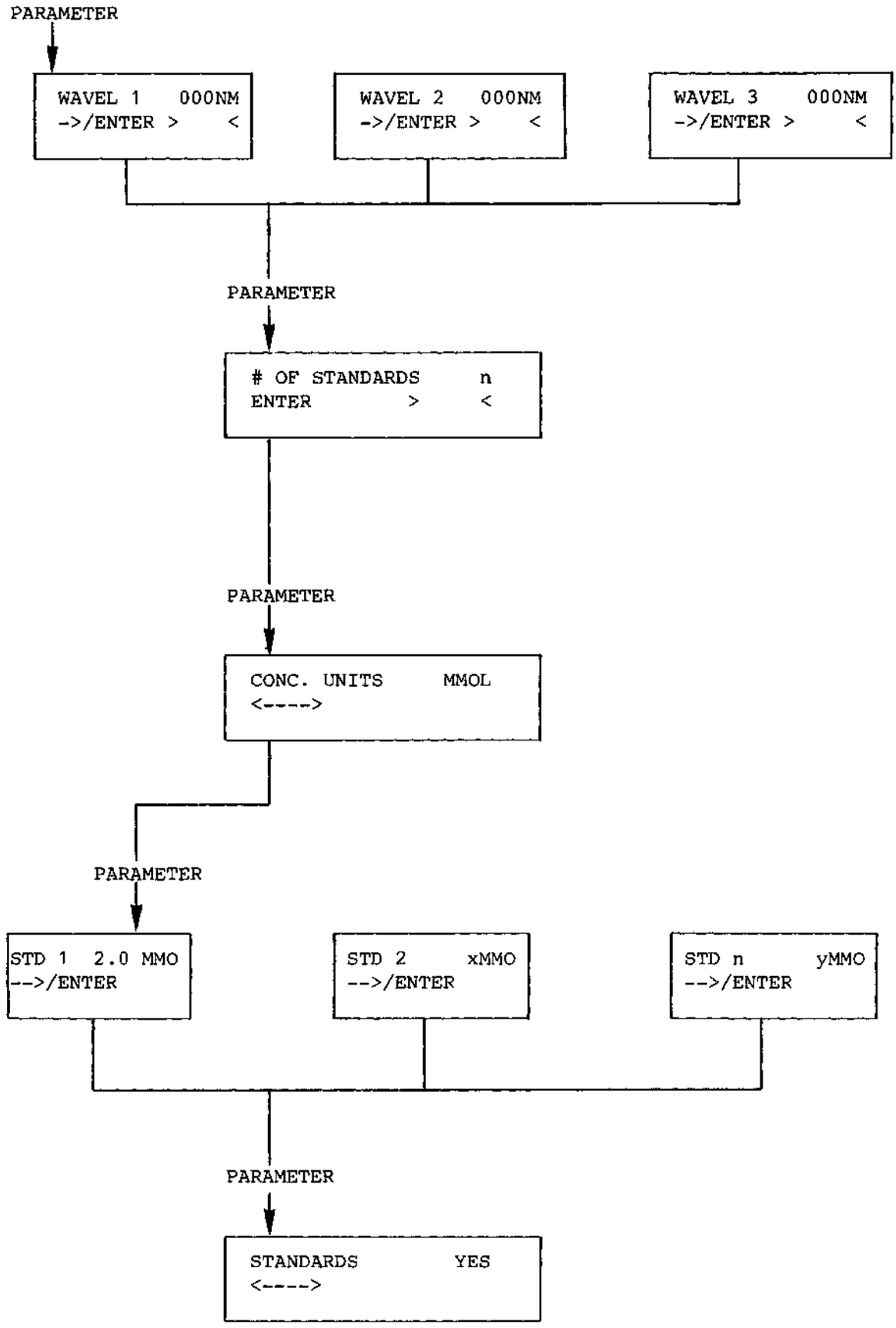



3WL ANALYS The absolute absorbance is calculated from 3 wavelengths to compensate for a sloping baseline

Select the required ordinate with the arrow keys.

(cont'd)

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**WAVEL 1 - 3** Selects the wavelengths at which absorbance is to be measured to calculate the concentration. If only one wavelength is required for calculation only one can be input.

If DELTA ABS has been chosen then two wavelengths must be input, and hence the arrow key prompt in the display.

If 3WL ANALYS has been chosen three wavelengths must be input and therefore there are arrow prompts when this analysis is being used.

Type in the wavelengths and press **ENTER**.

**# OF STANDARDS** Sets the number of standard solutions to be used to set up the standard curve.
Type in the number and press **ENTER**.

**CONC UNITS** Concentration can be calculated in a number of units:

MMOL = millimolar units, a solution with a concentration of 1 millimole substance per litre is a 1 millimolar solution.
G/L = grams of substance per litre.
MG/L = milligrams of substance per litre.
MOL = molar units, a solution with a concentration of 1 mole of substance per litre is a 1 molar solution.
C = is a general unit allowing the user to have results in other units from those above. If standard solutions in say µg/L are entered as C units, then the results output in C units will also be in µg/L.

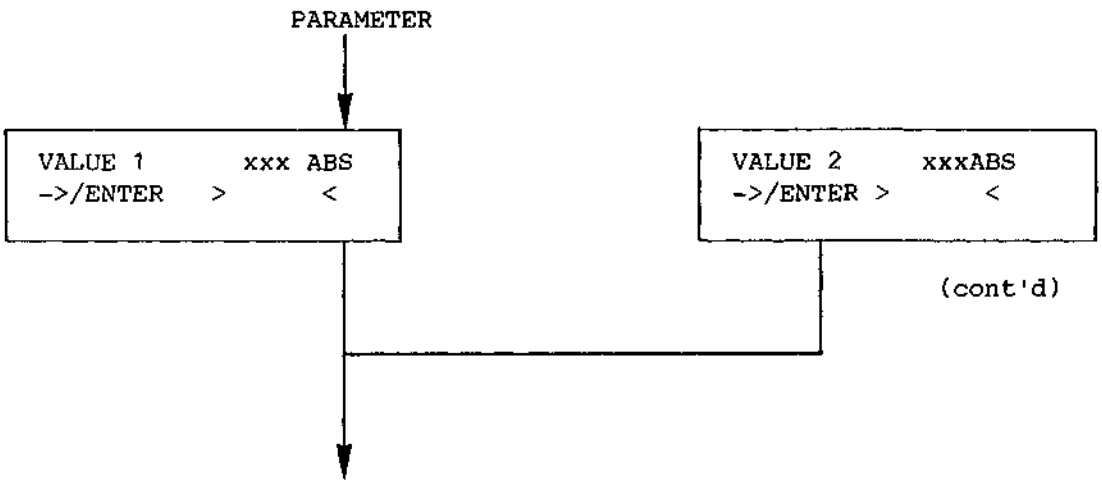
Select the required units with the arrow keys.

**STD 1 - n** The concentrations of the n standard solutions can be entered. Enter each concentration and then press an arrow key to access the next standard.

**STANDARDS** Selects whether a new set of standard solutions must be scanned upon starting each method. If the same standard curve is to be used repeatedly, set NO in the display. If many different standards are to be used then set YES in the display. Change between YES and NO with the arrow keys.

(cont'd)

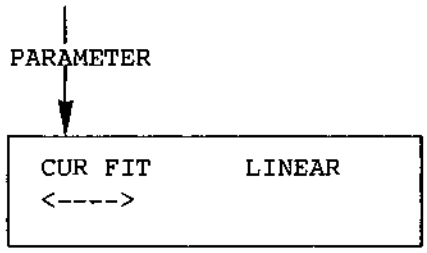
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**VALUE 1 - n** Calibration of the method's standard curve does not require the measurement of standard solutions if absorbance values are already known from the literature, or have been generated elsewhere in the laboratory.
These absorbance values can be input to the instrument on this parameter line.
Ensure that the ABS values are entered in the same order that the standard concentration values were entered.
After entering each absorbance value, press an arrow key > to access the next ABS value.
If the standard curve is to be set up using already known absorbance values, set the previous parameter, STANDARDS to NO. In this way the instrument will not request standard solutions when the method is started.

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**CUR FIT** Dependent upon the samples being analyzed, different standard curves can be calculated by the instrument:

LINEAR; Absorbance varies linearly with concentration throughout the whole range being dealt with. The standard curve passes through the origin.
A minimum of one standard is required.

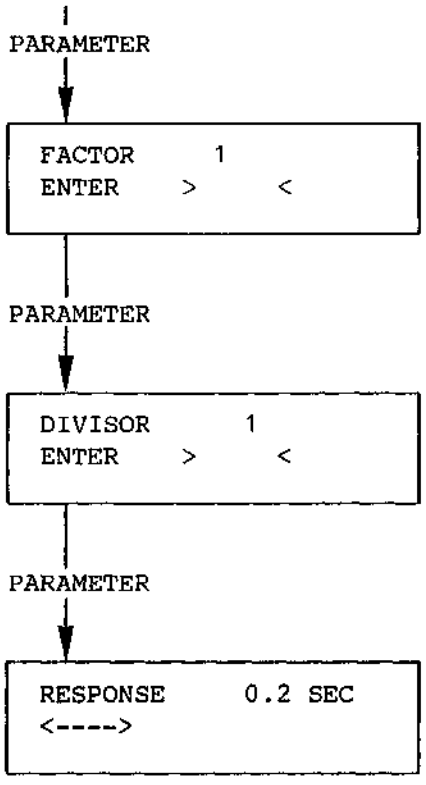
LINEAR INTERC; Absorbance varies linearly with concentration throughout the whole range being dealt with. However an intercept on the absorbance axis compensates for a consistent background absorption, due, for example, to cloudy solutions.
A minimum of two standards is required.

QUADRATIC; In some or all of the range being dealt with, absorbance no longer varies linearly with concentration.
A quadratic curve is required to fit the data. The curve passes through the origin.
A minimum of two standards is required.

QUADRATIC INTERC; As for the quadratic curve above, except that an intercept on the absorbance axis compensates for a consistent background absorption, due, for example, to cloudy solutions.
A minimum of three standards is required.

**RELIABILITY** In all cases the highest sample concentration should not exceed the highest standard concentration.
For maximum reliability of results with quadratic curve fits, Select the appropriate curve fit with the arrow keys.

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(common parameters follow: 5.4.6)

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**FACTOR DIVISOR**

Allow any dilutions that have taken place during sample preparation to be taken into account during setup of the method. By entering a Factor or Divisor, the displayed results can always be those relating directly to the original sample.
By setting the Factor/Divisor as a START parameter (see 5.4.8) a correction factor for each individual sample can be entered before analysis, giving results that are immediately comparable with one another.

For example:
A specific constituent in a powder is to be determined, and the results given as mg/g (mg of substance to g of powder).
The standard curve has however been established with pure substance in units of mg/L.
In order that the results are given as mg/g, regardless of the amount of powder actually weighed out, the actual weight of powder in each sample must be entered as divisor.
If dilution takes place to 0.25 liter rather than to 1 liter, then a dilution factor of 0.25 must also be entered.
The results are then automatically calculated as follows:

(F(L)/W(g)) * C(mg/L) = Result (mg/g).

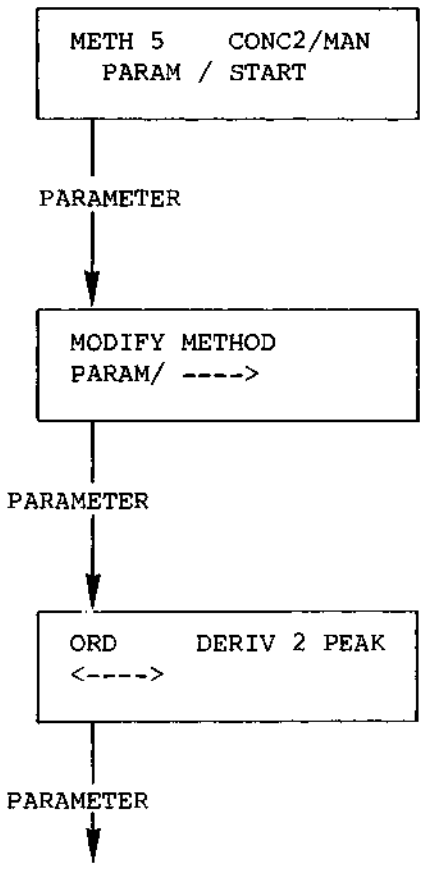
where F = dilution factor, W = Weight of powder,
C = measured concentration.

**RESPONSE**

Sets the time interval between registration of readings by the instrument.
Long response times lead to a good signal-to-noise ratio, but can result in a distorted signal. Choose as short a response time as possible without noise becoming a problem.
Select the required response time with the arrow keys.

(common parameters follow: 5.4.6)

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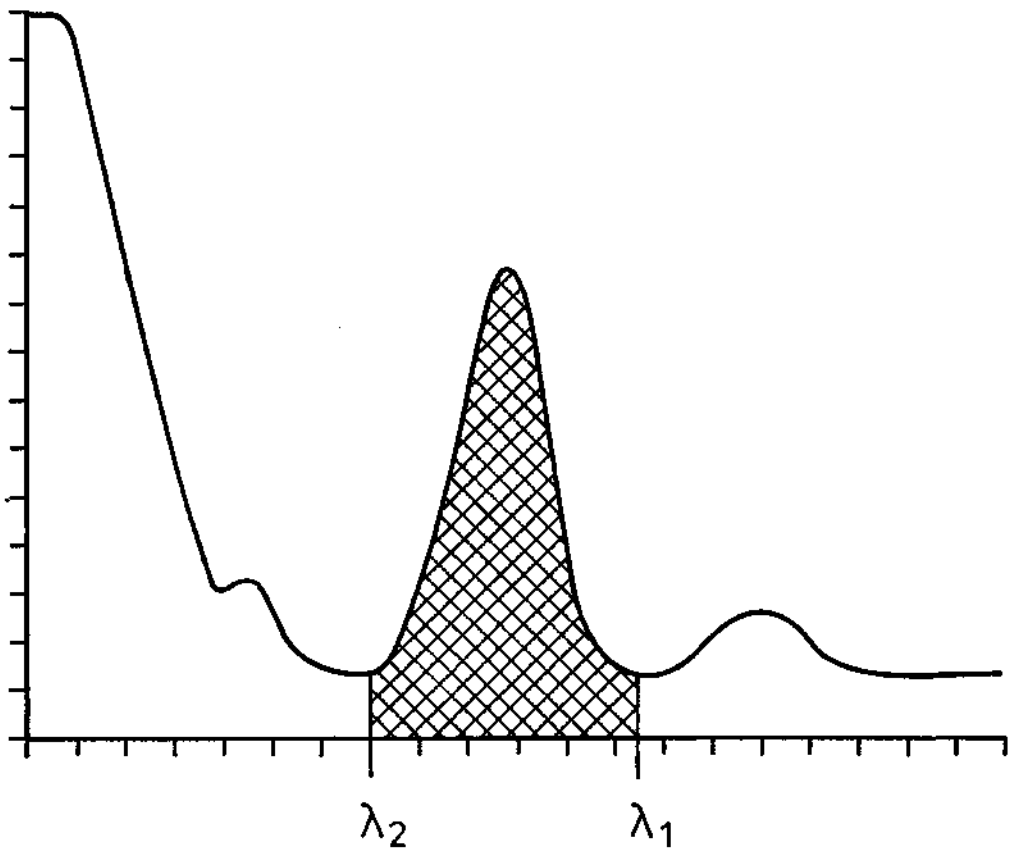
**METHOD HEADER** When the method header is in the display the method is in the active memory. Two options are available:

* Carry out an analysis run by pressing **START**
* Review or amend the method by pressing **PARAMETER**

**METHOD MODIFICATION** Using the arrow keys, select what type of task is to be carried out on the method. Select MODIFY method to review and amend the method.

**ORD MODE** Sets the process by which the instrument measures peak area or derivative magnitude to calculate concentration. Four options are available:

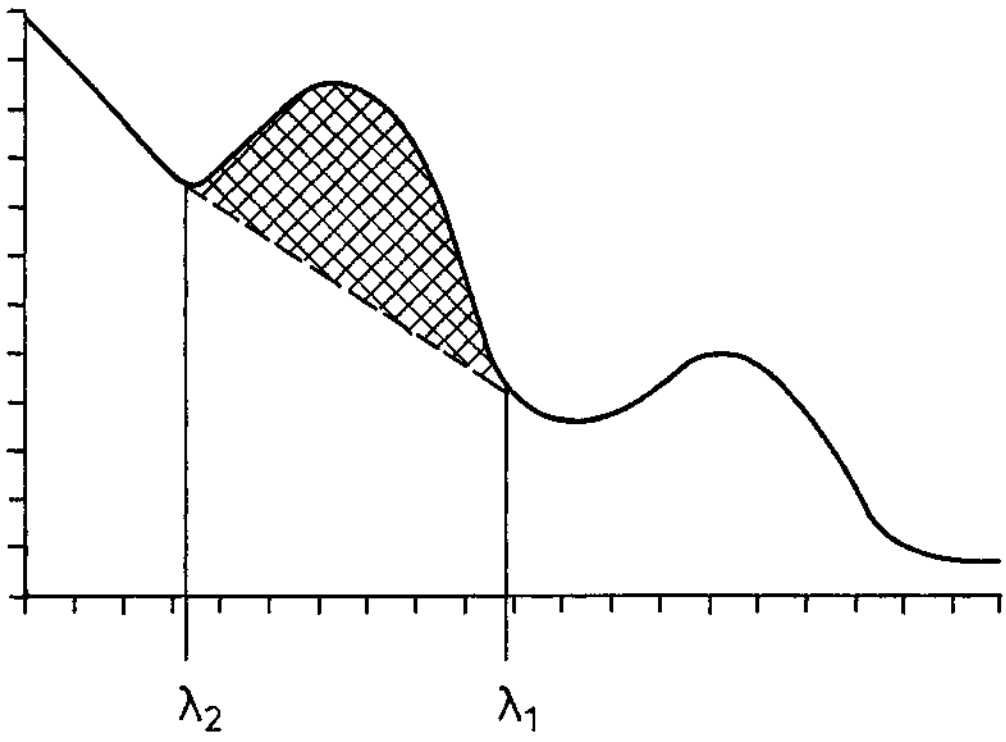
1.




TOTAL AREA: The total area below the curve within the range set by wav.min and wav.max is used for concentration measurement.

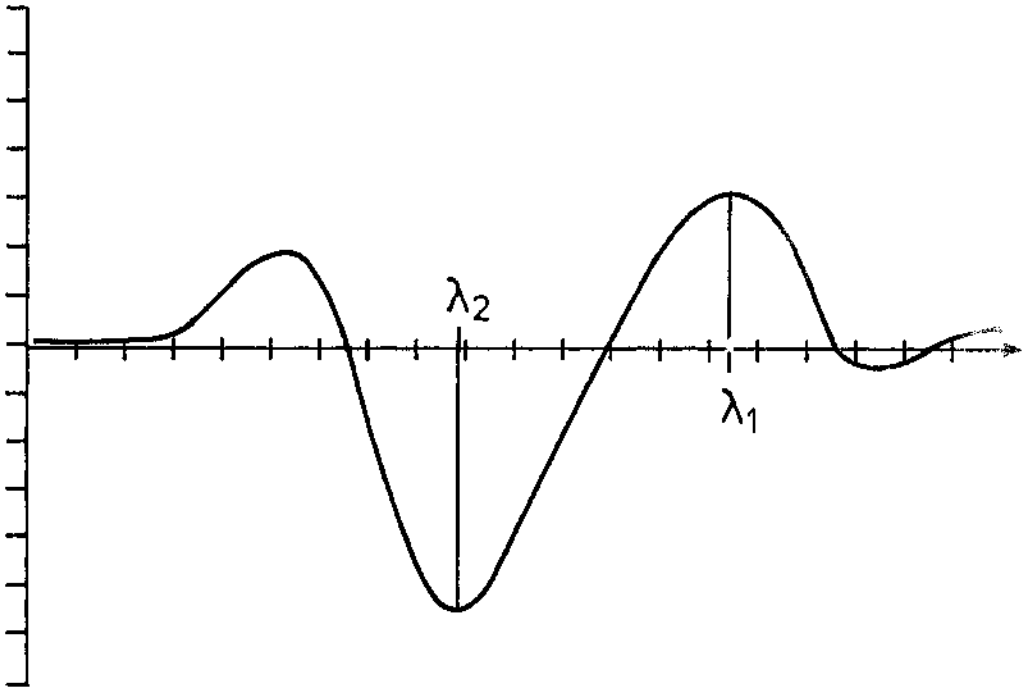
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2.




PEAK AREA: Concentration measurement with a sloping baseline. The area below a peak is measured and compensation for the sloping baseline is carried out in the range between wav.min and wav.max.

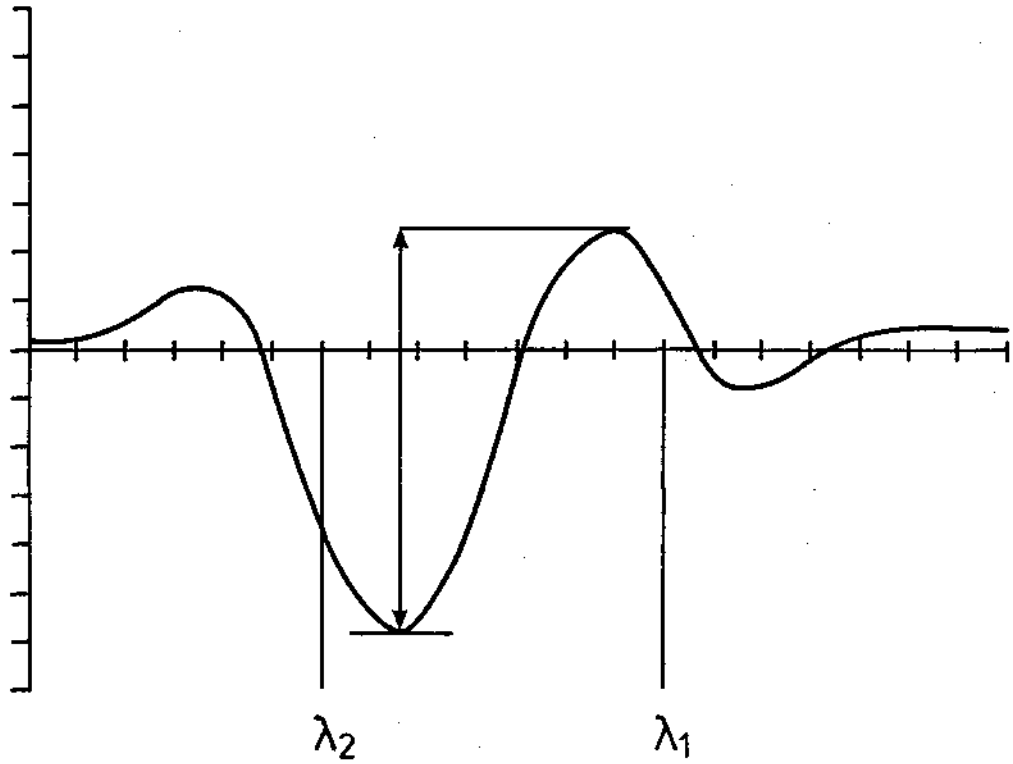
3.




DERIV 2 FIX: Concentration measurement based on the magnitude of the second derivative as measured at the two calculation wavelengths.

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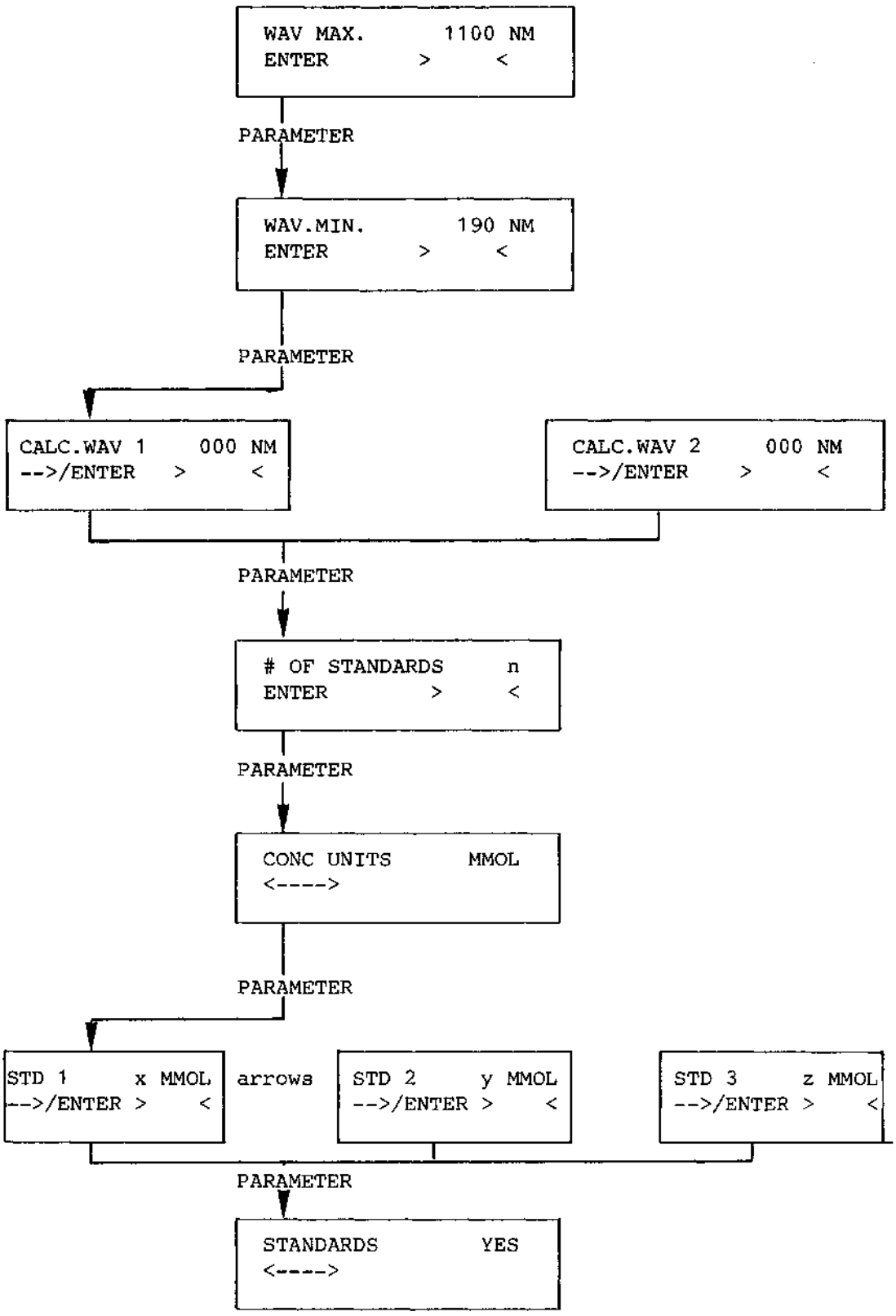
4.




DERIV 2 PEAK: Concentration measurement based on the magnitude of a second derivative peak.
The derivative is not measured at fixed wavelength.
A wavelength range is set by the two calculation wavelengths.
The instrument searches within the wavelength range for a derivative peak which exceeds a set threshold value.

**THRESHOLD** If Deriv2 Peak is chosen as the ordinate then the next parameter is the THRESHOLD parameter. This allows a threshold value to be specified. This enables the instrument peak detector to pick the correct peak in the calculation range.

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**WAV MAX**
**WAV MIN**

These two parameters set the scan range within which the sample spectrum is to be scanned.
WAV MAX is the top limit of that range.
WAV MIN is the bottom limit of that range.
Type in the values and press **ENTER**.
With the Area ordinate modes, the range specified by Wav. Max and Wav.Min is the range within which the area is calculated.
With the Deriv. ordinate modes the wavelength range is the range within which the derivative spectrum is determined.

**CALC.WAV 1 + 2**

These two parameters only appear when either of the derivative ordinate modes has been chosen.
Deriv2 Fix: calc.wav. 1 + 2 are the wavelengths at which the derivative magnitude is measured.
Deriv2 Peak: calc.wav. 1 + 2 set the range within which the instrument searches for the derivative peak.
Calc.Wav 1 + 2 must lie within the wavelength range specified by Wav.Max and Wav.Min.

**# OF STANDARDS**

Sets the number of standard solutions to be used to set up the standard curve. Type in the number and press **ENTER**.

**CONC UNITS**

Concentration can be calculated in a number of units:
MMOL = millimolar units, a solution with a concentration of 1 millimole substance per litre is a 1 millimolar solution.
G/L = grams of substance per litre.
MG/L = milligrams of substance per litre.
MOL = molar units, a solution with a concentration of 1 mole of substance per litre is a 1 molar solution.
C = is a general unit allowing the user to have results in other units from those above. If standard solutions in say µg/L are entered as C units, then the results output in C units will also be in µg/L.

Select the required units with the arrow keys.

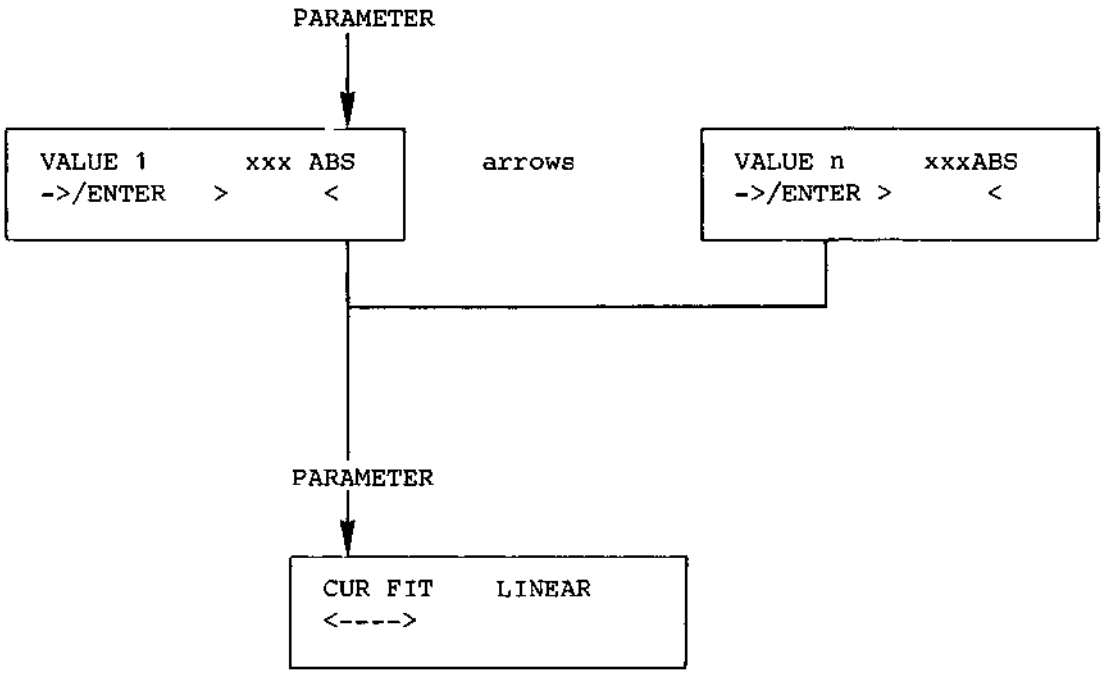
**STD 1 - n**

Allows entry of the concentrations of the standard solutions. Enter each concentration and then press an arrow key to access the next standard.

**STANDARDS**

Selects whether a new set of standard solutions must be scanned every time the method is started. If the same standard curve is to be used repeatedly, set NO in the display.
If many different standards are to be used, then set YES in the display. Change between YES and NO with the arrow keys.

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**VALUE 1 - n** Calibration of the method's standard curve does not require the measurement of standard solutions if absorbance values are already known from the literature, or have been generated elsewhere in the laboratory.
These absorbance values can be input to the instrument on this parameter line.
Ensure that the ABS values are entered in the same order that the standard concentration values were entered.
After entering each absorbance value, press an arrow key > to access the next ABS value.
If the standard curve is to be set up using already known absorbance values, set the previous parameter, STANDARDS to NO. In this way the instrument will not request standard solutions when the method is started.

**CUR FIT** Dependent upon the samples being analyzed, different standard curves can be calculated by the instrument:

LINEAR; Absorbance varies linearly with concentration throughout the whole range being dealt with. The standard curve passes through the origin.
A minimum of one standard is required.

LINEAR INTERC; Absorbance varies linearly with concentration throughout the whole range being dealt with. However an intercept on the absorbance axis compensates for a consistent background absorption due, for example, to cloudy solutions.
A minimum of two standards is required.

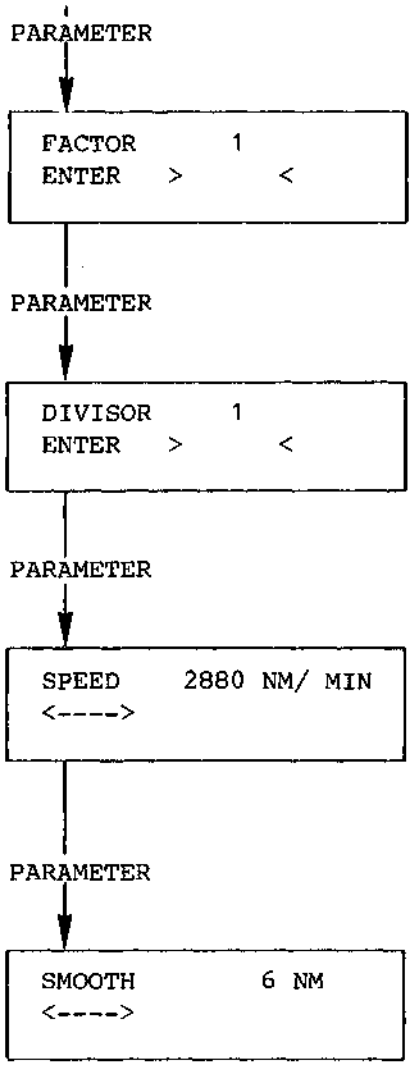
QUADRATIC; In some or all of the range being dealt with, absorbance no longer varies linearly with concentration.
A quadratic curve is required to fit the data. The curve passes through the origin.
A minimum of two standards is required.

QUADRATIC INTERC; As for the quadratic curve above, except that an intercept on the absorbance axis compensates for a consistent background absorption due, for example, to cloudy solutions.
A minimum of three standards is required.

**RELIABILITY** In all cases the highest sample concentration should not exceed the highest standard concentration.
For maximum reliability of results with quadratic curve fits, it is recommended that more standards from the non-linear region are used than from the linear region.

Select the appropriate curve fit with the arrow keys.

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(common parameters follow: 5.4.6)

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**FACTOR DIVISOR**

Allows any dilutions that have taken place during sample preparation to be taken into account during setup of the method. By entering a Factor or Divisor, the displayed results can always be those relating directly to the original sample. By setting the Factor/Divisor as a START parameter (see 5.4.8) a correction factor for each individual sample can be entered before analysis, giving results that are immediately comparable with one another.

For example:
A specific constituent in a powder is to be determined, and the results given as mg/g (mg of substance to g of powder).
The standard curve has however been established with pure substance in units of mg/L.
In order that the results are given as mg/g regardless of the amount of powder actually weighed out, the actual weight of powder in each sample must be entered as divisor.
If dilution takes place to 0.25 liter rather than to 1 liter, then a dilution factor of 0.25 must also be entered.
The results are then automatically calculated as follows:

$$(F(L)/W(g)) * C(mg/L) = Result\ (mg/g).$$

where F = dilution factor, W = Weight of powder,
C = measured concentration.

**SPEED**

If the peak being used to calculate the concentration is narrow, a slow scan speed should be selected so that the peak will be traced as faithfully as possible. A broader band will allow faster speeds to be selected.

**SMOOTH**

As small a SMOOTH as possible should be selected so that little smoothing of the peak takes place. Selection of the actual value is dependent upon the background noise.

(common parameters follow: 5.4.6)

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**NOTE:** The parameters listed under common parameters, LAMP through to CYCLES, are the same for all methods. However, the CONC methods have different graphic output parameters than the other methods:

The first graphics parameter in the conc methods is the PRINT DATA parameter. This allows the user to decide whether he wants the tabulated results of the analysis to be output or not.

PLOT STANDARDS. This parameter allows the user to decide whether the calibration curve is printed out before the start of every analysis.

PRINT STANDARDS. This parameter allows the user to decide whether the calibration data, (i.e. Standard solution concentrations and absorbances), is to be printed out at the start of every analysis.

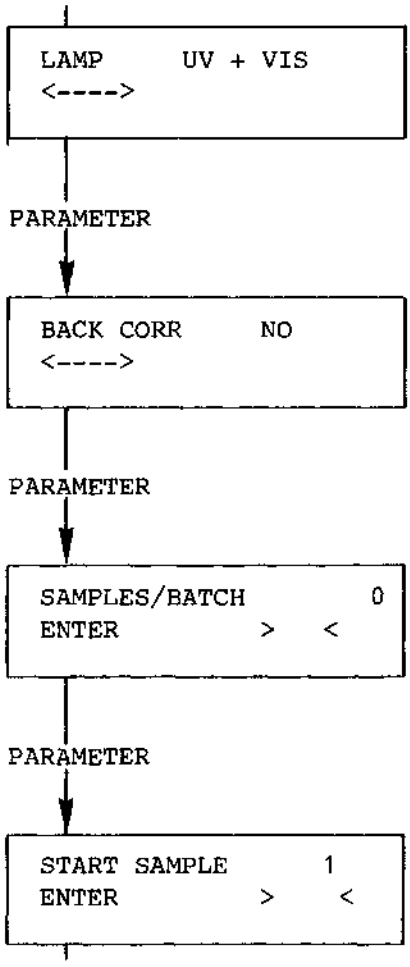
AUTOMETHOD. Allows the user to select whether the method parameters should be printed out at the start of every analysis run.

The final parameter, as with all other methods, is the operator I.D. parameter.

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**LAMP**

The lamps shown in the display are those which are turned on. If an analysis is undertaken outside the range of one of the lamps the redundant lamp may be turned off.
To prolong UV-lamp life the UV-lamp should only be turned off at the end of the working day.
After turning off the UV lamp it should be allowed to cool for about a minute before being turned back on.
The wavelength ranges of the lamps are as follow:
UV lamp : 190 - 342 nm
VIS lamp: 342 - 1100 nm

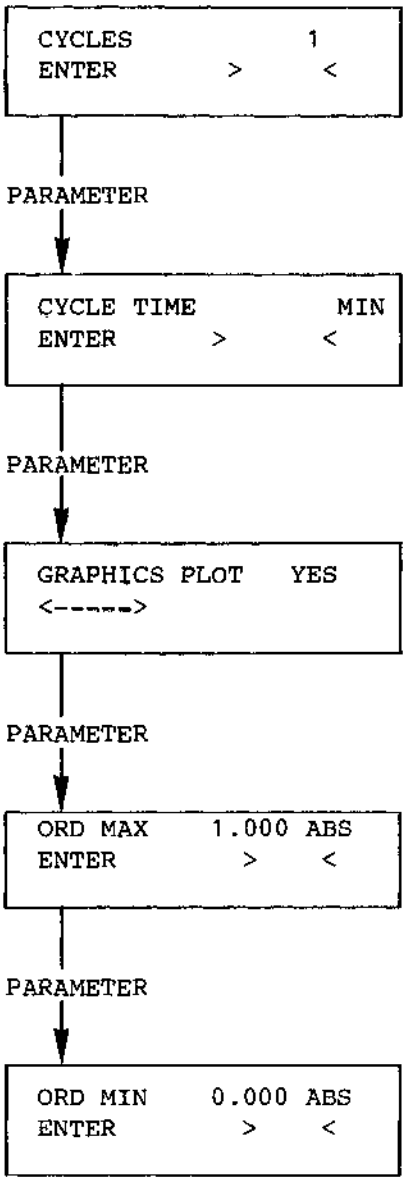
**BACKCORR**

If BACKCORR is YES it will be executed when the method is first started.
Timedrive methods: an autozero is carried out at the entered wavelength.
Wavprog and Conc1 methods: an autozero will be carried out at each of the entered wavelengths.
Scan and Conc2 methods: a blank baseline will be recorded between the scanning wavelengths.
In order for an autozero and a blank baseline to be recorded blank solution must be placed in both sample and reference cell holders.
Once an autozero or baseline has been recorded at the start of a work period, it should not be necessary to repeat it as long as the same solvent is being used.
Much flexibility can be gained from setting BACK CORR as a tagged parameter, (5.4.8.).
As a CALL parameter, a back corr can be selected the first time the method is started.
As a BATCH parameter, a back corr can be selected at the start of each batch.
As a START parameter, a back corr can be selected before each sample analysis.

**SAMPLES/BATCH**

For administrative purposes a number of samples can be processed as a batch. All the samples in a batch will undergo the same analysis.
Type in the number of samples, press **ENTER**.
**PARAMETER** accesses the START SAMPLE parameter. This is the identity of the first sample in the batch. Other samples in the batch will be numbered consecutively.

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If NO go straight to PRINT DATA parameter.

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**CYCLES** Selects how many times the same analysis will be carried out on each sample.

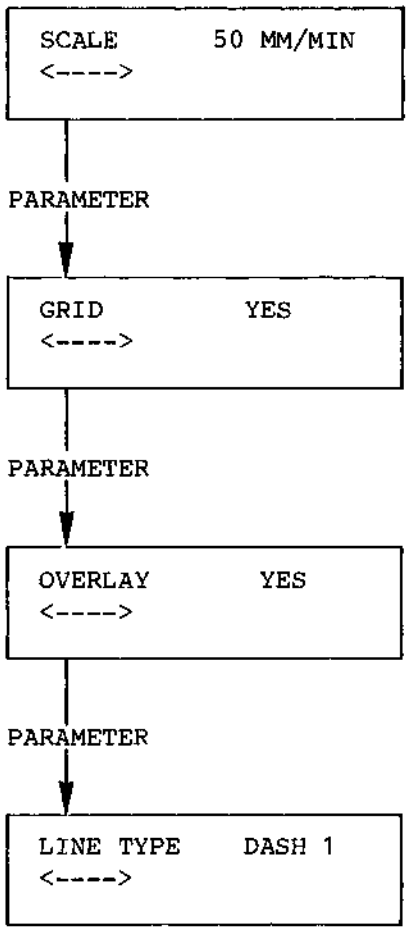
**CYCLE TIME** This is the time lapse between the start of one cycle to the start of the next

**NOTE:** Timedrive methods, analysis time = cycles * cycle time. Scan methods, cycle time should be longer than time taken to scan the spectrum. If not difficulties may arise when using automatic accessories.

**GRAPHICS PLOT** Selects whether the analysis results are output graphically or not. If GRAPHICS PLOT = NO then the software goes straight to the PRINT DATA parameter.

**ORD MAX/MIN** These two parameters set the minimum and maximum ordinate scale range of the graphic output.
Type in the required values and press **ENTER**.
Analytical readings may exceed the set values, leading to out of range graphical output.
If this is the case enter new values to obtain satisfactory output.
Derivative spectra take on both positive and negative ordinate values. Enter values accordingly.

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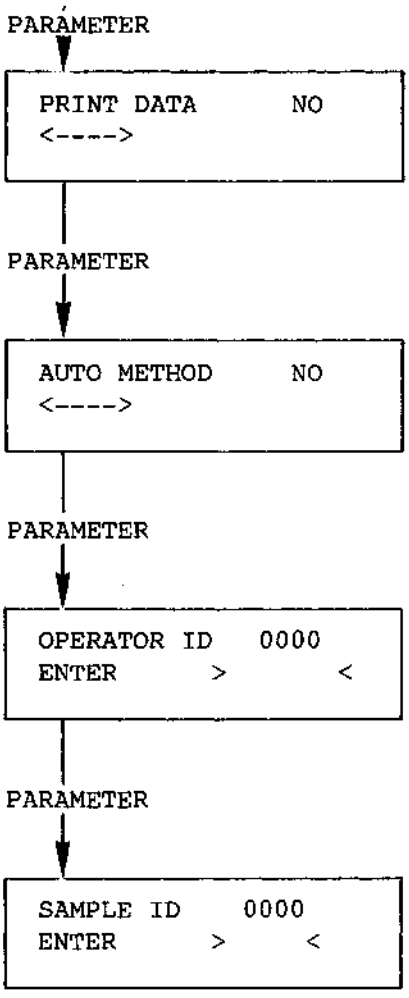
**SCALE** Allows the expansion of the abscissa scale to be selected. Choose the required value by pressing the arrow keys. Selectable values are:
Timedrive Methods: 1, 2, 5, 10, 20, 50, 100 mm/min.
Scan Methods: 0.5, 1, 2, 5, 10, 20, 50 nm/cm.

**GRID** Selects whether a grid is to be printed on the output. A grid can be useful for reading off values from spectra.

**OVERLAY** The overlay facility allows spectra from the same cycle to be printed out on the same plot. If the number of cycles is set to 1, then all the results from the same batch will be overlaid.
It is easier to compare differing absorbances in overlaid printout than in separate printouts.

**LINE TYPE** If overlaid printout has been selected 4 different line styles can be selected:
DASH 1 ——————————
DASH 2 — — — — — — — — —
DASH 3 ----------
DASH 4 _·_·_·_·_·_·_·
AUTO: If auto is selected, then for each different result a different line type will be selected in the order dash 1, dash 2, dash 3, dash 4, dash 1 and so on.

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**PRINT DATA** Selects whether analytical results will be printed out as data. If output is chosen the results are tabulated. If YES is selected, the next parameter will be THRESHOLD. This allows the user to select a threshold value so that the instrument only prints out results for those peaks which exceed the threshold. If too low a threshold is set then the results table will include not only the real peaks but also spikes due to noise.

**AUTO METHOD** Selects whether method parameters will be printed out at the beginning of an analysis run.

**OPERATOR ID** Allows the operator to enter an identification number.

**SAMPLE ID** Allows a sample identification number to be entered. Both identification numbers will be printed on any output. Up to 8 characters on the keyboard can be entered for ID.

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## 5.4.7. NUMBERED PARAMETERS

Each parameter in the software has a number. The same parameter in different methods will have the same number. When modifying or checking a method, rather then having to scroll through the whole list of parameters just to amend, say two, it is possible to go directly to the required parameter using the parameter number.
Once in the parameter list (i.e. at the first parameter), type in the parameter number and press **PARAMETER**. The parameter list will then go straight to the numbered parameter. (Numbered parameters do not necessarily appear in numerical order).

**PARAMETER NUMBERS**

The parameter numbers for each method are as follows:

**TIMEDRIVE METHODS**

| | |
|---|---|
| 1 | Ordinate Mode |
| 3 | Wavelength |
| 14 | Factor |
| 17 | Response |
| 18 | Lamp |
| 19 | Background Correction |
| 20 | Samples per Batch |
| 21 | Start Sample |
| 22 | Cycles |
| 23 | Cycle Time |
| 24 | Graphics Plot |
| 25 | Ordinate Max. |
| 26 | Ordinate Min. |
| 27 | Scale |
| 28 | Grid |
| 31 | Print Data |
| 35 | Auto Method |
| 36 | Operator ID |
| 37 | Sample ID |



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| SCAN METHODS | | WAVPROG METHODS | |
|---|---|---|---|
| 1 | Ordinate Mode | 1 | Ordinate Mode |
| 3 | Wavelength Max. | 2 | Number Of Wavelengths |
| 4 | Wavelength Min. | 3 | Wavelength (1-n) |
| 16 | Speed | 17 | Response |
| 17 | Smooth | 18 | Lamp |
| 18 | Lamp | 19 | Background Correction |
| 19 | Background Correction | 20 | Samples per Batch |
| 20 | Samples per Batch | 21 | Start Sample |
| 21 | Start Sample | 22 | Cycles |
| 22 | Cycles | 23 | Cycle Time |
| 23 | Cycle Time | 24 | Graphics Plot |
| 24 | Graphics Plot | 25 | Ordinate Max. |
| 25 | Ordinate Max. | 26 | Ordinate Min. |
| 26 | Ordinate Min. | 27 | Scale |
| 27 | Scale | 28 | Grid |
| 28 | Grid | 31 | Print Data |
| 29 | Overlay | 35 | Auto Method |
| 30 | Line Type | 36 | Operator ID |
| 31 | Print Data | 37 | Sample ID |
| 32 | Threshold | | |
| 35 | Auto Method | | |
| 36 | Operator ID | | |
| 37 | Sample ID | | |

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| CONC 1 | METHODS |
|---|---|
| 1 | Ordinate Mode |
| 3 | Wavelength |
| 7 | Number Of Standards |
| 9 | Concentration Units |
| 10 | Standard Concentrations |
| 11 | Standards |
| 12 | Absorbance Values |
| 13 | Curve Fit |
| 14 | Factor |
| 15 | Divisor |
| 17 | Response |
| 18 | Lamp |
| 19 | Background Correction |
| 20 | Samples per Batch |
| 21 | Start Sample |
| 22 | Cycles |
| 23 | Cycle Time |
| 31 | Print Data |
| 33 | Plot Standards |
| 34 | Print Standards |
| 35 | Auto Method |
| 36 | Operator ID |
| 37 | Sample ID |

| CONC 2 | METHODS |
|---|---|
| 1 | Ordinate Mode |
| 32 | Threshold |
| 3 | Wavelength Max. |
| 4 | Wavelength Min. |
| 5 | Calc. Wav 1 + 2 |
| 7 | Number of Standards |
| 9 | Concentration Units |
| 10 | Standard Concentrations |
| 11 | Standards |
| 12 | Absorbance Values |
| 13 | Curve Fit |
| 14 | Factor |
| 15 | Divisor |
| 16 | Speed |
| 17 | Smooth |
| 18 | Lamp |
| 19 | Background Correction |
| 20 | Samples per Batch |
| 21 | Start Sample |
| 22 | Cycles |
| 23 | Cycle Time |
| 31 | Print Data |
| 33 | Plot Standards |
| 34 | Print Standards |
| 35 | Auto Method |
| 36 | Operator ID |
| 37 | Sample ID |

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## 5.4.8. TAGGED PARAMETERS

Lambda 2 software allows parameters which need regular amendment (due, for example, to sample variations) to be tagged so that they appear on the display before the start of the analysis. In this way only those parameters which are likely to need amendment can be checked, the whole method does not need to be reviewed.

There are three levels of tag:

**CALL TAG &**

The tagged parameters will be displayed for amendment when the method is started for the first time after being recalled. CALL parameters are denoted by a & sign before them in the display.

**BATCH TAG !**

The tagged parameters will be displayed for amendment at the start of each new batch.
BATCH parameters are denoted by a ! sign before them in the display.

**START TAG ***

The tagged parameters will be displayed for amendment when each analysis is to be started, e.g. before the analysis of each sample in a batch.
START parameters are denoted by a * sign before them in the display.

For example, it may sometimes be necessary to carry out a back corr before some batches, but not before others.
By setting BACK CORR as a batch parameter the user can decide before each batch whether a background correction should be carried out.

In each case, analysis can only be continued by pressing **START**, whether the parameter has been amended or not.
Parameters in the method which are not tagged, and can only be amended by reviewing the method, are denoted as FIX parameters.
It is possible to have different tags within a method, and therefore, after a CALL tagged parameter has been checked, pressing **START** again will not necessarily start the analysis but may display a BATCH tagged parameter and then a START tagged parameter.
This allows tremendously flexible method operation.
(See Flowchart on page 6-3)

**SETTING TAGS**

To tag a parameter press |. | **PARAMETER** when the relevant parameter is in the display during a review. The screen will then display:

**PARAMETER DEFN. FIX**
< >

By pressing the arrow keys the correct tag can be called into the top line. Press **PARAMETER** to continue to next parameter.
The next parameter will then be shown in the display.
To tag this parameter it will be necessary to press
|. | **PARAMETER** again.

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## 5.5 CASSETTE OPERATION

**NOTE:** To operate with cassettes, the instrument must be fitted with the "cassette operating kit", B018-0206.

Cassettes provide 100 extra method files while plugged into the instrument. These method files are also saved when the cassette is removed from the instrument, allowing methods prepared on one instrument to be transferred quickly to other instruments and indeed from one lab to another.

**ACCESSING THE CASSETTE**

The cassette can be plugged in and unplugged from the instrument at any time.
When the cassette slot is opened, the software will wait until the cassette has been plugged in.
The display will read:

```
CASSETTE ACTION
   IS LOCKED
```

Once the cassette has been "recognized", the instrument effectively has two method branches:
that of the instrument and that of the cassette.

To access the cassette branch, type in

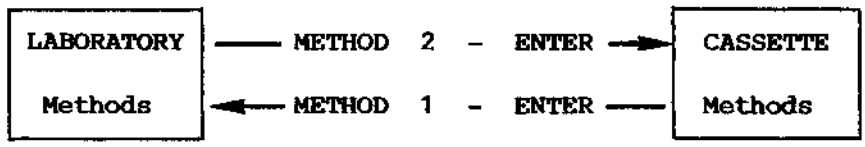
**METHOD 2 - ENTER.**

You are now in the cassette branch and a cassette method is in the active memory.
To call up further cassette stored methods, type in **METHOD n ENTER** (n = method number).
Active methods that have been called from the cassette branch are denoted by CMETH in the display.

**NOTE:** To call up a cassette method directly from the cassette branch, type in **METHOD 2 -** (method no.) **ENTER.**

To return to the instrument method branch, type in

**METHOD 1 - ENTER.**




Alternatively, press **STOP** to reach the LABORATORY or CASSETTE branch header and using the arrow keys move to the cassette or laboratory branch. Press **PARAMETER** to call up a method header.

(B2131-A2.2/4.89)

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**INSTRUMENT TO CASSETTE**

* Call from the laboratory branch the method which is to be copied.
* In the MODIFY METHOD options select **MARK FOR COPY** and press **PARAMETER**. The method is now marked.
* Access the cassette branch by typing **METHOD** 2 - (method no. to be copied to) **ENTER**.
* In the MODIFY METHOD option select NEW METHOD and press **PARAMETER**. Scroll through the new method options until NEW FROM MARK appears, press **PARAMETER**. The marked method will be copied over into this method file.

See also Section 5.3.

**CASSETTE TO INSTRUMENT**

To copy a method from cassette to instrument, the procedure is essentially the same as above except that a cassette method is marked and copied into an instrument method file.

**CASSETTE DIRECTORY**

In order to get a printed directory of the cassette methods type | . | **HELP** while in a cassette method header.

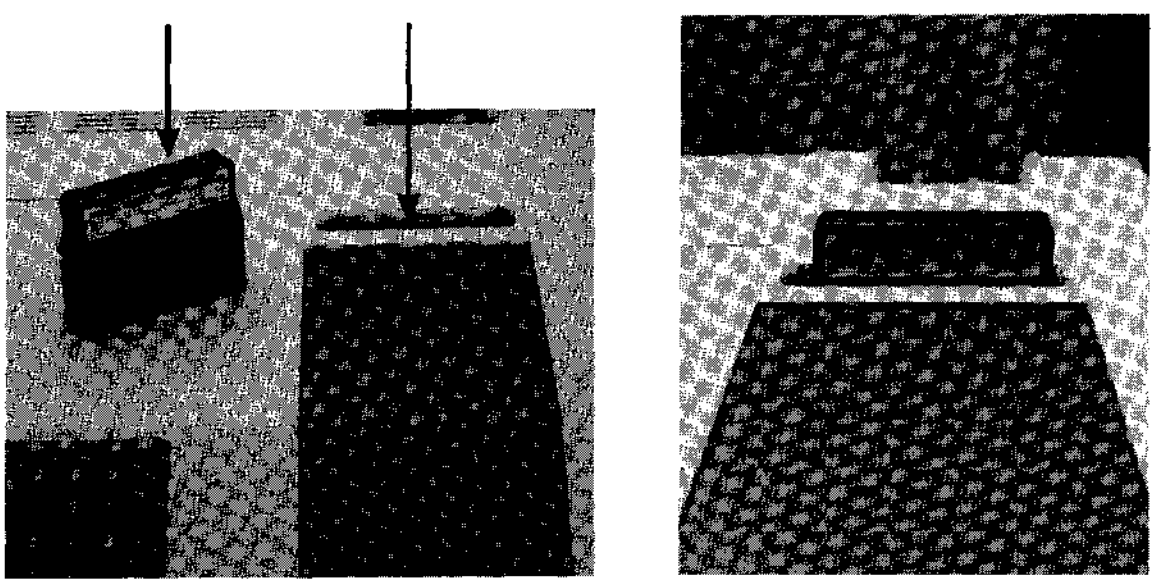


**Figure 5.3** a) Cassette and Cassette Slot
b) Cassette Inserted in Slot



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When fitted with the accessories board, the instrument automatically recognises accessories when they are installed. The accessory option in the method is automatically set for that accessory.
Methods can be written for other accessories by selecting a different accessory in the method. With no accessory attached, operation can be simulated with one sample. The method will run, and output will appear, as if an accessory were fitted.

A Lambda 2 without an accessory board can also be used to write methods for accessory operation. Accessory operation can be simulated. These methods can be transferred, on cassette, to an accessory equipped instrument.

**TURNING ON ACCESSORIES MODE**

A non-equipped Lambda 2 must be configured for accessory methods before these can be written.
To do this:

* Switch the instrument on in SuperUser mode. (section 8.1).

* Go to the Configuration branch using the arrow keys.

* Select method 10, Access Conf.
  Select to modify the method.

* Switch the ACCESSORIES ON ? parameter to YES.

The instrument is now configured for accessories.

* Turn the instrument off, and turn it on again so that it is not in SuperUser mode.

## 5.6.1 WRITING ACCESSORY METHODS

With an accessory installed, or with the accessory mode on, the parameter ACCESSORY will appear in the method parameters; immediately following BACK CORR.

The ACCESSORY parameter allows the user to select an accessory, and then to program the method for that accessory.

To select an accessory:
The ACCESSORY parameter will appear on the display.
(The accessory parameter number is 38).
Using the arrow keys select from the following options:

MANUAL: operation without accessories.
CELL5 : operation with a 5x5 cell changer.
CELL6 : operation with a 6x6 cell changer.
SI-SIP: operation with a single sipper.
SIS+MS: operation with a single sipper and multi-sampler.
SU-SIP: operation with a super- or econo- sipper.
SUS+MS: operation with a super- or econo- sipper and multi-sampler.
CEL13 : operation with a 13 cell carousel.

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If an accessory has been selected, parameters to control accessory operation will be displayed.
Accessory parameters are numbered for instant call up.

5.6.2
**CELL CHANGERS** If a cell changer is selected in a method then the parameter Samples/Batch does not appear.

**CELL5** Allows up to five sample and reference cells to be placed in a cell changer and analyzed sequentially.
After this accessory has been chosen, the next parameter is:

```
CELL 5     12345              (Parameter number 39)
ENTER      >     <
```

Enter the numbers of the positions in which sample cells are to be placed.
If only 1,2 and 3 are entered, only those positions will be moved into the radiation beam when the method is run.

If BACK CORR = YES then the background correction will be carried out with those cells in positions 1.
Hence even if only positions 2, 3 and 4 have been entered, if Back Corr = yes, then back corr will be carried out at positions 1.

Methods operating with the 5x cell changer are denoted by CEL5 in the method header following the method type.

**CELL 6** Allows up to six sample and reference cells to be placed in a cell changer and analyzed sequentially.
After this accessory has been chosen, the next parameter is

```
CELL 6     123456             (Parameter number 40)
ENTER      >     <
```

Enter the numbers of the positions in which sample cells are to be placed.
If only 1,2 and 3 are entered, only those positions will be moved into the radiation beam when the method is run.

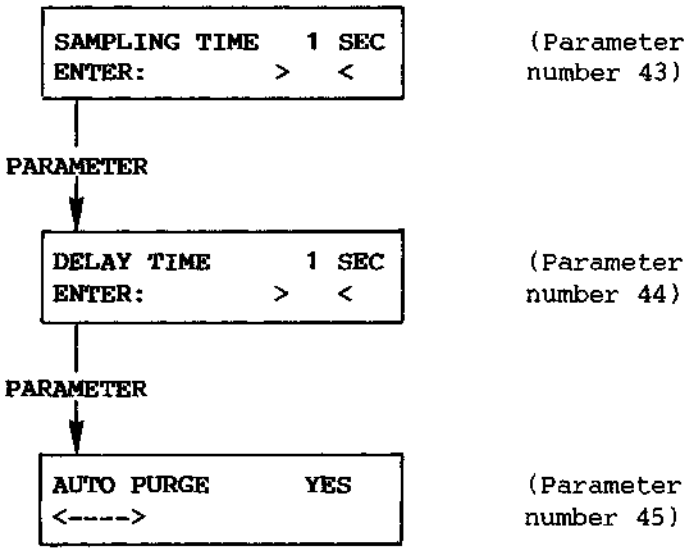
If BACK CORR = YES then the background correction will be carried out with those cells in positions 1.
Hence even if only positions 2, 3 and 4 have been entered, if Back Corr = yes, then back corr will be carried out at positions 1.

Methods operating with the 6x cell changer are denoted by CEL6 in the method header after the method type.

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5.6.3

**SI-SIPP** Allows operation with a single sipper.
After this accessory has been chosen the following parameters are displayed:




SAMPLING TIME — The length of time for which sample solution is aspirated into the flowcell. (Allowed range, 0.1 to 99.9 seconds).

DELAY TIME — Delay period to allow sample equilibration before the reading is made. (Allowed range, 0.1 to 99.9 seconds).

AUTO PURGE — Turns the auto purge function on and off.

Further information can be found in the handbook supplied with the accessory.

Methods operating with the single sipper are denoted by SIS in the method header after the method type.

**SIS+MS** Allows operation with a single sipper and multi sampler to give semi-automated analysis.

The parameters which are displayed when this accessory choice is made are the same as for operation with the single sipper alone.
Methods operating with the single sipper and multi sampler are denoted by SIM in the method header after the method type.

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5.6.4

**SU-SIPP** Allows operation with a super- or econo- sipper. Because operational parameters are set on the sipper control unit, no further sipper parameters can be set on the Lambda 2.

**WARNING:** Before using the super sipper, ensure that the modification to the control cable detailled in section 2.5.3 has been carried out. Without this modification damage to the Lambda 2 can result.

Methods operating with the super sipper are denoted by SUS in the method header after the method type.

**SUS+MS** Allows operation of a super- or econo- sipper with a multi sampler to give automated operation. Because operational parameters are set on the sipper control unit, no further sipper parameters can be set on the Lambda 2.

Methods operating with the super sipper and multi sampler are denoted by SUM in the method header after the method type.

5.6.5

**CEL13** Allows 13 sample cells to be placed in the sample compartment and analyzed sequentially. Two parameter lines allowing the occupied positions to be specified follow:

```
CELL 1 - 7  1234567          (Parameter number 41)
ENTER       >       <
```

This parameter allows the user to specify which of the first seven positions will be occupied.

```
CELL 8 - 13   123456         (Parameter number 42)
ENTER         >      <
```

This second parameter allows the user to specify which of positions 8 to 13 will be occupied. Position 8 is indicated by 1, and position 13 by 6.

**NOTE:** If you decide not to use any of the diplayed positions, type in 0; for example CELL 1 - 7 = 0.

All thirteen sample cells must be measured against the same reference cell. Position 1 is used for Background Correction.

A third parameter line turns the stirrer ON or OFF. The stirrer is positioned such that while one sample cell is being analysed, the next cell "in line" will be being stirred. Small stir bars must be placed in each cell.

```
STIRRER        ON            (Parameter number 46)
<---->
```

Methods operating with the 13 cell carousel are denoted by C13 in the method header after the method type.



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**DEFAULT METHODS**

Permanent default versions of the 5 analytical methods are stored in the laboratory branch.
Access default methods from the laboratory branch header by pressing | . | **PARAMETER** whilst in Super User mode (Ch. 8).

DMETH 1 TIMEDR/MAN
PARAM / START

will be displayed.
Further default methods can be accessed by scrolling with the arrow keys.
Default values are automatically read into a new method file before the parameters have been amended.

DEFAULT PARAMETERS:

DEFAULT METHOD 1 TIMEDRIVE MANUAL

| | | | |
|---|---|---|---|
| Ordinate: | ABS | Graphics plot: | Yes |
| Wavelength: | 500.0 nm | Ord max: | 1.000 ABS |
| Factor: | 1.0 | Ord min: | 0.000 ABS |
| Response: | 0.5 sec | Scale: | 20 mm/min |
| Lamp: | UV/Vis | Grid: | Yes |
| Back Corr: | Yes | Print Data: | No |
| Samples/Batch: | 0 | Auto Method: | No |
| Start Sample: | 1 | Oper. ID: | 0000 |
| Cycles: | 1 | Sample ID: | 0000 |
| Cycle time: | 0.10 min. | | |

DEFAULT METHOD 2 SCAN/MANUAL

| | | | |
|---|---|---|---|
| Ordinate: | ABS | Graphics plot: | Yes |
| Wav Max: | 1100.0 nm | Ord max: | 1.000 ABS |
| Wav Min: | 190.0 nm | Ord min: | 0.000 ABS |
| Speed: | 960 nm/min | Scale: | 50.0 nm/cm |
| Smooth: | 2 nm | Grid: | Yes |
| Lamp: | UV/Vis | Overlay: | No |
| Back Corr: | Yes | Print Data: | No |
| Samples/Batch: | 0 | Auto Method: | No |
| Start Sample: | 1 | Oper ID: | 0000 |
| Cycles: | 1 | Sample ID: | 0000 |
| Cycle time: | 0.1 min | | |

DEFAULT METHOD 3 WAVPROG MANUAL

| | | | |
|---|---|---|---|
| Ordinate: | ABS | Graphics plot: | No |
| # Wavelengths: | 3 | Print Data: | Yes |
| Wavel. 1: | 360.0 nm | Auto method: | No |
| Wavel. 2: | 418.5 nm | Oper ID: | 0000 |
| Wavel. 3: | 459.9 nm | Sample ID: | 0000 |
| Response: | 0.5 sec | | |
| Lamp: | UV/Vis | | |
| Back Corr: | Yes | | |
| Samples/batch: | 0 | | |
| Start Sample: | 1 | | |
| Cycles: | 1 | | |
| Cycle time: | 0.10 min | | |

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| | | | | |
|---|---|---|---|---|
| DEFAULT | Ordinate: | ABS | Standards: | No |
| METHOD 4 | Wavel. 1: | 500.0 nm | Value 1: | 0.5 |
| CONC1/MANUAL | # of Standards: | 3 | Value 2: | 1.0 |
| | Conc Units: | C | Value 3: | 1.5 |
| | STD 1: | 1.0 C | Cur Fit: | Linear |
| | STD 2: | 2.0 C | Factor: | 1.0 |
| | STD 3: | 3.0 C | Divisor: | 1.0 |
| | Response: | 0.5 sec | Print Data: | Yes |
| | Lamp: | UV/Vis | Plot Standards: | Yes |
| | Back Corr: | Yes | Print Standards: | Yes |
| | Samples/batch: | 0 | Auto method: | No |
| | Start sample: | 1 | Oper ID: | 0000 |
| | Cycles: | 1 | Sample ID: | 0000 |
| | Cycle time: | 0.10 min | | |

| | | | | |
|---|---|---|---|---|
| DEFAULT | Ordinate: | Peak Area | Standards: | No |
| METHOD 5 | Wav Max. | 600.0 nm | Value 1: | 0.5 |
| CONC2/MANUAL | Wav Min. | 500.0 nm | Value 2: | 1.0 |
| | # of standards: | 3 | Value 3: | 1.5 |
| | Conc Units: | C | Cur Fit: | Linear |
| | STD 1: | 1.0 C | Factor: | 1.0 |
| | STD 2: | 2.0 C | Divisor: | 1.0 |
| | STD 3: | 3.0 C | Print Data: | Yes |
| | Speed: | 960 nm/min | Plot Standards: | Yes |
| | Smooth: | 2 nm | Print Standards: | Yes |
| | Lamp: | UV/Vis | Auto Method: | No |
| | Back Corr: | Yes | Oper ID: | 0000 |
| | | | Sample ID: | 0000 |

| | | |
|---|---|---|
| DEFAULT | Date: | 000000 |
| METHOD 999 | Time: | 0000 |
| TIME/DATE | | |

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# 6 OPERATION

## INITIAL STARTUP

**IMPORTANT:** Before starting up the instrument make the following checks:

* Check that both SAMPLE and REFERENCE positions in the compartment are empty, or that sampling systems, if installed, are set up correctly.

* Ensure that there are no absorbing samples in either of the radiation beams.
  Absorbing samples could interfere with the startup routine.

Follow this routine to turn the instrument on:

1) Check that the power switch is **OFF**.
Connect the instrument to the electrical power supply.

2) Turn power switch to **ON**.
Wait for the instrument to complete initialization routine.
The display will show the status display:

**500 NM 0.000ABS**
**INPUT > <**

3) Turn on the printer. Ensure that it is ON-LINE.

4) Call a method by typing in the method number and pressing **ENTER**.
If the user is not sure which methods are in which method files, pressing | . | **HELP** will cause a method directory to be printed out.

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## 6.2 STARTING AN ANALYSIS

The method in the active memory can either be used to carry out an analysis, or the parameters can be reviewed and amended. (Amending parameters is dealt with in Chapter 5).

To start an analysis:

* place the sample and reference cells in the appropriate cell holders in the sample compartment.
* press **START**
* any CALL parameters will now be displayed; amend these if necessary.
* press **START**
* any BATCH parameters will now be displayed; amend these if necessary.
* press **START**
* any START parameters will now be displayed; amend these if necessary.
* press **START**

The analysis will now proceed.

If more than one cycle has been selected then the second cycle will start immediately the first one has been completed; and so on until all the cycles have been completed.

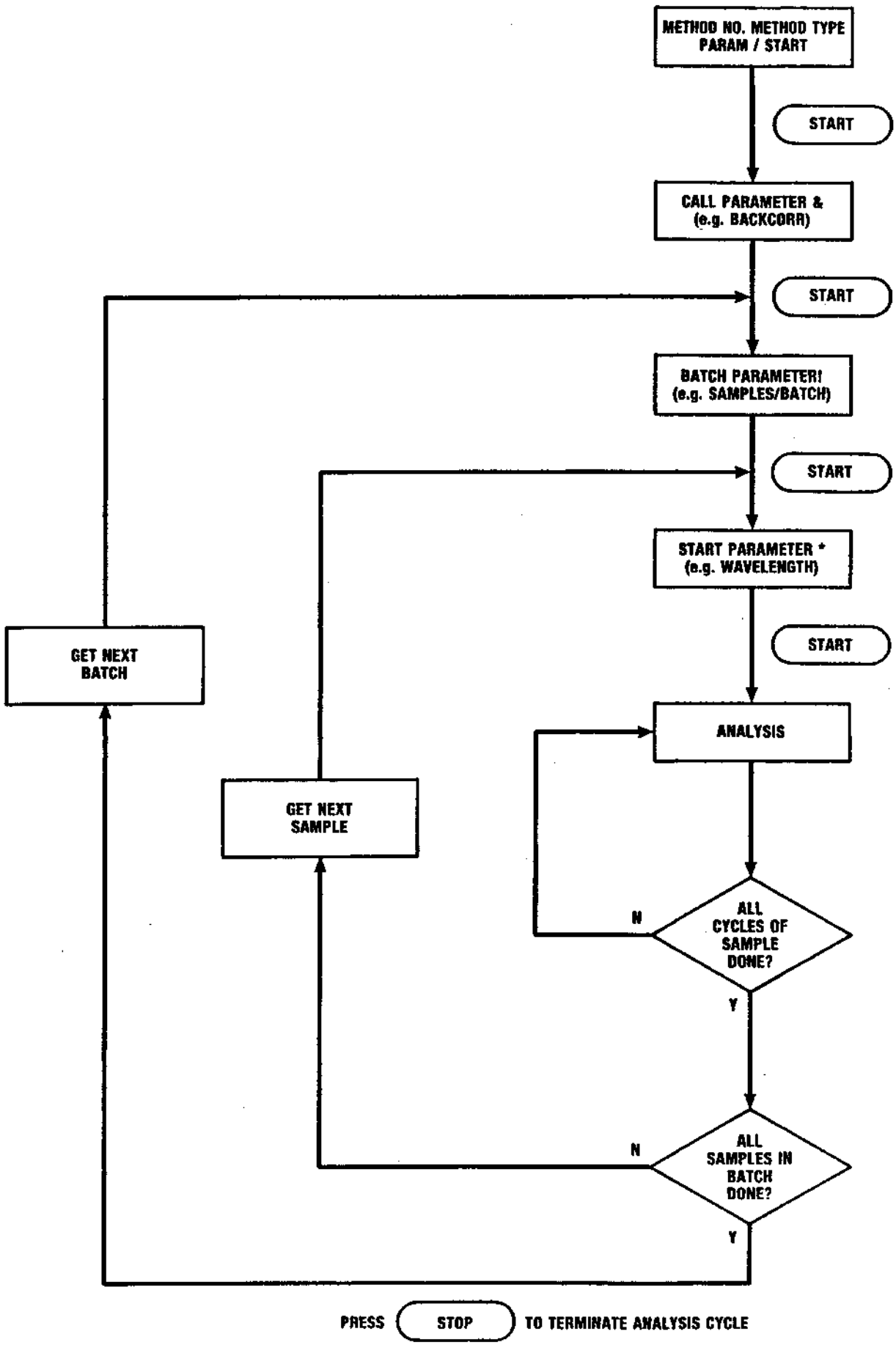
If a batch of more than one is being processed, then, after the analysis of the first sample is complete, the instrument will request the next sample. The next sample must then be placed in the sample cell holder, and **START** pressed. When all samples of a batch have been processed the next batch can be analysed. At the start of a new batch BATCH parameters will be displayed.

**BACK CORR** If BACK CORR = YES, it will be executed when the method is first started.
For Timedrive, WavProg and Conc1 methods, a background correction will be carried out at each of the entered wavelengths. For Scan and Conc2 methods, a baseline correction will be carried out over the wavelength range entered.
For a background correction, place cells containing blank solution in both sample and reference cell holders.
If BACK CORR is tagged then it will be executed dependant upon the tag. (See 5.4.6. and tags 5.4.8.).

**STOPPING AN ANALYSIS** At any time during an analysis, either during measurement or half way through a batch, pressing **STOP** twice will stop the method and return the display to the method header.

The flow diagram on the facing page shows the sequence of events during an analysis run.

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## 6.3 TIMEDRIVE METHOD OPERATION

1) Switch on the instrument as described in section 6.1.1.
Call up a Timedrive method.

2) If necessary amend the method parameters.
A Background correction can be selected dependent upon its tag level.

3) Press **START**, CALL parameters will be displayed for amendment.

4) Press **START**, if batches are being processed the display will read:

BATCH NUMBER 1
PRESS START

Press **START**, BATCH parameters will be displayed for amendment.

5) Press **START**, START parameters will be displayed for amendment.

6) Press **START**, the display will read:

**METHOD 1 SAMPLE 1** Go straight to sample
**PRESS START** X with **X ENTER.**

Place the sample solution in the sample cell holder, close the sample compartment cover and press **START.**
The Spectrometer will begin the analysis.
The display gives the following information:

**TD CYCLES**
**00.00 min xxx ABS**

**TD** indicates the method type running.
**CYCLES** indicates the number of cycles left to run. It will not be displayed when there is 1 cycle to run.
**min** indicates the time left to run for the cycle.
**ABS** indicates the current absorbance.
(Different units will be displayed dependent upon ordinate).

Analytical data will be output at the end of the analysis.

7) Analyze further samples in turn.

8) BATCH OPERATION:
After batch 1 has been analyzed, the instrument will request batch 2. Before analysis of the first sample, a background correction can be selected if BACKCORR is Batch tagged.

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## 6.4 SCAN METHOD OPERATION

1) Turn on the instrument as described in Section 6.1.1.
Call up a Scan method.

2) If necessary amend the method parameters.
A Background correction can be selected dependent upon its tag level.

3) Press **START**, CALL parameters will be displayed for amendment.

4) Press **START**, if batches are being processed the display will read:

BATCH NUMBER 1
PRESS START

Press **START**, BATCH parameters will be displayed for amendment.

5) Press **START**, START parameters will be displayed for amendment.

6) Press **START**, the display will read:

**METHOD 2 SAMPLE 1** Go straight to sample
**PRESS START** X with **X ENTER**.

Place the sample solution in the sample cell holder, close the sample compartment cover and press **START**.
The Spectrometer will begin the analysis.
The display gives the following information:

**SCAN CYCLES**
**0000 nm xxx ABS**

**SCAN** indicates the method type running.
**CYCLES** indicates the number of cycles left to run. It will not be displayed when there is 1 cycle to run.
**nm** indicates the wavelength currently being scanned.
(If the scan ends before the cycle time then time remaining will be displayed).
**ABS** indicates the absorbance at the wavelength shown.
(Different units will be displayed dependent upon ordinate).

Graphic output will be printed during the analysis run.
Analytical data will be output at the end of the analysis.

7) Analyze further samples in turn.

8) BATCH OPERATION:
After batch 1 has been analyzed, the instrument will request batch 2. Before analysis of the first sample, a baseline correction can be selected if BACKCORR is Batch tagged.

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## 6.5 WAVPROG METHOD OPERATION

1) Switch on the instrument as described in section 6.1.1.
Call up a WavProg method.

2) If necessary amend the parameters.
A Background correction can be selected dependent upon its tag level.

3) Press **START**, CALL parameters will be displayed for amendment.

4) Press **START**, if batches are being processed the display will read:

BATCH NUMBER 1
PRESS START

Press **START**, BATCH parameters will be displayed for amendment.

5) Press **START**, START parameters will be displayed for amendment.

6) Press **START**, the display will read:

**METHOD 3 SAMPLE 1** Go straight to sample
**PRESS START** X with **X ENTER**.

Place the sample solution in the sample cell holder, close the sample compartment cover and press **START**.
The Spectrometer will begin the analysis.
The display gives the following information:

**WAVP CYCLES**
**XXX nm y.yyyABS**

**WAVP** indicates the type of method running.
**CYCLES** indicates the number of cycles left to run. It will not be displayed if there is 1 cycle to run.
**nm** indicates the wavelength being scanned.
**ABS** indicates the current absorbance.
(Different units will be displayed dependent upon ordinate.
%T for Percent Transmission,
RAT for ratio operation,
DIF for difference operation).

Analytical data will be output at the end of the analysis.

6) Analyze further samples in turn.

7) BATCH OPERATION:
After batch 1 has been analyzed, the instrument will request batch 2. Before analysis of the first sample, a background correction can be selected if BACKCORR is Batch tagged.

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## 6.6 CONC METHOD OPERATION

**General**

Operation of Conc methods is as straightforward as the previous methods. However, setting the parameters to suitable values does require care.
Before setting up the method, it is advisable to first scan the spectrum of the sample to find suitable absorbance bands upon which calculation can be based.

If calibration data, concentrations and absorbance, are already known these can be entered when the method is being set up. By setting the parameter STANDARDS to NO, then sample analysis can begin immediately.
If the analytical curve calculated from one set of standards is to be used repeatedly, setting STANDARDS to NO will mean that standards do not have to be analyzed each time the method is run.
It may be helpful to set STANDARDS as a Tagged Parameter.

**STANDARDS AND CURVES**

Should a standard not lie on the calculated curve, it is possible to recalculate the curve using a different curve fit.
Set STANDARDS to NO, change the curve fit and start the method. The data will be fitted to the new curve algorithm.
Alternatively, it is possible to reanalyse that standard again and the curve be recalculated.
To reanalyse one standard:

1. Ensure that STANDARDS = YES, start the method.

2. The display will show:

   **STD 1 (conc)**
   **PRESS START**

3. Type in the number, X, of the standard to be reanalysed, press **ENTER**. The display will read:

   **STD X (conc)**
   **PRESS START**

4. Place standard X in the sample compartment and press **START**.

5. After analysis press **STOP**, set STANDARDS to NO, and start the method. If the curve is satisfactory, samples can be analyzed. If the curve is still unsatisfactory, repeat steps 1 to 4.

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**REMOVING STANDARDS**

A Standard that does not lie on the curve can also be removed from the method. This can be done in 2 ways:

1. If the curve is linear, passing through the origin, the incorrect standard can be deleted by entering the concentration of the standard as 0.000 and the value as 0.000. In this way the incorrect standard is deleted.

2. If the curve is not linear reduce the number of standards by 1. When this is done the last standard will be deleted. Enter the concentration and value of this deleted standard in place of the incorrect standard X. In this way the incorrect is deleted.

**BACK CORR**

Normally a Back Corr will only be presented for amendment dependant upon its tag.
In Conc methods however, Back Corr is presented for amendment before the standards analysis, regardless of the tag set on Back Corr. This contributes to more reliability since a background correction can always be carried out before standard analysis.

More detailed information about individual parameters can be found in chapter 5.
Specific information concerning operation with the various Conc methods can be found on the following pages.

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## 6.6.1.
## CONC 1 METHOD OPERATION

**CALCULATION**
**WAVELENGTH**
* **Scan the spectrum** of the sample using a SCAN method.
* **Choose a strong absorption band:**
The wavelength at the peak apex will be WAVEL.1 for all analysis methods.

**DELTA ABS**
If the baseline is flat but offset, choose a second wavelength, WAVEL.2, where the baseline has a minimum, to compensate for the baseline offset.

**3WL ANALYS**
If the baseline is sloping, choose a second and a third wavelength at the beginning and end of the peak, WAVEL.2 and WAVEL.3, to compensate for the sloping baseline. See Section 5 for details.

**METHOD MODIFICATION**
* **Call up a CONC1 method** or create a new CONC 1 method. Select to **MODIFY the parameters.**

* **Select the ordinate mode** ABS, DELTA ABS or 3WL ANALYS.

* **Enter the analysis wavelengths.** WAVEL.1 is the peak apex wavelength, WAVEL.2 and WAVEL.3 are the secondary wavelengths to compensate for baseline offsets.

**STANDARDS**
* **Enter the number of standards and the concentrations** of each standard.

* **Enter known absorbance values,** in the VALUES parameter line. Set the STANDARDS line to NO.
**For unknown absorbance values,** ignore the VALUES parameter and set STANDARDS to YES, standards' absorbance will be measured during analysis.

* **Set further parameters to their required value.**
Press **STOP** to return to the method header.

(cont'd)



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**ANALYSIS**

* Press **START**, if STANDARDS was set to YES, the first standard solution will be requested:

**STD 1 [xxx]**
**PRESS START**

**Place the first standard in the sample cell holder,** press **START.**
Once the first standard has been analyzed the second standard will be requested, and so on.

* If PLOTSTANDARDS was set to YES, the calculated standard curve will now be printed out.
(If STANDARDS was set to NO, then the standard curve will be output immediately **START** is pressed).

* The instrument will request the first sample:

**METHOD 6 SAMPLE 1**
**PRESS START**

Place the sample in the sample cell holder, press **START.**
Further samples will be requested as the instrument requires them.
Sample concentration will be printed out after each analysis, if PRINTDATA is set to YES.

**NOTE:** A CONC method is stored complete with its calibration data. If the method is to be used repeatedly with the same calibration data, set the STANDARDS parameter to NO.
When the method is called up, analysis can begin immediately, without having to enter values or analyze any standards.



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## CONC2 METHOD OPERATION

Peak Area and Total Peak

**CALCULATION WAVELENGTH**

* **Scan the spectrum** of the sample using a SCAN method.
* **Select a strong absorption band.**
  The wavelengths corresponding to the start and end of the peak will be WAV.MAX and WAV.MIN.

**METHOD MODIFICATION**

* **Call up a CONC2 method** or create a new CONC2 method.
  Select to **MODIFY the parameters.**
* Select the required **ordinate mode.**
* **Enter the wavelength range** within which the peak area is to be calculated. (WAV.MAX and WAV.MIN).

**STANDARDS**

* **Enter the number of standards and the concentrations** of each standard.
* **Enter known absorbance values,** in the VALUES parameter line. Set the STANDARDS line to NO.
  **For unknown absorbance values,** ignore the VALUES parameter and set STANDARDS to YES, standards' absorbance will be measured during analysis.
* **Set further parameters to their required values.**
  Press **STOP** to return to the method header.

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**ANALYSIS**

* Press **START**, if STANDARDS was set to YES, the first standard solution will be requested:

**STD 1 [xxx]**
**PRESS START**

**Place the first standard in the sample cell holder,** press **START.**
Once the first standard has been analyzed the second standard will be requested, and so on.

* If PLOTSTANDARDS was set to YES, then the standard curve will now be printed out.
(If STANDARDS was set to NO, then the standard curve will be output immediately **START** is pressed).

* The instrument will request the first sample:

**METHOD 6 SAMPLE 1**
**PRESS START**

Place the sample in the sample cell holder, press **START.**
Further samples will be requested as the instrument requires them.
Sample concentration will be printed out after each analysis, if PRINTDATA has been set to YES.

**NOTE:** A CONC method is stored complete with its calibration data. If the method is to be used repeatedly with the same calibration data, set the STANDARDS parameter to NO.
When the method is called up, analysis can begin immediately, without having to enter values or analyze any standards.



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**CONC 2 METHOD OPERATION**

Deriv 2 Fix and Deriv 2 Peak

**CALCULATION WAVELENGTHS**

* **Scan the spectrum** of the sample with Abs ordinate. Select a strong absorption band.

* **With D2 ordinate, scan the absorption peak closely.** With PRINTDATA = YES the maxima and minima of the derivative spectrum will be printed out.

* **Select the largest peak** and note the wavelengths. These will be CALC.WAV. 1 + 2. (CALC.WAV. 1 the higher wavelength).

DERIV 2 PEAK

(Scan the most dilute standard with D2 ordinate. Note the size of the peak in the derivative peak in the spectrum. Set the CONC2 threshold a little below this value).

**METHOD MODIFICATION**

* **Call a CONC2 method,** or create a new CONC2 method. Select to **MODIFY the method.**

* **Select the required ordinate mode.**

* DERIV 2 PEAK operation requires entry of a threshold value. This is entered immediately after the ordinate.

* Enter the wavelength range over which the samples are to be scanned.

* **Enter CALC.WAV. 1 and 2,** the wavelengths at which the derivative is to be calculated.

**STANDARDS**

* **Enter the number of standards and the concentrations** of each standard.

* **Enter known absorbance values,** in the VALUES parameter line. Set the STANDARDS line to NO.
**For unknown absorbance values**, ignore the VALUES parameter and set STANDARDS to YES, standards' absorbance will be measured during analysis.

* **Set further parameters to their required values.** Press **STOP** to return to the method header.



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**ANALYSIS**

* Press **START**, if STANDARDS was set to YES, the first standard solution will be requested:

**STD 1 [xxx]**
**PRESS START**

**Place the first standard in the sample cell holder,** press **START.**
Once the first standard has been analyzed, the second standard will be requested, and so on.

* If PLOTSTANDARDS is set to YES the calculated standard curve will be printed out.
(If STANDARDS was set to NO then the standard curve will be output immediately **START** is pressed).

* The instrument will request the first sample:

**METHOD 6 SAMPLE 1**
**PRESS START**

Place the sample in the sample cell holder, press **START**
Further samples will be requested as the instrument requires them.
Sample concentration will be printed out after each analysis, if PRINTDATA is set to YES.

**NOTE:** A CONC method is stored complete with its calibration data. If the method is to be used repeatedly with the same calibration data, set the STANDARDS parameter to NO.
When the method is called up, analysis can begin immediately, without having to enter values or analyze any standards.

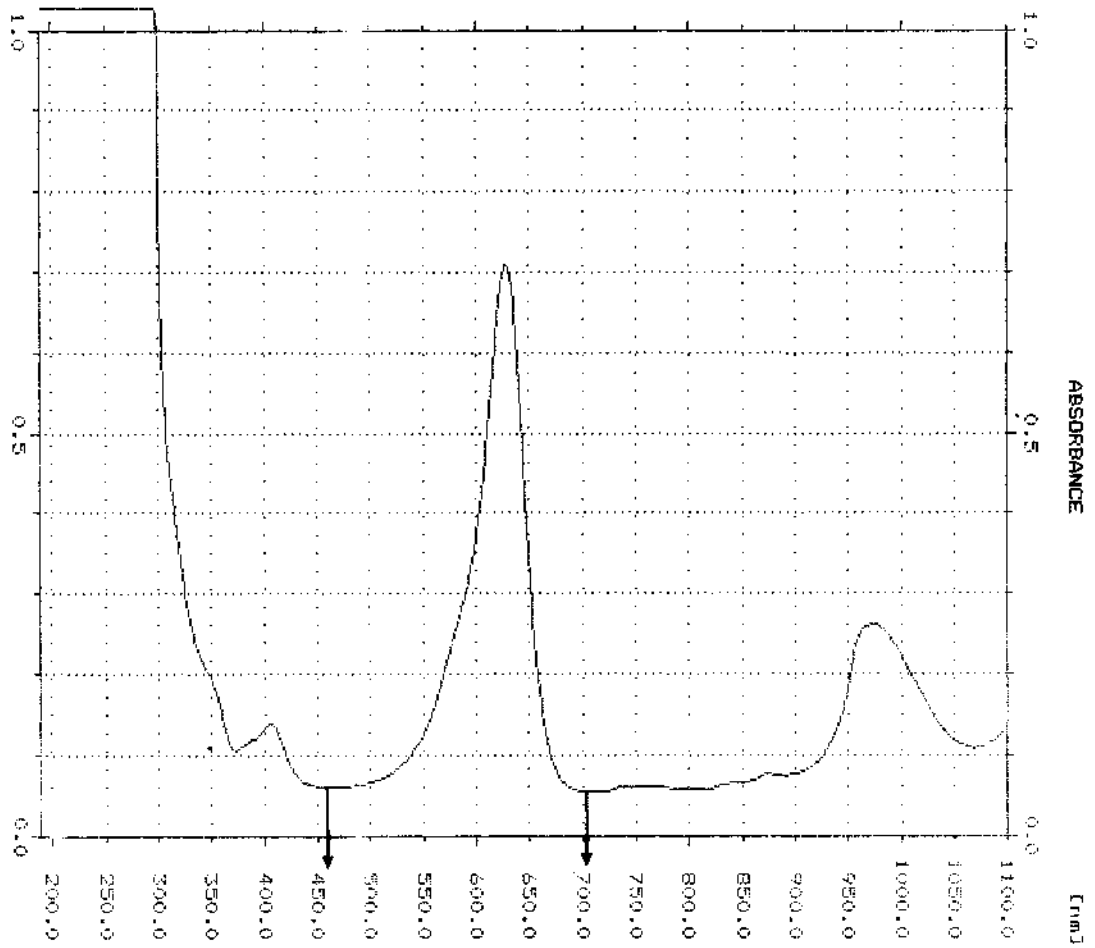


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The following is an example of setting up a Conc 2 method with Deriv 2 Peak ordinate. First optimum parameters are found by scanning Absorbance and derivative spectra, the Conc 2 method is then set up and utilized.

**SCAN THE STANDARD**

First scan the standard over a wide wavelength range to identify suitable absorption bands.



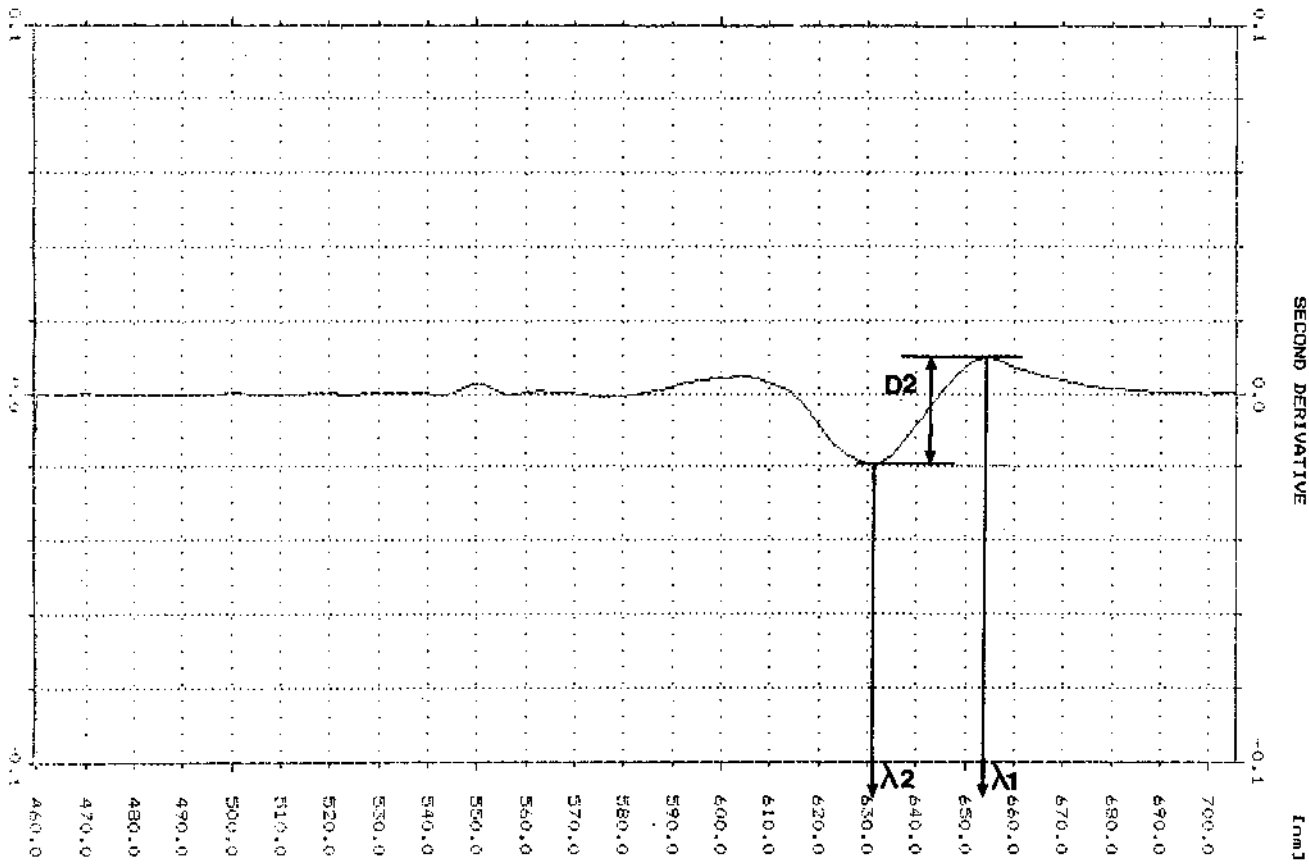

Note the wavelengths corresponding to the beginning and end of the band of interest:

| WAVELENGTH | DATA |
|---|---|
| 974.4 nm (MAX) | 0.263 ABS |
| **705.6 nm (MIN)** | **0.056 ABS** |
| 628.8 nm (MAX) | 0.709 ABS |
| **460.0 nm (MIN)** | **0.063 ABS** |
| 292.0 nm (MAX) | 4.213 ABS |
| 287.2 nm (MIN) | 3.858 ABS |
| 281.6 nm (MAX) | 4.321 ABS |

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**DERIVATIVE SPECTRUM**

Now scan the second derivative spectrum of the standard, with a wavelength range corresponding to the beginning and end of the band of interest. Set the PRINT DATA parameter to YES so that data about the peaks is output.




| | WAVELENGTH | DATA |
|---|---|---|
| λ1 | 656.2 nm (MAX) | 0.010 D2 |
| λ2 | 630.8 nm (MIN) | -0.020 D2 |
| | 606.2 nm (MAX) | 0.005 D2 |
| | 556.6 nm (MIN) | -0.001 D2 |
| | 550.6 nm (MAX) | 0.006 D2 |

From the derivative spectrum choose the largest peak to trough difference, D2. Refer to the analytical data printed after the spectrum to find the wavelengths at which the peak and trough occur. These wavelengths will be CAL.WAV. 1 and 2.

Scan the 2nd derivative spectrum of the most dilute standard. Note the magnitude of the derivative peak chosen for calculation. The threshold value in the Conc2 method should be set a little below this noted value.

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**SETTING THE METHOD PARAMETERS**

Set the relevant method parameters to the values chosen above.

The scanned wavelength range must be larger than the range covered by Calc.Wav. 1 and 2.

```
Calc.Wav. 1 =    1 = 656.2 nm          Wav.Max. = 700 nm
Calc.Wav. 2 =    2 = 630.8 nm          Wav.Min. = 600 nm
Threshold   =      0.005.
```

```
PERKIN-ELMER
LAMBDA 2 UV/VIS SPECTROMETER

METH  5        CONC 2


SAMPLE ID    ...................

OPERATOR ID  ...................
```

| | | | |
|---|---|---|---|
| WAV. MAX | 700.0NM | ORD | DERIV2 PEAK |
| WAV. MIN | 600.0NM | THRESHOLD | 0.005 D2 |
| # OF STANDARDS | 3 | CALC.WAV 1 | 656.2NM |
| STD 1 | 5.0 C | CALC.WAV 2 | 630.8NM |
| STD 2 | 15.0 C | CONC UNITS | C |
| STD 3 | 25.0 C | STANDARDS | YES |
| SPEED | 480 NM/MIN | VALUE 1 | 0.0125 |
| BANDWIDTH | 10 NM | VALUE 2 | 0.0396 |
| LAMP | UV+VIS | VALUE 3 | 0.0624 |
| BACK CORR | NO | CUR FIT QUAD | INTERC |
| SAMPLES/BATCH | 0 | FACTOR | 1 |
| START SAMPLE | 1 | DIVISOR | 1 |
| CYCLES | 1 | PRINT DATA | YES |
| CYCLE-TIME | 0.1MIN | PLOT STANDARDS | YES |
| | | PRINT STANDARDS | YES |
| | | AUTO METHOD | NO |
| | | OPER. ID | 0000 |
| | | SAMPLE ID | 0000 |

(B2131-A1/R)

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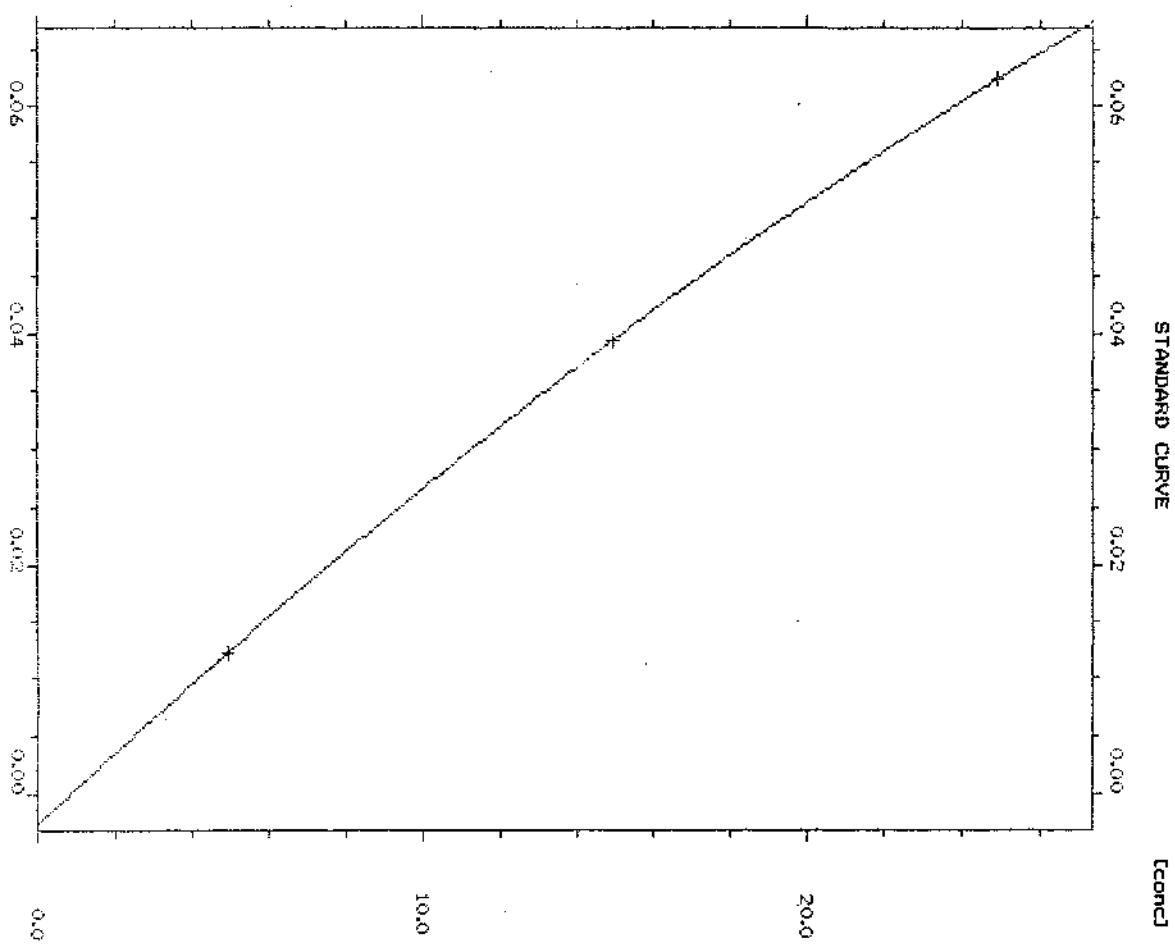
**STANDARD MEASUREMENT**

Start the Conc2 method. The instrument will request the standards. After the standards have been analyzed, the standard curve will be output with curve parameters.

```
STANDARD CURVE PARAMETERS :
     ABSC          0.000 -   27.500
     ORD          -0.003 -    0.067
     PRMS      -0.00266 ,  0.00314 , -0.00002
     RESID ERR   0.00000


SCAN - LIMITS     : 600.0 nm   700.0 nm
MEASUREMENT WL'S : 656.2 nm   630.8 nm

STD.-NR.    TIME          DERIV2          CONCENTRATION
   01       00:28         0.0125           5.0000
   02       00:28         0.0396          15.0000
   03       00:28         0.0624          25.0000
```




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**SAMPLE ANALYSIS**

Samples can now be analyzed.

Results:

FACTOR : 1.000000
DIVISOR : 1.000000

THRESHOLD : 0.005

| SAMPLE | CYCLE | DERIV2 | CONCENTRATION |
|---|---|---|---|
| 001 | 00:29 | 0.022 | 8.376 C |
| 002 | 00:30 | 0.038 | 14.558 C |
| 003 | 00:30 | 0.052 | 20.297 C |
| 004 | 00:31 | 0.022 | 8.263 C |
| 005 | 00:32 | 0.038 | 14.446 C |
| 006 | 00:32 | 0.053 | 20.433 C |

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## 6.7 OPERATION WITH ACCESSORIES

**GENERAL**

Operation with accessories is as straight forward as manual operation.
Details of individual accessory operation can be found in the accessory manuals.

Care should be taken when using cell changers however; depending on the tag status of the Back Corr parameter, not all positions in the cell changer can be used.

**BACK CORR FIX/CALL TAG**

Blank solution for the background correction should be placed in position 1.
Thereafter, all positions can be used for sample analysis.

**BACK CORR BATCH TAG**

Blank solution for the background correction should be placed in position 1.
If Back Corr = Yes then position 1 cannot be used for sample analysis.
If Back Corr = No then all positions can be used for sample analysis.

**BACK CORR START TAG**

Blank solution for the background correction should be placed in position 1.
Sample solutions can only be placed in positions 2 onwards, regardless of whether Back Corr is on or off.

### 6.7.1 CONC METHOD OPERATION WITH ACCESSORIES

Care must be taken regarding cell positions when placing standards in the cell changer, and also dependent upon the Back Corr tag status.
When a back corr is to be carried out blank solution should always be placed in position 1.
When standards are placed in the cell changer for analysis, all positions in the cell changer can be used.
Standard 1 should be placed in position 1 and so on.
If 10 standards are used with a 6x6 cell changer, after the first 6 standards have been analyzed, then standard 7 should be placed in position 1 and so on.

The Back Corr tag conditions above apply to conc methods too.

As with all conc methods, if Back Corr = Yes then it will be carried out before the Standards analysis regardless of the tags allocated to the two parameters Back Corr and Standards.

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## 7.1 UNUSUAL SAMPLES

If a sample is chemically stable and undergoes no physical or chemical change other than to absorb incident radiation, errors in photometric values should not be caused by the sample. Many samples are not this stable, and special consideration must be given to them.

**VOLATILE LIQUIDS**

Some sample solutions containing volatile components can vary in concentration during recording. If this occurs, the resulting photometric data will lack reproducibility. When such a sample is to be analyzed, use stoppered cells.

**SAMPLES NOT OBEYING THE BEER-LAMBERT LAW**

Quantitative analyses utilizing the absorption of radiation are based on the assumption that sample absorptance remains constant with changes in concentration. For some samples this assumption is incorrect. It should be borne in mind that absorptance can change during sample preparation, depending upon the volume of reagent added for color development or precipitation. For details, refer to reference books dealing with these subjects. Since absorptance is also temperature dependent to varying degrees, samples should be checked for effect if non-repeatable results are obtained. If a sample has a highly temperature-dependent absorptance, wait until thermal equilibrium is attained, or else use a thermostatted cell or cell holder to maintain constant temperature.

**CHEMICALLY REACTIVE SAMPLES**

In the event that a reaction has taken place between the sample material and the solvent in the cell, spectral data based on that sample cannot always be expected to have sufficient stability or reproducibility. For these samples, use of a quantative analysis that takes advantage of the change in transmittance may yield better results. For details refer to reference books dealing with this specific subject.

**PHOTO-ACTIVE SAMPLES**

Some samples are known to be photo-active in that they fluoresce upon absorbing light. Since a small amount of the fluorescence will be measured by the detector, a higher apparent transmittance will often result. Samples are also known to exist that will undergo photochemical reactions as they absorb light. With these samples, which are mostly biochemical, lack of reproducibility will characterize the resultant data.

**OTHER SAMPLE PROPERTIES**

Samples that are polarizing in nature, or have a double index of refraction, are often difficult to measure accurately. The emerging monochromatic light is slightly polarized because of having been refracted. Thin-film samples also pose a problem, since optical interferences may develop, causing a regular interference pattern to be superimposed on the spectral curve.

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## 7.2 SOLVENT - SAMPLE RELATIONSHIP

Apart from the ability of the solvent to dissolve the sample without reacting with it, the following requirements should be met:

* The radiation absorption in the scanning region should be small. High absorption by the blank reduces the reference energy, thus increasing noise.
  The use of solvents showing extreme absorption should, however, be avoided.

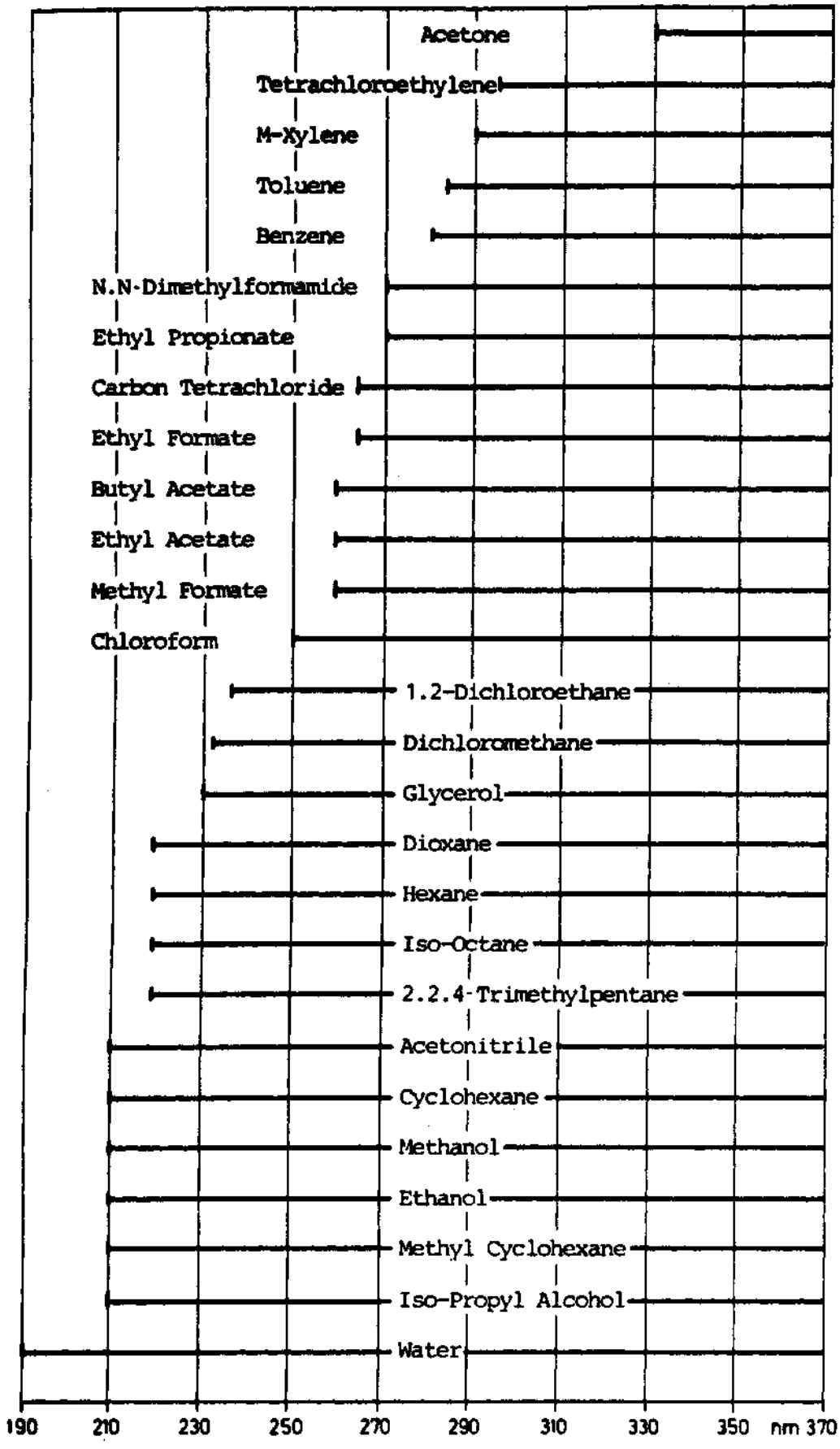
* Evaporation should be fairly low at ambient temperature.

Table 7-1 shows the usable wavelength limits of a number of commonly used solvents. The lower limit has been defined as that at which 10 mm of solvent has a transmittance of 10%.

**NOTE:** In general, aromatic compounds exhibit high absorbance in the UV region and hence are not suitable as solvents for these wavelengths.
Whenever a solvent of unknown absorbance is to be used, its spectrum should first be scanned to determine the useful wavelength range.

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Table 7-1. Usable Wavelength Limits of Solvents




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## 7.3 MICROCELL ALIGNMENT

When using microcells it is essential that the cell holders are aligned to allow the maximum possible transmission of radiation.

To carry out the alignment:

* Using the GOTO LAMBDA key slew the monochromator to 0.0 nm. (Zero order, white radiation).

* Close off the exit windows on the right hand side of the sample compartment with a piece of card.

* Place a microcell in each of the cell holders.
  The cells must be pushed down fully.
  The cells should be filled with a low absorbing solvent, (e.g. water or ethanol).

**NOTE:** Alignment is effective for each cell holder/microcell combination only. The same microcell must always be placed in the cell holder in which it was aligned.
It is recommended that the cells be marked SAMPLE and REFERENCE using adhesive labels.

* Holding a piece of matt white paper behind the cell holder, visually check horizontal alignment of the light beam in the cell sample area.

* If the light beam is not centred exactly, loosen the two locking screws on the cell holder, and with a small screw-driver, rotate the horizontal adjustment screw right or left to center the light beam. Tighten the two locking screws.

* Visually check the vertical alignment of the light beam in the cell sample area. Correct alignment is achieved when the light beam is just above (2 - 3 mm) the floor of the cell sample area.
  Vertically align the cell using the lifter at the rear of the cell holder. Turn the screw clockwise to raise the cell, anticlockwise to lower the cell.

* Remove the card blocking the exit windows and close the sample compartment cover.

* Slew the monochromator to any wavelength above 190 nm.

This completes the microcell alignment procedure.

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## 7.4 ERROR MESSAGES

**DON'T PROTECT ALL METHODS**

The instrument must always have access to at least one method. If all methods are protected then the instrument cannot function. This error message appears when an attempt is made to ALL protect all methods.
Set some other protection on this last method.

**DON'T PROTECT ALL BRANCHES**

If all branches are ALL protected then there is no access to any branch and the instrument cannot function.
This error message appears when an attempt is made to ALL protect a final branch when all other branches have ALL Protect.
Set some other protection on this final branch.

**ERROR: LAST METHOD**

The instrument must always have at least one stored method in each branch. This message is displayed if an attempt is made to delete the last method stored in a branch.

**PROBLEM: ACCESSORY NO INITIALIZATION**

This error message is displayed during instrument startup when the instrument gets no response from the fitted accessory. This may be due to the fact that the accessory is not properly plugged in, or not powered up. A less likely cause is a mechanical fault in the accessory stopping the normal functioning.

**PROBLEM: BRANCH WRITE PROTECTED**

If a branch is write protected then no methods in that branch can be changed.
This error message appears when an attempt is made to change one of the methods.
To be able to change method parameters, switch on the instrument in super user mode and change the branch protection.

**PROBLEM: CASSETTE SENSOR MALFUNCTION**

When the cassette slot is opened, a sensor is activated and the instrument waits for the cassette. If the sensor is defective this will be noticed during startup and this error message is displayed.

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**PROBLEM: MARK NOT SET**

This error message appears when COPY FROM MARK in the New Method Options is activated but no mark has been set.
Set a mark first and then use Copy From Mark.

**PROBLEM: METHOD NO. LIMITS 1 - 999**

Methods can only be numbered from 1 - 999.
This error message is displayed when an attempt is made to call a method number outside these limits.
Call a method number within the limits.

**PROBLEM: METHOD NOT FOUND**

A mark has been set, but the marked method can no longer be found, e.g. a cassette method has been marked but the cassette is removed before the method has been copied.
Replace the cassette and redo copy from the start.

**PROBLEM:METHOD PROTECTED**

The method number called exists but is ALL protected and hence cannot be accessed.
To access the method, change the method protection while in SuperUser mode.

**PROBLEM: NO CASSETTE**

A cassette method is called (2, -, n,), but no cassette is plugged in.
Plug in a cassette and recall the method, or use a method stored in the instrument register.

**PROBLEM: NO ENERGY REFERENCE**

Not enough energy is being transmitted.
Possible causes are:
Lamps not illuminated due to lose connection.
Lamps are burnt out.
The radiation beam is obstructed in the sample compartment.
The detector is defective.

**PROBLEM: NO ENERGY UV LAMP**

Not enough energy is being transmitted.
Possible cause:
Lamp connection is lose.
Lamp is burnt out.
Radiation obstructed in sample compartment.
The detector is defective.

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**PROBLEM: NO ENERGY VIS LAMP**

Not enough energy is being transmitted.
Possible cause:
Lamp connection is lose.
Lamp is burnt out.
Radiation beam obstructed in the sample compartment.
The detector is defective.

**PROBLEM: TRANSFER NOT ALLOWED**

Some specialized methods on cassette cannot be copied over into the instrument method register.
This error message appears when this is attempted.
These methods can only operate when the cassette is plugged into the instrument.

**SYSTEM ERROR**

When an internal system error occurs, the instrument operating software "crashes" and a full reset is automatically carried out. After the reset is complete a System Error message is shown on the display. Make a note of this message.
After the full reset the instrument should operate without problem. Press PARAMETER to continue after the system error has been displayed.
However, if further operation is not possible, contact your nearest Perkin-Elmer service representative and inform him of the error message which has been displayed. This will help him to solve the problem as quickly as is possible.

**PROBLEM: DIRECTORY FULL**

It was attempted to create a new method in a software branch. However, the method allocation for that branch is full and hence no more methods can be created.

Example: Twenty methods are already stored in the Laboratory Branch. An attempt is made (using the new method options) to create another new method. This is not possible since the branch is full and the above error message will be displayed.
If a new method is required, you should overwrite an existing method that is no longer needed.

**PROBLEM: MEMORY FULL**

If the majority of stored methods are CONC2 methods that use a large number of standards, it is possible that the available memory space will be filled before 20 methods (or 100 methods for cartridge) have actually been stored. If it is attempted to create or store new methods, this error message will be displayed. If this occurs, you should overwrite an existing method that is no longer needed.



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Some error messages are output on the printer:

**TIMEDRIVE/SCAN/WAVPROG METHODS**

**Baseline Corrector Data Do Not Fit.**
**Start Baseline Correction!**

This error message appears during Timdrive/Scan/Wavprog method operation.
The data relating to a baseline correction in a scan method and an autozero in a timedrive/wavprog method are interlinked. Hence if a baseline correction is recorded for a scan method, but then an autozero is recorded for a timedrive/wavprog method, the first baseline correction will no longer be valid. Hence, to run another scan method a new baseline correction must be run first.
Alternately, if an autozero is recorded for a timedrive method and then a baseline correction for a scan method, the first A/Z will no longer be valid.

**Wavelength Data Do Not Fit.**
**Start Background Correction!**

This error message appears during wavprog method operation. When background correction has been recorded for all of one set of wavelengths this background correction will no longer be valid if the set of wavelengths is changed. A new background correction must be recorded for the new wavelength set.

**CONC METHODS**

**Value Not Within Valid Limits.**
**Check Standards or Change Curve Fit Algorithm!**

The measured value for the sample is outside the range covered by the standard analytical curve. By including further standards in the curve the sample value may then fall within the range.
Alternately, changing the curve fit algorithm may also cause the sample to be accomadated by the curve.

**Cannot Approximate Standard Curve.**
**Check Standards or Change Curve Fit Algorithm!**

Non-linearity of the samples is so great that a curve cannot be fitted by the present curve fit algorithm. Try a different curve fit. E.g. if only one standard is measured a quadratic curve fit cannot be used, a linear curve fit must be chosen.

**Two Solutions For 1 ABS Value.**
**Change Curve Fit Algorithm!**

Due to non-linear absorption, two samples of different concentration may exhibit the same absorbance. This cannot be fitted by a linear algorithm. Use a quadratic curve fit.

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**CONC 2 DERIV2 PEAK OPERATION**

**More Than One Peak Within Wavelength Limits.**
**Change Threshold or Measurement Wavelengths.**

The peak detector has detected more then one peak within the wavelength range. This problem can be eliminated by increasing the threshold value so only one of the peaks is detected, or the wavelength range can be narrowed so that only one peak is found in the wavelength range.

**No Peaks Detected.**
**Change Threshold**

No peaks were detected by the peak detector. Reduce the threshold value so that the peaks are detected.

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# 8 FURTHER INSTRUMENT FUNCTIONS

In everyday use the user only sees the Laboratory branch of the instrument software.
However in SuperUser mode it is possible to set method "protection" and to access other branches.
Some branches may occassionally be of interest to the user, e.g. for setting accessory configuration.
Other branches are for service purposes, the user has access to these but cannot use the methods stored.

SuperUser mode, setting protection, and other branches are described in the following sections.

## 8.1 SUPERUSER MODE

To operate the instrument in SuperUser mode, the switch S1 on the main circuit board must be at position 8. (At any other position SuperUser mode cannot be accessed).
Now access SuperUser mode as follows:

* Before switching the instrument on, depress the keys | 1 | 5 | - all at once, and keep them depressed while the instrument is switched on.
* Release the keys about 5 seconds after the software version is shown on the display.
* Once initialization is complete the instrument is in SuperUser mode.

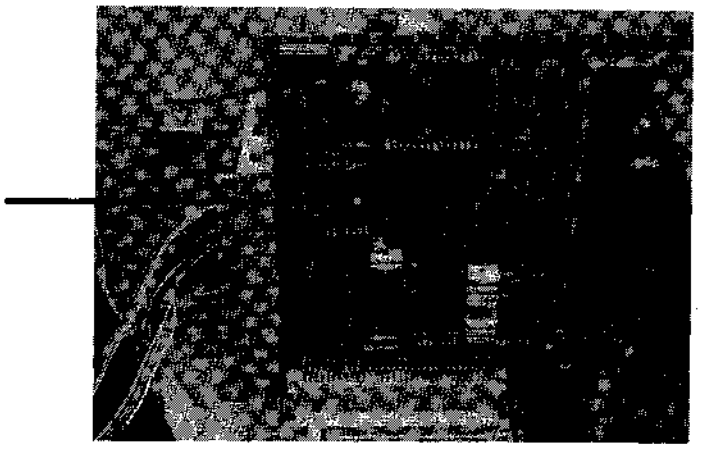


**Figure 8-1. Main Circuit Board and Switch S1**

**WARNING:** Disconnect the instrument from the electrical supply before opening the cover.
Do not touch any components within the instrument.
The settings on switch S1 should only be adjusted with a small screwdriver.

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## 8.2 SETTING PROTECTION

Setting protection on branches and methods will possibly be the most frequent reason for using superuser mode.
Protection can be set on branches and/or methods.
The protection options are:

* Write Protect, preventing method parameters from being overwritten.
* Read/Write Protect, preventing method parameters from being read or overwritten. (RD/WR abbreviation appears on screen).
* Execute Protect, preventing the method from being run. (EXE abbreviation appears on screen).
* All Protect, the method, or branch is inaccessible and cannot be called.

When protection is set on a method the protection applies to that method only.
However, when protection is set on a branch, that protection is valid for all the methods stored in that branch.
Hence, when Read/Write protection is set for a branch, all methods will be read/write protected.
It is possible to override branch protection for individual methods if a higher category protection is set for the method.

e.g. If Read/Write protection has been set for the branch, then methods can be individually set with ALL protection. However, it is not possible to set individual methods with only WRITE protection since this is of a lower category than the Read/Write protection set on the Branch.

If branch protection has been set as ALL, then that branch cannot be accessed.

**LOCKING THE INSTRUMENT**

Through the considered use of protection, it is possible to "lock off" parts of the instrument software for which the everyday user has no requirement.
Indeed, protection can be set such that, for routine sample checking, only the method dedicated to that task is accessible, and that method can only be started.

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**SETTING METHOD PROTECTION**

In superuser mode the protection options for setting method protection are found in the METHOD MODIFICATION options:




When CHANGE PROTECTION appears on the display press **PARAMETER** and the current protection set on the method will be displayed. This protection can be changed by pressing the arrow keys until the required option is scrolled into the display. Press **PARAMETER** to confirm the selection.

**SETTING BRANCH PROTECTION**

In superuser mode the options for setting branch protection can be accessed from the branch header by pressing
**|** . **|** **METHOD**
The display will then show CHANGE PROTECTION .
Press **PARAMETER** and the current branch protection will be displayed. This protection can be changed by pressing the arrow keys to scroll the required option into the display.
Press **PARAMETER** to confirm the selection.
If a branch is no longer ALL protected it can be accessed when SuperUser mode is off.

**LOCKING THE INSTRUMENT**

In order to lock the instrument so that only one method is accessible, and can only be started, follow the procedure below:

* On the branch header level, set branch protection on branches other than the laboratory branch to ALL.
* Set laboratory branch protection to Read/Write protection.
* Access the laboratory branch and set protection on all the methods which are not required to ALL.
* The required method automatically has RD/WR protection since this is the branch protection.
* When the instrument is now powered up out of superuser mode the only task that the user can carry out is to start the method that is read/write protected.

**NOTE:** Any CALL, BATCH and START parameters in the method can be changed when displayed during a method run, regardless of whether the method is Write or Read/Write protected.

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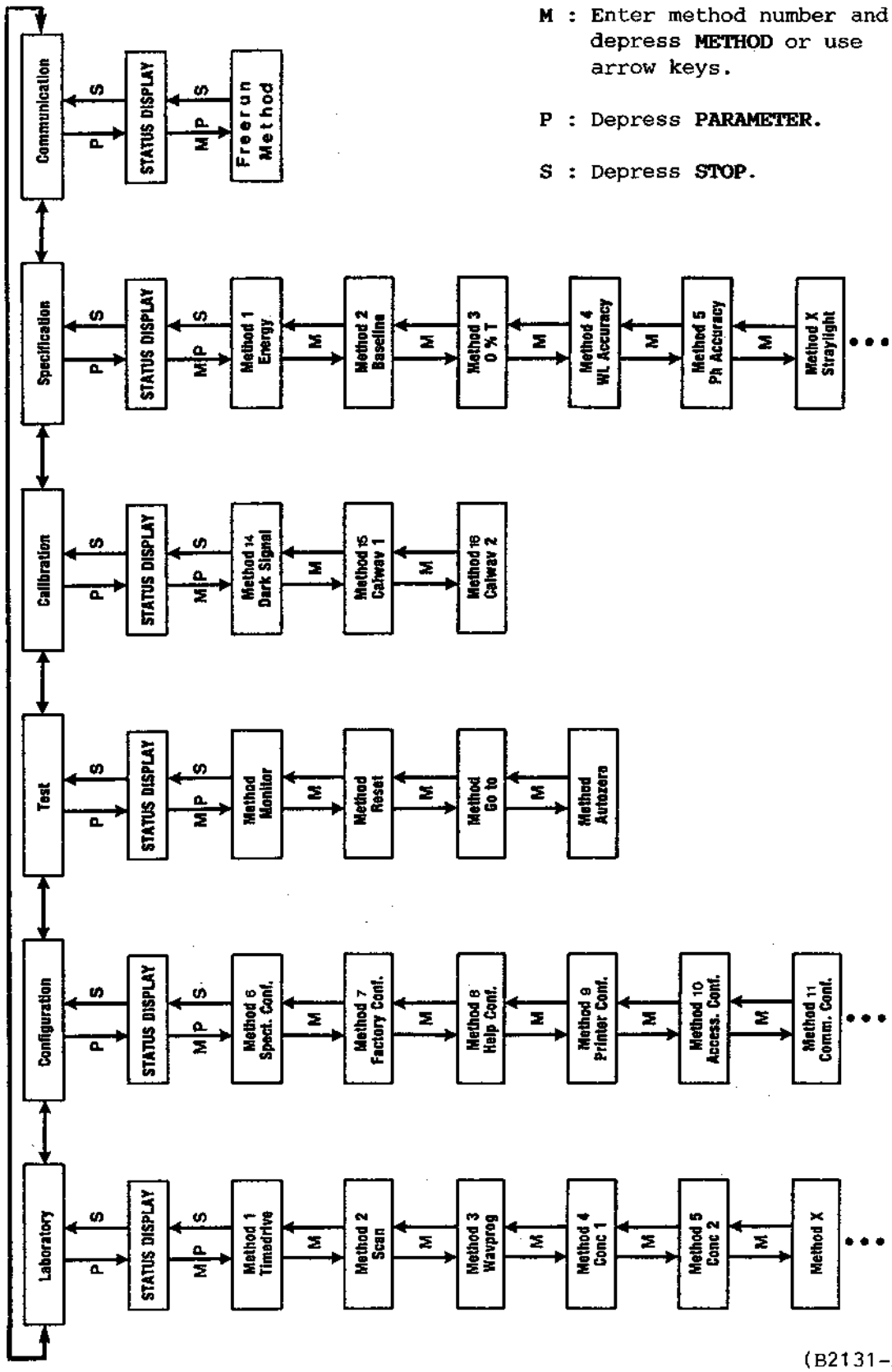
## 8.2.1. EXITING SUPERUSER MODE

In order that any protection set becomes effective, and the everyday user has no opportunity to access other software branches, the instrument must no longer be in superuser mode. The only way to exit superuser mode is to turn the instrument off and then turn it on again without entering the superuser code. While the instrument is turned off, if necessary the setting of switch S1 can be altered so that even use of the code will not access superuser mode.

**WARNING:** Disconnect the instrument from the electrical supply before opening the cover.
Do not touch any of the electrical components within the instrument. Switch S1 settings should only be adjusted with a small screwdriver.

**NOTE:** Turning the instrument on and off too often will shorten lamp-life.
The deuterium lamp will not always re-illuminate if turned on while hot. Hence, it is advisable to allow the lamps to cool for 2 minutes or so before turning the instrument back on.

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## 8.3 OTHER BRANCHES

The other branches of the instrument software contain methods affecting instrument set-up and various diagnostic routines for instrument service.
To access the branches the instrument must be in SuperUser mode. When the status display is shown, press **STOP** and the branch header will be displayed. Different branch headers can be scrolled onto the display with the arrow keys.
When the required branch header is on the display, press **PARAMETER** and the branch status display will be shown.
Methods can be called from here as in the laboratory branch.
It is only possible to amend existing methods. No new methods can be created.

The branches are as follows:

**COMMUNICATION** Method for use when the spectrometer is connected to a PC.

**CALIBRATION** Methods to calibrate the optical system of the instrument. Wavelength calibration and dark current compensation methods.

**CONFIGURATION** Methods allowing various universal instrument parameters to be amended and then incorporated into instrument operation.

**TEST** Diagnostic methods allowing service personnel to check all aspects of instrument operation. The user is not able to use these methods.

**SPECIFICATION** Methods which allow service personnel to check that instrument performance falls within the specified limits.

In everyday use the user should have little need of the functions offered in the above mentioned branches.
The Communication and Configuration branches may be necessary when the instrument is being set up for a specific purpose.

Methods in the TEST and SPECIFICATION branches should only be executed by a Perkin-Elmer Service representative, should a problem arise.

The various branches and methods are described in more detail below.
To get a directory of methods stored in the various branches press | . | **HELP** when a branch header is on the display.

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8.3.1

**COMMUNICATION** This branch stores the operating routines allowing an external computer to operate the spectrometer.
It is recommended that PECSS software (rev.3.1 or higher) be installed in the computer.

**CONNECTING THE PC**

* Before connecting the PC, ensure that PORT USAGE (8.3.3) has been set to 'Computer'.

* Connect the computer to the standard RS-232-C interface at the right of the spectrometer.

* Switch the computer on and call up PECSS.

* Spectrometer operation can now be controlled via the PC operating PECSS software.

**NOTE:** If other software is written for the instrument, it must use the routines contained in the communication branch.



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8.3.2.

**CALIBRATION** Stores methods for instrument calibration, such as dark signal compensation, 1 wavelength calibration and 2 wavelength calibration.

**DARK SIGNAL METHOD 14** A dark signal compensation is carried out when a method is started, and when more than 10 minutes passes since the last one.
The TEST OUTPUT parameter determines whether the result of the compensation is printed out. Set the parameter to YES or NO.
If necessary the compensation routine can be turned off.
E.g. to avoid disturbance during sensitive kinetic analyses.
Set the DARK SIGNAL parameter to NO.
In most circumstances the routine should be left ON.
Each photodiode detector for the sample and reference beams has a residual current flowing through it, even when no radiation is falling on it. This residual current is called the dark signal. The compensation routine ensures that this residual current is accounted for during analysis.

**CALWAV 1 METHOD 15** A routine for wavelength calibration based on a peak at a specified wavelength. The default wavelength is at 0.0 nm.
To check wavelength calibration, scan the spectrum of a known wavelength standard, e.g. holmium oxide.
If the spectrum wavelengths do not correspond to the actual wavelengths then the instrument should be calibrated again.

Calibration can take place using the internal 0.0 nm or the 656.1 nm deuterium emission peak, (selected after AUTOPEAK), or a peak in the spectrum of an external wavelength standard can be used.
If using a wavelength standard:
* select to modify the method.
* select SPEC PEAK using the arrow keys. Press ARAMETER
* OLD PEAK = wavelength for peak in spectrum scanned above.
* NEW PEAK = actual wavelength for this peak.
* press **START** the instrument will be calibrated.

**CALWAV 2 METHOD 16** Wavelength calibration routine based on two peaks at known wavelengths.
To check wavelength calibration, scan the spectrum of a known wavelength standard, e.g. holmium oxide.
If the peak wavelengths do not correspond to the known wavelengths then recalibration is necessary.

Calibration can take place using the two internal peaks at 0.0 nm and at 656.1 nm (AUTO PEAK); or using two symmetrical peaks in the spectrum of a wavelength standard.
If using a wavelength standard:
* Select to modify the parameters.
* Select SPEC PEAK using the arrow keys. Press **PARAMETER**
* OLD PEAK 1 = wavelength of peak 1 in spectrum above.
  NEW PEAK 1 = actual wavelength of peak 1.
* OLD PEAK 2 = wavelength of peak 2 in spectrum above.
  NEW PEAK 2 = actual wavelength of peak 2.
* Press **START**, the instrument will be calibrated.

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8.3.3.

**CONFIGURATION** This branch allows the user to tailor aspects of instrument operation to his own needs.

The following methods are stored in the configuration branch:

Spectrometer configuration.
Factory configuration.
Help configuration.
Printer configuration.
Accessories configuration.
Communication configuration.
Port Usage.
Date time.

**SPECTROMETER CONFIGURATION METHOD 6**

Allows the user to specify whether the spectrometer operates in double beam (DB) mode or single beam mode (single beam passing through either reference (SBR), or sample (SBS), cell holder).
Select the required mode using the arrow keys when modifying the method.
Single beam operation allows the user to check lamp energy.
To get valid readings with single beam always set %T ordinate.
The baseline correction routine can also be turned off by setting that parameter to NO.

**FACTORY CONFIGURATION METHOD 7**

Allows the user to set offset values for the 0.0 nm and 656.1 nm wavelength calibration peaks. This allows compensation for minimal inherent wavelength offset due to monochromator curvature.
The user can also alter the wavelengths at which different filters are brought into the radiation beam.

To change any of the above parameters, select to modify the method.
To enter new values for any of the parameters, scroll the parameter into the display and enter the new value.
Filter changes can only take place within a specific range and if an out of range entry is made an error message will be displayed. Enter a new value within the allowed range.

**HELP CONFIGURATION METHOD 8**

Allows the user to select the language in which HELP texts appear on the display, and the level of assistance given in the texts.
Text can be displayed in either English or German.
The level of assistance can vary between 0 and 2.

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**PRINTER CONFIGURATION METHOD 9**

Allows printer output to be turned on and off, and spacing between output pages to be varied.
If no printout is required, set the PRINTER ON? line to NO.
If printout is required set the parameter to YES.

The spacing between output page can be selected from 4 lines, 8 lines or Formfeed. Select the required value using the arrow keys.
If a colour printer is available turn the COLOUR ON parameter to YES.

**ACCESSORIES CONFIGURATION METHOD 10**

Turns the accessory option ON or OFF.
With the option ON, the ACCESSORY parameter is displayed when methods are being modified. This parameter allows the user to specify whether the method is to be used manually or with any of the various accessories.
ANALOG REC parameter turns the optional analog recorder output ON or OFF.

**NOTE:**

To operate with accessories an accessory control board must be installed in the instrument.
It is, however, possible to amend methods for use with accessories on an instrument without the accessories board. These methods can then be transferred, on cassette, to other instruments fitted with the accessories board.

**COMMUNICATION CONFIGURATION METHOD 11**

Allows the user to set the communication protocol for communication with an external computer.
Default values are set for operation with PECSS software running on an Epson PC.
(If operation with a different computer is required, different settings can be selected with the arrow keys).

The communication parameters are as follows:

| **Parameter** | **Default value** |
|---|---|
| Port Enable | NO |
| Respond | PROMPT |
| Baud rate | 4800 |
| Bits/Character | 8 |
| Stopbits | 1 |
| Parity | NONE |
| Terminator | CRLF |
| ETXT Character | 015 |
| Prompt- Character | 021 |
| Break- Character | 043 |
| Erase- Character | 010 |
| Kill- Character | 010 |
| Range | 2 |

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**PORT USAGE METHOD 12**

Specifies the external device which can be connected to the RS-232 C port.
The port can be configured for either a printer or an external computer.
Select PRINTER or COMPUTER using the arrow keys.
If an optional second interface has been installed, PORT USAGE should be set to COMPUTER. The printer can then be connected to the second interface.
For any change in Port Usage to be registered by the instrument, return to the method header and press **START**.

The test options are primarily for use by service personnel should a problem arise. They can also be used to connect the computer should a fault occur with the main interface.
For further information see Maintenance chapter.

TEST 1
TEST 2
TEST 3
TEST 4

**DATE/TIME METHOD 13**

Allow the date and time to be entered.
* Select to modify the method:

* Enter the date in the DATE line.

* Press **PARAMETER**

* Enter the time in the TIME line.

* Press START. The clock will run while the instrument is powered up.

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8.3.4.
**TEST BRANCH** The methods in this branch are for use by service personnel only. Protection is set at EXECUTE protect. This protection should not be altered.

**MONITOR** This method allows a monitor to be connected to the instrument to check the functioning of the software in the instrument.

**RESET** A full reset of instrument parameters and methods to default values is possible when FULL RESET = YES. With FULL RESET = NO only instrument parameters (e.g. filter wheel change points and monochromator settings) are reset to default values. Analytical method parameters are not affected.

**GOTO** This method slews the monochromator to a specified wavelength. It is usually activated by the GOTO LAMBDA key on the keyboard when the status display is shown.

**BACK CORR** This method carries out an autozero at the wavelength shown on the status display. It is normally activated using the **BACKCORR** key on the keyboard when the status display is shown.

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**8.3.5. SPECIFICATION BRANCH**

This branch contains methods that permit service personnel to check that instrument performance conforms to the specifications.
The methods stored in this branch are as follows:

| Method No. | Measurement | Tolerance |
|---|---|---|
| Method 1 | Energy for single sample beam, Wavelength range 1100 - 190 nm. | Energy maximum 40 - 60% |
| Method 2 | Baseline correction Wavelength range 1100 - 190 nm. | ±0.001 A at 0A |
| Method 3 | 0 %T Line Wavelength range 1100 - 190 nm. | |
| Method 4 | Wavelength accuracy with holmium oxide glass. At wavelengths: 637.5; 536.4; 459.9; 360.8 and 287.6 nm. | ± 0.5 nm |
| Method 4 | Wavelength reproducibility for 5 measurements. | ± 0.2 nm |
| Method 5 | Photometric accuracy with NBS filters at 1000, 635, 546.1 and 440 nm. | ± 0.01 A at 1000 nm<br>± 0.005 A at 635 nm<br>at 1 A 546.1 nm<br>and 440 nm |
| Method 5 | Photometric Reproducibility for 5 measurements, at wavelengths 1000; 635; 543.1 and 440 nm. | ± 0.002 at 1 A |
| Method 6 | Stray light with Didymium glass at 805, 742 and 585 nm. | |
| Method 7 | Stray light with NaNO2 at 370 and 340 nm. | < 0.02 %T |
| Method 8 | Stray light with NaI at 220 nm. | < 0.03 %T |
| Method 9 | Absorbance with KCl (1.2%) against distilled water at 200 nm. | > 2 A |
| Method 10 | Noise at 500 nm, 0 A. | < 0.0002 A |
| Method 10 | Stability (drift) at 500 nm. | < 0.0003 A/h |
| Method 11 | Stability (drift) at 1100 nm. | |
| Method 12 | Stability (drift) at 200 nm. | |

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**DEFAULT METHODS**

Permanent default versions of all methods are stored in the instrument. The default methods for each branch can be accessed from the branch header by pressing |·||**PARAMETER**| simultaneously.
The first default method will then be displayed, denoted by DMETH. Other default methods can be accessed by scrolling with the arrow keys.
Default methods are copied into the normal method files when a Full Reset is carried out (8.3.4).



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# 9 MAINTENANCE INFORMATION

## 9.1 DAILY CARE

The spectrometer is constructed with high quality components and requires little maintenance other than to keep it clean and free from dust.

Keep the sample compartment cover closed at all times. The sample compartment windows should be kept installed at all times. These measures will keep the instrument optics free from dust and fumes.

To keep the instrument in good condition follow the care instructions below:

* Immediately wipe up any spillages in the sample compartment. Dry with lintless cloth or paper.
When wiping the compartment windows clean make sure that the windows are not scratched (windows are optical components).
* Do not leave samples in the sample compartment longer than necessary. This applies particularly to volatile and fuming samples.
* If a sampling system is installed and parts are left in the sampling compartment they should be cleaned at the end of the working day. It is general practice to fill such systems with deionized water if left overnight.
* Do not place beakers and cells containing solution on top of the spectrometer, this is bad laboratory practice. Any spillage could cause expensive damage to the instrument's electronics and optics.

## 9.2 CLEANING THE SAMPLE COMPARTMENT

Clean the sample compartment every time spillage occurs. This preserves the matt black finish and prevents corrosion and contamination.
Each standard sample compartment baseplate has a drainage hole to run any spilled liquids off to the bench top. Place some thick filter paper under the instrument to soak up any run off.

To clean the sample compartment:
* Remove the cell holder or other accessory from the compartment.
* Rub away all foreign matter using a soft cloth and mild detergent.
* Rinse the cleaned surfaces with a clean damp cloth.
* Dry with lintless cloth or paper.

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## 9.3 USE AND CARE OF-

**CELLS**

A good spectrometer cell is an optical device, forming a part of the optical system of the instrument with which it is used. It must be accorded the same careful treatment applied to any optical component. Optical faults of a minor nature, scratches, lint, fingermarks, etc., can easily introduce substantial analytical errors.

The following list of cell handling rules must be followed to prevent analytical errors and to achieve the utmost precision.

* Hold cells only by the matt finish surfaces. These are not optical surfaces.
* Protect cells from scratches and never permit them to rub against one another or against other hard surfaces.
* Avoid abrasive, corrosive or stain producing cleaning agents, and ensure that the exposed surfaces of cells are optically clean.
* Before placing cells in the cell holder, always wipe the optical surfaces dry and free from finger marks using a soft cloth or cleaning tissue.
* Condensation can form on the optical surfaces if cold solutions are being scanned.
* Ensure that no bubbles cling to the inner surface of the cell, particularly when dealing with cold solutions.
* For maximum precision and accuracy, standardize and test with cells of the same type, and always insert cells into the holders with the same orientation ( e.g. cell inscriptions to the light beam).
* The transmission of glass decreases rapidly below 340 nm. If a great deal of work is performed in the lower wavelength range, quartz cells should be used.
  Avoid the use of glass cells exhibiting less than 70% transmission when filled with deionized water.

**AND WINDOWS**

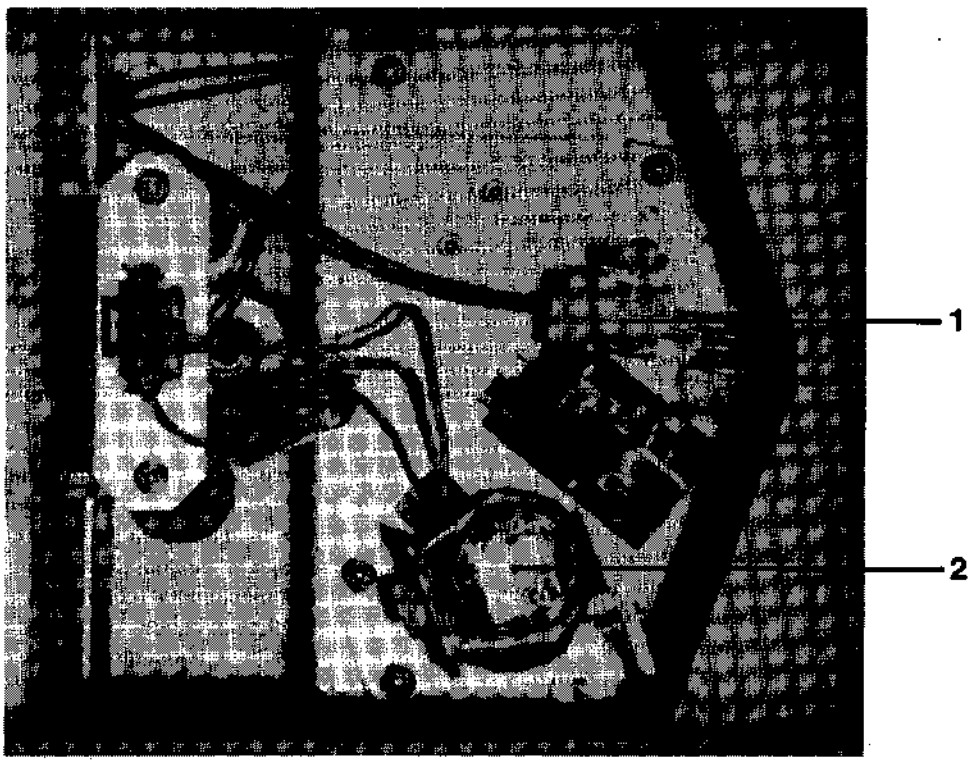
Two quartz windows are fitted in the entrance apertures to the sample compartment. The windows may be used in the entire spectral range of the spectrometer. The windows seal the sample compartment and thus protect the instrument's optics from dust and fuming or aggressive samples.

The windows are optical components and demand the same care and handling as the cells. The appropriate cell handling rules above should be closely adhered to. Windows are most suitably cleaned with a soft cloth moistened with alcohol.

The windows of each set for the instrument are checked at 195 nm at the manufacturing site and are not permitted to vary more than maximum 1 %T from each other. They can thus be installed in any order in the sample compartment.

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**Figure 9-1. Radiation Source Compartment**




1) Halogen Lamp Assembly

2) Deuterium Lamp Assembly

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9.4.1

**HALOGEN LAMP REPLACEMENT**

If the lamp burns out or if the bulb becomes blackened after prolonged use, it should be replaced. Replacement lamp assemblies are complete with prealigned mounts. (Part number B011-4620)

**CAUTION:** If the old lamp was lighted, allow it to cool before proceeding with replacement.

**WARNING:** **Switch OFF the spectrometer and remove the plug from the electrical supply before proceeding with the replacement.**

1) Open the lamp compartment by sliding the cover (at the rear left hand corner of the instrument) off to the left.

2) Pull the white ceramic connector from the rear of the halogen lamp.

3) Remove the lamp assembly from the bracket by undoing the thumbscrew and pulling the lamp mount vertically upward. Save the thumbscrew for use with the new assembly.

4) Unpack the new lamp assembly, taking care to hold it only by the metal mount to prevent fingermarks on the bulb.

5) Slip the slot at the base of the lamp mount over the stud on the bracket in the lamp compartment and then secure with the thumbscrew.

6) Push the ceramic connector firmly onto the pins at the base of the lamp.

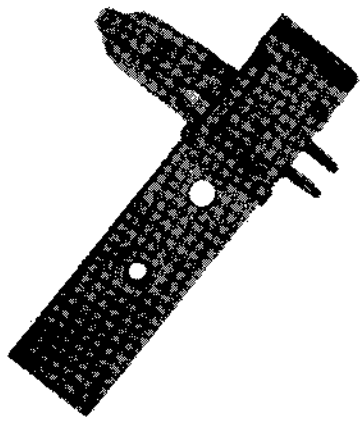
7) Wipe the lamp with a soft cloth moistened with alcohol to remove dirt, since this would otherwise be burned in when the lamp is hot.

8) Replace the lamp compartment cover.

This completes the halogen lamp replacement procedure.

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**Figure 9-2. Prealigned Halogen Lamp Assembly**



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9.4.2
**DEUTERIUM LAMP REPLACEMENT**

If the lamp burns out or indicates falling energy after prolonged use, it should be replaced. Replacement lamp assemblies are complete with prealigned mounts. (Part number B016-0917).

**NOTE:** An operating hours counter is incorporated in the red deuterium lamp lead.
By means of the gap between the two display bars it is possible to read off the number of hours that the lamp has been in operation.
One scale division corresponds to approximately 100 hours.
After a total operating time of more than 2000 hours the lamp still exhibits more than 50% of its original radiant intensity.
To prolong lamp-life, the lamp should only be switched off at the end of the working day and not during.
Turning on a hot lamp immediately after it has been switched off will markedly reduce lamp-life. Allow the lamp to cool for about 2 minutes before turning back on.

**CAUTION:** If the old lamp was lighted, allow it to cool before proceeding with the replacement.

**WARNING:** **Switch OFF the spectrometer and remove the plug from the electrical supply before starting the replacement.**

1) Open the lamp compartment by sliding the cover (at the rear left hand corner of the instrument) off to the left.

2) Unplug the deuterium lamp leads from the terminal board.

3) Remove the lamp assembly from the bracket by undoing the thumbscrew and pulling the lamp mount vertically upward. Save the thumbscrew for use with the new lamp assembly.

4) Unpack the new lamp assembly, taking care to hold it only by the metal mount to prevent fingermarks on the lamp window.

5) Slip the slot at the base of the lamp mount over the stud on the bracket in the lamp compartment and then secure with the thumbscrew.

6) Plug the deuterium lamp leads into the terminal board.

7) Wipe the lamp window with a soft cloth moistened with alcohol to remove dirt, since this would otherwise be burned in when the lamp is hot.

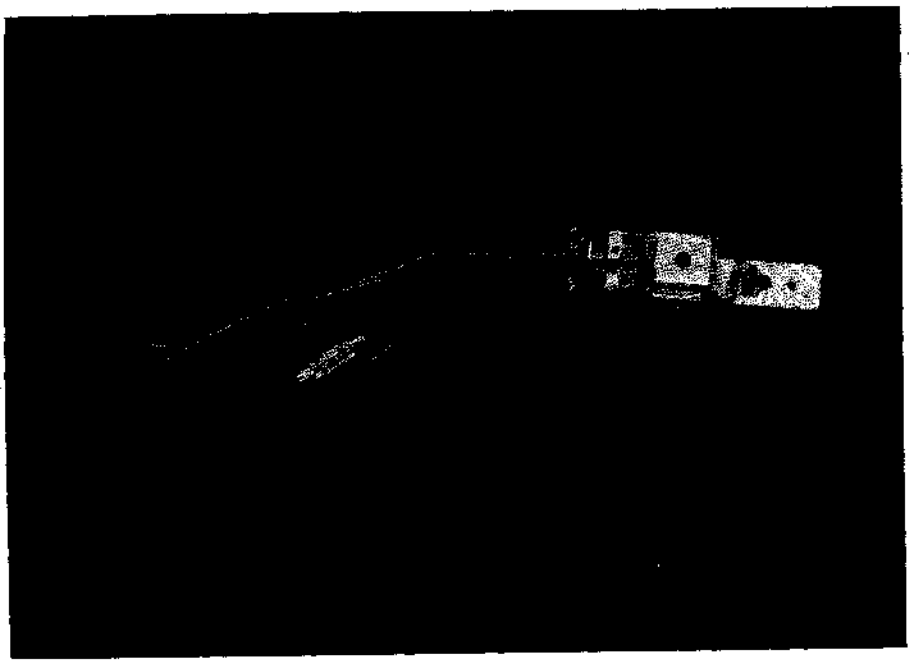
8) Replace the lamp compartment cover.

This completes the deuterium lamp replacement procedure.

**WARNING:** Never look into a lighted UV lamp. DAMAGE TO THE EYES can result. Wear protective glasses.

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Figure 9-3. Prealigned Deuterium Lamp Assembly



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## 9.5 INTERFACE FAULTS

During operation, the instrument generates three output signals; printer output, computer output and test output. To cope with these three data sets the instrument effectively has three output ports:
the standard RS-232-C interface,
the second optional RS-232-C interface,
a third internal interface which can only be accessed by service personnel.

The data sets are directed to specific outputs dependent upon the mode chosen in the PORT USAGE method (8.3.3). The table below lists the port usage options, and shows which data sets are directed to which output interface.

| OPTION | OUTPUT | DATA SET SENT |
|---|---|---|
| PRINTER | STANDARD | Printer Signal. |
| | OPTION | Computer Signal. |
| | TEST | Test Signal. |
| COMPUTER | STANDARD | Computer Signal. |
| | OPTION | Printer Signal. |
| | TEST | Test Signal. |
| TEST 1 | STANDARD | Test Signal. |
| | OPTION | Computer Signal. |
| | TEST | Printer Signal. |
| TEST 2 | STANDARD | Printer Signal. |
| | OPTION | Test Signal. |
| | TEST | Computer Signal. |
| TEST 3 | STANDARD | Computer Signal. |
| | OPTION | Test Signal. |
| | TEST | Printer Signal. |
| TEST 4 | STANDARD | Test Signal. |
| | OPTION | Printer Signal. |
| | TEST | Computer Signal. |

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Should the Standard interface become defective, it is possible to connect either the computer or printer to the accessory interface using one of the test options.

**EXAMPLE:** If PRINTER mode is set and the standard interface is defective, the printer will not function.
Switch to TEST 4 or COMPUTER and connect the printer to the second interface. The printer should function normally.

If COMPUTER mode is set and the standard interface is defective, the computer will not be able to function.
Switch to TEST 1 or PRINTER and connect the computer to the second interface. The computer should function normally.

**NOTE:** Everyday operation should not be carried out in one of the test modes.
Operation in Test mode allows the instrument to be used should one of the interfaces fail, but a service engineer should be called, as soon as possible, to correct the fault.

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## 9.6 CHANGING FUSES

Power fuses are located in a fuse holder integrated into the power receptacle at the rear of the instrument.
If a fuse should blow, replace it as follows:

* Switch OFF the instrument and remove the power cord from the instrument power receptacle.

* Grip the fuse holder at the top and bottom and pull it out. (Or use a small screwdriver to lever it out).

* Remove the spent fuse and replace it with one of the same type and rating. (A spare fuse is contained in the holder).

* Insert the fuse holder back into the receptacle.

* Reconnect the power cord and switch the instrument on.

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# 10. HAZARDS

Any hazards that may arise through use of the instrument are mentioned at appropriate places in the manual.

The pink sheet at the front of the manual should also be read carefully before using the instrument.

**ULTRA-VIOLET RADIATION**

UV radiation emitted by the deuterium lamp is damaging to the human eye.
DO NOT LOOK INTO A LIGHTED UV LAMP.
SERIOUS DAMAGE TO THE EYES COULD RESULT.

WEAR PROTECTIVE UV ABSORBING GLASSES, should any work have to be carried out with the instrument cover open and the lamps illuminated.

**HIGH VOLTAGES**

High voltages can reside on circuitry if the instrument is turned off but still connected to the electrical supply.
If work is to be carried out inside the instrument pull the plug from the wall socket.
If work is to be carried out inside the instrument while it is switched on work should be carried out by a suitably qualified person who is aware of the hazards.

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# Lambda 2 Accessory Supplement
# Lambda 2 Zubehör-Installation

B 2134 – A 1.1 – M 1244 / 2.89 Printed in Federal Republic of Germany B019-0898

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# L A M B D A  2

## ACCESSORY SUPPLEMENT

Supplementary Information
for the
Installation and Operation
of Accessories

**Publication:** B2134

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**Author** : Simon Morris

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# A T T E N T I O N

BEFORE USING ANY OF THE INSTRUMENTS MENTIONED IN THIS MANUAL IT IS ESSENTIAL TO READ THE RESPECTIVE DIRECTIONS CAREFULLY AND TO PAY PARTICULAR ATTENTION TO ANY ADVICE CONCERNING POTENTIAL HAZARDS THAT MAY ARISE FROM THE USE OF ANY INSTRUMENT.

## ELECTRICITY

If any part of a system is not installed by a Perkin-Elmer Service Representative, ensure that the corresponding plug on the line cord is correctly wired, as follows:

| | Cord Lead Colors | |
|---|---|---|
| | International | U.S.A. |
| Live terminal | Brown | Black |
| Neutral terminal | Blue | White |
| Earth-grounded terminal | Green/Yellow | Green |

To ensure satisfactory and safe operation of the instruments it is essential that the green/yellow lead of each line cord is connected to true electrical ground (earth).

To prevent interferences due to earth loops, all instruments should be connected to the same phase of the electrical supply.

Always switch off instruments and disconnect them from the electrical supply before connecting or disconnecting any units. Permanent damage to circuit boards can result if connections or disconnections are made while instruments are switched on.

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# LAMBDA 2 ACCESSORY SUPPLEMENT

## 1. INTRODUCTION

The Lambda 2 Spectrometer utilises a Lambda series sample compartment and hence can be fitted with all Lambda series accessories, except for integrating spheres and the Scattered Transmission Accessory.
This supplement details any installation advice peculiar to Lambda 2 which you may not find in the accessory manuals.

Lambda 2 operates some methods that are not available on other Lambda Series spectrometers.
Details concerning the operation of accessories using these methods are also listed in this supplement.

**CIRCUIT BOARD**

To operate accessories with the instrument it is essential that Lambda 2 is fitted with the Accessory Circuit Board (B017-9708).

**CONTROL**

To control the accessories you will find the ACCESSORY parameter immediately following the BACK CORR parameter. This parameter allows you to select the accessory to be used with the instrument. Depending upon the accessory chosen, certain further accessory-specific parameters may follow. These are detailed in this supplement as well as in the Lambda 2 Operator's Manual.

The accessory configurations included in the supplement are:

1) Cell Changers (B012-7391 Multicell Programmer and Manual Cell Changer Assemblies)

2) Single Sipper (B012-7390)
3) Single Sipper and Multisampler

4) Super Sipper (unheated B012-6829, and heated B012-6830)
5) Super Sipper and Multisampler

6) Econo Sipper (B013-8597)
7) Econo Sipper and Multisampler

8) Multisampler (B012-6831)

9) Analog Chart Recorders

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## 2. SAMPLE COMPARTMENT

**BASEPLATES**

Lambda 2 has a one-piece baseplate covering the floor of the sample compartment.
For this reason, procedures for installing certain accessories vary slightly from the directions given in the respective accessory manual.
For some accessories you must additionally order an extra half baseplate, while other accessories require the Sample Compartment Kit.

The accessories affected are:

**Manual Cell Changers (B009-9356 and B008-9416)**

An extra half baseplate (B012-6705) must be ordered to cover the rear half of the sample compartment floor.
The half baseplate must be installed before the accessory.
Screw the half baseplate down securely with the socket head screws provided.

**LC Micro-Flowcell (B011-0662)**

To install this accessory, the Sample Compartment Kit (B018-0210) is required.

**Light Pipe Accessory (B016-6476, B016-6511, B016-6512)**

To install this accessory, the Sample Compartment Kit (B018-0210) is required.

**Thermostatted Super Sipper (B012-6830)**

Remove the cell holder from the baseplate supplied with the accessory and screw it directly onto the Lambda 2 baseplate.

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## CABLES/HOSE GUIDES

Most accessories have control cables to connect them to the instrument. Certain thermostatted accessories additionally have tubing connections.
You should arrange these cables/tubes carefully to ensure that they do not obscure the radiation beams and that they do not allow light to enter the sample compartment.

Cables for cell changers should be led from the bottom of the sample compartment and along under the instrument to the connection panel.

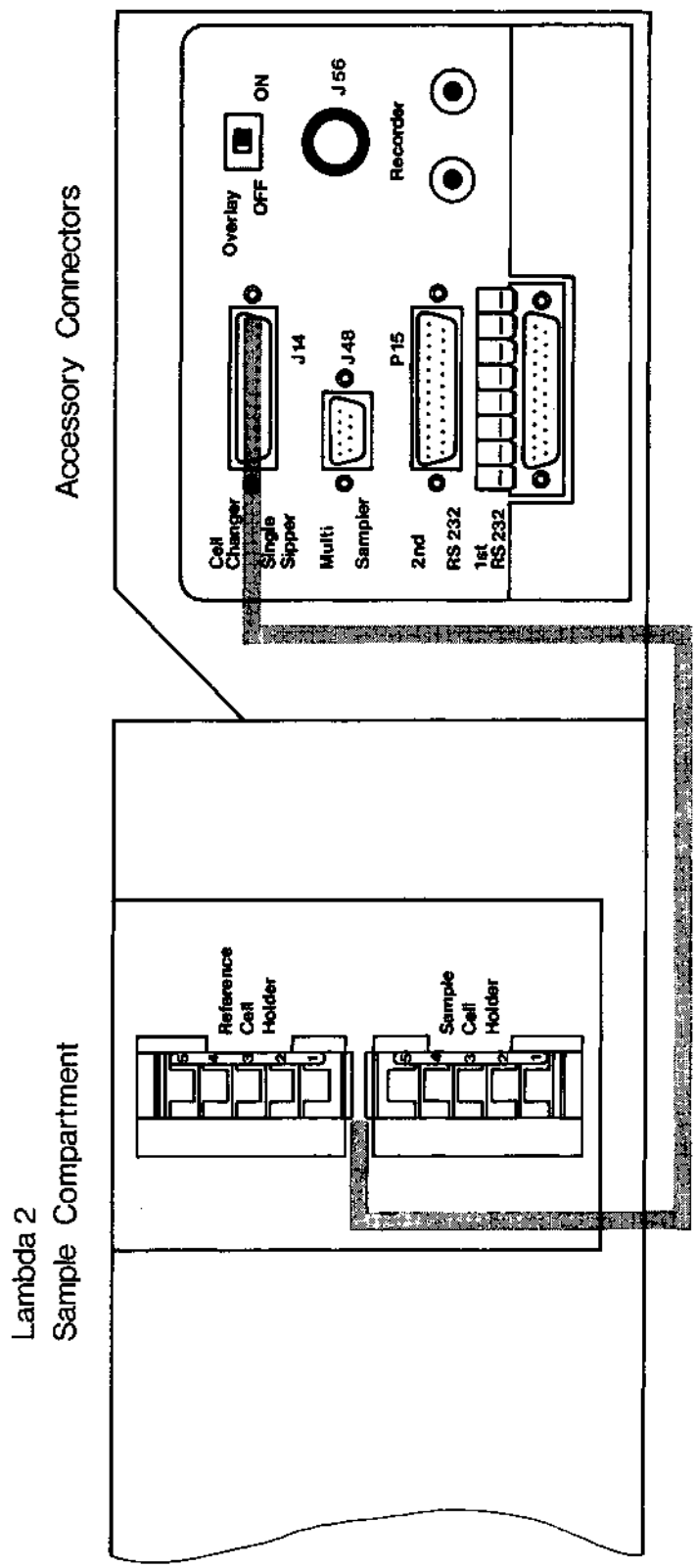
Cables from the sipper units should be led from the front of the instrument and along under the instrument to the connection panel.

Keep tubes out of harm's way in the sample compartment by affixing them to the walls using magnetic clips.
You can then lead them out of the sample compartment through the four hose guides at the front of the sample compartment.

**CAUTION:** Never connect or disconnect any cables while the Spectrometer or the ancillary instrument is switched on.
Damage to circuit boards may otherwise result.

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## 3. CELL CHANGERS




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**INSTALLATION** When installing the cell changer, lead the control cable from the bottom of the sample compartment under the instrument to the accessory connection panel.
Connect the plug to socket **J14** (marked 'Cell Changer').

**GENERAL** After selecting the cell changer at the ACCESSORY parameter, there will be a further parameter allowing you to select which cell positions you will be using. There is one parameter for 5x5 and 6x6 cell changers, allowing the selection of cells 1 to 5 or 6.

**BACK CORR** Position 1 is always used for BACK CORR, regardless of whether that position has been selected in the method.

Depending upon the Tag status of BACK CORR, not all positions can be used:

FIX/CALL tag: Blank solution for background correction must be placed in position 1; after the background correction, any positions can be used for samples.

BATCH tag: If BACK CORR = YES then position 1 will be used exclusively for Blank solution. Samples must be placed in positions 2 onward.
If BACK CORR = NO then all positions can be used for sample analysis.

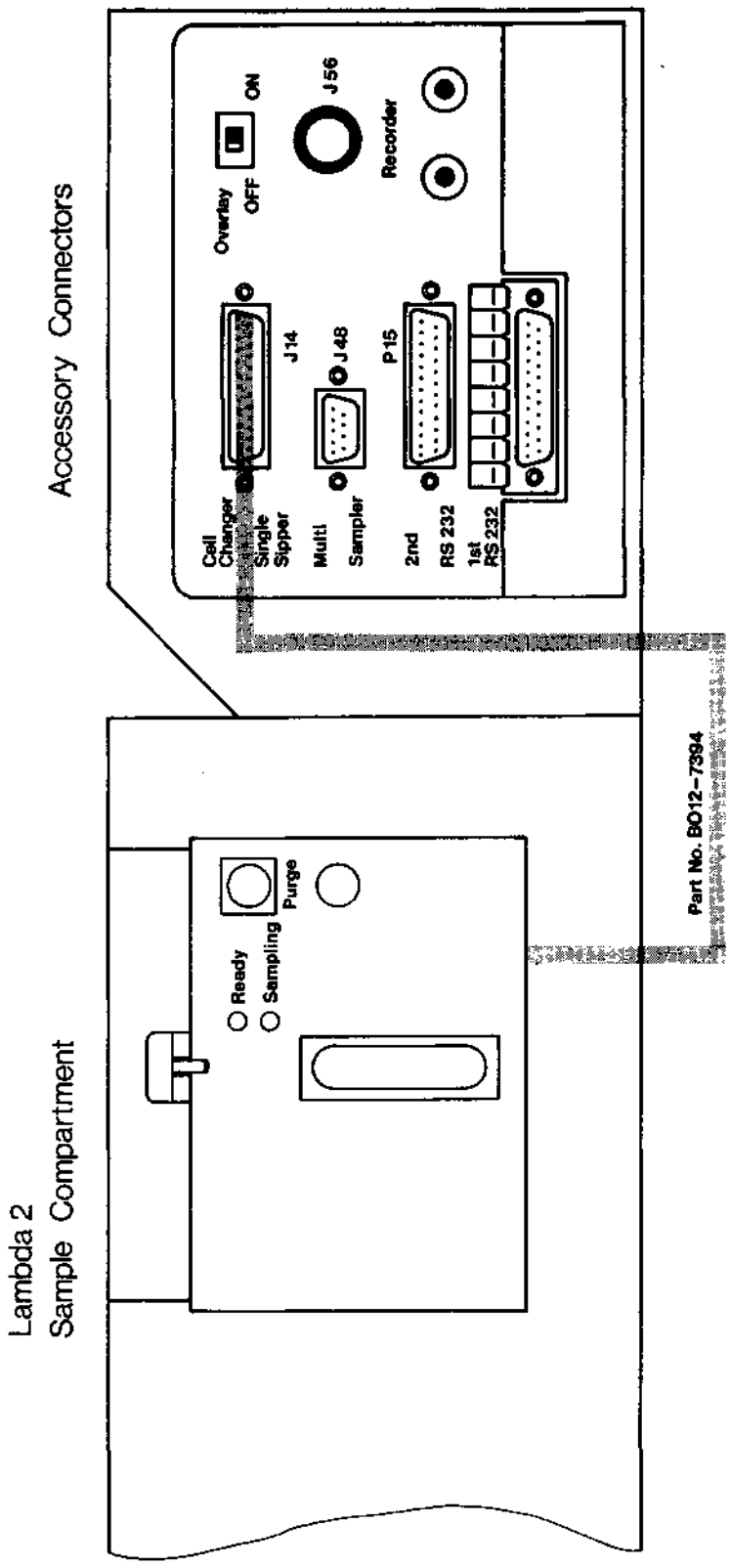
START tag: Blank solution should be placed in position 1, which is used exclusively for background correction. Samples can only be placed in positions 2 onward.

**CONC METHODS** Care must be taken when operating concentration methods with a cell changer. The above BACK CORR tag conditions apply to concentration methods, with the extra condition that if BACK CORR = YES, a background correction will always be carried out before the standards are analyzed.

Once background correction has been completed, standards can be determined.
Standards can be placed in all cell positions.
If 10 standards are to be analyzed using a 6x6 cell changer, place standards 1 to 6 in positions 1 to 6.
Press **START** and the first 6 standards will be analyzed.
When the instrument requests the next standards, place standards 7 to 10 in positions 1 to **4**.
Press **START** and standards 7 to 10 will be analyzed.
The instrument will now request samples. Samples can only be placed in those positions selected when setting up the method.
Also take into account the Back Corr tag conditions listed above.

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## 4. SINGLE SIPPER




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**INSTALLATION** Follow the instructions supplied with the Single Sipper to install it in the sample compartment, and to fit the necessary tubing.
The control cable should be led under the instrument to the right-hand panel where it should be plugged into the socket marked for 'Single Sipper' (**J14**).

**CONTROLLING THE SIPPER** The Spectrometer recognizes the Single Sipper automatically when it is plugged into socket J14.
The ACCESSORY parameter is displayed immediately following the BACK CORR parameter. It will be set at SI-SIPP.
The next three parameters are specific to the Single Sipper:

SAMPLING TIME The length of time during which sample solution is aspirated into the flowcell. (Allowed range, 0.1 to 99.9 seconds.)
This is dependent upon flowcell volume and sample viscosity.
To directly access this parameter, use parameter No. 43.

DELAY TIME Delay period to allow sample equilibration before the reading is made. (Allowed range, 0.1 to 99.9 seconds.)
To directly access this parameter, use parameter No. 44.

AUTO PURGE Turns the auto purge function on and off.
To directly access this parameter, use parameter No. 45.

Methods operating with the Single Sipper are denoted by SIS in the method header after the method type.

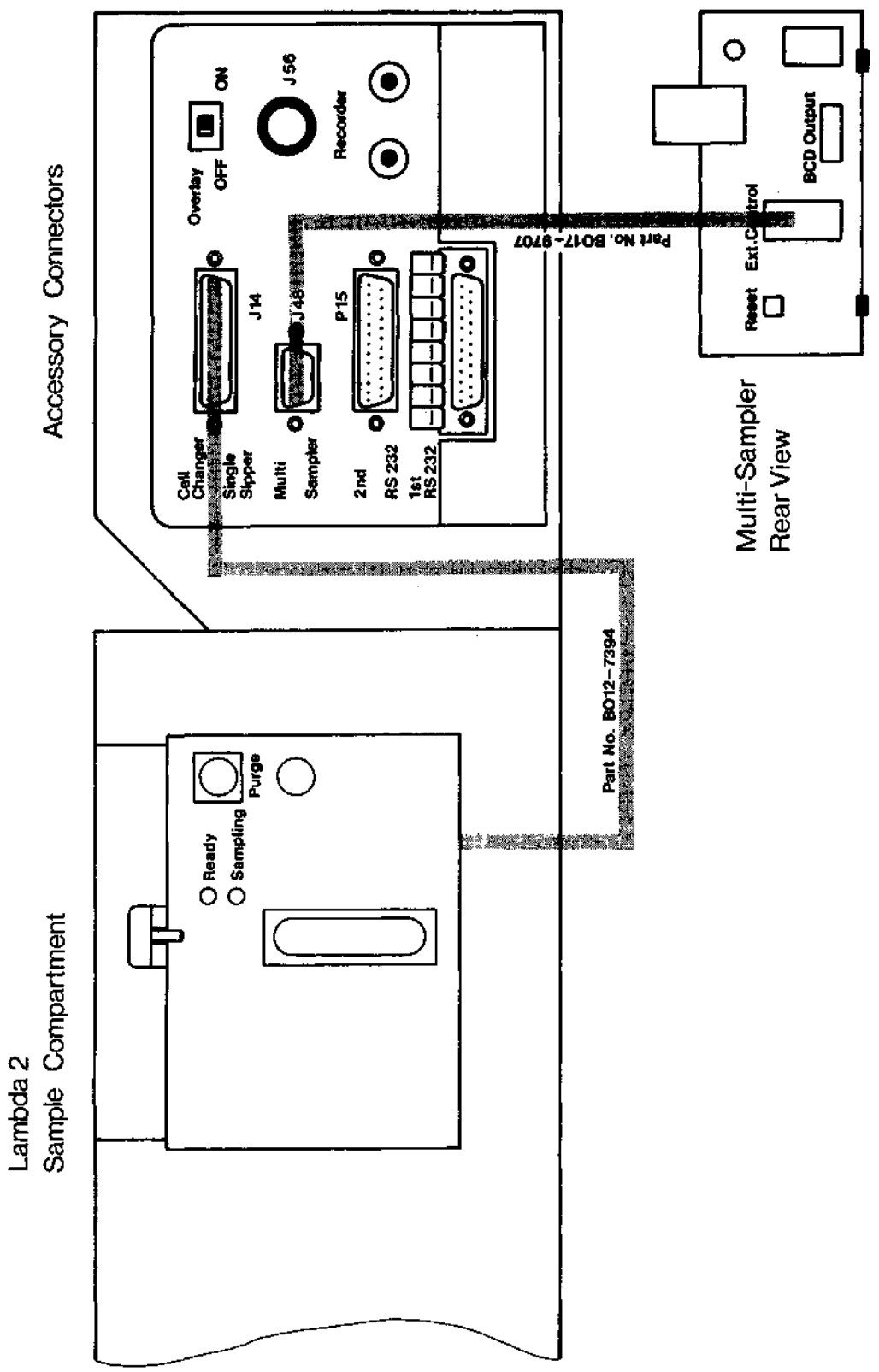
**OPERATION** Operation with the Single Sipper is as straightforward as operating the instrument manually, just much quicker.

* If selected, BACK CORR will be requested when the method is started.
  - Place the sipper tube in the blank solution and press the actuator button.
  - Blank solution will be aspirated and a background correction carried out.

* The instrument will request the next sample when ready.
  - Place the sipper tube in the sample solution and press the actuator button.
  - Sample will be aspirated and analyzed.

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## 5. SINGLE SIPPER AND MULTISAMPLER




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**INSTALLATION** Install the Single Sipper in the sample compartment as described in the previous section. Install the Multisampler as described in the directions provided with the Multisampler. The Single Sipper is connected to socket **J14**, the Multisampler is connected to **J48**.

**CONTROL** Always wait until the instrument startup routine has been completed before switching on ancillary instruments.
You can set single sipper/multisampler parameters in the same way as the sipper alone (see above).

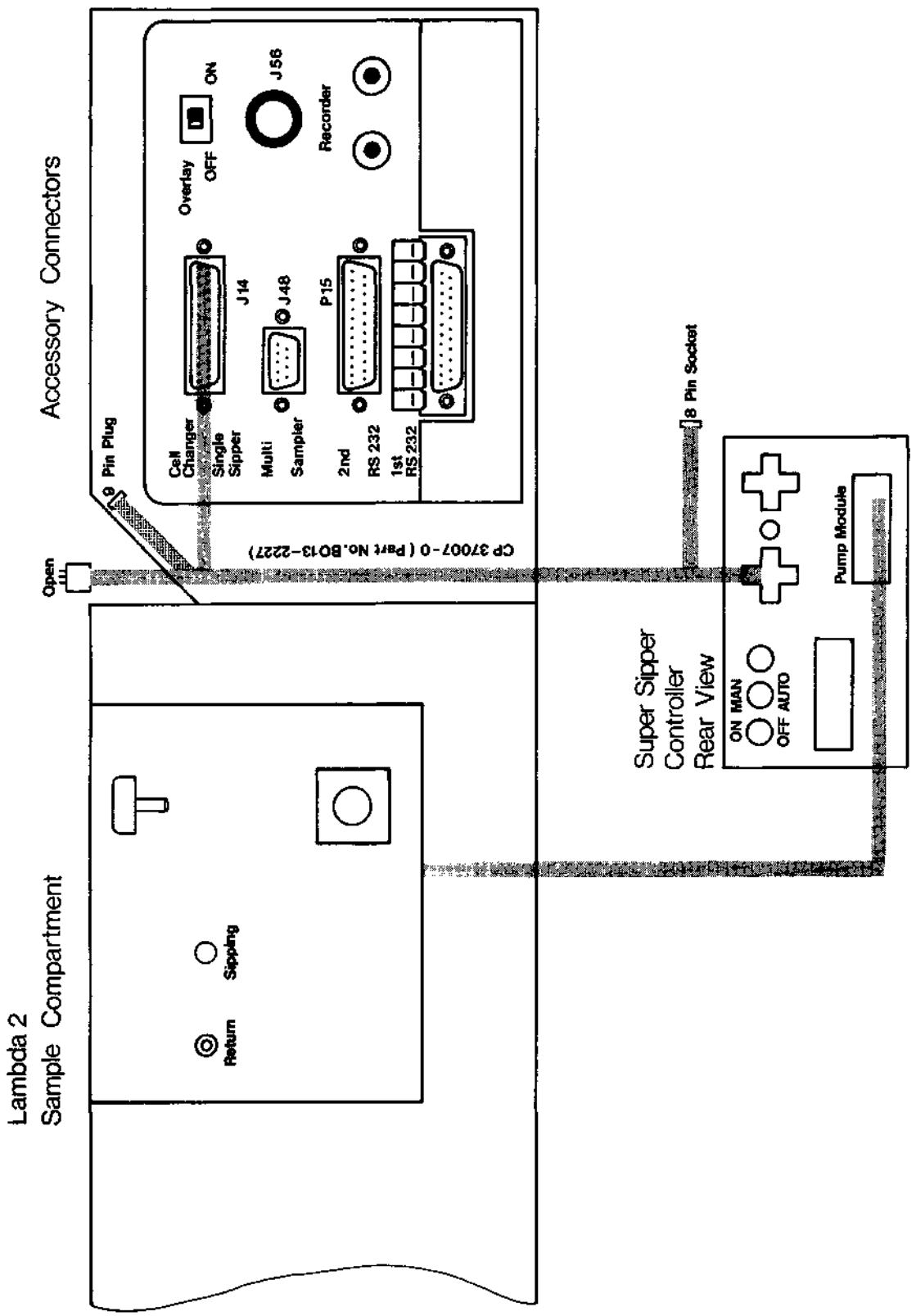
**OPERATION** Operation with the Single Sipper/Multisampler combination is semi-automated.
Set the Multisampler controls to AUTOMATIC and COMPLETE CYCLE. When the instrument requires a sample, the Multisampler advances the next sample and the sipper tube is lowered into the sample solution. You only need to press the sampler MANUAL ADVANCE key. The sample will be aspirated and analyzed. After analysis the Multisampler advances the next sample solution.
If you want to skip a sample position, switch the sampler to MANUAL and TUBE ADVANCE, and press MANUAL ADVANCE.

BATCH PROCESSING It is possible to set up your samples in the Multisampler so that you can carry out batch analyses.
Read the instructions supplied with the Multisampler concerning the loading order of the sampler.
If you want to carry out background corrections before each batch, BACK CORR must be set as a Batch parameter.
Do not turn it off when it is presented for amendment on the Lambda 2 display, you will upset the order of your samples.
Place a tube of blank solution before each batch of sample tubes.
After each batch the display will read ACCESSORY START.
To start the next batch press the MANUAL ADVANCE switch on the Multisampler front panel. The next batch will be run.

CONC METHODS Take care when loading the Multisampler with standards and samples.
If BACK CORR = YES then, regardless of tag, a background correction will be carried out before the standards are analyzed. Place a tube of blank solution in the Multisampler before the standards.
If BACK CORR has a batch tag it will be presented for amendment before each batch of samples.
Do not turn BACK CORR off, otherwise your samples will get out of order.
Place a tube of blank solution before each batch of samples.

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## 6. SUPER (RETURN) SIPPER




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**CAUTION** **BEFORE INSTALLING THE SUPER SIPPER:**

* If the Super Sipper has never been used with Lambda 2 before, or has been used only with other Lambda Series instruments, an alteration must be made to the connecting cable (B013-2227). The connection to PIN 14 of the 25-pin Cannon socket must be interrupted, and the wire connected to the cable shielding.

  If this is not done, damage to the internal circuitry of Lambda 2 can occur.
  This work should be carried out by a suitably qualified person.

  Mark this cable for use with Lambda 2 only.

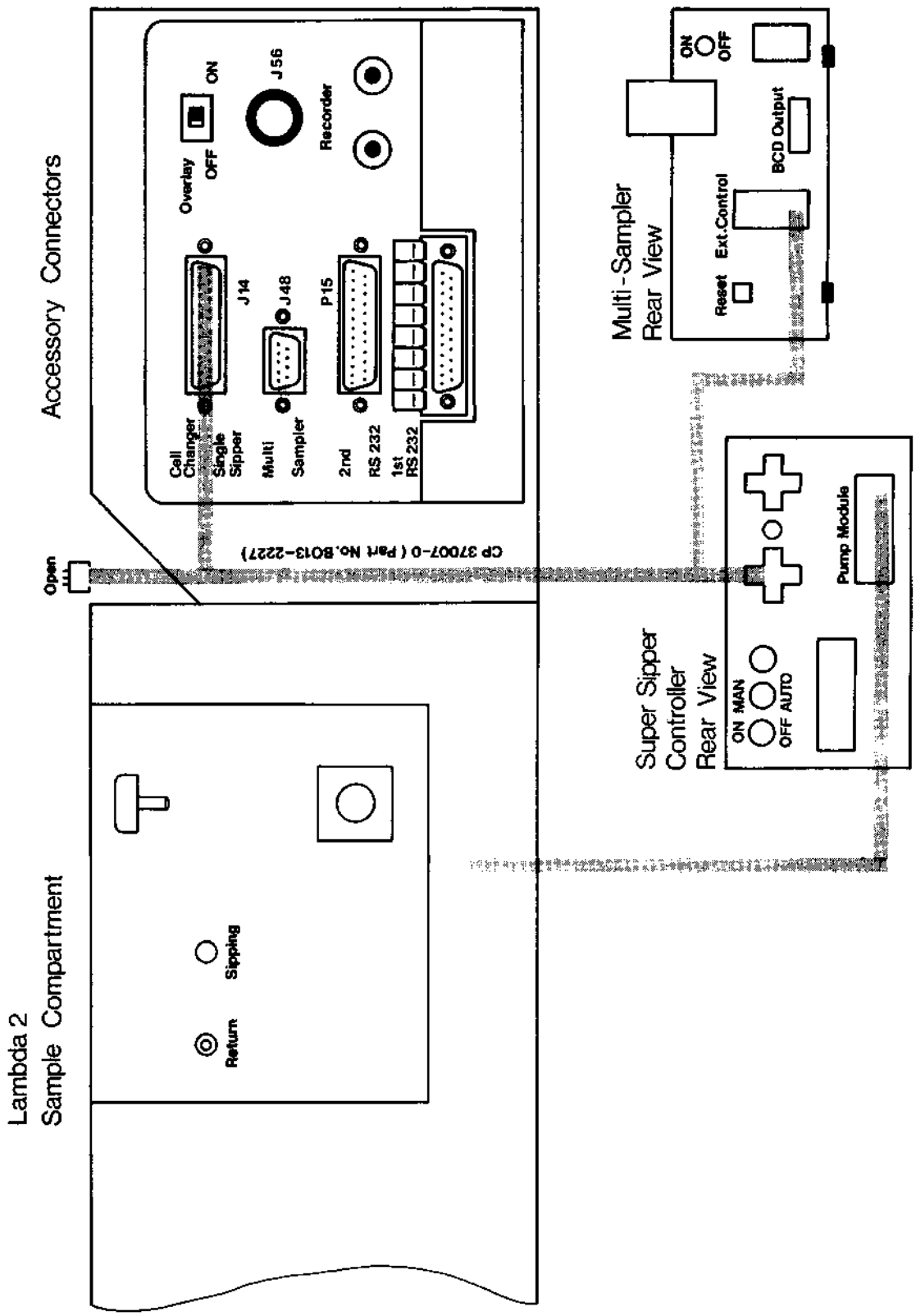
**INSTALLATION** Install the Super Sipper pump unit in the Lambda 2 sample compartment as described in the super sipper handbook.
Connect the sipper pump unit to the sipper control unit.
Connect the sipper control unit to socket **J14** of Lambda 2.

**CONTROL** Always wait until the instrument startup routine has been completed before switching on ancillary instruments.
The Super Sipper is controlled from the sipper control unit. Hence you cannot enter any super sipper parameters from the Lambda 2 keyboard. Any parameter changes must be made on the sipper control unit.

**OPERATION** Operation with the Super Sipper is exactly the same as for the Single Sipper. Refer to that section of this supplement.

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## 7. SUPER SIPPER AND MULTISAMPLER




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**IMPORTANT:**

Before installing the Super Sipper, read the 'CAUTION' notice in Section 6.

**INSTALLATION** Install the super sipper pump unit in the sample compartment as directed in the super sipper manual.
Connect the pump unit to the control unit.
Connect the multisampler to the control unit using the 8-pin plug on the cable. Plug the 25-pin connector into socket **J14** and the 9-pin plug into socket **J48** on Lambda 2.

**CONTROL** Always wait until the instrument startup routine has been completed before switching on ancillary instruments.
As explained previously, no entries need to be made on Lambda 2 for sipper operation. Make these entries on the sipper control unit.

**OPERATION** Operation with the Super Sipper/Multisampler system is fully automatic. Switch the sampler to COMPLETE CYCLE and AUTOMATIC. You only need to press the MANUAL ADVANCE key to start analyses. To skip a sample position, select MANUAL and TUBE ADVANCE, and press MANUAL ADVANCE.

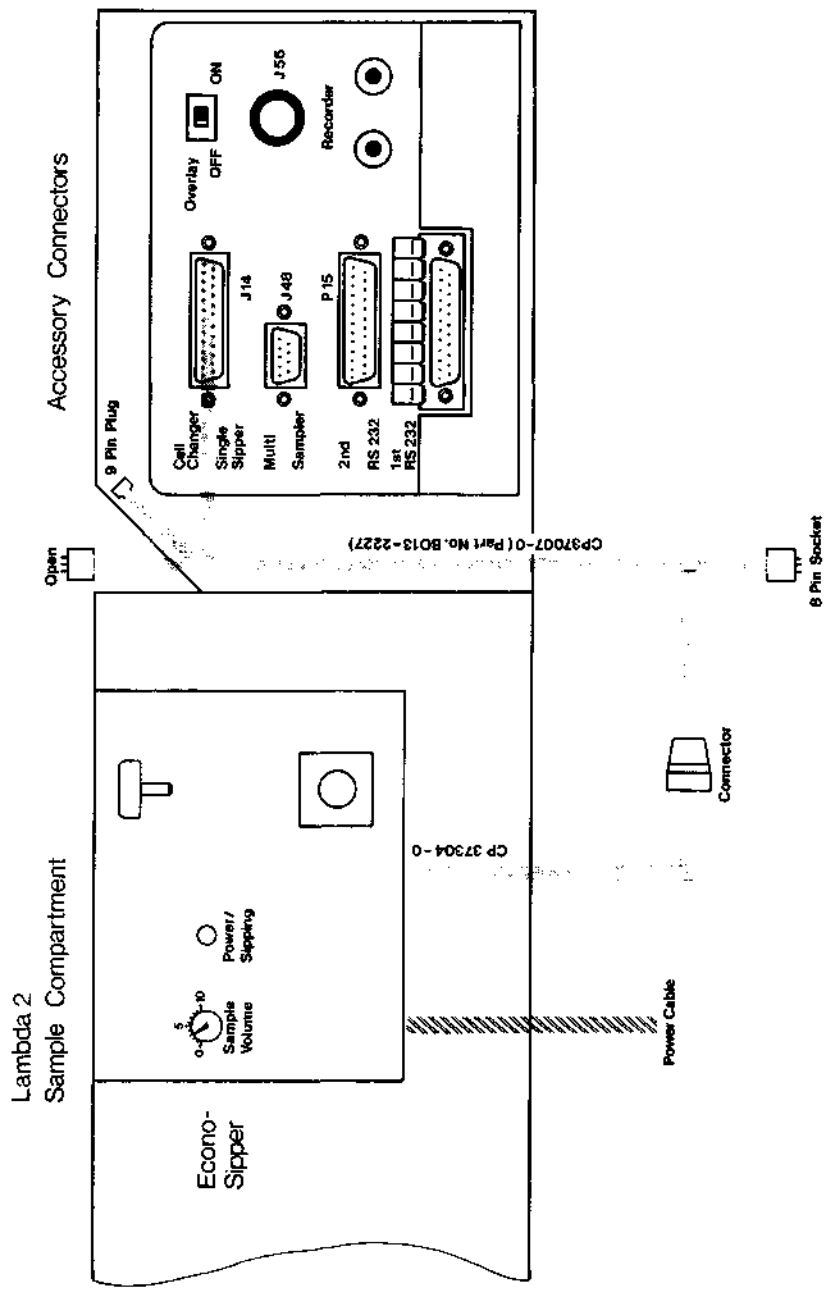
BATCH PROCESSING It is possible to set up your samples in the Multisampler so that you can carry out batch analyses.
Read the instructions supplied with the Multisampler concerning the loading order of the sampler.
If you want to carry out background corrections before each batch, BACK CORR must be set as a Batch parameter.
Do not turn it off when it is presented for amendment on the Lambda 2 display, you will upset the order of your samples.
Place a tube of blank solution before each batch of sample tubes.
After each batch the display will read ACCESSORY START.
To start the next batch press the MANUAL ADVANCE switch on the multisampler front panel. The next batch will be run.

CONC METHODS Take care when loading the Multisampler with standards and samples.
If BACK CORR = YES then, regardless of tag, a background correction will be carried out before the standards are analyzed. Place a tube of blank solution in the Multisampler before the standards.
If BACK CORR has a batch tag it will be presented for amendment before each batch of samples.
Do not turn BACK CORR off, otherwise your samples will get out of order.
Place a tube of blank solution before each batch of samples.

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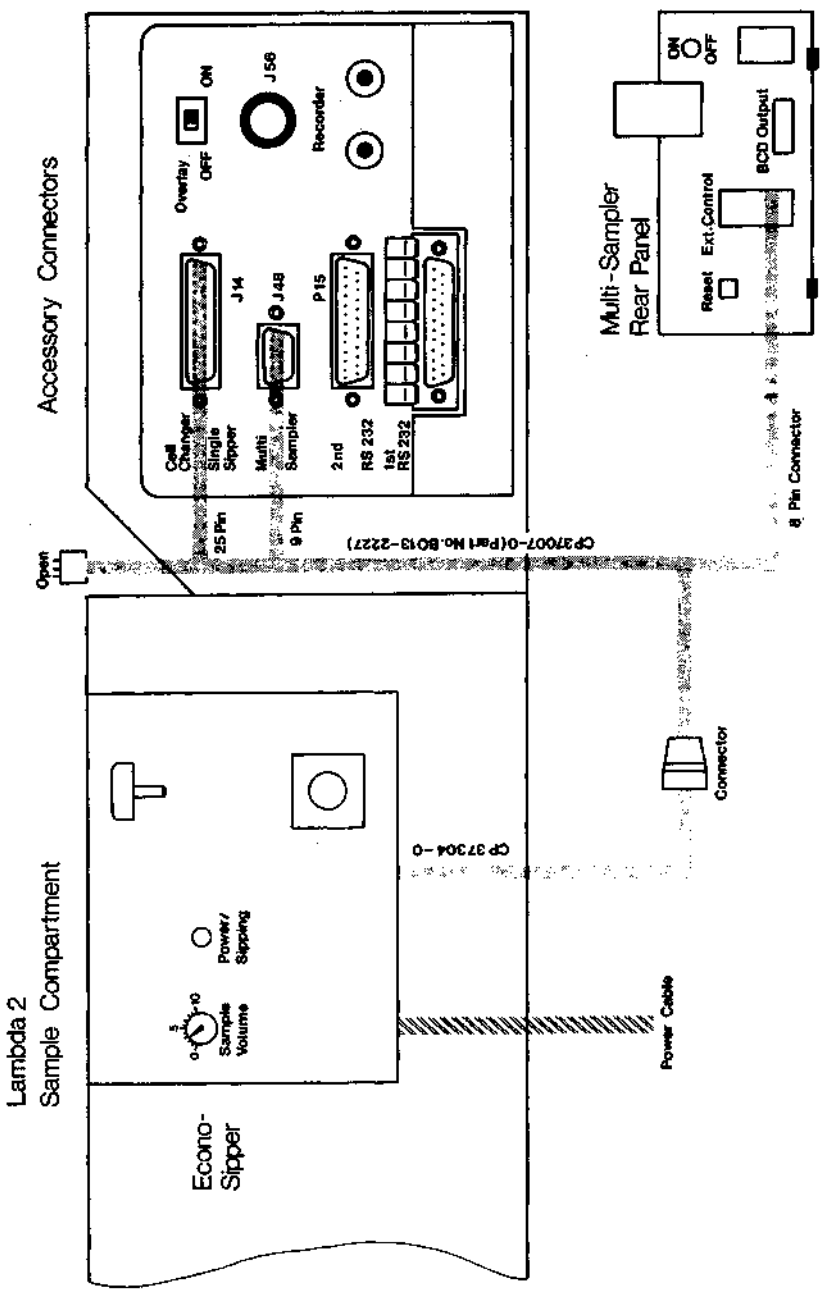
## 8. ECONO SIPPER

The Econo Sipper is an accessory very similar to the Super Sipper. However, it does not have a separate control unit. The only parameter which can be altered is sample volume. This is altered with the control on the front of the unit. No entries need be made on Lambda 2.
Operation is exactly the same as for the Super Sipper and you are referred to that section.



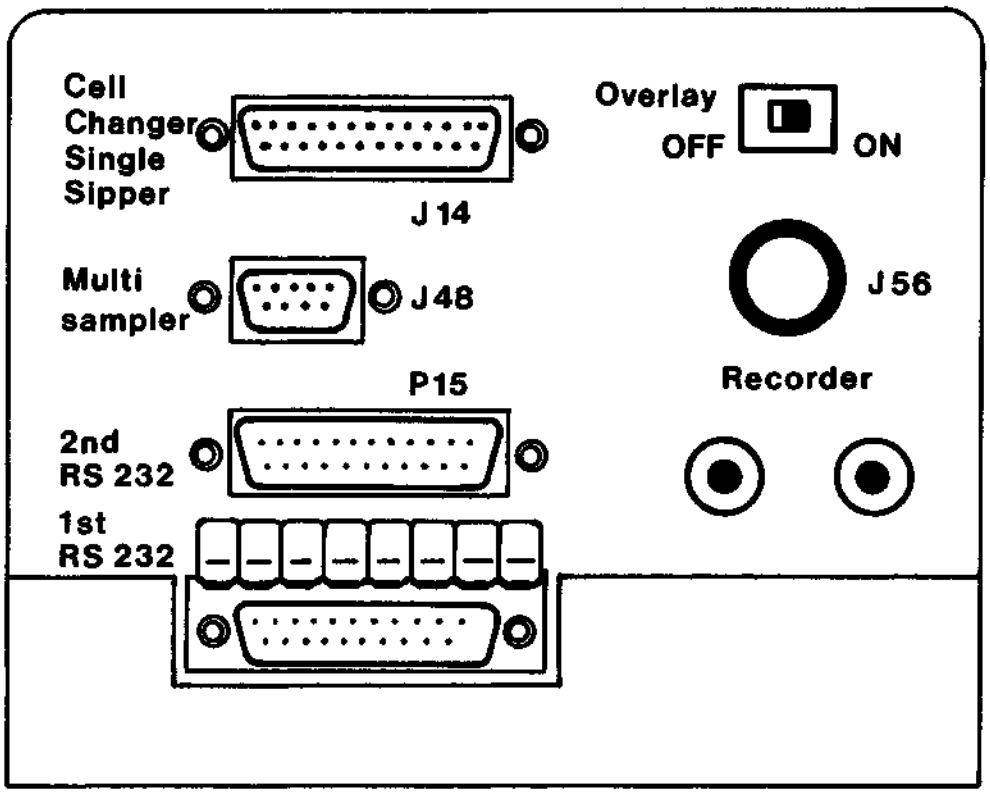

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## 9. ECONO SIPPER AND MULTISAMPLER




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## 10. ANALOG CHART RECORDERS




### CONNECTING AN ANALOG CHART RECORDER

The black and red terminals on the Lambda 2 accessory connector panel are for output of the analog signal to an ancillary strip chart recorder. These terminals must be connected to the corresponding terminals on the recorder. Range 0 - 1 V, corresponding to the ordinate range set on Lambda 2.

Socket J56 is for the connection of the recorder control cable. Connect the corresponding DIN plug to this socket.
Two relays are switched from this socket:
5G = START signal, pins 1 + 3, starts recording.
5F = RESPOOL signal, pins 4 + 5, respools paper to allow overlay plotting.
These relays can be controlled via the RS-232-C interface. Refer to the Interface Description (Publication B2133.)

The requisite cables for connecting a recorder to Lambda 2 are supplied with the Model 561 and R100A Recorders.

The OVERLAY ON/OFF switch allows the overlay facility available on the R100A Recorder to be switched on and off.

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# L A M B D A 2

# Z U B E H Ö R - I N S T A L L A T I O N

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# A C H T U N G

**BITTE LESEN SIE VOR DER INBETRIEBNAHME DER GERÄTE DIE BEDIENUNGSANLEITUNGEN. BEACHTEN SIE INSBESONDERE ALLE HINWEISE UND WARNUNGEN ÜBER DIE GEFAHREN BEI DER BENUTZUNG DER GERÄTE.**

Diese Sicherheitshinweise sollen die in den einzelnen Ländern gültigen Sicherheitsbestimmungen nicht ersetzen, sondern nur ergänzen.

## STROMVERSORGUNG

Die Geräte werden mit einem Schutzkontaktstecker nach VDE-Vorschrift geliefert. Beim Verwenden anderer Stecker vergewissern Sie sich bitte, daß diese Stecker für die Netzstromversorgung richtig verdrahtet sind:

Brauner Anschlußdraht an die stromführende Klemme (P),
Blauer Anschlußdraht an die Sternpunktklemme (MP),
Grün-gelber Anschlußdraht an die Erdklemme (Schutzleiter).

Das Leitungsnetz zur Versorgung des Systems mit Netzstrom sollte entprechend den einschlägigen VDE-Vorschriften installiert und abgesichert sein.

Um eine optimale Entstörung aller verwendeten Geräte zu erreichen, sollten alle Geräte des Meßplatzes aus dem gleichen Stromkreis versorgt werden.

Wartungs- und Reparaturarbeiten sollten nur vom Perkin-Elmer Kundendienst oder von entsprechend ausgebildeten Fachkräften ausgeführt werden.

Vor dem Öffnen der Geräte unbedingt alle Geräte ausschalten und die Netzstecker ziehen.
Ausschalten allein genügt nicht, da auch dann noch an einigen Stellen der Geräte gefährliche Spannung anliegen kann.

**WARNUNG:** Die Geräte dürfen nicht in explosiver Atmosphäre betrieben werden. Leicht entflammbare Chemikalien und Lösemittel dürfen nicht in der unmittelbaren Umgebung des Gerätes aufbewahrt werden.

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# 1 ALLGEMEINE HINWEISE

**VORSICHT:** Vor Installation oder Ausbau eines Zubehöres unbedingt

- das Lambda 2 und sämtliche Zubehöre ausschalten
- und die Netzstecker der Geräte ziehen.

So vermeiden Sie Schäden durch elektrische Spannung.

Der Probenraum des Lambda 2 ist baugleich mit allen Geräten der Lambda-Serie. Somit können alle Lambda-Zubehöre verwendet werden, mit Ausnahme der Integrationskugeln und dem Zubehör für trübe Proben (B013-6851).

Die Installation der Zubehöre ist in den entsprechenden Handbüchern eingehend beschrieben.

Diese Anleitung enthält:
- Hinweise speziell für die Zubehörinstallation am Lambda 2
- Hinweise zum Betrieb von Zubehör mit Methoden, die nur am Lambda 2 verfügbar sind.

**Leiterplatte** Für den Betrieb mit Zubehör muß das Lambda 2 mit der Zubehörleiterplatte (B017-9708) ausgerüstet sein.

**Zubehör-parameter** Nach dem Parameter **BACK CORR** folgt der Parameter **ACCESSORY**. Mit **ACCESSORY** kann das verwendete Zubehör gewählt werden. Je nach Zubehör erscheinen weitere, spezielle Parameter, vergl. auch Gerätehandbuch Lambda 2.

**Zubehör** Diese Anleitung enthält Hinweise zu folgenden Zubehören:

Küvettenwechslersysteme (B012-7391)

Single Sipper (B012-7390)
Single Sipper mit Multisampler

Super (Return) Sipper (B012-6829,
B012-6830 elektr. thermostatisierbar)
Super (Return) Sipper mit Multisampler

Econo Sipper (B013-8597)
Econo Sipper mit Multisampler

Multisampler (B012-6831)

Analogschreiber

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**Probenraum-platten**

Das Lambda 2 hat eine 1-teilige Probenraumplatte.
Einige Zubehöre müssen daher anders installiert werden, als in den entsprechenden Anleitungen beschrieben:
Zum Teil wird die zusätzliche halbe Probenraumplatte benötigt, z. T. einen anderen Probenraumdeckel.

**Manuelle Küvettenwechsler (B009-9356 und B008-9416)**
Benötigen eine zusätzliche halbe Probenraumplatte für den hinteren Teil des Probenraumes.
Zuerst die halbe Probenraumplatte einsetzen und mit den mitgelieferten Schrauben sichern, dann den Küvettenwechsler einsetzen.

**LC Mikro-Durchflußküvette (B011-0662)**
Benötigt den Probenraumdeckel B018-0210.

**Lichtleiter-Zubehör (B016-6476, B016-6511, B016-6512)**
Benötigt den Probenraumdeckel B018-0210.

**Thermostatisierbarer Super Sipper (B012-6830)**
Den Küvettenhalter von der mitgelieferten Probenraumplatte entfernen und direkt auf der Probenraumplatte des Lambda 2 anbringen.

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**Kabel und Schläuche**

Die Zubehöre werden z. T. über Steuerkabel mit dem Spektrometer verbunden, z. T. sind Schlauchverbindungen nötig (z. B. bei thermostatisierten Zubehören).

Kabel und Schläuche sollten so verlegt werden, daß sie nicht beschädigt werden, den Meßstrahl nicht stören und der Probenraumdeckel richtig geschlossen werden kann.

Die Kabel der Küvettenwechsler vom Boden des Probenraumes, unter dem Spektrometer hindurch, zum entsprechenden Anschluß führen.

Die Kabel der Sipper von der Vorderseite des Spektrometers, unter dem Gerät hindurch, zum entsprechenden Anschluß führen.

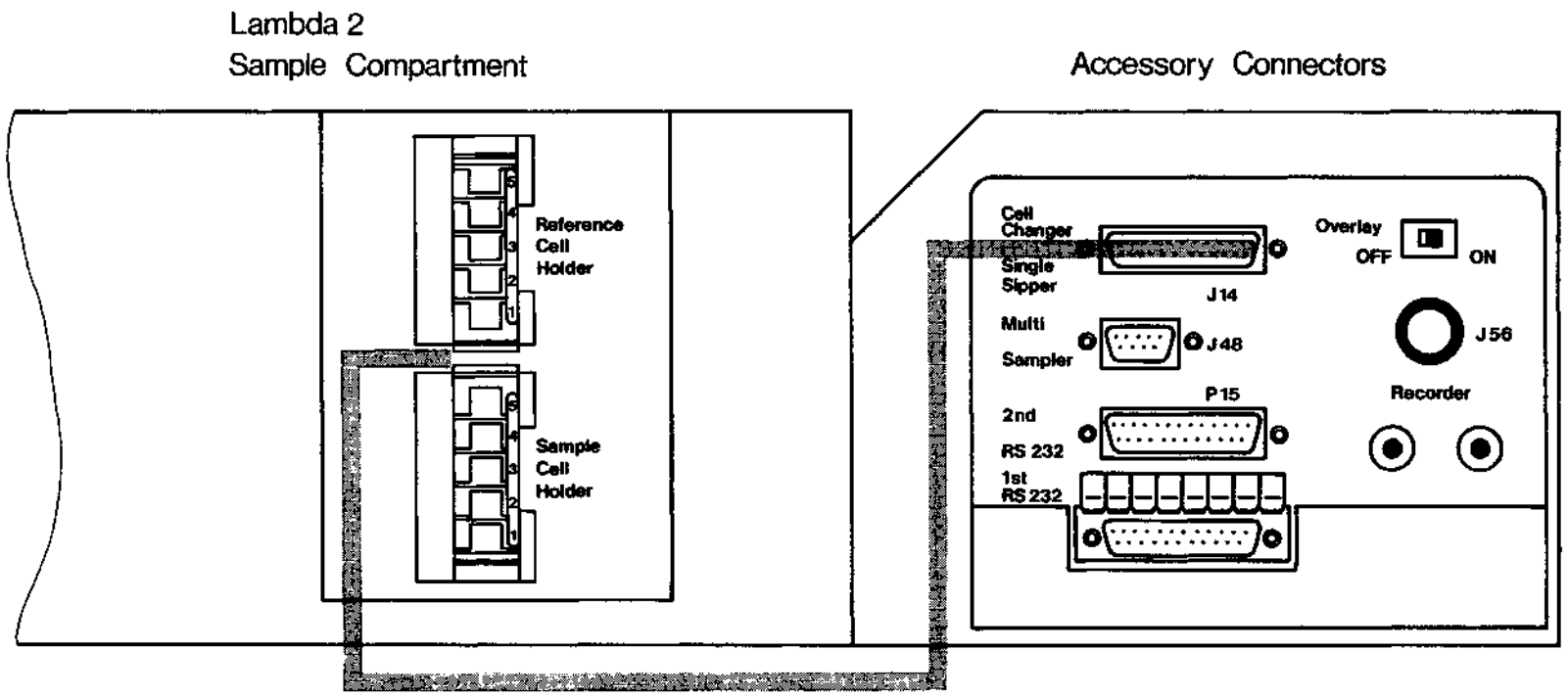
Schläuche so im Probenraum verlegen, daß sie nicht beschädigt werden (z. B. mit Magnethaltern an den Wänden befestigen). Die Schläuche durch die 4 Bohrungen in der Vorderseite des Probenraumes herausführen.

**VORSICHT:** Vor Installation oder Ausbau eines Zubehöres unbedingt

- das Lambda 2 und sämtliche Zubehöre ausschalten
- und die Netzstecker der Geräte ziehen.

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# 2 KÜVETTENWECHSLER

**Installation** Das Steuerkabel des Küvettenwechslers vom Boden des Probenraumes zur rechten Geräteseite führen (Zubehöranschlüsse). Den Kabelstecker in Buchse J14 (Cell Changer) einstecken.

**Parameter** Mit dem Parameter **ACCESSORY** den Küvettenwechsler wählen. Mit dem nachfolgenden Parameter die benutzten Probenpositionen wählen: Position 1 - 5 (5x5-Halter) bzw. Positon 1 - 6 (6x6-Halter).

**BACK CORR** Für den Nullabgleich wird grundsätzlich Position 1 benutzt. Je nach Markierung des Parameters **BACK CORR** kann daher die Position 1 u. U. nicht für Probenmessungen benutzt werden:

FIX-/CALL-Markierung: Die Blindlösung für den Nullabgleich muß in Position 1 eingesetzt werden.
Nach erfolgtem Nullabgleich können alle Positionen für Probenmessungen benutzt werden.

BATCH-Markierung: Wenn **BACK CORR** = **YES**, wird Position 1 ausschließlich für den Nullabgleich benutzt. Proben können erst ab Position 2 eingesetzt werden.
Wenn **BACK CORR** = **NO**, können alle Positionen für Probenmessungen benutzt werden.

START-Markierung: Position 1 wird ausschließlich für den Nullabgleich benutzt. Proben können nur ab Position 2 eingesetzt werden.

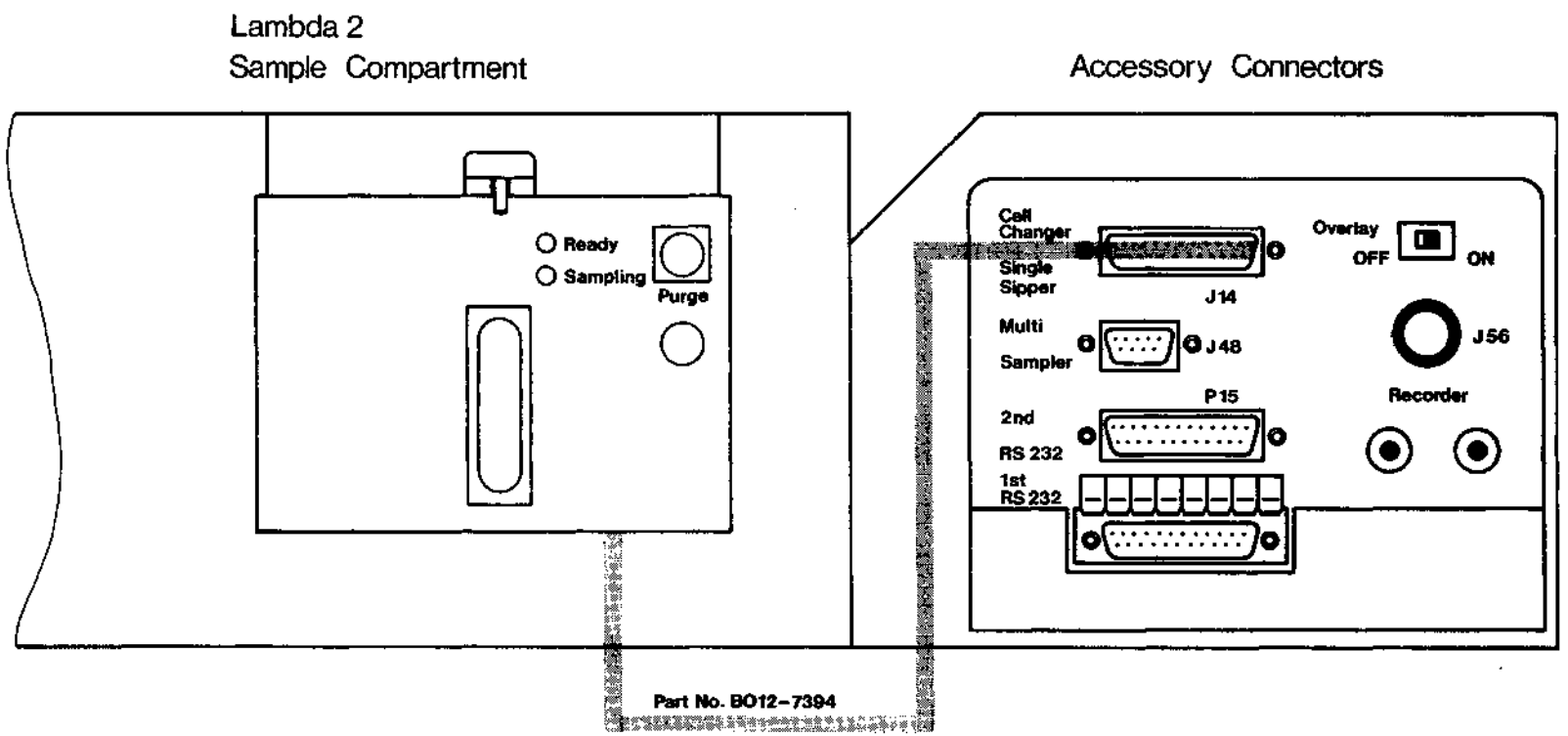
**Conc-Methoden** Bei Conc-Methoden im Zusammenhang mit Küvettenwechslern bitte folgende Punkte beachten:

Wenn **BACK CORR** = **YES**, wird vor der Messung der Standards grundsätzlich ein Nullabgleich durchgeführt.
Daneben gelten die o. g. Bedingungen für den Nullabgleich.

Nach dem Nullabgleich können die Standards gemessen werden. Dazu können alle Positionen benutzt werden.

Sollen 10 Standards im 6x6-Halter gemessen werden,
Standard Nr. 1 - 6 in Position 1 - 6 einsetzen, **START** drücken.
Wenn das Gerät die nächsten Standards fordert,
Standard Nr. 7 - 10 in Position 1 - 4 einsetzen
und **Start** drücken.
Nach der Messung der Standards fordert das Gerät Proben.
Die Proben nur in die Positionen einsetzen, die in der Methode vorgewählt wurden. Dabei die Anmerkungen zum Nullabgleich beachten!

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# 3 SINGLE SIPPER

**Installation** Bei der Installation die Anleitung zum Single Sipper beachten.
Das Steuerkabel unter dem Spektrometer hindurch zur rechten Anschlußleiste führen.
Den Kabelstecker in Buchse J14 (Single Sipper) einstecken.

**Sipper-steuerung** Das Spektrometer erkennt den Sipper als Zubehör, sobald er an die Buchse J14 angeschlossen ist.
Den Parameter **ACCESSORY** auf **SI-SIPP** setzen (**ACCESSORY** folgt direkt nach **BACK CORR**).
Die folgenden 3 Parameter sind typische Sipperparameter:

SAMPLING TIME — Zeitraum, über den die Probe in die Durchflußküvette eingesaugt wird (erlaubter Wertebereich 0,1 ... 99,9 s).
Die zu wählende Zeit ist abhängig vom Küvettenvolumen und von der Probenviskosität.
Parameter-Nr. 43 ruft den Parameter direkt auf.

DELAY TIME — Equilibrierzeit (erlaubter Wertebereich 0,1 ... 99,9 s).
Parameter-Nr. 44 ruft den Parameter direkt auf.

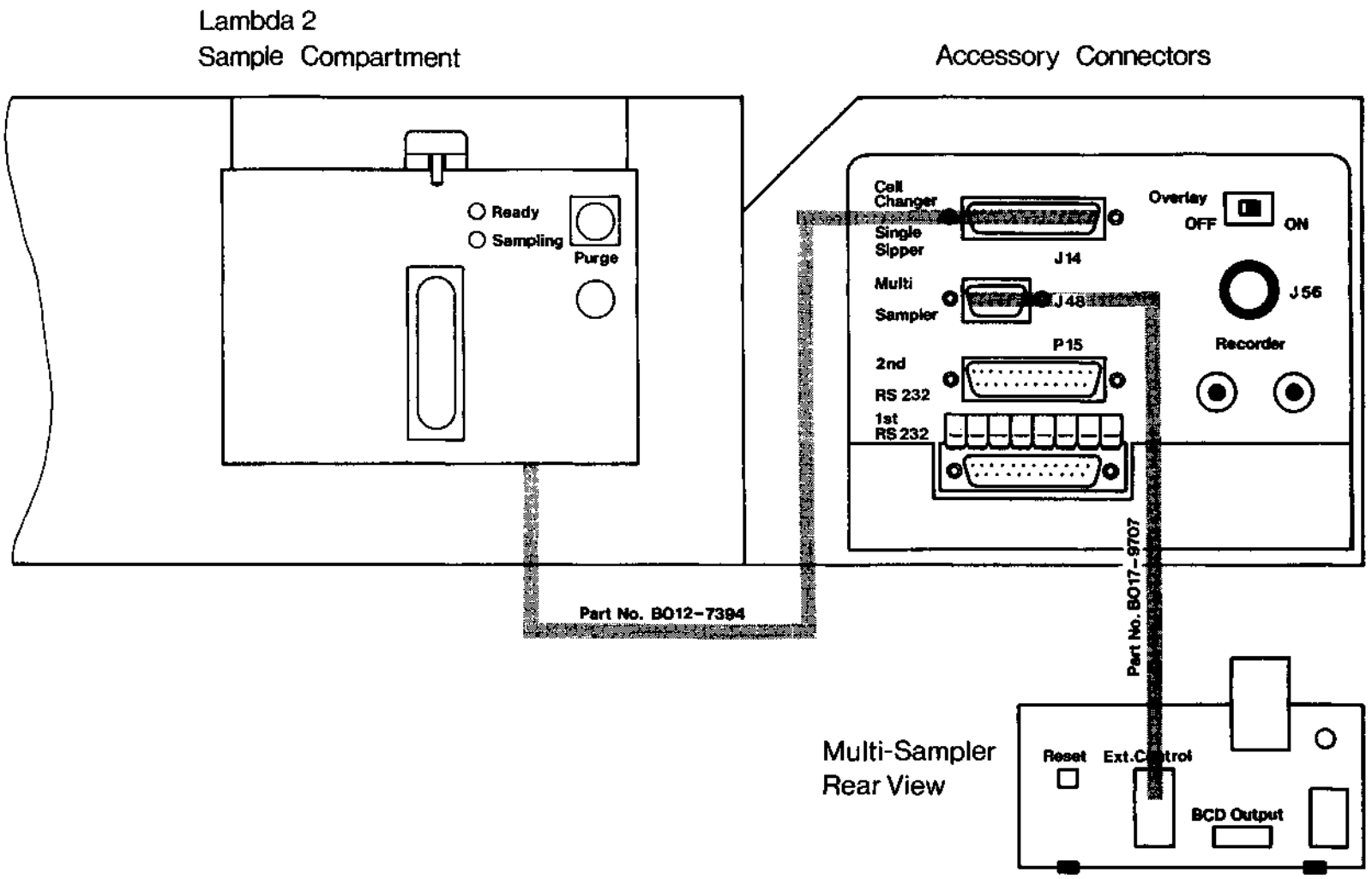
AUTO PURGE — Ein- und Ausschalten der AUTO-PURGE-Funktion (automatisches Spülen).
Parameter-Nr. 45 ruft den Parameter direkt auf.

**Betrieb** Das Spektrometer arbeitet mit dem Single Sipper wie im manuellen Betrieb, nur sehr viel schneller.
Folgende Punkte sind dabei zu beachten:

* Wenn **BACK CORR** entsprechend gesetzt ist, kann zu Beginn der Methode ein Nullabgleich durchgeführt werden:
- Den Ansaugschlauch in die Blindlösung halten und die Start-Taste am Sipper drücken.
  Blindlösung wird angesaugt und ein Nullabgleich durchgeführt.

* Nach dem Nullabgleich fordert das Spektrometer die 1. Probe:
- Den Ansaugschlauch in die Probenlösung halten und die Start-Taste am Sipper drücken.
  Probenlösung wird angesaugt und gemessen.

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## 3.1 Single Sipper und Multisampler

**Installation** Bei der Installation die Anleitung zum Single Sipper und zum Multisampler beachten.
Den Single Sipper an Buchse J14 anschließen, den Multisampler an Buchse J48 anschließen.

**Steuerung** Die Zubehöre erst einschalten, wenn das Spektrometer die Einschaltroutine beendet hat.
Für die Kombination Single Sipper/Multisampler werden die gleichen Parameter angezeigt wie für den Single Sipper.

**Betrieb** Mit Single Sipper und Multisampler ist automatischer Betrieb möglich.

- Am Multisampler AUTOMATIC und COMPLETE CYCLE wählen. Fordert das Spektrometer eine Probe, schaltet der Multisampler zur nächsten Probe, und der Ansaugschlauch taucht in die Probe ein.
- Am Multisampler die Taste MANUAL ADVANCE drücken. Probenlösung wird angesaugt und gemessen. Nach der Messung schaltet der Multisampler zur nächsten Probe.
- Um eine Probe zu überspringen, am Multisampler MANUAL und TUBE ADVANCE wählen, anschließend MANUAL ADVANCE drücken.

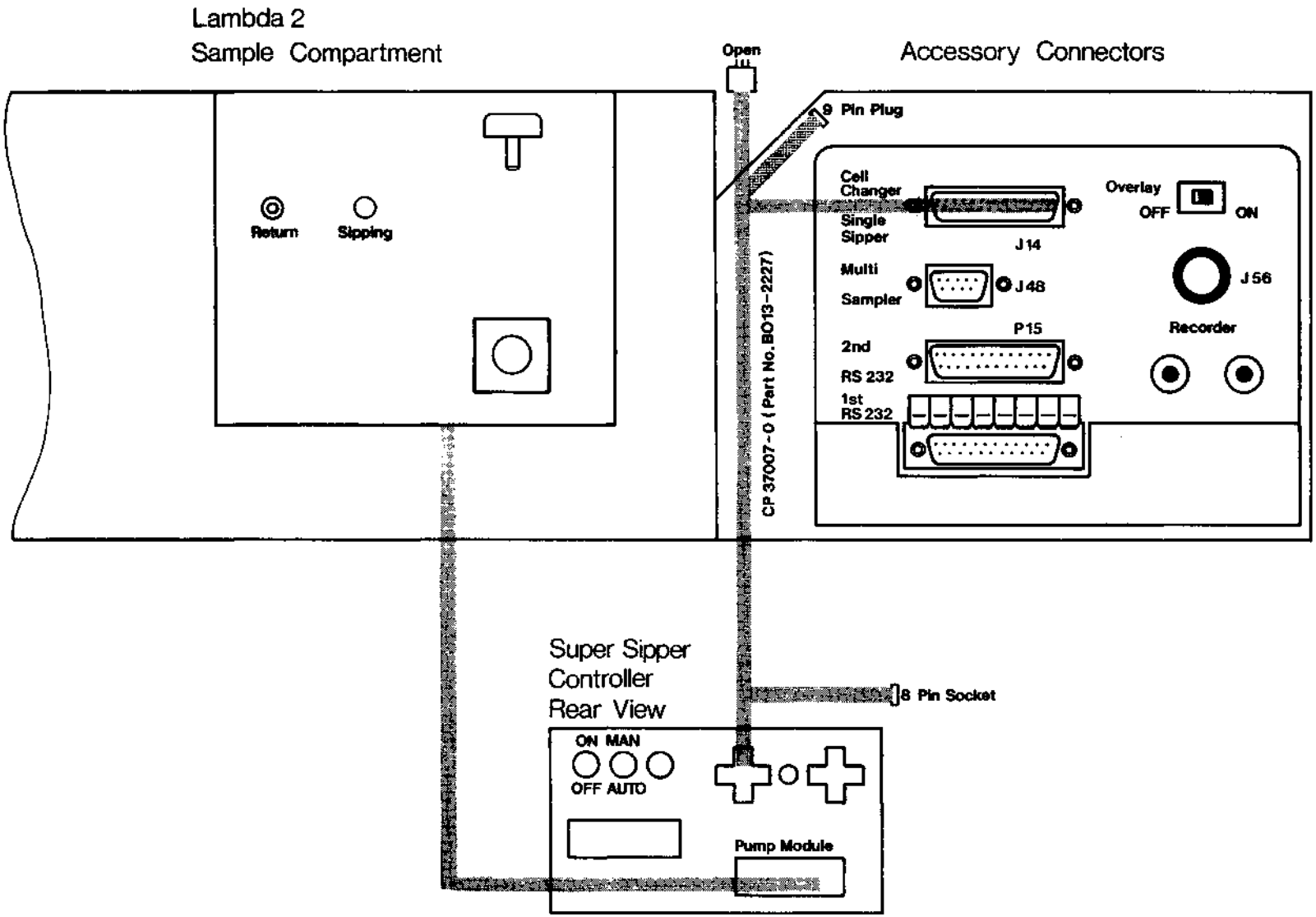
BATCH-Betrieb Der Multisampler kann auch für BATCH-Betrieb benutzt werden. Beim Bestücken des Multisamplers die entsprechenden Abschnitte der Multisampler-Anleitung beachten.

- Soll vor jeder Probenreihe (BATCH) ein Nullabgleich durchgeführt werden, **BACK CORR** mit der BATCH-Markierung versehen. Vor jede Probenreihe eine Blindlösung einsetzen. Auf keinen Fall den Parameter ändern, wenn er während des Betriebes angezeigt wird: Die Reihenfolge der Proben gerät sonst durcheinander.
- Nach jeder Probenreihe erscheint auf der Anzeige "ACCESSORY START". Die nächste Probenreihe mit der Taste MANUAL ADVANCE (am Multisampler) starten.

Conc-Methoden Den Multisampler sorgfältig bestücken:

- Bei **BACK CORR** = **YES** wird vor den Standards ein Nullabgleich durchgeführt, unabhängig von der Parametermarkierung. Vor die Behälter mit den Standards eine Blindlösung setzen.
- Ist **BACK CORR** mit der BATCH-Markierung versehen, vor jede Probenreihe eine Blindlösung setzen. Auf keinen Fall den Parameter ändern, wenn er während des Betriebes angezeigt wird: Die Reihenfolge der Proben gerät sonst durcheinander.

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# 4 SUPER (RETURN) SIPPER

**ACHTUNG:** Vor der Installation des Super Sippers bitte unbedingt beachten:

Das Steuerkabel B013-2227 muß geändert werden.
Dazu am 25-poligen Stecker die Verbindung von **Pin 14** zum Kabelschirm auftrennen.
Ohne diese Änderung wird die Leiterplatte im Spektrometer beschädigt.

Diese Arbeit sollte nur von einem Fachmann ausgeführt werden.

Das geänderte Kabel markieren und nur am Lambda 2 benutzen.

**Installation**

Die Pumpe des Super Sippers im Probenraum des Lambda 2 installieren, dabei die Anleitung im Handbuch zum Super Sipper beachten.

- Die Pumpe mit der Steuereinheit des Sippers verbinden.
- Die Steuereinheit an Buchse J14 (Lambda 2) anschließen.

**Sipper-steuerung**

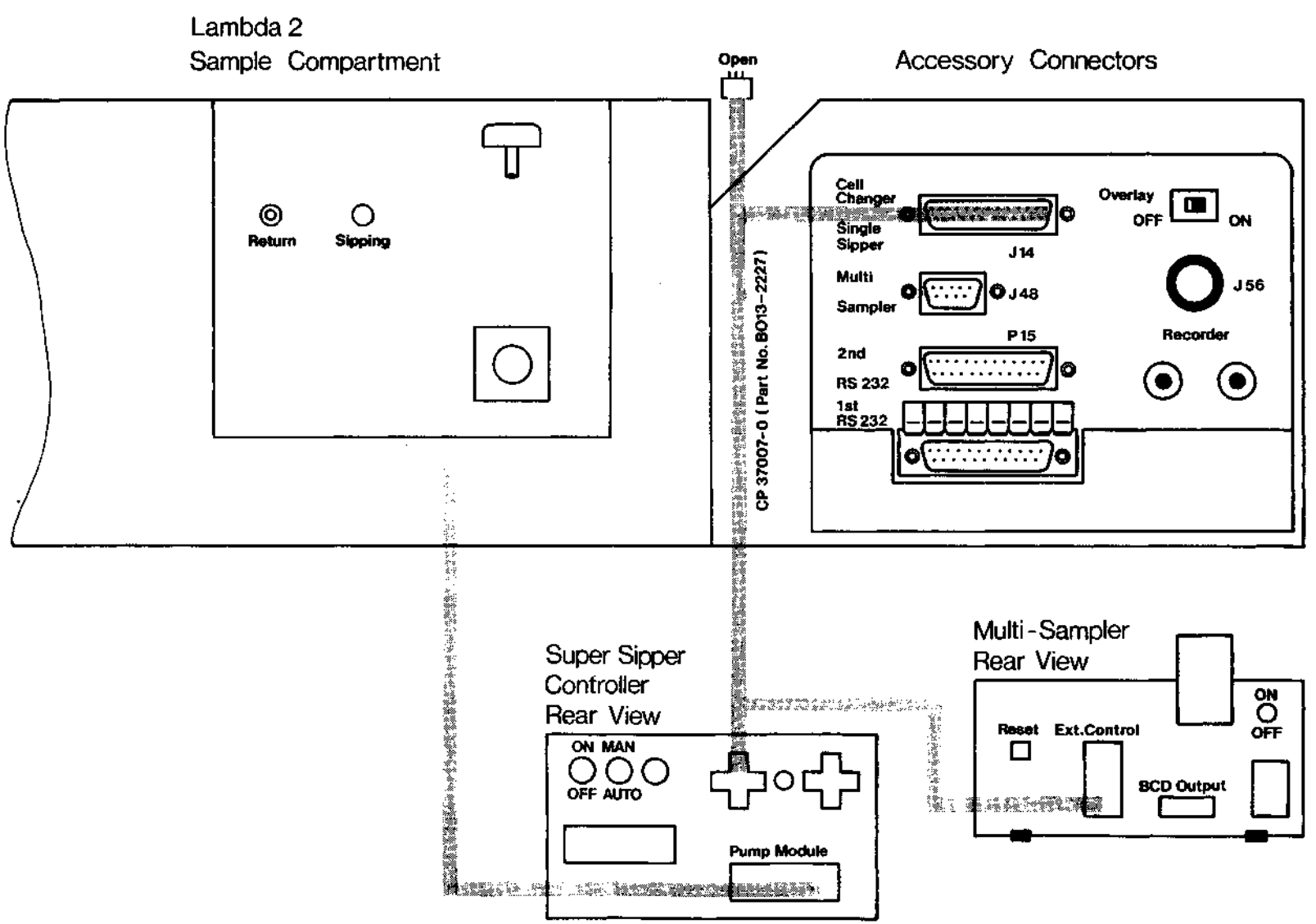
Den Sipper erst einschalten, wenn das Spektrometer die Einschaltroutine beendet hat.

- Der Super Sipper wird über die Steuereinheit kontrolliert. Sipperparameter können nur an der Steuereinheit geändert werden, die Eingabe über die Spektrometertastatur ist nicht möglich.

**Betrieb**

Für den Betrieb mit Super Sipper gelten die gleichen Bedingungen wie für den Betrieb mit dem Single Sipper, vergl. Abschnitt 3.

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## 4.1 Super (Return) Sipper und Multisampler

**ACHTUNG:** Vor der Installation des Super Sippers die Anleitung in Abschnitt 4 beachten.

**Installation** Die Pumpe des Super Sippers im Probenraum des Lambda 2 installieren, dabei die Anleitung im Handbuch zum Super Sipper beachten.

- Die Pumpe mit der Steuereinheit des Sippers verbinden.
- Mit dem 8-poligen Stecker den Multisampler an die Steuereinheit anschließen.
  Den 25-poligen Stecker in Buchse J14 des Lambda 2 einstecken, den 9-poligen Stecker in Buchse J48 des Lambda 2 einstecken.

**Steuerung** Die Zubehöre erst einschalten, wenn das Spektrometer die Einschaltroutine beendet hat.
Die Sipperparameter können nur an der Steuereinheit eingegeben und geändert werden.

**Betrieb** Mit Super Sipper und Multisampler ist vollautomatischer Betrieb möglich.

- Am Multisampler AUTOMATIC und COMPLETE CYCLE wählen.
- Um die Messungen zu starten, die Taste MANUANL ADVANCE am Multisampler drücken. Alle weiteren Vorgänge laufen automatisch ab.
- Um eine Probe zu überspringen, am Multisampler MANUAL und TUBE ADVANCE wählen, anschließend MANUAL ADVANCE drücken.

BATCH-Betrieb Der Multisampler kann auch für BATCH-Betrieb benutzt werden. Beim Bestücken des Multisamplers die entsprechenden Abschnitte der Multisampler-Anleitung beachten.

- Soll vor jeder Probenreihe (BATCH) ein Nullabgleich durchgeführt werden, **BACK CORR** mit der BATCH-Markierung versehen. Vor jede Probenreihe eine Blindlösung einsetzen. Auf keinen Fall den Parameter ändern, wenn er während des Betriebes angezeigt wird: Die Reihenfolge der Proben gerät sonst durcheinander.
- Nach jeder Probenreihe erscheint auf der Anzeige "ACCESSORY START". Die nächste Probenreihe mit der Taste MANUAL ADVANCE (am Multisampler) starten.

Conc-Methoden Den Multisampler sorgfältig bestücken:

- Bei **BACK CORR** = **YES** wird vor den Standards ein Nullabgleich durchgeführt, unabhängig von der Parametermarkierung. Vor die Behälter mit den Standards eine Blindlösung setzen.
- Ist **BACK CORR** mit der BATCH-Markierung versehen, vor jede Probenreihe eine Blindlösung setzen. Auf keinen Fall den Parameter ändern, wenn er während des Betriebes angezeigt wird: Die Reihenfolge der Proben gerät sonst durcheinander.

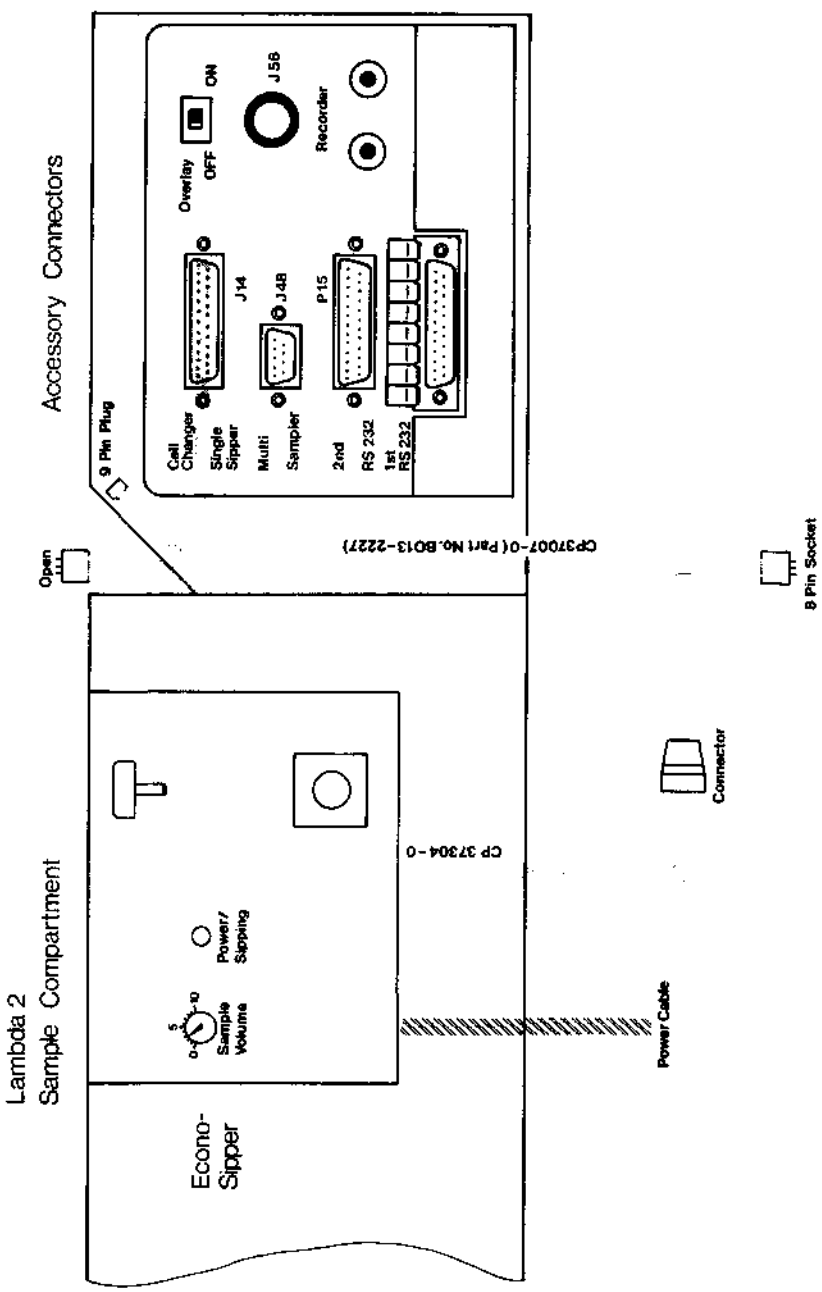
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# 5 ECONO SIPPER

Der Econo Sipper arbeitet nach dem gleichen Prinzip wie der Super Sipper, nur die separate Steuereinheit fehlt. Der einzige einstellbare Sipperparameter ist das Probenvolumen (Drehknopf an der Vorderseite des Sippers).

Am Lambda 2 sind keine Eingaben nötig.

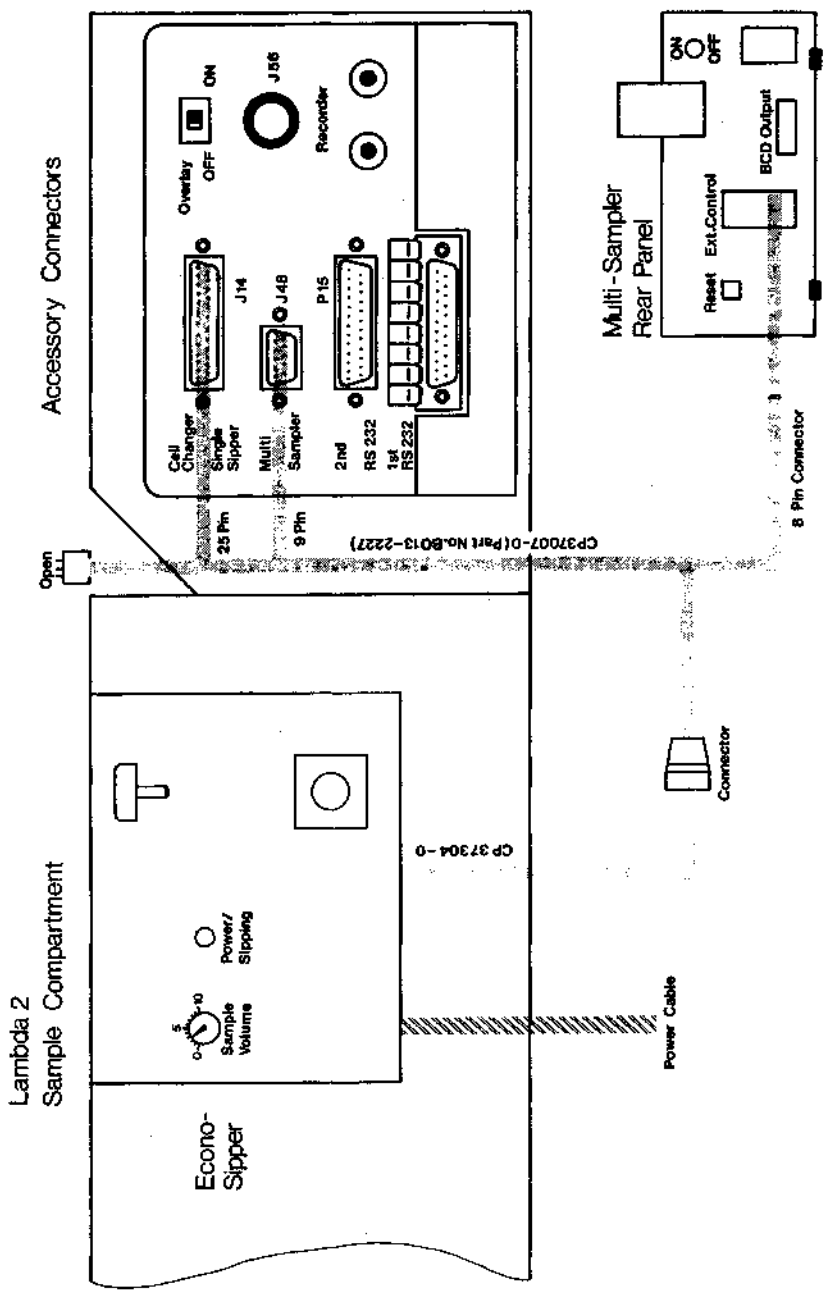
**Betrieb** Siehe Abschnitt 4 (Super Sipper).




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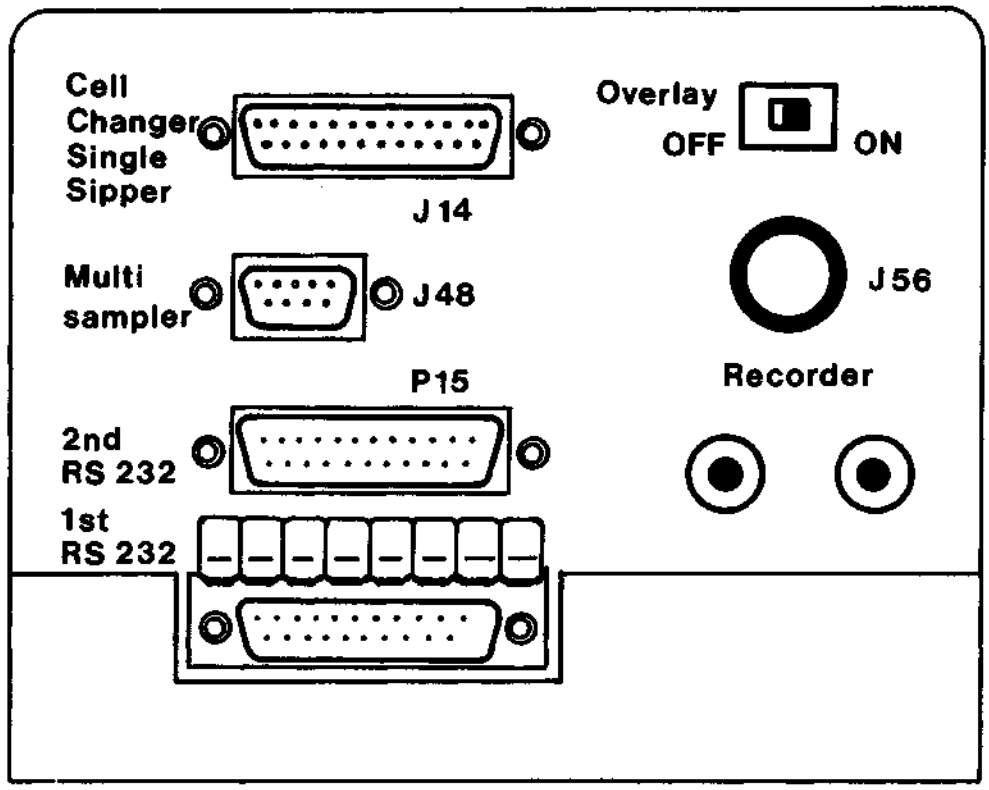
## 5.1 Econo Sipper und Multisampler

Siehe Abschnitt 4.1 (Super Sipper und Multisampler).




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# 6 ANALOGSCHREIBER




**Anschluß**

Die rot und schwarz markierten Buchsen sind die Ausgänge für das Analogsignal (Schreiberausschlag).
Diese Buchsen mit den entsprechenden Anschlüssen am Schreiber verbinden.
Registrierbereich 0 - 1 V, entsprechend dem gewählten Ordinatenbereich am Lambda 2.

An Buchse J56 wird das Steuerkabel zum Schreiber angeschlossen: Den entsprechenden DIN-Stecker des Steuerkabels in die Buchse einstecken.

2 Relais werden über den Anschluß J56 geschaltet:
5G = START-Signal (Pin 1 + 3); startet die Aufzeichnung.
5F = RESPOOL-Signal (Pin 4 + 5); transportiert das Papier zurück und ermöglicht so die Aufzeichnung mehrerer Spektren übereinander (Mehrfachaufzeichnung, OVERLAY).
Die Relais können auch über die RS232-Schnittstelle geschaltet werden (s. Schnittstellenbeschreibung B2133).

Der Schalter **Overlay** schaltet die Mehrfachaufzeichnung am Schreiber R100A ein (ON) bzw. aus (OFF).

Die benötigten Schreiberkabel sind im Lieferumfang der Schreiber R561 und R100A enthalten.

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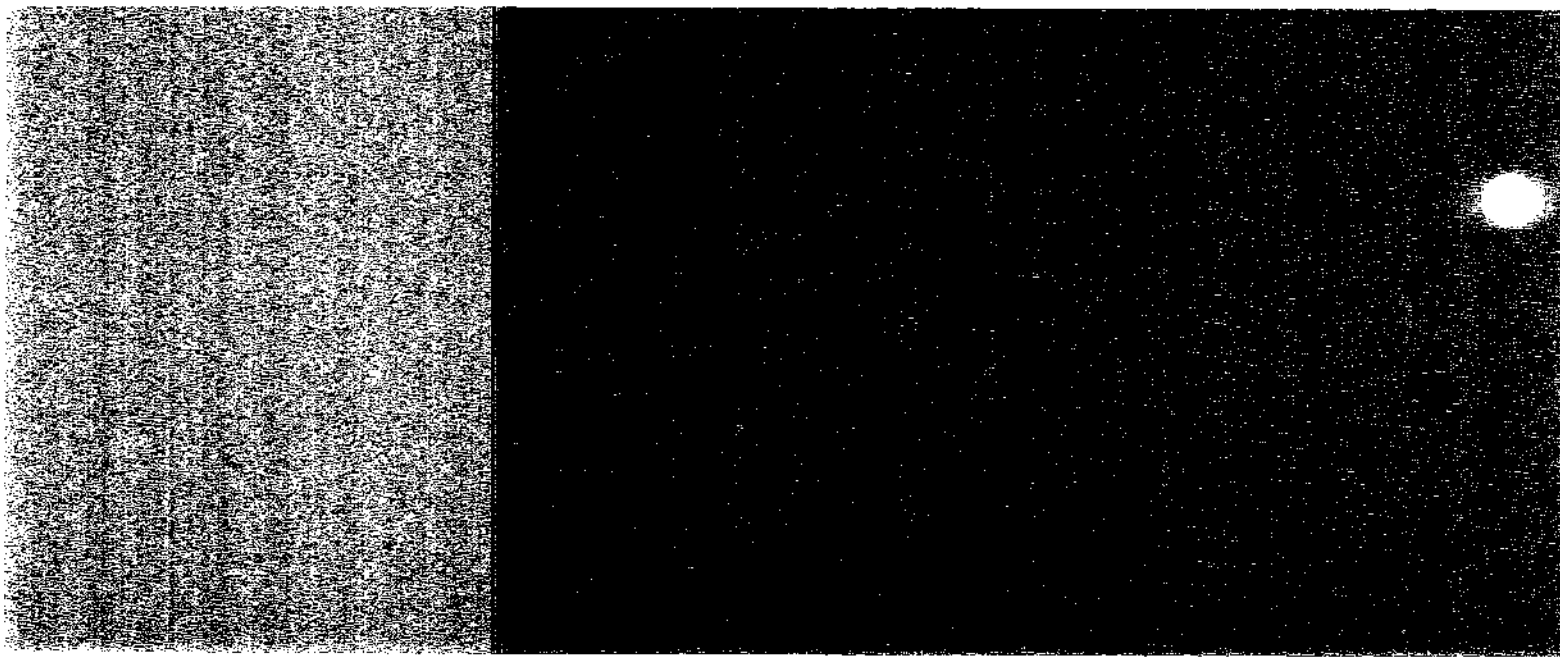
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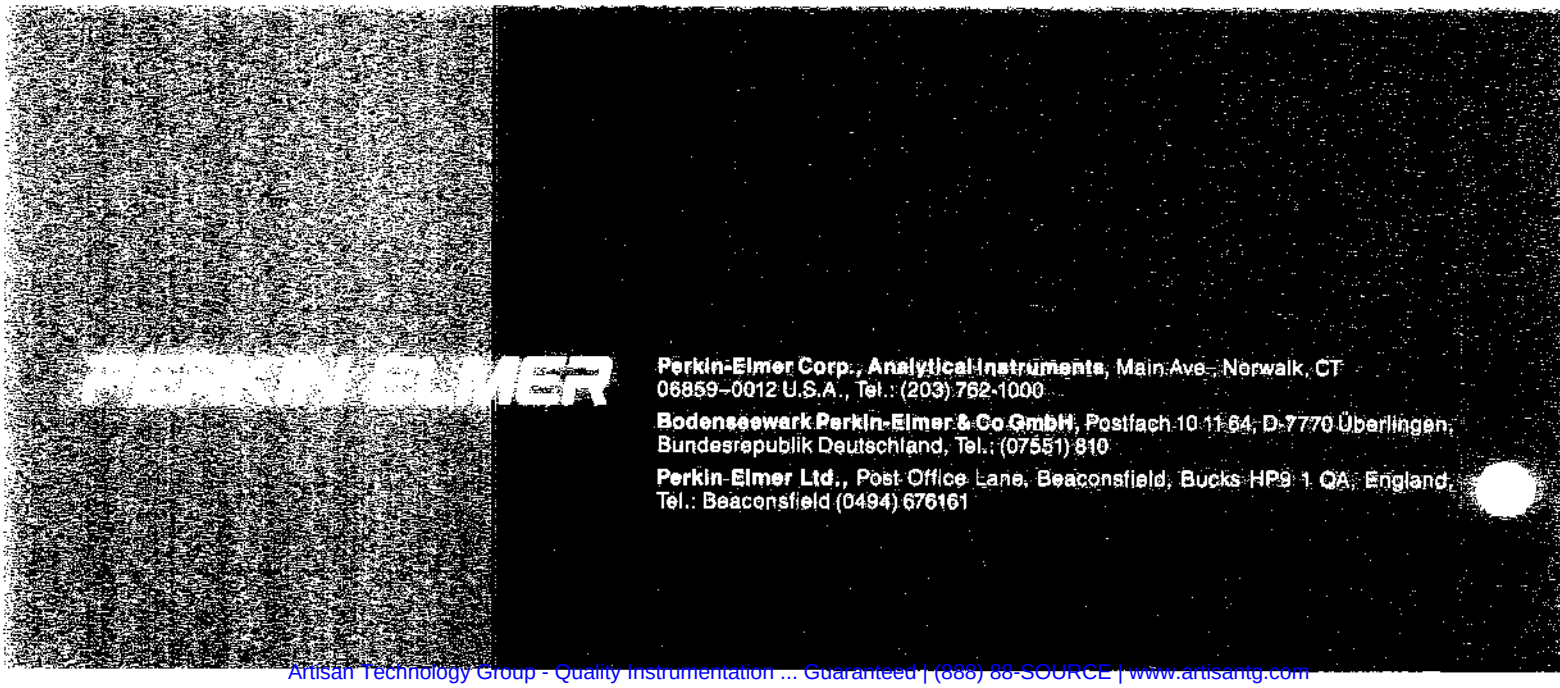
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