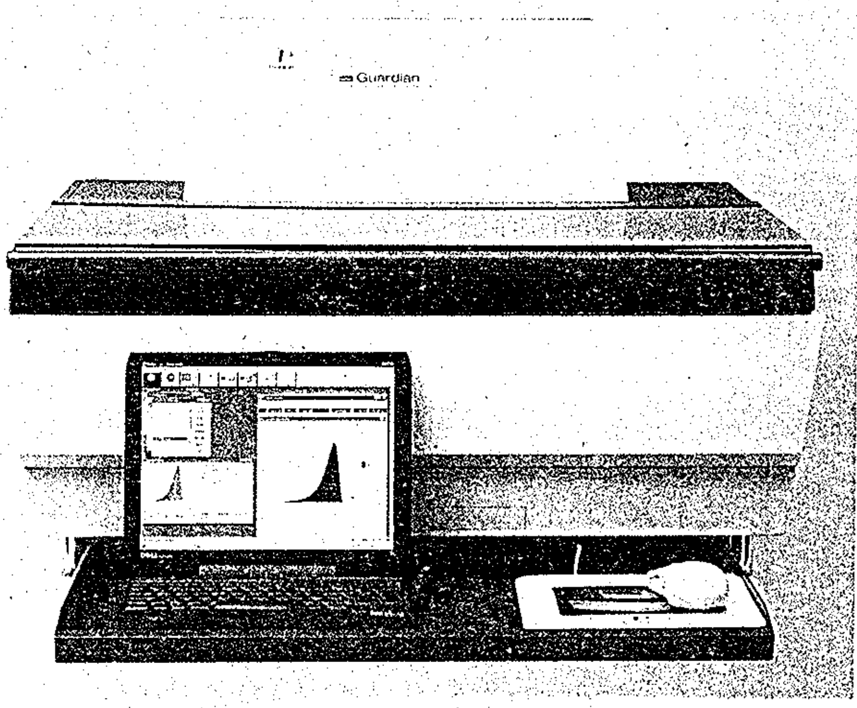
INSTRUMENT MANUAL

1414-932-05
November 2001

WALLAC WinSpectral™

Liquid Scintillation Counter



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Wallac 1414

WinSpectral™

Digital Spectrum Analysis (DSA) based liquid scintillation counter

For instruments with software version 3.0 including WinSpectral α/β and Guardian

PerkinElmer™
life sciences.

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Website: www.perkinelmer.com/lifesciences

Warning

This equipment must be installed and used in accordance with the manufacturer's recommendations. Installation and service must be performed by personnel properly trained and authorized by PerkinElmer Life Sciences.

Failure to follow these instructions may invalidate your warranty and/or impair the safe functioning of your equipment.

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Contents

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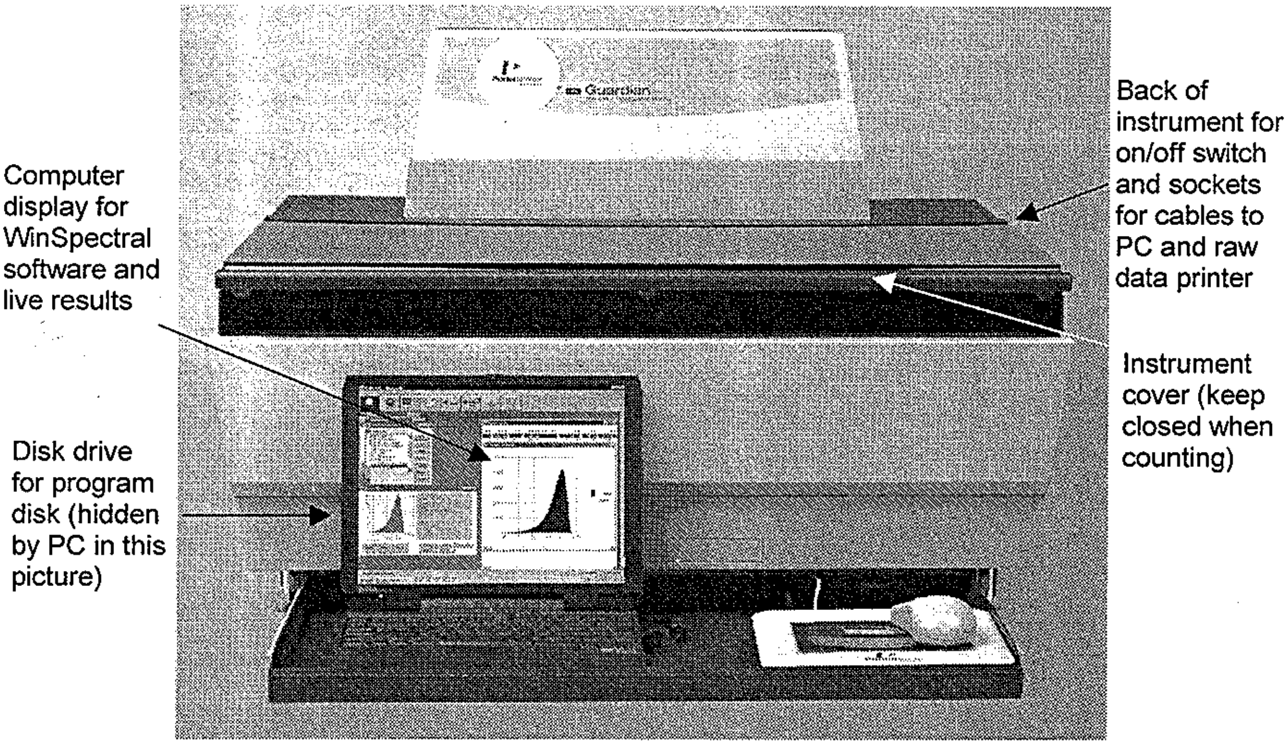
1. Functional description

Introduction

A new LS counter

The Wallac 1414 range of DSA liquid scintillation counters, WinSpectral, WinSpectral α/β and Guardian, is created to bring the best in LSC and PC technology to your laboratory. The combination of Digital Spectrum Analysis (DSA) with the Microsoft Windows graphical user interface as well α/β separation (WinSpectral α/β and Guardian) and sophisticated shielding (Guardian) makes an unbeatable combination.

The figure below shows the instrument and identifies the different parts of the system.



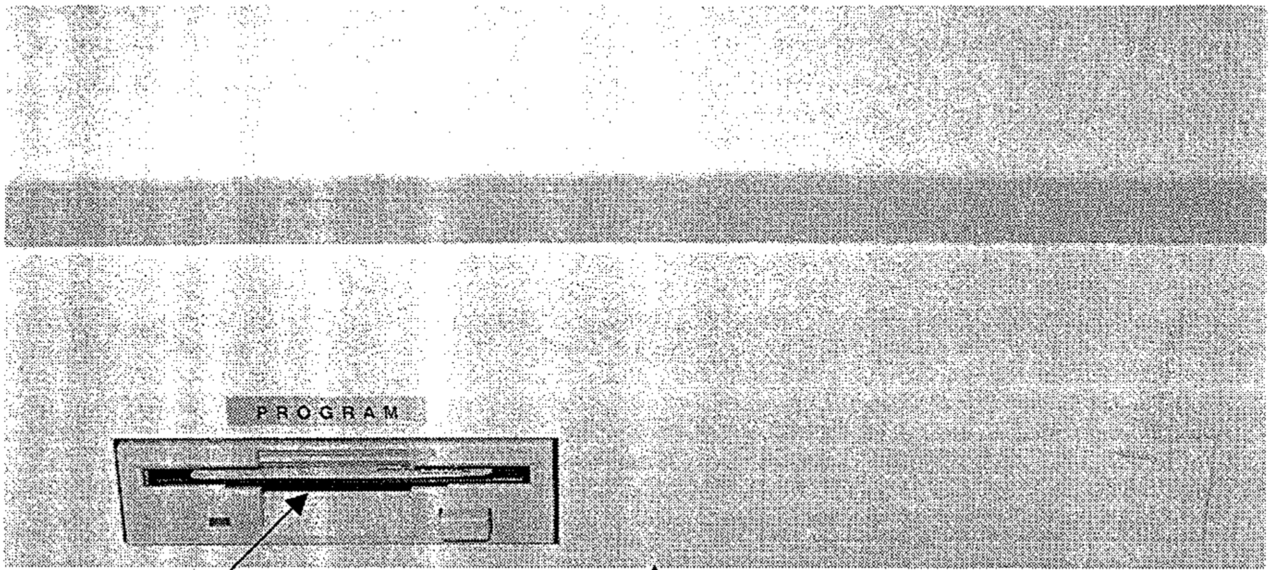
Wallac 1414 Guardian and computer

In this manual you will find a description of what you can do with your counter (whichever model you have), including the various options available, an explanation of how to use the instrument, as well as background information about how it works and how to install it.

Note: in this manual the name WinSpectral is normally used and no distinction is made between the three different models except where they differ.

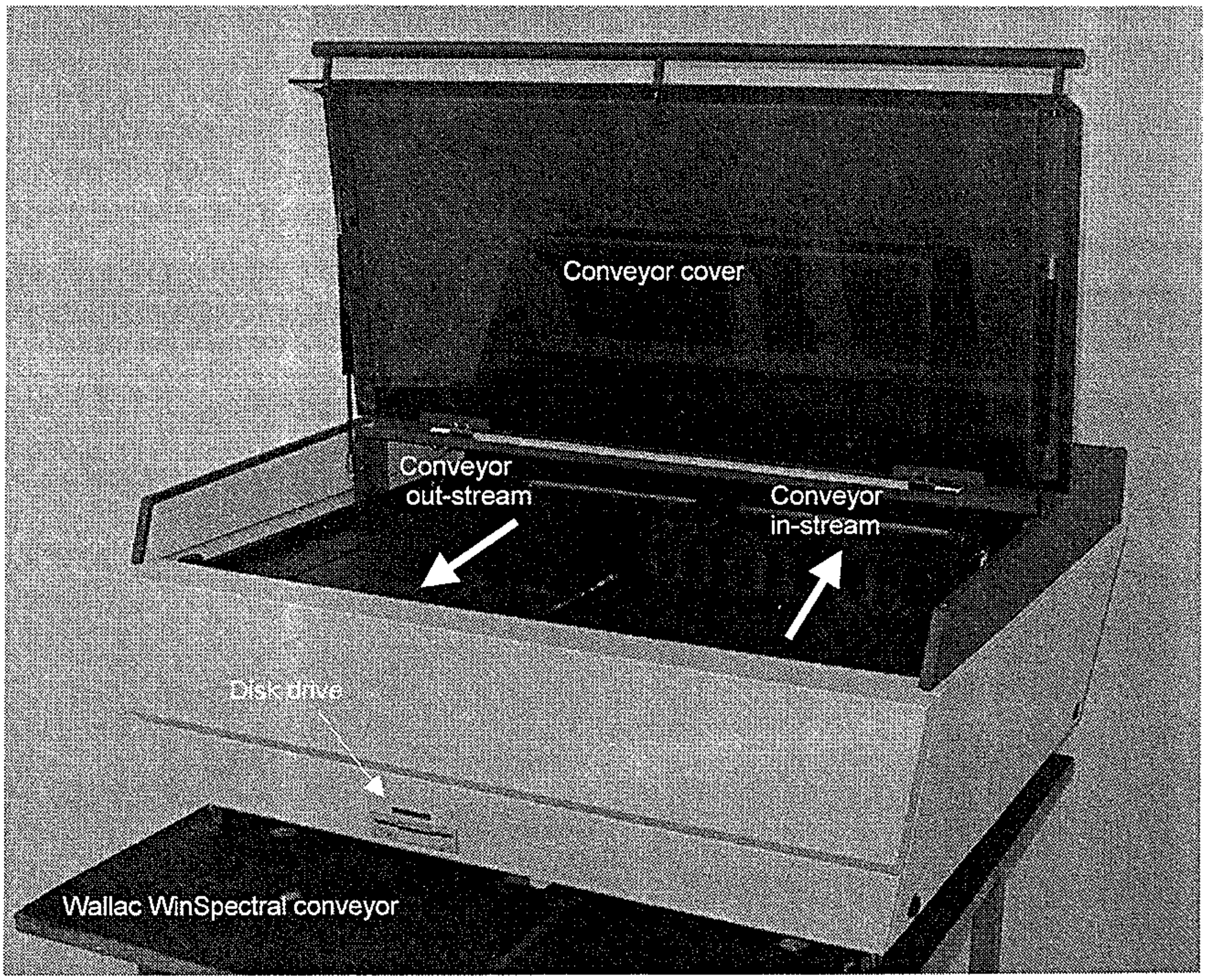
Reset button

If it is necessary to reboot the WinSpectral, there is, as an alternative to the On/Off switch at the back, a reset button located underneath the instrument at the front near the disk drive. Press this to reboot the instrument.



Hardware features and benefits

FlexiRack™ sample changer



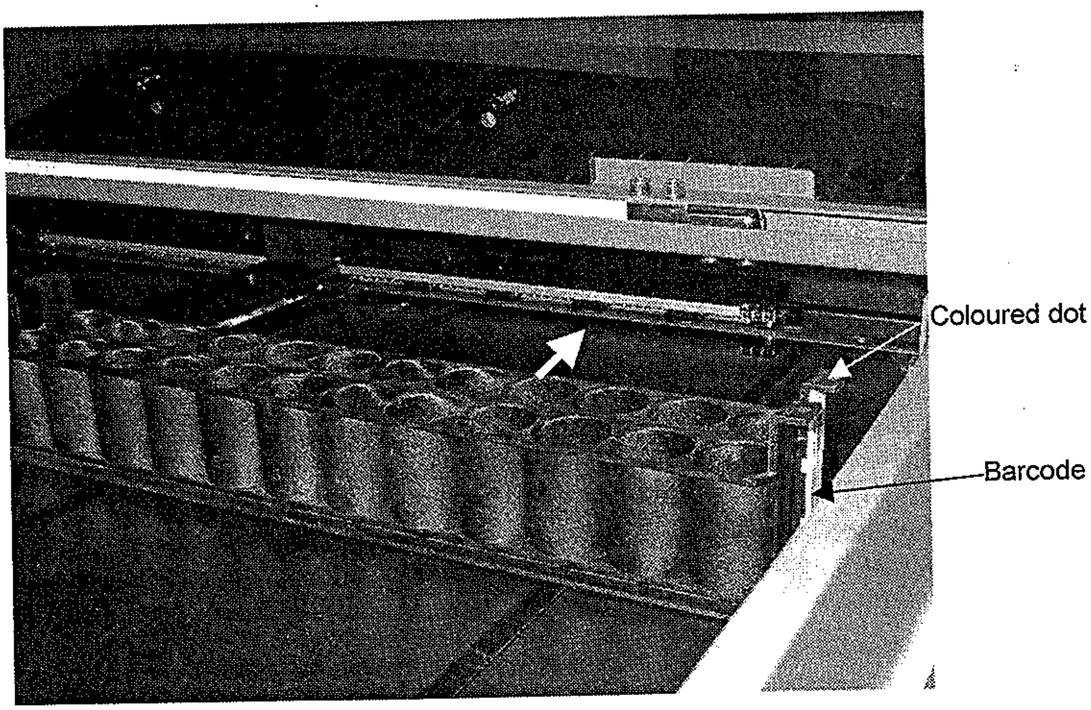
The conveyor is designed to give the maximum visibility to the user. You can easily see the sample racks wherever they happen to be on the conveyor. The cover of the conveyor is made of a plastic which both allows a clear view of the conveyor yet shields the samples from ultra-violet light and thus reduces the problem of luminescence. The cover is supported by a spring-loaded levers so that it opens and closes smoothly.

The sample conveyor has two lanes for sample racks, an in-stream (on the right) and an out-stream, as well as two transfer lanes, one of which is the counting lane. Sample racks glide along the in-stream and out-stream of the conveyor, moved by friction contact with two rubber belts. Movement along the transfer lanes is by precision-made toothed belts. The conveyor is coated by an electrically conductive teflon layer to reduce friction to a minimum and to prevent the generation of static electricity on the sample racks. The conveyor motors run very quietly to avoid disturbance to users and others in the laboratory.

All movement is controlled by stepper motors and directly triggered optical sensors. This removes the danger of sensors jamming and increases the reliability of the whole conveyor system. Sample change time is about 9 s.

Conveyor movement is fully bi-directional. With the OneRack function counting is interrupted, the rack in the counting position is driven backwards so that you can load a single rack for stat counting. After this rack has been counted, counting of the other racks resumes automatically. You do not have to remember to put the interrupted rack back at the head of the in-stream later on because it is already in the right position to resume counting.

Racks are loaded with the coloured dot at the right hand end. This is the end where barcodes are fixed.



WinSpectral conveyor showing how racks should be loaded

Multi-size racks and vials

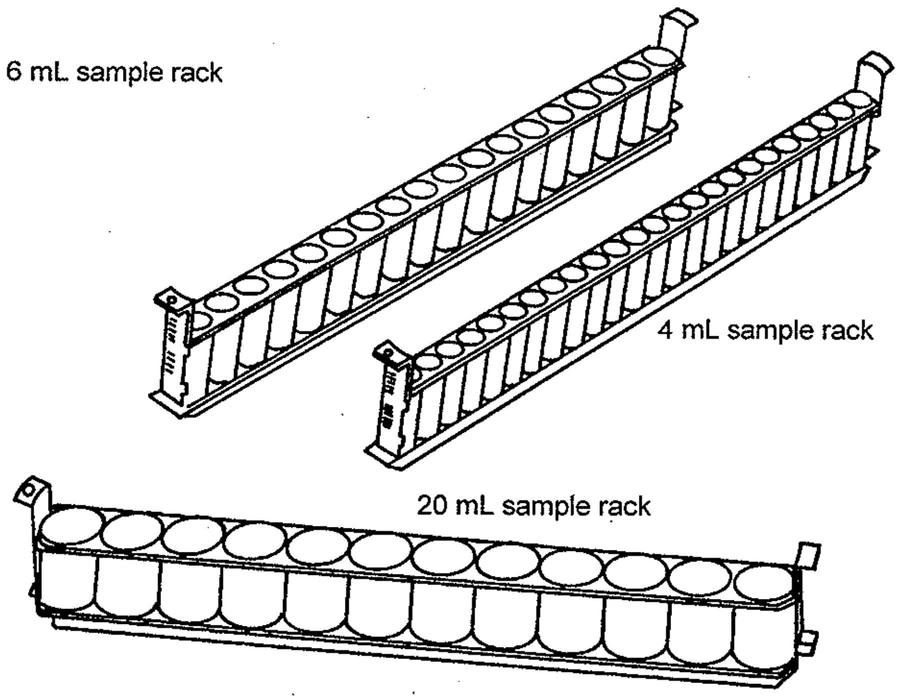
You are not limited to using only one size of rack on the conveyor if you have the appropriate options:

With FlexiRack I you can load racks of twelve 20 ml samples along with racks of eighteen 6 ml samples.

With FlexiRack II you can not only load racks of the above two types but also racks of twenty-four 4 ml samples.

These can be in any order.

A 20 ml sample rack can hold twelve samples with a maximum vial diameter of 28.4 mm. The conveyor will accept 28 racks making a full load of 336 samples.



Racks for different vial sizes accepted by the FlexiRack system

The corresponding figures for 6 ml vials are 18.4 mm diameter, 18 samples per rack and 40 racks on the conveyor making 720 samples in all.

For 4 ml vials the figures are 13.4 mm diameter, 24 samples per rack, 52 racks per conveyor load, i.e. 1248 samples.

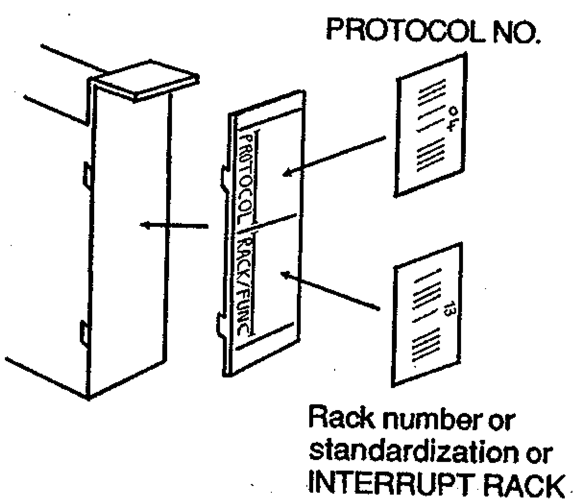
The maximum height allowed for the top of a vial above the conveyor is 78 mm. This means that for a vial of the maximum diameter in any of the categories its length can be up to 68 mm. Smaller vials in any category can be a few millimetres longer.

Not only flat bottomed vials can be accepted but also round bottomed and conical vials. For example adapters allow Eppendorf tubes and Microfuge tubes to be counted directly without the need for carrier vials.

Users with different vial sizes can load their own batches of samples on the same conveyor at the same time.

PosIden™ ID reader

The purpose of the ID system is to allow 'hands off' performance of the instrument and to enable total results identification. 'Hands off' means that counting procedures such as changes of protocol, quench fine tuning or stat counting, can be initiated automatically. Good laboratory practice requires that the results are identified in data files or on printouts with ID numbers. The Wallac WinSpectral PosIden ID reader enables all this.



PosIden system

PosIden uses a barcode reader and ID labels. These are self-adhesive and attach to a strip of plastic, the ID Clip which is then attached to the end of the sample rack. The position of the clip is such that the coding can be easily read without moving the rack, wherever the rack is on the conveyor. The combination of ID labels and clips allow each laboratory to prepare the needed combination of coded ID Clips to meet its individual requirements.

The ready-to-use ID Clip can have labels on it as follows:

The clip for the first rack in the assay has up to two labels

- a label with the protocol number.

- an optional label with the rack number for the first rack.

The clips for other racks in the assay need only to have the rack No. label fixed them

If the rack contains quenched standards the ID Clip is coded with a label showing the quench protocol number (1-99) and instead of rack No. a special label which identifies the samples in the rack as samples for spectrum library fine tuning.

For automatic interrupt counting the rack is coded with a clip with the protocol number and a special label indicating the interrupt function.

The ID labels are supplied in a binder with 20 sheets of labels. The ID Clips are supplied in a package of 100. The box on the PC keyboard shelf can be used to hold the ID binder and clips.

Elevator reliability

The elevator system is designed to handle a whole variety of sample vials without the danger of the vials falling over or getting jammed. The key to this is the small sized elevator head combined with the centralizing counterweight. The counterweight meets samples before they are raised from the rack thus ensuring that they remain vertical all the time they are on the elevator.

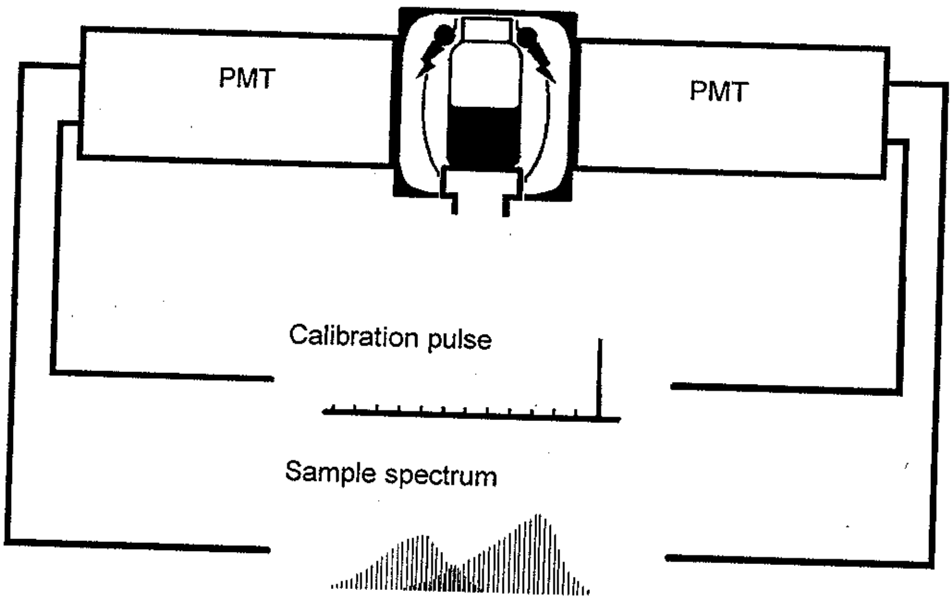
Static elimination

Plastic vials are widely used in liquid scintillation counting and manufacturers tend to use plastic also as a rack material. Conditions are often optimum for the build-up of static charge, especially when relative humidity is low (central heating) and lab technicians have to use plastic gloves during sample preparation (as when using conventional toluene, xylene and cumene based cocktails).

Static build-up on the sample vials is minimized both by the presence of ionizers around the sample elevator and by the fact that at no point does the vial come into contact with any material which might tend to deposit charge on it since a vial in the FlexiRack™ system is raised directly into the measuring chamber without making further contact with other materials. The rack design also reduces static charge build-up, because the samples are located in round holes in the rack without any sharp edges which tend to collect static charges more easily. The light shutter is in the form of two plates which close around the stem of the elevator after the vial is in position.

There is also a Statistic Monitor which flags any samples which show signs of static electricity discharges.

Automatic continuous spectrum stabilization (ACSS)



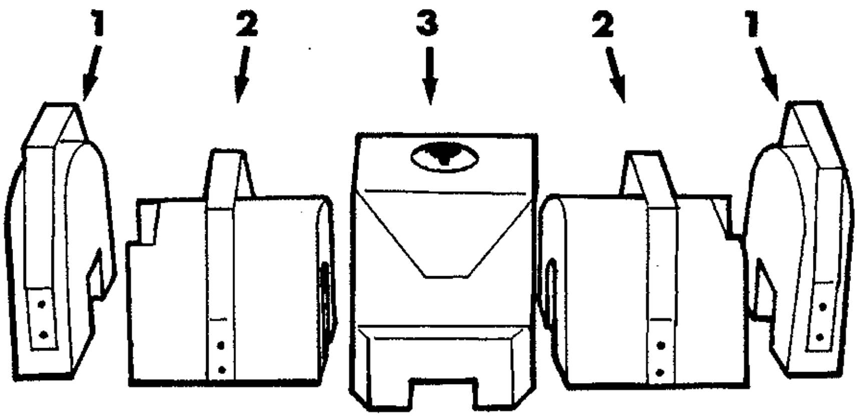
Automatic continuous spectrum stabilization (ACSS) system

You need to be sure that your instrument is stable. You do not want changes in high voltage, temperature, detectors or aging to affect the accuracy of your results. The patented built-in "automatic continuous spectrum stabilization" system ACSS offers gain stability for your Wallac WinSpectral counter without the need to measure reference samples. This system is completely automatic and requires no user intervention.

The principle is to detect when a change that would affect the gain of the instrument occurs and by means of a feedback loop to correct for the change so that the gain remains stable. Each photomultiplier has a temperature compensated reference light emitting diode (LED) which is fixed close to each detector and is used to check the gain of the PMT, see the figure above. A reference pulse is produced in the photomultiplier 10 times a second. The ratio of the pulse produced at the first dynode to that produced at the anode is measured. This ratio should be constant even if the LED output changes. If the ratio changes then the gain has changed. To compensate, the high voltage is adjusted until the reference pulse ratio is back to what it was. This takes care of changes to the high voltage and detectors. A second feedback loop monitors the signal at the first dynode to ensure that the whole system is temperature stabilized.

ACSS also acts as a protection against any damage to detectors through light leaks. If a leak occurs, the voltage to the detectors is automatically reduced thus protecting them from overloading.

Multi-section passive shielding



Order for removing shield (WinSpectral and WinSpectral α/β)

The massive lead shielding combined with the coincidence electronics for the pairs of detectors reduces the background to a low level. However this shielding does not make for an instrument too heavy to be conveniently handled because you can easily remove the lead shielding and then reassemble it when the instrument has been moved to its new position. The figure above shows the pieces and the order in which they should be removed from WinSpectral. Assembly is performed in the opposite order. See the installation chapter for more information about shielding.

Guardian guard detector

The Wallac 1414 Guardian low level counter is equipped with an external guard detector around the sample chamber and phototubes. The guard has its own phototubes and is optically isolated from the sample chamber and thus offers true background event detection. It does not cause observable counting efficiency losses in low level counting. A sample event that is simultaneous with the guard event is rejected by the electronics since the event is then most probably caused by a particle or photon from the environment. No limitation in sample fluor selection is introduced by this background subtraction method.

The guard is switched on automatically, and remains on except when the external standard is being used. Usage of Wallac Guardian is thus identical to that of Wallac 1414 WinSpectral α/β, but the performance is improved due to the lower background rate.

Temperature control for sample homogeneity

When dealing with the maximum sample holding capacity of scintillators it becomes very essential that the temperature remains practically constant through the whole sample batch. Proper temperature control guarantees that all samples are counted at the same temperature in spite of variation of ambient conditions (if for instance the air conditioning is turned off during the night time). Thus the risk of phase separation in samples due to, for example, a temperature drop during overnight counting, is eliminated.

The conveyor can be temperature stabilized. This is achieved by installing the optional Temperature Control unit which can be set to maintain the temperature at a selected value in the range 5°C below ambient to 10° C above. The temperature control system is based on two thermoelectric (Peltier) modules, which enables users to adjust and control the temperature via software.

A more powerful external cooling system (10° C below room temperature) can be added later if needed.

A parameter called TEMP in the programmable output allows you to output the temperature at which sample measurements were made.

External standard for higher precision DPM

The wide range of vial types accepted by Wallac WinSpectral counters puts greater demands on the external standard parameters than ever before. If the count rate of the external standard depends on the sample volume, the few microlitres in a Microfuge tube will give a low count rate. To avoid this the isotope of the external standard is ^{152}Eu, which belongs to a lower toxicity class than previously used external standard isotopes, which means a higher activity can be used thus allowing the external standard count time to be reduced and the counting precision increased.

The external standard source is mounted on a wire which moves it from its rest position in its own shielding to just beneath the sample vial. This arrangement allows the external standard to be positioned very accurately beneath the sample vial (it actually moves through the sample elevator shaft to a position beneath the sample).

The external standard quench parameter value SQP(E) is the endpoint of the spectrum. The use of ^{152}Eu means that you get high precision for the SQP(E) due to the well defined end of the ^{152}Eu Compton spectrum and also high sensitivity for the colour correction feature due to the relatively high energy of the isotope.

All of these features contribute to high precision DPM results.

Electronic convenience

So far we have been concentrating on the "body" of the instrument. Now we turn our attention to the "brains" - the electronics.

This is basically found in three racks and the detector unit. The proven approach of mounting the electronics in racks allows easy upgrading and service thus reducing down time when any changes are made. The central processor unit uses a 16 bit microprocessor to ensure speedy response and data handling.

The electronics not only controls operation of the instrument but it communicates with PC to allow control of the counter from the PC running the Windows WinSpectral software.

Multiple programmable MCA

Wallac WinSpectral DSA counters have a multiple programmable multichannel analyser (MCA). The isotope spectrum is divided into 8 logarithmically set spectral ranges of 4096 discrete channels. The tritium range offers up to 0.0038 keV resolution, this is equivalent to a 520,000 channel linear MCA. The appropriate number of linear channels are summed into 1024 logarithmic channels. These retain the precision of the original analogue to digital conversion of 0.0038 keV per channel.

This feature makes possible the DSA features such as ChemiStrip method of chemiluminescence correction, spectrum stripping background correction and DOT DPM calculation.

Internal disk drive

The counter has an internal disk drive with a 3.5" microfloppy disk which contains the program, see the picture on page 10. The program disk can easily be changed to accommodate program changes and updates. This is a much simpler procedure than changing chips on an electronic circuit board.

External personal computer

The WinSpectral Windows software runs on any 386 (SX), 486 (SX) or PENTIUM computer.

Bench top or floor standing

A Wallac WinSpectral DSA LSC can be either bench top or floor standing. You can select which ever is most convenient for your working environment. The Wallac Floor Stand allows the counter to become floor standing and easily movable without having to collect together lots of separate pieces of equipment.

The monitor arm and a pullout shelf for the PC keyboard and mouse are standard features whether or not you use the Floor Stand.

These all make for ease and convenience in operating the system while keeping the footprint in the laboratory to a minimum.

Software features and benefits

Windows graphical user interface

Older types of LSC software still run under MS-DOS a character based operating system. Since MS-DOS does not allow the possibility of multitasking, any application program or commercial software must be run after the counting protocols. This kind of sequential operation inhibits the effectiveness of a busy research team.

The new generation WinSpectral software running under Microsoft Windows offers dozens of features. No longer are you limited to 640 kB of memory or character based software packages. You can exchange data between WinSpectral and any Windows compatible software.

The graphical user interface itself is intuitive and easy to use. Operation involves clicking buttons or icons with a mouse pointer or selecting items from menus on the PC screen.

Hypertext help

All steps of operation are guided by comprehensive context sensitive help screens conforming to the style you are used to with other Windows software packages. Simply press the F1 key to get help for just the point of operation you are at. You can use the help to search for information on any feature you like. The hypertext links enable you to instantly jump in the help to other related pieces of information. The hypertext help is truly a manual at your fingertips

Easy Count

If you do not want to set parameters then you can use the Easy Count feature. You simply load the samples and click the Easy Count button. The DSA feature allows the counter to determine automatically which isotope you are using from a selection of three (^{3}H or ^{14}C are the defaults) and then counts the samples. An Easy Count protocol can also be initiated by using a special ID label on the racks. Easy Count is described in detail in the User manual.

Easy GLP

Compulsory daily calibration, the so-called GLP (Good Laboratory Practice) feature has proved inadequate in older generation counters since minor temperature fluctuations or voltage changes can affect the response at any time.

The basis for the superior performance of Wallac WinSpectral is the Wallac patented ACSS feature (see the previous chapter on Hardware features).

The GLP protocol allows you to quickly set parameters for the monitoring of up to 8 performance parameters for GLP and to get a ready formatted report after the run.

CPM counting

Introduction

CPM counting is used when sample preparation is expected to yield samples with close to constant counting efficiency. This means that the results of the samples in an assay can be compared with each other and used in further data analysis. A typical CPM application is filter counting.

Wallac WinSpectral allows you to select from six common isotopes plus an additional 69 others.

One error source in CPM counting is that of unexpected counting efficiency variations, e.g. partial elution of the sample from the filter disk. Your Wallac counter includes features to detect and inform the user of counting efficiency variations as described below.

CPM monitor

The CPM Monitor is another feature of your counter. It allows you to be confident of the consistency of the quench level of your samples.

The CPM Monitor checks the SQP(I) value of each sample. If this is within 10% of the SQP(I) of the first sample then there is no flag. If the difference is larger it then calculates and outputs the percentage ratio of the sample SQP(I) to the first sample SQP(I). The acceptance limit (default 10%) is a system parameter and can be changed there, see the User manual.

Spectrum stripping background correction

The traditional methods for background correction are: a typed in background CPM value or a CPM value measured from a background sample in a counting window. However the intensity of the background radiation is different at different energy levels so these methods do not give the best result. The new calculation methods in Wallac WinSpectral DSA counters do not utilize counting windows, but use digital spectrum analysis (DSA) to calculate the CPM/DPM values over the MCA area covered by the sample spectrum.

The background counting time is selected to be either the same as or five times the length of the sample counting time.

Background correction proceeds as follows. Firstly, one or more background samples are measured and a background spectrum accumulated. Then using the spectrum stripping technique the background spectrum is subtracted channel by channel from the sample spectrum. This gives you a reliable background correction.

Note: If you use both background samples and reference samples then the positions of the background samples must be immediately before those of the reference samples and start from position 1. If there are only reference samples they must begin from position 1. No empty positions are allowed.

DPM counting - DOT DPM

Why the need for DPM correction?

A fact which makes LS counting special among analytical methods is that each sample is also a unique detector. Usually analytical methods are based on the principle that the sample emits radiation or some form which is measured in an external detector, e.g. gamma counter or an external source sends radiation which is absorbed by the sample, e.g. spectrophotometric methods.

In LS counting, each sample consists of the actual sample mixed with the detector, the scintillation cocktail. Thus counting efficiency is dependent on sample type, sample to cocktail ratio, volume, colour of the sample, cocktail type etc. The counting efficiency variation from sample to sample must be corrected for to allow comparisons and analysis of the samples in a batch. To do this several methods have been developed during the lifetime of LS counting.

Other methods

Several methods are offered for DPM calculations in LSCs e.g. methods based on counting windows, methods based on isotope spectrum quench parameters, methods based on external standard quench parameters, methods based on extrapolation (Efficiency Tracing), methods based on factory installed quench curves etc.

Common to these methods is that they are not universal, this means they are good for some applications but give bad results for others. This requires a good knowledge to select the right method for any specific application.

Usually the methods require special calibrations, quench curves, to be measured which are dedicated to a specific experimental situation, isotope pair, cocktail, colour or chemical quench etc. Preparation of quenched standards, checking of quench curves etc. is time consuming. In dual label counting, counting window settings are critical and different isotope ratios demand different window settings. Separate standardizations must be done for different isotope combinations. Possible occurrence of colour quench will cause systematic errors.

The Wallac solution, one universal method

DPM counting in Wallac WinSpectral uses the DSA features, the Digital Overlay Technique, DOT, and the use of spectrum libraries. DOT is the most general method available today for quench correction in LS counting.

Digital Overlay Technique - DOT

DOT is used to reconstruct a standard spectrum at the same quench level and intensity as the unknown sample. When the standard spectrum has been fitted, the DPMs are calculated.

The procedure is the same for dual or triple label counting except that when the standard spectrum for each isotope has been reconstructed these spectra are combined and fitted to the composite spectrum of the unknown sample. After a successful fit the isotope ratios and intensities are established and the DPM values calculated.

Spectrum library

A Wallac WinSpectral with DOT DPM has a library of spectrum data comprising information about isotope/cocktail/vial type combinations for about 100 quench levels (10 chemical x 10 colour). This library is built-in to the counter during its production. Along with each combination the SQP(E) and colour index are determined and saved. WinSpectral then works not with quench curves, but with quench surfaces which represent both chemical and colour quenching.

In sample counting, the SQP(E) and colour index are measured and the corresponding point on the quench surface is determined. This is then combined with the counting mode, isotope and vial type specified in parameter setting to get a specification for the spectrum data to be selected from the library.

Advantages of DOT DPM

- the Wallac library provides Easy Count DPM results without quench curve measurements.

- no quench curve measurements are needed for specific isotope combinations in dual or triple label counting.

- no window settings are needed

- result quality is not dependant on isotope ratios

- a successful fit gives assurance of sample quality

- no systematic errors caused by colour quench

- no plastic vial effect

- no volume dependence

DOT allows for analysis of the fine structure in the isotope spectrum which can be used to warn the user of phase separation, contamination or other phenomena which will give erroneous values.

Accuracy Enhancement for even better results

A typical DPM measurement includes determination of quench level with the help of the external standard, calculation of the counting efficiency corresponding to the quench level with the help of a quench curve, counting of the sample to obtain sample CPM and then the final DPM calculation.

The critical factor which determine the quality of the DPM results is how correctly the counting efficiency was determined. The accuracy of the counting efficiency value totally depends on how accurate the External Standard Quench parameter is and how exactly the quench curve used represented the unknown samples.

The external standard values suffer from counting error due to the counting statistics. The modern external standard quench parameters are usually calculated from the endpoint of the external standard spectrum and depend on the shape of the spectrum. With higher quench levels and smaller sample volumes the shape becomes more and more undefined and the counting error greater. Consequently there will be bigger errors in the external standard quench parameter and bigger DPM errors.

In the DOT method the SQP(E) value is only the starting point for an iterative procedure which searches the spectrum library for the spectrum which gives the best possible fit to the measured unknown spectrum. The Accuracy Enhancement gives the following features:

Accuracy Enhancement minimizes the DPM errors caused by erroneous external standard quench parameter values.

Savings in time, only a few seconds counting time is needed for the external standard even for samples with small volumes.

Normally the counting efficiency - quench parameter relation is also dependent on the quenching agent and the cocktail used. Thus, if the samples are quenched by another quencher than the one used for the quench curve, systematic errors will occur. With Accuracy Enhancement systematic errors due to 'Quenching agents' or different cocktails are minimized, thus the applicability of the Wallac libraries is increased.

When using Easy Count it is not necessary to specify spectrum library or vial type, Accuracy Enhancement will search through all variations to find the best fit.

SQP(I) DOT, Single Label DPM

The SQP(I) DOT option offers you single label DPM results without the use of the external standard. The method is based on the Digital Overlay Technique. This means that no counting window is required.

To use the SQP(I) DOT you need to make a quench standardization or fine tuning of one of the HiSafe or Xylene quench data sets in the Wallac library. The standard spectra and the counting efficiency are stored as a function of the SQP(I) instead of the external standard quench parameter.

The fine tuned spectrum library can then be combined with counting protocols to obtain DPM results of unknown samples.

Depending on the number of protocols (15 or 100) the SQP(I) DOT option allows you to make 14 or 99 quench standardizations.

Dual DOT CPM/DPM

This offers you dual label counting without using the external standard. You need to make two fine tunings, one for each isotope to be used to label unknown samples. For each fine tuning you must only use one standard and the chemical and colour quenching of these two standards must be the same. Samples can have quench levels which differ to some extent from those of the standards because the Accuracy Enhancement procedure takes account of this.

When you define the DPM counting protocol you specify the two isotopes to be used, then the program prompts you to give the numbers of the two sets of fine tuned data.

Alpha/beta separation and background reduction (option)

Note: this feature is an option for 1414 WinSpectral and can be installed later; for 1414 WinSpectral α/β and 1414 Guardian it is built-in.

Introduction

Electrons from beta decays and electromagnetic (gamma, X-ray) interactions as well as Cerenkov phenomena produce pulses consisting mainly of prompt or fast fluorescence. On the other hand, the heavily ionizing particles, such as alpha particles or neutrons produce pulses with a more delayed (slow) component and are thus longer than those produced by beta particles. WinSpectral with alpha/beta separation option utilizes Pulse Shape Analysis, PSA, to separate between the long pulses typical for alpha decay and the shorter pulses typical for the gamma or beta background radiation.

The other function of this option is called PAC (pulse amplitude comparison). PAC rejects more background counts than sample counts because background pulses have greater amplitude disparity than do sample counts. Adjustment of the PAC can lead to a better figure of merit.

Benefits

The background of glass vials exhibits some slow fluorescence which is induced by cosmic and other environmental radiation and the inherent radioactivity of glass material. Pulse shape analysis is therefore a useful method of reducing this background in beta counting. The glass fluorescence appears mainly in the spectrum region of low energy beta particles such as those from tritium. The simultaneous alpha/beta separation allows you to measure alpha radiation with a background of less than 1 CPM. The sensitivity for alpha detection approaches the detection limit for semiconductor alpha particle detectors but with considerably simpler sample preparation.

The ability to simultaneously measure gross alpha and gross beta activity reduces the workload in the laboratory because the alpha detection level is sufficient to detect alpha activity at the picocurie activity levels which are the allowed activity limits.

Moreover simultaneously with the measurement of alpha radiation, beta radiation from the decay chain can be detected and analyzed. The beta spectrum may contain also Cerenkov radiation, conversion electrons, Compton electrons, X-rays and Auger electrons if they are being produced in the sample.

The PSA/PAC features can also be used to discriminate a part of the background pulses from the sample pulses. The best benefit of this is obtained when glass vials and a fast scintillator are used. Especially in low energy beta counting considerable reduction of glass vial background can be achieved by using the PSA.

Due to this alpha/beta separation feature Wallac WinSpectral with this option has a lower background than a standard LS counter. Thus the improved sensitivity can be used to minimized sample volumes and specific activity, with savings in consumables and waste as a result.

Note: vial carriers as an guard for small vials can be used to further improve background values.

How does PSA work?

The PSA integrates the delayed (tail) and the prompt (peak) light pulse from a sample producing both types of radiation. If the ratio of the delayed to prompt component exceeds the pre-set ratio, the pulse is directed into the long pulse or alpha spectrum. If the ratio does not exceed the pre-set ratio, the pulse is directed into the short pulse or beta spectrum. Very low background count rates are achieved for alpha particles, since most of the LS background is composed of short pulses.

The above mentioned pre-set ratio is controlled by the user with the PSA level, whose range is from 0 to 255 in steps of 1. The higher the PSA level is set, the more counts will be directed into the long pulse spectrum. At PSA level = 255 all counts are directed into the long pulse spectrum, and at 0 all counts go into the short pulse spectrum. An optimum value for the best alpha/beta separation can be found somewhere between these two extremes as explained in the next paragraph. PSA levels higher than the optimum reduce beta counting efficiency and background leading often to better figures merit. Lower PSA levels than optimum do same for alpha counts.

Determining the PSA level

Cocktails produce different pulse lengths; HiSafe cocktails show relatively slow pulses while xylene and toluene based cocktails are fast. The PSA option allows the optimum PSA level to be found to match the cocktail speed. This is done by stepping through a range of PSA values.

Ascertaining the correct PSA level is ideally done with two reference samples: one that emits alphas (e.g. Am-241) and the other that emits betas in the alpha spectrum range (e.g. Cl-36, Sr-90, P-32). Count rates should be kept below a reasonable limit by dilution if necessary (less than 10 000 CPM is recommended). The reference samples must be made in the same type of vial, same cocktail and same mixing ratio as the actual unknown samples to be measured.

A protocol can be created which steps PSA levels through a range which is appropriate for the cocktail. See the User manual for details. There are two extra windows that can be set which in range are typical for the alpha region but the first one is for measuring the beta spectrum (short pulses) while the second one is for the alpha spectrum (long pulses). The results are printed, each line contains the CPM value for both windows, the PSA level used and a calculated field, 'RATIO' which is the CPM value in the alpha window divided by the total CPM value. For example, you can select the final PSA level to allow from 0.5 to 5 % loss of alpha counting efficiency (alphas spilling into the beta channel). In this way an almost pure alpha spectrum is acquired with the minimum loss of counting efficiency. In the same way, when you are only interested in beta counting in the presence of alphas (or glass originated background) you may allow some percentage loss of beta counting efficiency, (betas spilling into the alpha channel).

Note that the slower the cocktail the lower will be the optimum PSA level. You may use a more limited window to include the main alpha emission range, e.g. for Ra-226 and Rn-222 Ch 600-800 is good with water miscible HiSafe cocktails.

A printout and a plot of 'RATIO' vs. repeat number is generated in the run and the spectra are saved.

The optimum PSA level for glass vial background reduction in beta counting is found in a similar way. You prepare two samples, one "hot" and one background sample. You then step over the desired PSA levels and select the final level with e.g. 10 % of "hot" counts spilling into the alpha category.

For rapid use without stepping you can select LOW as the default PSA level for slow cocktails and HIGH for fast cocktails.

How does PAC work?

The Pulse Amplitude Comparator (PAC), can be used for background reduction in uncoloured samples. The sample pulse amplitudes from the left and right phototubes differ less from each other than do the background pulse amplitudes since quite many of the latter ones are generated in the phototubes themselves by environmental and internal radiation.

Determining the PAC level

To find the suitable PAC level for optimum background reduction an active beta sample and a blank sample are made, matching the samples to be measured in the type of cocktail, mixing ratio and vial.

A protocol can be created which scans the PAC levels over a range which is appropriate for the cocktail, see the User manual for more details. Both the active sample and the background are measured. The count rates in the windows of the beta emitter are printed.

There are two extra windows, both wider than the whole spectrum, but the first one is for the beta spectrum while the second one is for the PAC rejected beta spectrum. The results are printed; each line contains the CPM value in each window, the PAC level used and a calculated field, 'RATIO' which is the CPM value in the beta window (the pulses with less amplitude disparity than defined by the PAC level) divided by the total CPM value. The higher the PAC level the closer to each other must the amplitudes be for the pulse pair to be accepted. A greater number of pulses will be rejected at high PAC values than at low ones.

A printout and a plot of 'RATIO' vs. repeat number is generated in the run and spectra saved.

The optimum PAC level is the one at which E*E/B, beta counting efficiency squared over background or figure of merit in the beta window, is at a maximum.

PAC does not reject alpha pulses as much as beta pulses since the number of photons from an alpha decay is very much greater than that from a beta decay. The variation of the left and right pulse amplitudes is thus less for an alpha decay event than for a beta decay.

Chemiluminescence

Why is chemiluminescence a problem?

About two thirds of all samples in LSC are of biological origin which means that they comprise long macromolecules. In order to get them soluble in organic scintillator solution these macromolecules have to be hydrolysed to smaller fragments. This process often results in the release of chemiluminescence in samples.

How is it solved?

There are two levels of solution to the chemiluminescence problem, the first of which is common to most beta counters and the other is specific for Wallac WinSpectral DSA counters.

Chemiluminescence events are "single photon" ones. Each time chemiluminescence occurs a single photon is emitted in one direction. In contrast, a normal radioactive decay releases a burst of several photons in different directions (a "multiple photon" event). A beta counter has two detectors viewing the sample vial from opposite sides. A chemiluminescence event (or any other single photon background event or random noise event in a detector) will trigger only one of the detectors whereas a radioactive decay will trigger both detectors almost simultaneously. By requiring that a signal be received from both detectors within a few nanoseconds and by rejecting signals that only come from one detector or the other, most of the chemiluminescence and random background can be cut out.

However there are limits. The period of a few nanoseconds referred to above is called the coincidence resolving time of the detectors. If two chemiluminescence or other random events occur within the coincidence resolving so that each triggers one of the detectors then the result will look like a true multiple photon event and will be counted. If there is a high rate of chemiluminescence events many of these "random coincidences" will occur resulting in false count results. The DSA feature ChemiStrip™, a unique patented spectrum stripping chemiluminescence correction method, solves this problem.

The counter has five 1024 channel MCAs. ChemiStrip uses one MCA to store the combined sample and chemiluminescence spectrum and another the chemiluminescence alone. This second spectrum is obtained by making use of the difference between the chemiluminescence and sample spectra. The latter comes from discrete bursts of photons which occur within the coincidence resolving time of the detectors. If the signal from one detector is delayed relative to the other then no coincidence will occur and no count will be recorded in the "delayed spectrum" MCA. In the case of chemiluminescence the spectrum is formed by an almost continuous flood of photons. This means that there is just as much chance of a random coincidence between two single photon events occurring at the same time as there is between one event and the delayed signal of a previous event. The chemiluminescence events will thus contribute to the delayed spectrum whereas the sample events will not.

Channel by channel stripping (subtraction) of chemiluminescence from the sample + chemiluminescence spectrum is then done. This maintains the exact shape of the sample spectrum by removing the exact chemiluminescence spectrum from the combined one. The corrected CPMs are then used in further calculations. The result is that you can cease to worry about chemiluminescence. Just select chemiluminescence correction "on" and your Wallac WinSpectral will look after the rest.

Isotope decay

The problem of decay

If you are counting samples labelled with an isotope such as phosphorus 32 with a half life of 14.2 days you are likely to face the situation where the count rate for samples drops over the course of an assay. In this case there will be about a 1% change from the first to the last samples in three hours. With longer counting runs or repeat runs over several days this could introduce significant errors in your results.

The solution

When you are setting your LSC or measurement parameters protocol you select half-life correction as part of the advanced mode parameter. Since you will have already specified the isotope(s) used the counter will know exactly what correction to make. In addition, you can specify if you want the initial time for the correction to be the start of the counting of the assay or a date which you set. See the User manual for details of parameter setting.

Sample quality monitor

Sample inhomogeneity

One problem in liquid scintillation counting is inhomogeneity of the mixture composed of the sample being analysed and the liquid scintillation cocktail. If sample and cocktail are not adequately mixed some of the beta particles will be absorbed in the sample phase and thus not reach the scintillation liquid phase. This will appear as a reduced counting efficiency for the sample.

This happens because the external standard spectrum which is produced by the Compton electrons resulting from the gamma radiation from the external standard, is not affected by the inhomogeneity of the sample and cocktail mixture. This is because the Compton electrons are generated throughout the whole volume and thus will produce scintillation effectively. The counting efficiency might even be improved for the external standard as the quenching agents are in the sample phase.

Thus the counting efficiency obtained from the measurement of the external standard is not correct if the sample and cocktail mixture is two phased.

The sample and liquid scintillator mixture can be homogeneous when the counting starts, but as time goes the last samples can be inhomogeneous if the samples are close to the sample holding capacity of the cocktail. When using an emulsifying cocktail, the sample is suspended in "drops", micelles, in the cocktail. The micelles are small compared to the range of the beta particle energy so that absorption in the micelles is minimized. However if the sample amount is big close to the sample holding capacity the micelle start to grow, which will affect the counting efficiency.

Sample Quality Monitor

Wallac WinSpectral DSA counters include a Sample Quality Monitor which can be chosen in output selection (see Output items in the User manual).

In the DPM case this monitor evaluates the amount the spectrum of the external standard and the unknown samples deviate from the library values you are using. If the deviation is not significant then the monitor gives the output "100".

If there is a significant deviation but one that might be accounted for by some fine tuning of the library then the sample quality monitor is below 95. You should check your sample and if it seems all right you should consider fine tuning the library you are using.

The Sample Quality Monitor warns you of problems with your sample as well as indicating when fine tuning is necessary.

Sample count rate variation monitor (Statistics Monitor)

The problem

Plastic vials are widely used in liquid scintillation counting and manufacturers tend to use plastic also as a rack material. Conditions are often conducive for the build-up of static charge, especially when relative humidity is low (central heating) and lab technicians have to use plastic gloves during sample preparation (as when using conventional toluene, xylene and cumene based cocktails).

The solution

Wallac WinSpectral has a built-in ionizer unit which removes static charges by sending positive and negative ions towards the vial. Since the vials in the FlexiRack system are raised directly into the measuring chamber without making further contact with other materials, the ionizer is very effective. The rack design also reduces static charge build-up, because the samples are located in round holes in the rack without any sharp edges which tend to collect static charges more easily.

In addition to the ionizer there is the Statistics Monitor. The purpose of this monitor is to warn of unacceptable count rate variation in a sample. The sample count rate in different periods of the sample counting time is measured to determine the sample count rate variation. A χ^2 test is used to see if the count rate variation for each sample lies within the range expected from the statistical nature of radioactive decay. Sometimes it may not lie within this range due to, for example, chemiluminescence, radio frequency signals, static or extreme cases of normal statistical variation. In such cases of unexpectedly high sample count rate variation the sample is recounted.

The test is applied to both the sample alone and the sample with the external standard in position. A maximum of two recounts are made for each of these cases. The various flags that can appear are given in Output items in the User manual.

After a second recount (if there is one) the result of the second recount will be appear as output then counting of the next sample or repeat will begin.

The chemiluminescence monitor and correction feature as well as the special static eliminator, allow the correction of chemiluminescence if it persists, and static is eliminated anyway. However, the Statistics Monitor gives you an independent check on sample count rate variation and avoids results based on unexpectedly high sample count rate variation being accepted undetected.

Advanced statistical result evaluation

If you want to do statistical analysis on your results, as many do especially in a research environment, then your Wallac WinSpectral provides you the tools you need. You can get quench monitoring errors, DPM errors, theoretical errors, observed errors, chi-squares, to mention but a few. In addition you can select trend plots and frequency distributions. You do not need to go to an external statistics program to do the analysis.

The Spectrum Plot option allows the printout of the sample spectrum after the numerical results. The Spectrum Plot option also allows the sample spectra to be sent and stored on the datalogger, PC or Mainframe, if corresponding options are installed.

The Statistical Plot option allows a graphical presentation of the result to be added to the printout extras, e.g. sample CPM as a function of sample number. The Statistical Plot also include Levy-Jennings type plot of repeat or replicate measurements.

Alternatively you can output results to MultiCalc or Excel for further evaluation.

Other computer interfaces

Wallac WinSpectral can also save data in ASCII format files or binary WKS type files. The WKS type files can be read directly into spread sheet programs such as LOTUS 123, Symphony or EXCEL.

Spectrum analysis (option)

Note: this feature is an option for 1414 WinSpectral and can be installed later; for 1414 WinSpectral α/β and 1414 Guardian it is built-in.

Alpha counting and low level beta counting applications include spectrum analysis as a final step.

The counter saves the measured sample spectra in the connected PC. The spectra can then be analyzed off-line using the Wallac Spectrum Analysis program.

The Spectrum analysis program can be installed in the PC connected to the counter but also in a PC in the researcher's office. The program offers:

- off line optimization of counting windows,

- display of up to three spectra simultaneously on screen

- smoothing and zoom function for analysis of details,

- colour graphics

- calculations of Figure of Merit, Counting Efficiency, during on screen optimization

- calculation of radio-carbon age

- calculation of tritium units/litre for ^3H in water

- statistical calculation and Levy-Jennings plots of repeats, replicates or cycles

- high resolution printout of measured spectra.

For the laboratory with recurring routines the Wallac Spectrum Analysis Macro Program allows macro programming of often repeated analysis routines and summing/subtraction of spectrum files.

Password protection

You can set a password to protect any protocol - LSC, fine tuning or Easy GLP. This will mean that the protocol can be used by those not knowing the password but it cannot be changed. To unlock a protocol so that you can change it you must give the password first. If the password is forgotten, your local service representative can get access to the protocol.

How to proceed

Part 2 tells about the User manual which you will find in the package with the program disks. This gives you all the information you need to operate Wallac WinSpectral.

Part 3 describes the methods used for calculating results.

Part 4 contains some of the Quality control information that comes with the counter.

Part 5 gives you information on how to install and start the instrument.

Part 6 presents the instrument specifications, gives safety information and describes routine maintenance that can be performed by the user.

Part 7 is the alphabetical index for the Instrument manual excluding the User manual, which has its own index.

We at Wallac trust that you will find your Wallac WinSpectral counter to be a powerful and reliable aid to enable you to achieve the results you want in your work.

Appendix 1 Isotopes

Number	Name	Window		Half-life	Number	Name	Window		Half-life
1	H3	5	350	12.43y	39	K40	5	1010	1.26E9y
2	I125	5	550	60d	40	K42	5	1024	12.36h
3	C14	5	660	5730y	41	K43	5	980	22.4h
4	S35	5	750	87.4d	42	Kr85	5	890	10.73y
5	Ca45	5	750	164d	43	La140	5	1023	40.27h
6	P32	5	1010	14.3d	44	Lu176	5	950	3.7h
7	Ag110	5	850	249.8d	45	Lu177	5	840	6.7d
8	Ag111	5	940	7.5d	46	Mo99	5	980	66.2h
9	As74	5	1010	17.7d	47	Na22	5	850	2.6y
10	As76	5	1024	26.5h	48	Na24	5	1010	15.02h
11	Au198	5	930	2.696d	49	Nb95	5	660	35d
12	Au199	5	830	3.13d	50	Ni63	5	530	100y
13	Ba140	5	940	12.8d	51	Os193	5	1010	31h
14	Br82	5	900	36h	52	P33	5	750	25d
15	Ca47	5	1023	4.54d	53	Pb210	5	520	21y
16	Cd115	5	1010	44.6d	54	Pm147	5	750	2.623y
17	Ce141	5	870	32.5d	55	Pr142	5	1023	19.2h
18	Ce144	5	770	284.3d	56	Rb86	5	1010	18.7d
19	Cl36	5	900	3.07E5y	57	Ru103	5	725	39.26d
20	Co57	100	750	271.7d	58	Ru106	5	500	369d
21	Co58	5	900	70.8d	59	Sb122	5	1023	2.8d
22	Co60	5	770	5.27y	60	Sb124	5	1024	60.2d
23	Cr51	5	770	27.8d	61	Sb125	5	870	2.77y
24	Cs134	5	870	2.06y	62	Sc46	5	790	83.3d
25	Cs137	5	940	30.17y	63	Sc47	5	870	3.43d
26	Cu64	5	870	12.9h	64	Sr89	5	1010	50.5d
27	Eu152	5	1023	13.3y	65	Sr90	5	850	28.6y
28	Eu154	5	1010	16y	66	Tb160	5	900	72.3d
29	Eu155	5	750	1.81y	67	Tc99	5	660	2.13E5y
30	Fe55	5	750	2.69y	68	Tl204	5	900	3.78y
31	Fe59	5	980	44.6d	69	Tm170	5	930	128.6d
32	Ga72	5	1024	14.1h	70	W185	5	820	75.1d
33	Ge77	5	1000	11.3h	71	Xe133	5	790	5.25d
34	Hg203	5	720	46.57d	72	Y90	5	1023	64.1h
35	I130	5	1010	12.3h	73	Y91	5	1010	58.5d
36	I131	5	870	8.04d	74	Zn65	5	780	243.6d
37	I132	5	1023	2.3h	75	Zr95	5	900	64d
38	Ir192	5	890	74d					

2. Operating information

Operating information

The information you need to operate Wallac WinSpectral can be found in the User manual 1414-920 which is included with the program disks.

There is also a built-in help which can be accessed while running the workstation. This contains similar information to the User manual.

3. Calculation methods

Calculation methods

Introduction

This chapter describes the path by which the Wallac 1414 WinSpectral DSA counter arrives at the final isotope activity values using the Digital Spectrum Analysis (DSA) features such as Digital Overlay Technique (DOT). Measured count values are combined with built-in reference spectrum information which takes account of both chemical and colour quench, as well as cocktail type in order to arrive at the final activity values. Corrections such as those for chemiluminescence and half-life etc. are also included if required.

Activity

The DPM (or activity a) is defined as

$$a = x / e = (y / t) / e$$

in which x is the count rate (CPM), e is the counting efficiency and y is the number of counts collected during counting time t. The error in a, da, can be written as

$$da = \left(\frac{1}{e}\right)\sqrt{dx^2 + a^2 \cdot de^2}$$

Count rate

For a multi-label sample with m isotopes, the measured spectrum is the sum of m individual pure isotope spectra. Hence, for each channel j $(1 \leq j \leq n)$ in the measured composite spectrum, the total count rate c_j is equal to the sum of the individual count rates x_{ij} where i $(1 \leq i \leq m)$ designates each of the m pure isotope spectra. In mathematical terms this can be expressed as a set of linear equations:

isotope i →

$$\begin{array}{ll} 1 \quad\; 2 \qquad\quad m & \\ x_{1,1} + x_{2,1} + \ldots + x_{m,1} = c_1 & \text{channel 1} \\ x_{1,2} + x_{2,2} + \ldots + x_{m,2} = c_2 & \text{channel 2} \\ \vdots & \\ x_{1,n} + x_{2,n} + \ldots + x_{m,n} = c_n & \text{channel } n \end{array}$$

For each isotope i, the individual count rate $x_{i,j}$ is a product of the (unknown) activity of the isotope a_i, the counting efficiency e_i and a scaling factor $s_{i,j}$ telling which proportion of the spectrum is in each channel j. While the activity and the efficiency are the same for all channels, each channel has its own scaling factor. The array of scaling factors $s_{i,j}$ is in fact equal to the reference spectrum of the isotope i, normalized so that the sum of all scaling factors equals 1.0. Thus,

$$x_{i,j} = s_{i,j} \cdot a_i \cdot e_i = s_{i,j} \cdot x_i$$

where x_i denotes the unknown count rate of isotope i. Using this relationship, the above set of n linear equations can be written as:

$$s_{1,1} \cdot x_1 + s_{2,1} \cdot x_2 + ... + s_{m,1} \cdot x_m = c_1$$

$$s_{1,2} \cdot x_1 + s_{2,2} \cdot x_2 + ... + s_{m,2} \cdot x_m = c_2$$

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$$s_{1,n} \cdot x_1 + s_{2,n} \cdot x_2 + ... + s_{m,n} \cdot x_m = c_n$$

Introducing matrix notation, this set of n linear equations can be written as

$$S*X = C$$

where S denotes an m-by-n matrix containing the m reference spectra s_i. C is an array (or column vector) comprising the n measured count rates c_j and X is an array (or row vector) comprising the m unknown count rates x_i to be determined. In the case of single label counting, the matrix S is reduced to a column vector of length n.

As the number of equations n is much larger than the number of unknowns m, this set must be solved by using the method of least squares. In practice, the method of weighted least squares is preferred in which the statistical accuracy of each count rate in C is taken into account. The solution can be written as

$$X = (S^T \cdot W^{-1} \cdot S)^{-1} \cdot (S^T \cdot W^{-1} \cdot C) = G \cdot (S^T \cdot W^{-1} \cdot C)$$

where S^T represents the transpose of the model matrix S and W^{-1} represents the inverse of the weight matrix W. The matrix W is an n-by-n matrix in which the diagonal elements are equal to the weight of each channel j and all other elements are equal to zero. A suitable weight value w_j is the inverse of the number of counts in channel j.

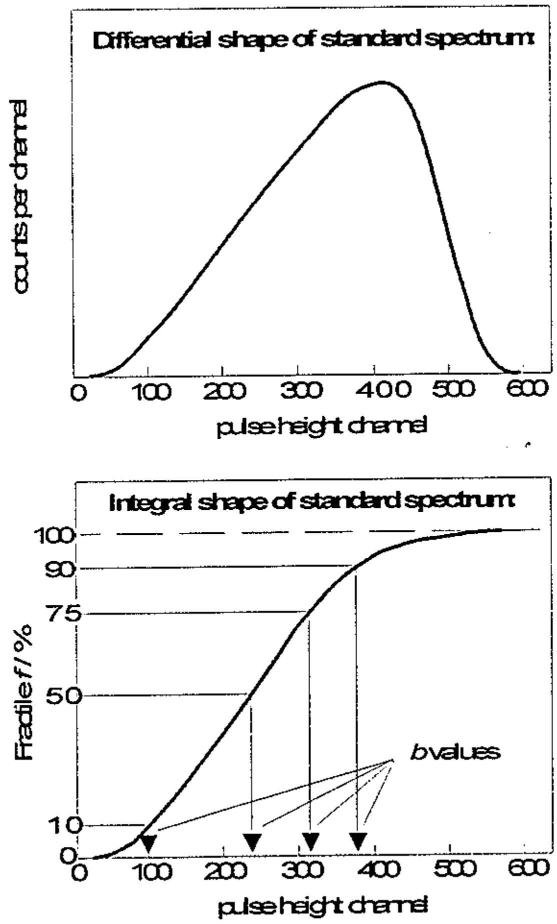
The errors in the vector *X*, dx_i, due to the reference spectrum fitting, are given by the equation

$$dx_i = \sqrt{|g_{i,i}|} \qquad (1 \leq i \leq m)$$

in which $g_{i,i}$ denotes a diagonal element in the matrix *G* defined above.

Reference spectra

Conversion of spectrum to FIP format

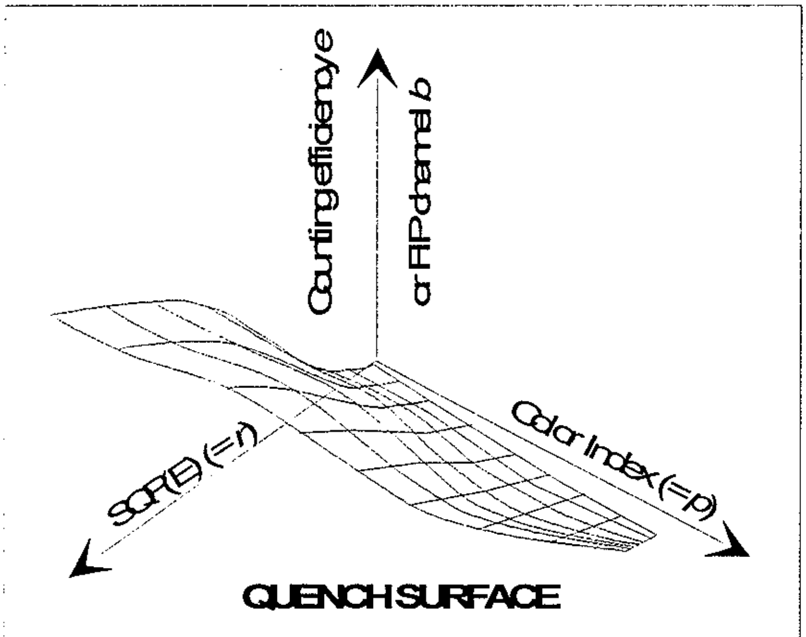


The built-in reference spectra s_i are stored in the spectrum library by using Fractile Integral Presentation (FIP). The spectrum library data for an isotope is produced by measuring in the factory from 60 to 80 standards of the isotope. In this standard set there is one subset with only chemical quench, one subset with only colour quench and one subset with a mixture of both. The spectra are stored in integral form reduced to a limited number of channel values (real numbers b_u, $1 \leq u \leq q$) corresponding to *q* predetermined percentage values (fractiles f_u) of the total integral intensity (=100%). Thus each spectrum is reduced to *q* fractile channels with value b_u. The b_u values depend on the quench level in a well behaved manner which can be described as a function of a suitable quench index.

In Wallac WinSpectral, two quench indices are used: the total quench level (SQP(E) or p) and the colour quench level (Colour or r). The variable p is equal to the overall quench level index determined from the end-point of the external standard spectrum (see below), while r is equal to the colour index determined from the left-right dispersion of the external standard pulses (see below). Thus each fractile channel b_u can be described by a surface function of the form

$$b_u(p,r) = \sum_{k=1}^{l} h_{u,k} \sqrt{(p-p_k)^2 + (r-r_k)^2 + h_0} \qquad \text{(for } 1 \leq u \leq q\text{)}$$

where $h_{u,k}$ are l parameters defining the surface, h_0 is a constant, and p_k and r_k are l coordinate pairs defining a grid (lattice) on the surface. The constant h_0 and the grid points p_k and r_k are the same for all b_u parameters.



A complete reference spectrum is arrived at by first computing the b_u parameters, interpolating this array and differentiating the result. Notice that errors in the reference spectra are considered to be unimportant.

Counting efficiency

The counting efficiency e is also a function of the two quench indices, p and r. The counting efficiency e is a surface function defined as

$$e(p,r)=\sum_{k=1}^{l} h_k\sqrt{(p-p_k)^2+(r-r_k)^2+h_0}$$

where h_k are l parameters defining the shape of the surface, h_0 is a constant, and p_k and r_k are l co-ordinate pairs defining a grid (lattice) on the surface. The error in e, de, is a function of the errors in p and r (dp and dr).

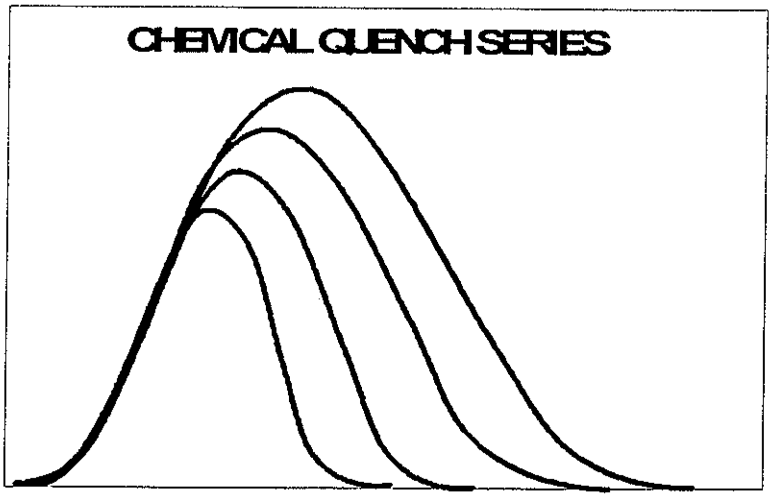
$$\frac{\partial e}{\partial p}=\sum_{k=1}^{l}\left\{\frac{h_k(p-p_k)}{\sqrt{(p-p_k)^2+(r-r_k)^2+h_0}}\right\}$$

$$\frac{\partial e}{\partial r}=\sum_{k=1}^{l}\left\{\frac{h_k(r-r_k)}{\sqrt{(p-p_k)^2+(r-r_k)^2+h_0}}\right\}$$

and, finally

$$de^2=\left(\frac{\partial e}{\partial p}\right)^2\cdot dp^2+\left(\frac{\partial e}{\partial r}\right)^2\cdot dr^2$$

Total quench index



The total quench index p is defined by the expression

$$\sum_{j=p}^{ul} c_j = z \cdot c_{tot} = z \cdot \sum_{j=ll}^{ul} c_j$$

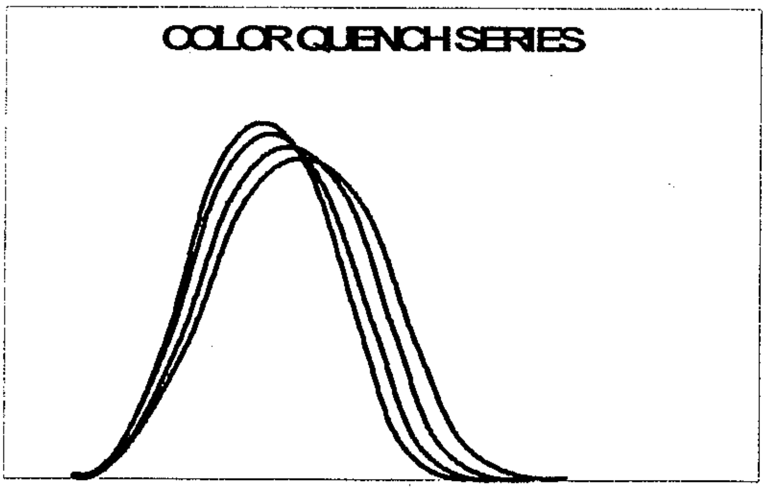
in which i denotes the pulse height channel, ul denotes the upper pulse height limit, ll denotes the lower limit and z is a constant (=0.01). The external standard spectrum is here rather treated as a continuous distribution $c(j)$ instead of a discrete distribution c_j in order to facilitate greater detail in determining p which is hence not an integer but a real number:

$$\int_p^{ul} c(j) \cdot dj = z \cdot c_{tot}$$

The error dp is given by the expression

$$dp = \left(\frac{1}{c_j} \right) \sqrt{(1-z) \cdot z \cdot c_{tot}} \qquad \text{(for } j=int(p)\text{)}$$

Colour index



The colour index r is defined by the equation

$$r = 1 + \frac{y_a}{y_b}$$

where y_a denotes the number of counts above the left/right or right/left pulse height criterion limit and y_b denotes the number of pulses below this limit. Hence, the error dr is given by

$$dr = (r-1)\sqrt{\frac{1}{y_a} + \frac{1}{y_b}}$$

Spectrum quench index

The spectrum quench index SQP(I) can be used as an alternative to SQP(E) and Colour index for quench monitoring and activity calculations. SQP(I) is defined by the equation

$$SQP(I) = \frac{\sum_{i=ll}^{ul} i \cdot y_i}{\sum_{i=ll}^{ul} y_i}$$

where ll and ul are the lower and upper limits of the spectrum. When using SQP(I), the Colour index is assumed to be equal to the value for an uncoloured sample.

The error in SQP(I) is expressed as:

$$dSQP(I) = \sqrt{\left\{\frac{1}{c_{tot}}\sum_i i^2 \cdot c_i - SQP(I)^2\right\}\frac{1}{c_{tot}}}$$

$$= \sqrt{\frac{1}{c_{tot}^2}\left(\sum_i i^2 \cdot c_i - c_{tot} \cdot SQP(I)^2\right)}$$

$$= \frac{1}{c_{tot}}\sqrt{\left(\sum_i i^2 \cdot c_i - c_{tot} \cdot SQP(I)^2\right)}$$

where $c_{tot} = \sum_i c_i$

Background correction

If a background sample is measured, then the count rate c_j is corrected by subtraction

$c_{jcorr}=c_j-c_{jbkg}$

where c_{jcorr} is the corrected count rate in channel j and c_{jbkg} is the measured count rate in channel j. In this case the correction is made before spectrum fitting.

If a background value is submitted, then the count rate x_i is corrected after spectrum fitting.

Half-life correction

Half-life correction is performed on the count rate x and the activity a after spectrum fitting by using the equation:

$$x_{corr}=x\cdot\frac{t_2-t_1}{t_{1/2}}\left[e^{-\ln(2)\cdot t_1/t_{1/2}}-e^{-\ln(2)\cdot t_2/t_{1/2}}\right]$$

where ln(2) is the natural logarithm of 2, t_1 is the time elapsed at the beginning of the counting period since the reference time, t_2 is the time elapsed at the end of the counting period since the reference time and $t_{1/2}$ is the half-life.

Chemiluminescence correction

A chemiluminescence spectrum is recorded at the same time as the normal (uncorrected) spectrum by using the delayed coincidence principle. After measurement, the measured spectrum is corrected by subtracting the delayed spectrum from the normal spectrum, channel by channel. The percentage of chemiluminescence (CLM%) is computed by the formula

$$CLM\%=100\%\cdot\frac{y_{del}}{y}$$

where y_{del} is the number of counts in the delayed spectrum and y is the number of counts in the normal spectrum.

Statistics of repeat/replicate counting

The mean value of n count values y_i is

$$y=\frac{1}{n}\sum_i y_i$$

Note: It is assumed that the counting times of all n repeats/replicates are the same.

The theoretical standard error σ_i (or standard deviation) of each measurement is equal to $\sqrt{y_i}$. The standard error σ of the n measurements is equal to

$$\sigma = \sqrt{\frac{1}{(n-1)} \cdot \sum_i (y - y_i)^2}$$

while the observed standard error σ_{y_o} of the mean is equal to

$$\sigma_{y_o} = \frac{\sigma}{\sqrt{n}}$$

The theoretical standard error σ_{y_i} of the mean is equal to

$$\sigma_{y_i} = \frac{1}{n}\sqrt{\sum_i \sigma_i^2} = \frac{1}{n}\sqrt{\sum_i y_i}$$

The theoretical standard error σ_{y_i} can be compared to the observed standard error σ_{y_o} by using the reduced χ_r^2 ("Chi-square") value:

$$\chi_r^2 = \frac{\sigma_{y_o}^2}{\sigma_{y_i}^2}$$

The degree to which χ_r^2 deviates from unity is a direct measure of the extent to which the observed error deviates from the theoretical. χ_r^2 multiplied by the 'degrees of freedom' (= *n-1*) can be used to determine a probability that a random sample from a normal distribution would have a larger (or smaller) value of χ^2 than the observed value.

List of symbols used

a = activity (DPM)
b = FIP channel
c = measured total count rate (CPM)
c_j = measured channel count rate (CPM)
C = an array (or column vector) comprising the measured count rates cj
d = error in function or parameter
e = counting efficiency
f = fractile value
$g_{i,i}$ = diagonal element of matrix G
G = the matrix $(S^T * W^{-1} * S)^{-1}$
h = surface function parameter
i = index to isotope
j = index to channel
k = index to surface function parameter
l = number of parameters defining the surface
m = maximum number of isotopes
n = maximum number of channels or number of repeats/replicates
p = total quench index (SQP(E))
q = number of fractiles f
r = colour quench index (Colour)
s_i = reference spectrum, a column in S
$s_{i,j}$ = channel value in reference spectrum, an element in the matrix S
S = a matrix containing the reference spectra s_i.
t = counting time or elapsed time
$t_{1/2}$ = half-life
u = index to fractile f and FIP channel b
w_j = weight value for channel j, a diagonal element in the matrix W
W = the weight matrix
x_i = unknown total count rate for isotope i, an element in the array X
$x_{i,j}$ = unknown channel count rate for isotope i
X = an array (or row vector) comprising the count rates x_i
y = number of counts
z = fractile for total quench index p
σ = standard error at 68.3% confidence limit (= standard deviation)
χ_r^2 = reduced chi square

4. Quality control information

QC Contents

Evaluation of the safety of the Eu-152 external standard source in Wallac 1400 series LS counters

Isotope

Europium 152.

Maximum activity

12 microcuries (half-life 13.5 years).

Origin of radioactive material

The Eu-152 radioactive material is made by the Radiochemical Centre, Amersham, England.

Manufacturer of the standard capsules

The Eu-152 standard capsules are manufactured by Wallac Oy, Finland.

Manufacturing process

The manufacturing process by which the radioactive standards are produced involves two steps: the preparation of the radioactive material and the encapsulation of this material with a metal cover.

In the manufacturing process the active Eu-152 solution (1 mCi/ml) is absorbed homogeneously into crystalline synthetic zeolites in such a way that the activity of each active source is 12 μCi.

The active source is enclosed in a stainless steel shield of thickness 0.35 mm. The source is sealed first with silicon rubber and then the shield is closed by using a stainless steel pin, which is held in place by pinching the shield with a special tool. The wire that moves the capsule is then inserted into the shield and fixed in place by again pinching the shield.

ISO-test

The capsule has been tested by the Quality Assurance department of Wallac Oy according to the ISO 2919 standard. The rating C43434 has been achieved.

External standard transport system

The transport system is illustrated in document 1036 1092.

The standard is mounted on a metal wire which is moved by two friction wheels controlled by a stepper motor and two sensors.

The capsule has two stable positions in the transport system; one in the storage shielding and the other in the measuring chamber inside the lead shielding. The storage shield is made of lead (thickness 50 mm) and is equipped with a radiation hazard warning label. This includes a definition of the isotope and its activity.

The capsule is transported to the measuring chamber along a PA (polyamide) tube and a stainless steel elevator tube. The transport time is less than 3 seconds.

Disposal of the capsule

When the instrument is no longer in use, the Eu-152 standard capsule must be removed and sent to the appropriate radiation safety authorities or to Wallac for safe disposal. This procedure must be performed only by a qualified Wallac service engineer.

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life sciences.

1096 1060

Quality Control Report

Name of Product:	1400-Series Liquid Sintillation Counters
Manufacturer:	Wallac Oy, Turku, Finland
Function Tested:	Classification of External Standard Capsule Eu-152 according to ISO 2919.
Activity of the capsule:	440 kBq

CERTIFICATION

Eu-152 External standard source has been tested according to ISO 2919 - 'Sealed Radioactive Sources - Classification'.

The tests are:

Test	Class	Short definition
Temperature	4	- 40 °C (20 min) + 400 °C (1 h) and thermal shock to 20 °C
Ext. pressure	3	25 kPa abs. to 2 MPa abs.
Impact	4	2000 g from 1 m
Vibration	3	2 times 10 min, two axes 25 Hz to 50 Hz/5G, 50 Hz to 90 Hz/0.635 mm amplitude, 90 Hz to 400 Hz/10G
Puncture	4	50 g from 1 m

Leakage tests according to ISO TR 4826 have been performed after the classification tests. The tests show no significant leakage.

The source fulfils ISO 2919 requirements and is therefore classified as C43434.

Date: 25 Nov. 1994

Signature of controller Raimo Kananen

Raimo Kananen, M.Sc.
Physicist
Quality Assurance Department, Instruments

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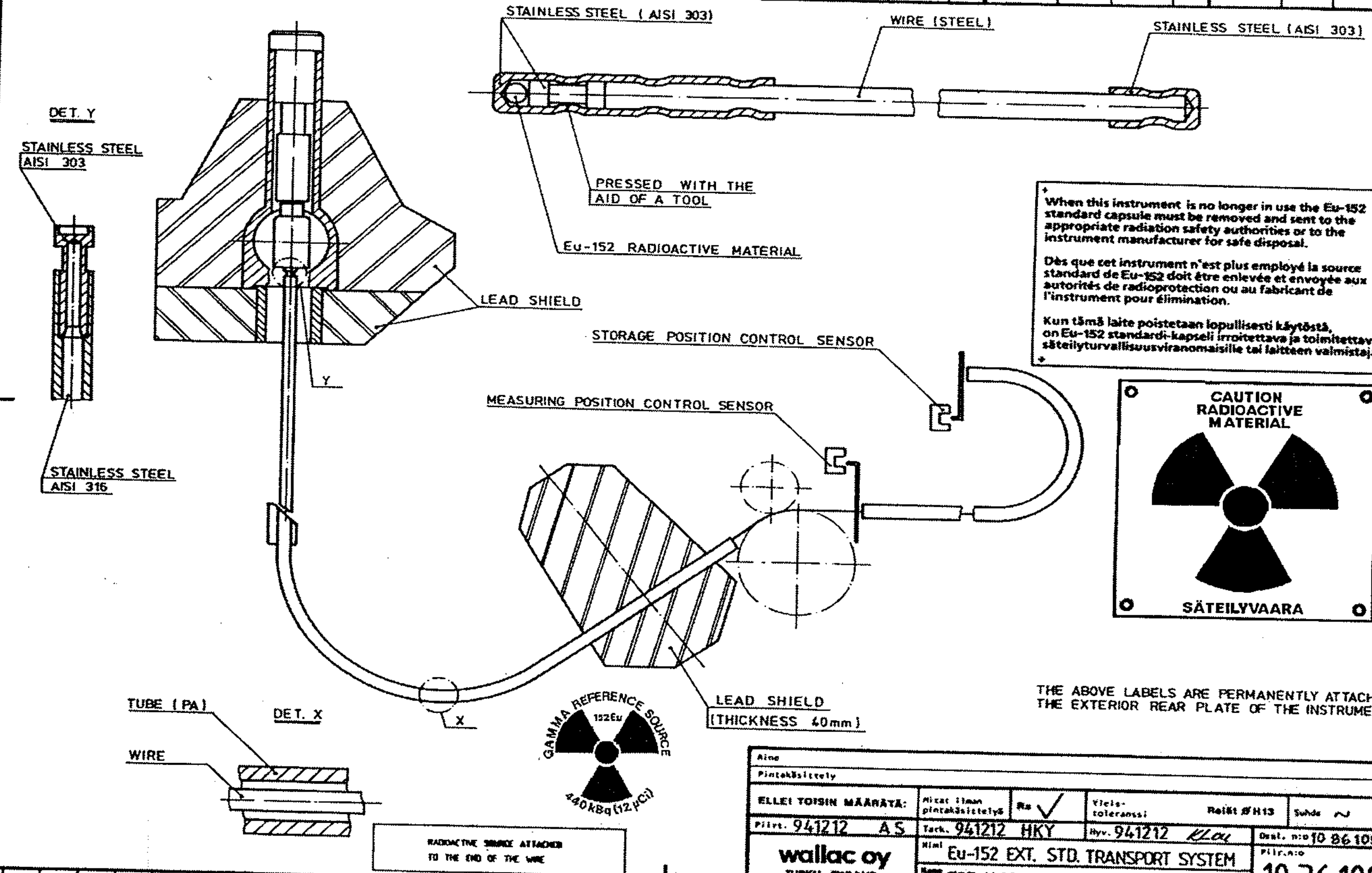
Asema	KT n:o	Päiväm.	Piirt.
	2279	941212	AS



When this instrument is no longer in use the Eu-152 standard capsule must be removed and sent to the appropriate radiation safety authorities or to the instrument manufacturer for safe disposal.

Dès que cet instrument n'est plus employé la source standard de Eu-152 doit être enlevée et envoyée aux autorités de radioprotection ou au fabricant de l'instrument pour élimination.

Kun tämä laite poistetaan lopullisesti käytöstä, on Eu-152 standardi-kapseli irroitettava ja toimitettava säteilyturvallisuusviranomaisille tai laitteen valmistajalle.

THE ABOVE LABELS ARE PERMANENTLY ATTACHED TO THE EXTERIOR REAR PLATE OF THE INSTRUMENT

Projektio

Mitoituksen puuttuessa käytetään.

Sisäkulman jyrsintä

Tappien ja vastaavien sorvaus

Levyn ja nauhan taivutus

Aine

Pintakäsittely

ELLEI TOISIN MÄÄRÄTÄ:	Mitat ilman pintakäsittelyä	Ra ✓	Yleistoleranssi: Reiät Ø H13 / Suhde ~
Piirt. 941212 A S	Tark. 941212 HKY	Hyv. 941212 KLau	Osat. n:o 10 86 1081
wallac oy TURKU - FINLAND	Nimi Eu-152 EXT. STD. TRANSPORT SYSTEM / Name FOR 1400-SERIES LSC-COUNTERS		Piir. n:o 10 36 1092

1390 3693

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DECLARATION OF CONFORMITY FOR CE-MARKING
INSTRUMENTS

We

Supplier's name
WALLAC OY
Address
PL 10, 20101 TURKU, FINLAND

declare under our sole responsibility that the product

Name, type or model, lot, batch or serial number, possibly sources and numbers of items	
1414 Liquid Scintillation counter	Valid from serial number 4140044

to which this declaration relates is in conformity with the following standard(s) or other normative document(s)

Title and/or number and date of issue of the standard(s) or other normative document(s)
EN 50082-1 :1992; EN 50081-1 :1992 EN 61000-3-2 :1995 + A1 :1998 + A2 :1998 + A14 :2000; EN 61000-3-3 :1995 EN 61010-1 :1993

(if applicable) following the provisions of the following directives

Electromagnetic compatibility (EMC), 89/336/EEC Low voltage (LV), 73/23/EEC

Date and place of issue
01 June 2001 TURKU, FINLAND
Name and signature or equivalent marking of authorized person
Pekka Mäkinen, Quality Assurance Manager

5. Installation information

Installation

Environment

Normal clean laboratory conditions usually provide a satisfactory operational environment, but the following points should be taken into consideration.

Ideally a separate room should be provided for your Wallac 1414 WinSpectral counter as this allows the best control over the immediate environment. Ventilation should be adequate for all conditions of use, the temperature should be reasonably constant at about 22°C, relative humidity should not be excessive, and direct sunlight should not be able to reach the instrument. It is also important that the various isotopes are stored well away from the instrument in another room. Only those radioactive samples that are actually being measured should be in the laboratory at any time in order to keep the background at a low level.

Electric power

Three electrical outlets should be available, with, if possible, a separate power line for the instrument itself having an isolation switch and a fuse box. If excessive fluctuations in the mains voltage are anticipated, a mains stabilizer may be necessary.

Unpacking

To unpack the instrument proceed as follows (see fig. 5.1).

Cut off and remove the binding bands. Open the clinching nails and lift off the cover. Unpack the separate packages. Then open the lower clinching nails and remove the box. Unscrew the fixing screws. Lift the instrument up from the shipping package base and unscrew the suspension bushes from the frame.

Check all units and accessories against the packing list. Note any possible transport damage.

Move the instrument to its place of operation.

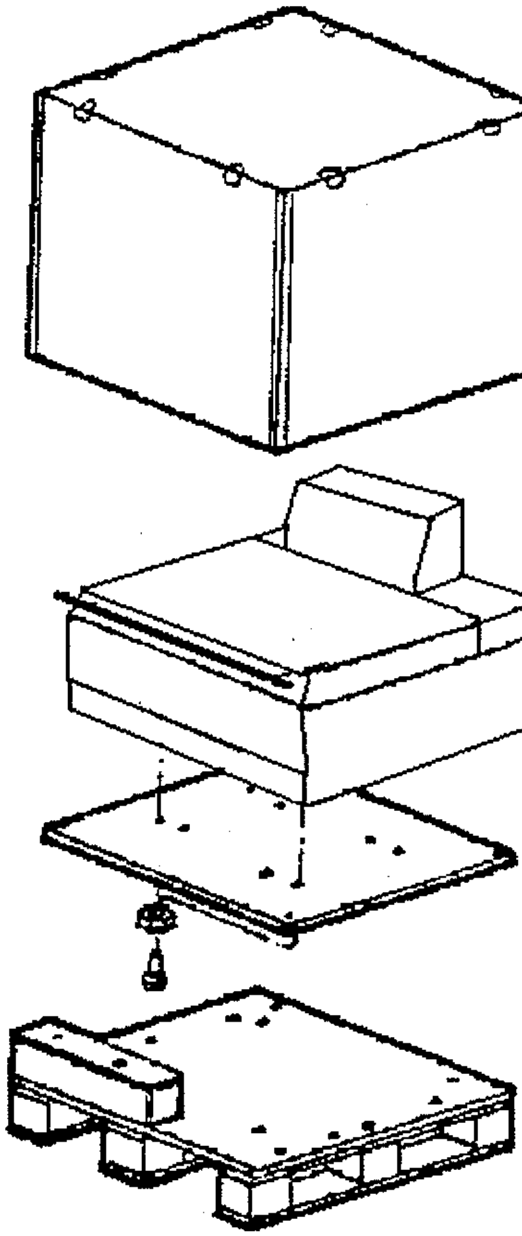


Fig. 5.1 Unpacking the instrument and accessories

Installing the lead shielding

Remove the screws holding the back panel (fig. 5.2).

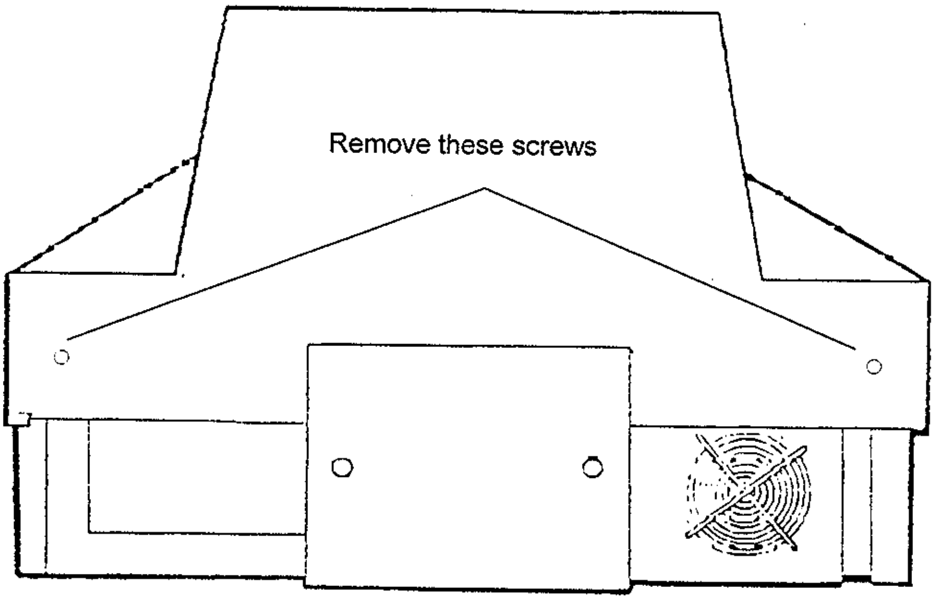


Fig. 5.2 Removing the screws

Remove the back panel (fig. 5.3)

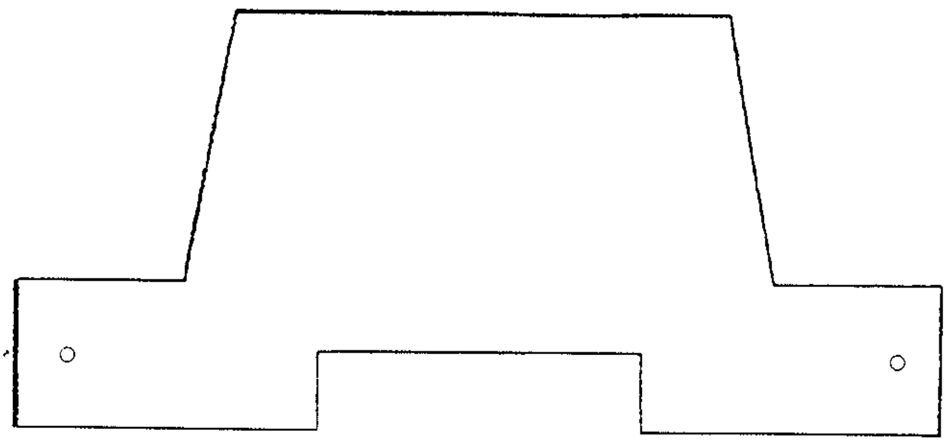


Fig. 5.3 Removing the back panel

Unscrew and remove the PC board (fig. 5.4).

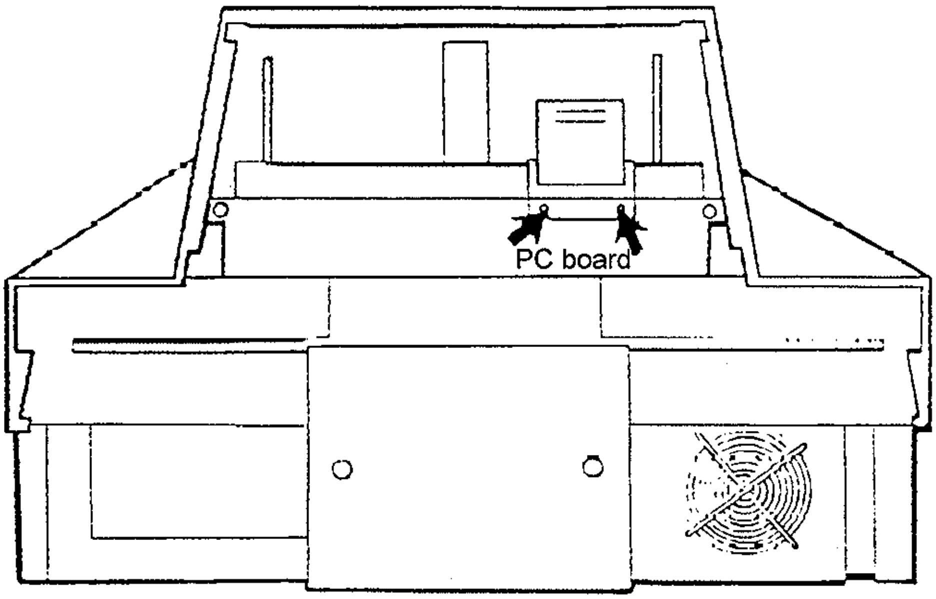


Fig. 5.4 Removing the PC board

Unscrew the top cover screws: two at the back (fig. 5.5).

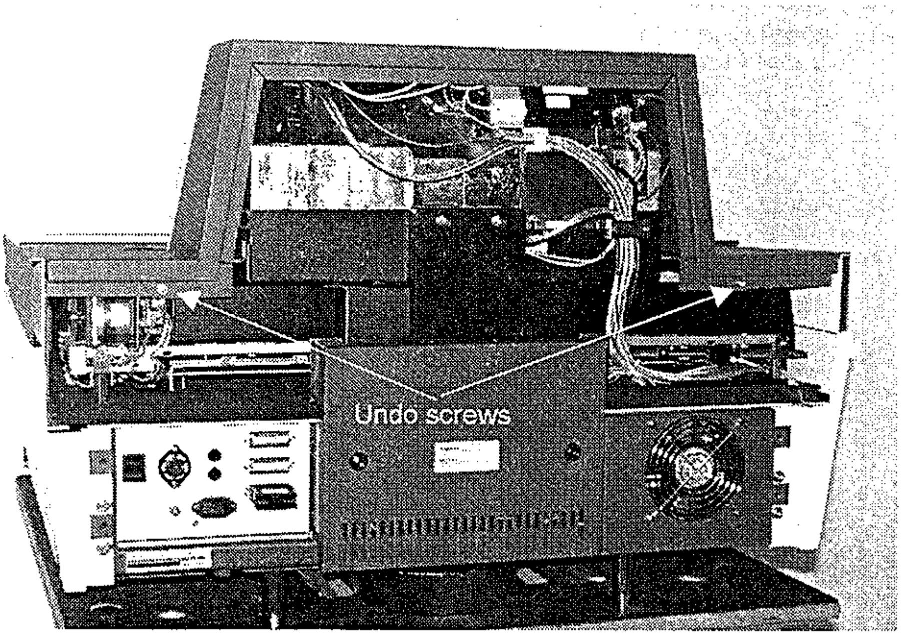


Fig. 5.5 Removing the two screws at the back

Remove the four screws on top (two on each side) securing the top cover (fig. 5.6).

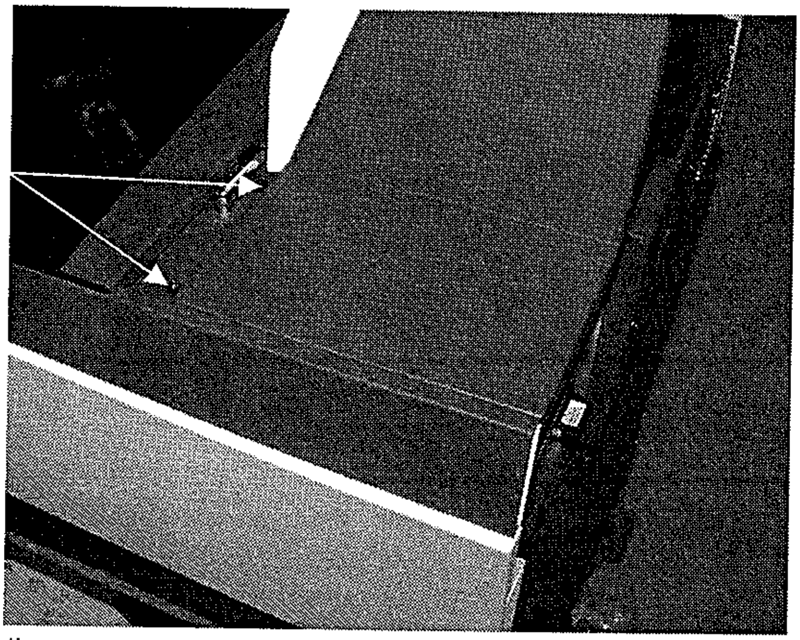


Fig. 5.6 Removing the screws on top

Lift the top cover off and remove it (fig. 5.7).

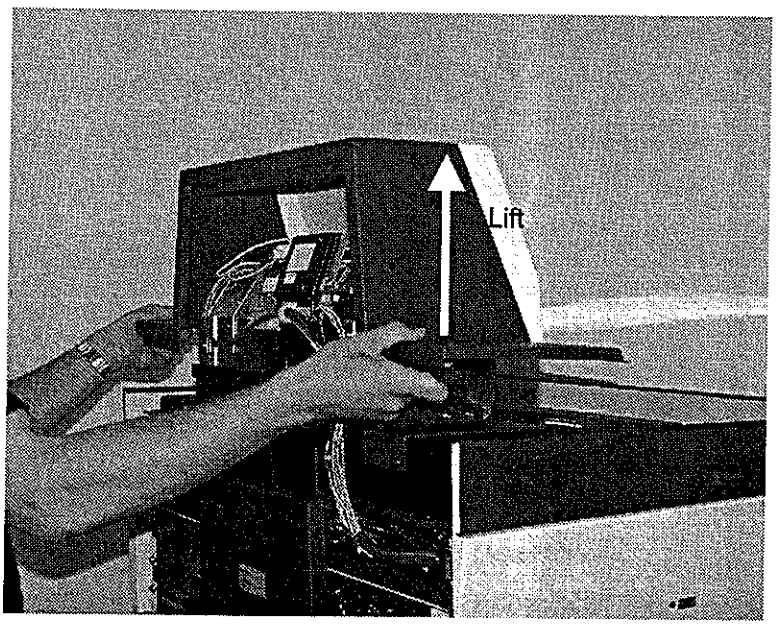
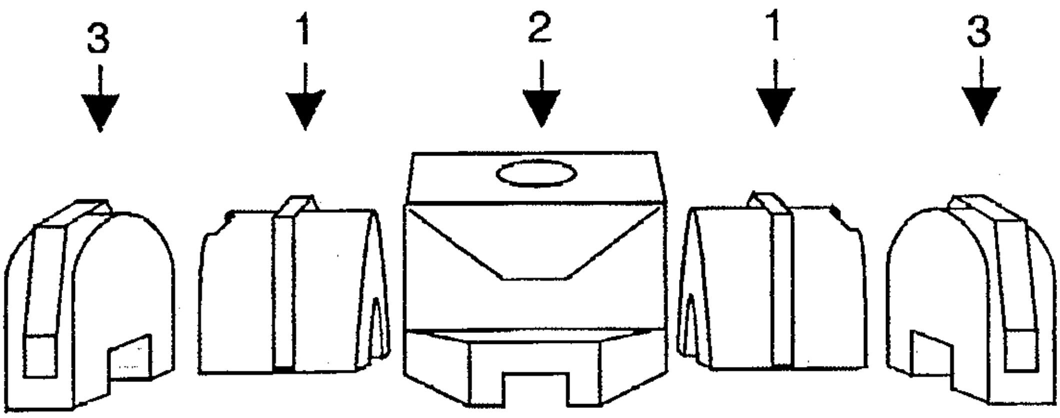


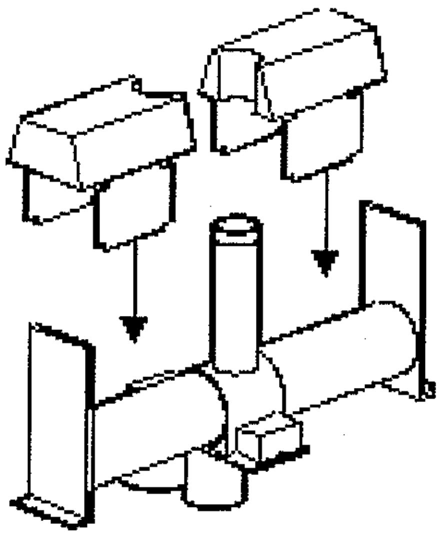
Fig. 5.7 Lifting the top cover off

Install the lead shielding for 1414-001 WinSpectral as shown (fig. 5.8) the lead sections are numbered.



5.8 Installing the shielding for WinSpectral

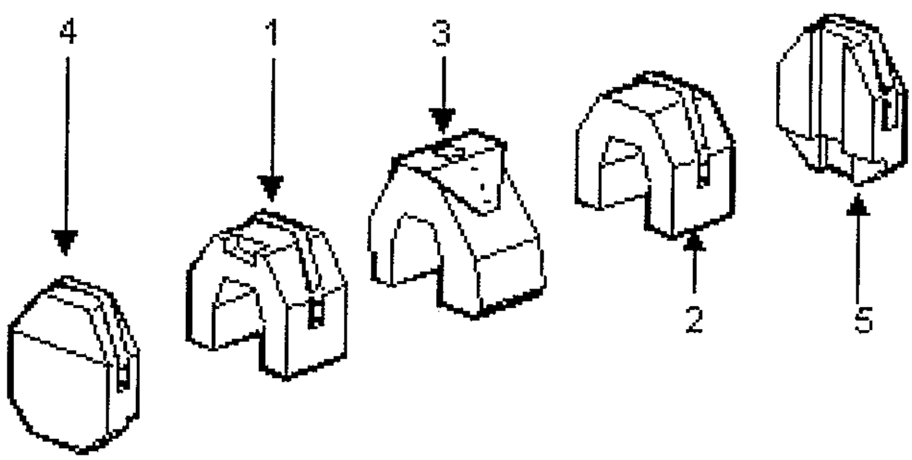
In the case of 1414-002 WinSpectral α/β, first place the two identical pieces of lead shielding with metal supports on top of the PMTs around the tube for the counterweight, see fig. 5.9.



5.9 Installing the shielding for WinSpectral α/β

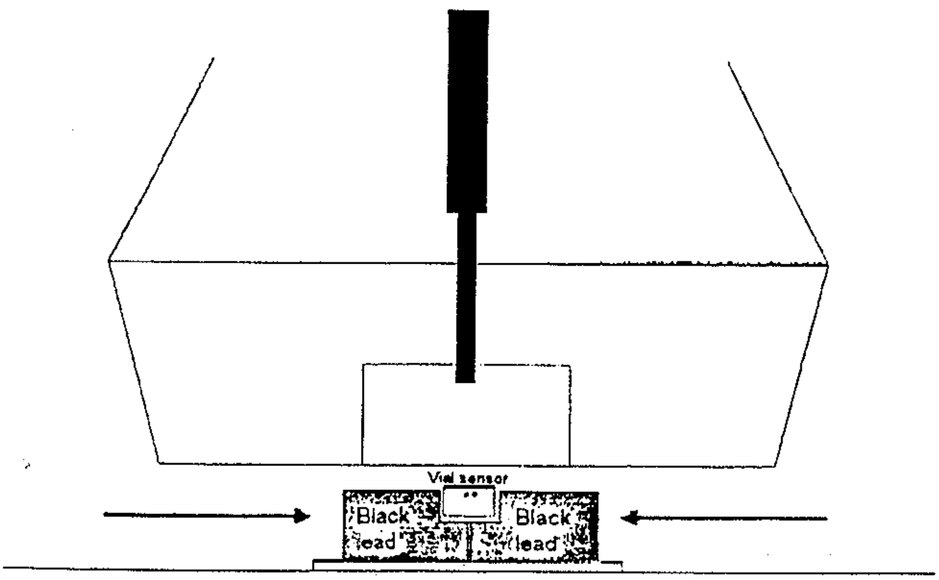
When you have done this, proceed with the following instructions, which are the same for 1414-002 WinSpectral α/β and 1414-003 Guardian; see the following three figures.

The shielding should be installed in the order shown in fig 5.10.

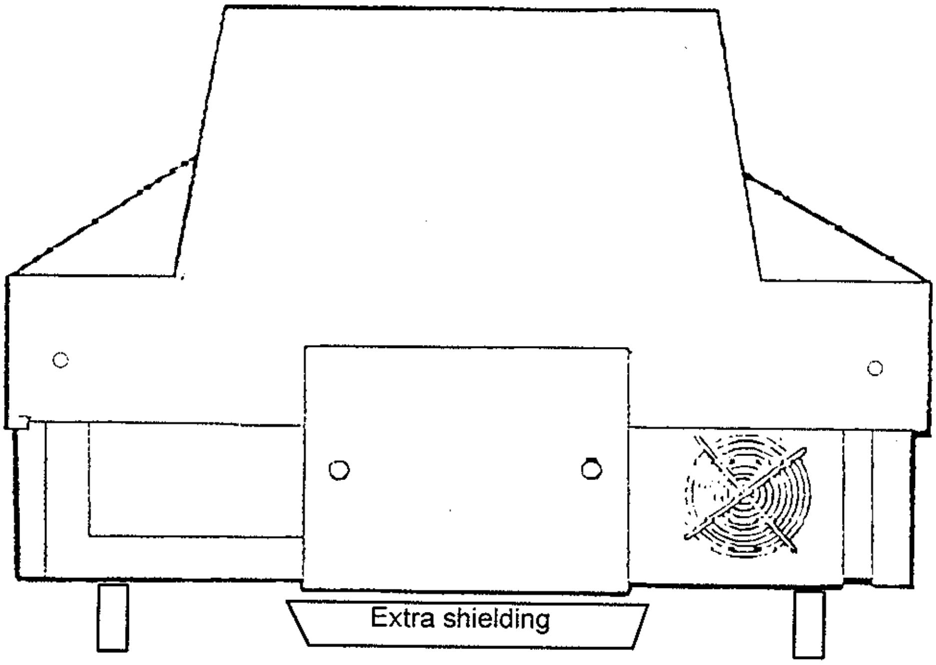


5.10 Installing the shielding for WinSpectral α/β and Guardian

There is additional shielding to install around the vial sensor (fig. 5.11) and under the instrument on the bench (centred on the elevator), see fig. 5.12.



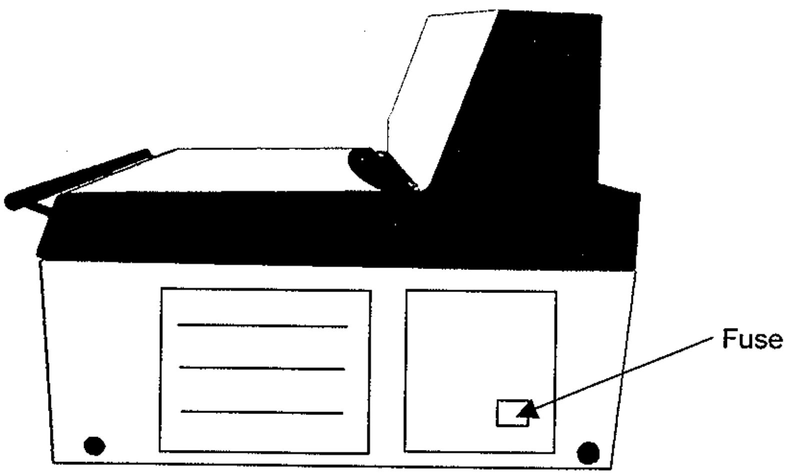
5.11 Installing the extra shielding for WinSpectral α/β and Guardian



5.12 Installing the extra shielding underneath for WinSpectral α/β and Guardian

Installing the stand-by power supply fuse

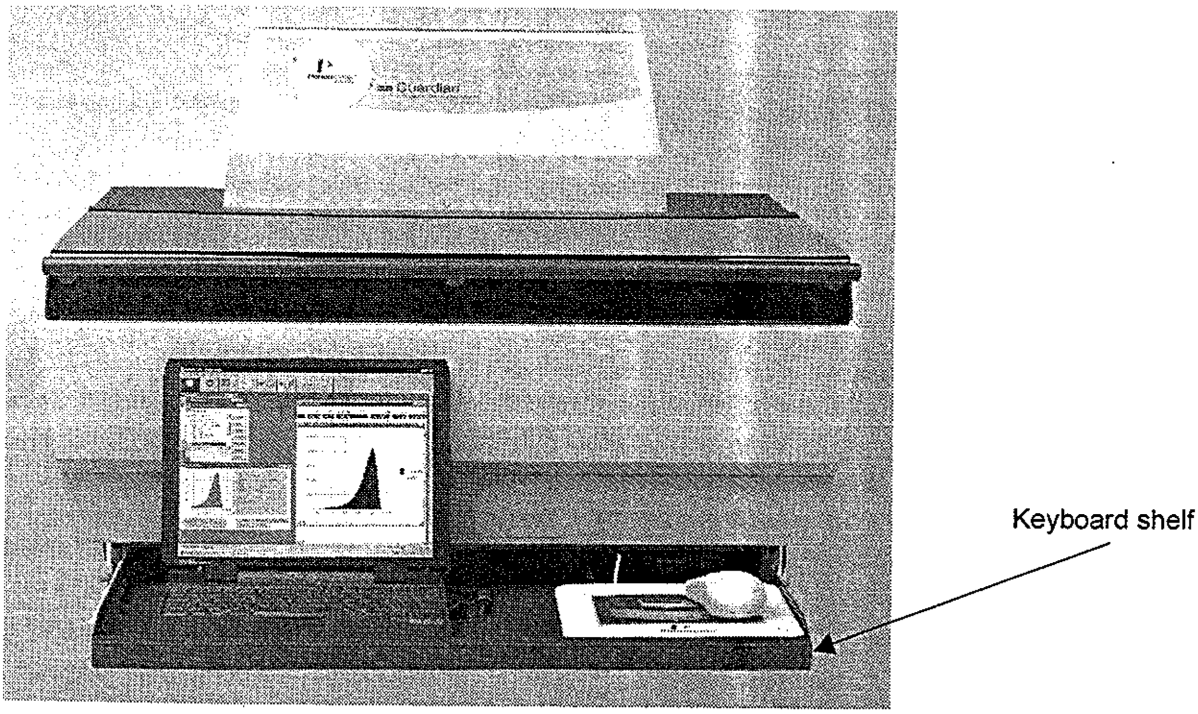
During transportation the stand-by power supply is disconnected. Install the fuse after the lead shield installation (fig. 5.13). The replace the instrument covers.



5.13 Stand-by power supply fuse

Installing the PC keyboard shelf

Assemble and fix the PC keyboard and mouse shelf (product number 10860862) and the monitor stand (1414-160) following the instructions supplied with them, see fig. 5.14.



5.14 Installing the keyboard shelf

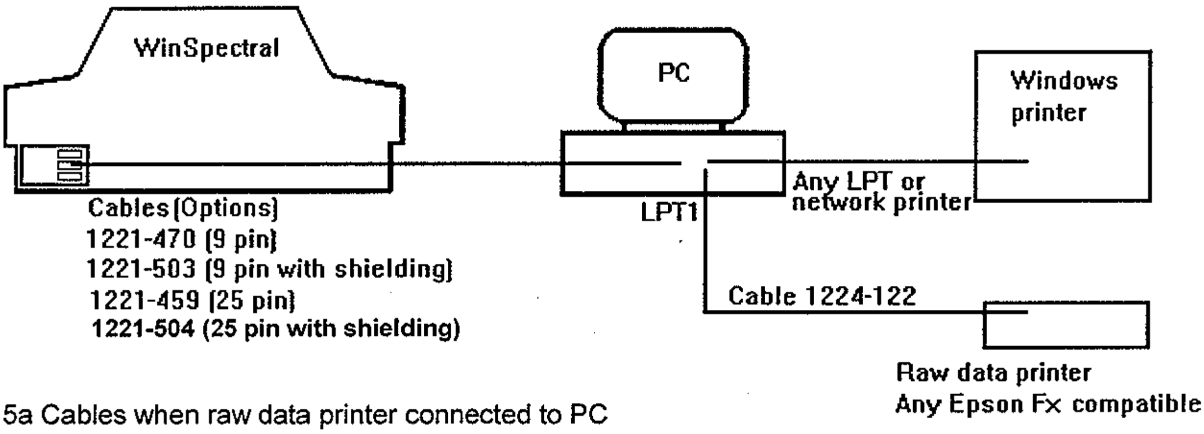
Checking the mains voltage setting

Measure and note the mains voltage at the outlets to be used.

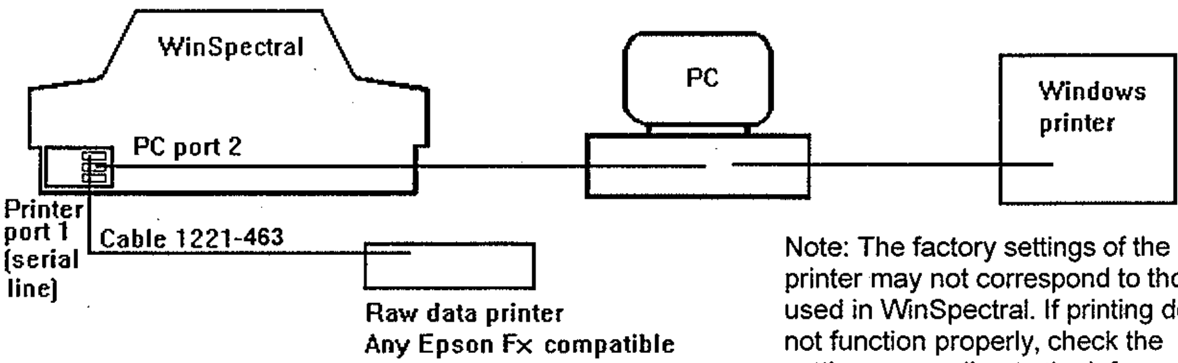
Locate the mains selector switch, this is on the left side when looking at the rear of the instrument. If necessary adjust the mains selector switch to correspond with the measured supply. For supplies with a nominal voltage of 230 V it is recommended that the selector be set to 240 V.

Check that the fuses fitted in the fuse carriers on the back panel are of the correct rating for the local supply, and according to the label.

Connecting the counter and peripherals



5.15a Cables when raw data printer connected to PC



Note: The factory settings of the printer may not correspond to those used in WinSpectral. If printing does not function properly, check the settings according to the information given in Serial port settings

5.15b Cables when raw data printer connected to WinSpectral

Connect the counter to the PC and the printer(s) using the cables shown in fig. 5.15 above. As well as the normal PC Windows printer e.g. a network laser printer, you can also have a raw data printer. Normally this will be connected to the PC (fig. 5.15a), but you can have it connected to WinSpectral directly in port 1 (fig. 5.15b).

The PC is connected to port 2 of the counter.

The third port is not used and should have a blanking connector (1086 0713).

Further details of the settings for the printer are given in its own operating manual.

Plug in the power cables for each device.

Installing the PC software

Install the WinSpectral following the instructions in the User manual.

After installation, start the program by double clicking the 1414 WinSpectral icon in the WinSpectral window.

When the WinSpectral main window appears, you will see at the bottom the status bar; this is divided into four sections. In the third and fourth there are the date and time respectively. In the second there is a message about the communication status. If the counter is not switched on then there will be a message "No response from counter".

Starting the counter

Lift the instrument cover and then the diskette drive cover. This latter should be lifted straight up first to release it. Then insert the instrument program diskette into the diskette drive in the counter and replace the cover. Do not remove the program diskette.

Switch on the counter. The message on the status bar will change to "Counter online". During the loading process the status bar will show the following messages: "Loading DOT", "Loading texts" and "Loading 1400 program". When the program is completely loaded, the message "Counter idle" will appear. Loading takes some 2 minutes.

Note: The program diskette labelled BACKUP may also be used but be sure to always have a program diskette in addition to the one in the instrument. When needed, make a backup copy using the "Backup instrument disk" function in the System menu. It is also recommended that from time to time you make a backup copy of your program diskette to save your own protocols and spectrum libraries.

Note: You should also make backup copies of the WinSpectral program diskettes before use. Use e.g. the Copy disk function in your Windows File manager.

Carrying out a functional check of the instrument

For checking the instrument mechanically and for checking the function of the ID reader proceed as follows:

Load some racks with ID labels, then start counting and check that the instrument changes the protocol as required by the labels.

Performance test with Easy GLP

To define the proper values for GLP you have to make an initial measurement of the local background. Make sure that all stationary and moving gamma sources are removed from the neighbourhood of the instrument. Use at least 300 s measurement time.

Start the editing of the Easy GLP protocol (see page 65 in the User manual). Type the values of the supplied standards into the text box marked Reference samples. The isotope windows are 5-350 for ^3H and 5-650 for ^{14}C. The measurement time should be 300 s or longer and the precision 0.5%.

Test values are as follows (where FTDS means the Final Test Data Sheet included with this Instrument manual):

	Min	Max	Comment
^3H background			Use the local background*
^3H efficiency	-3%	+1%	From FTDS value
^3H SQP(I)	-5 ch	+3 ch	From FTDS value
^{14}C background			Use the local background*
^{14}C efficiency	-2%	+1%	From FTDS value
^{14}C SQP(I)	-5 ch	+3ch	From FTDS value

*The minimum and maximum background warning limits for GLP are calculated from "the value measured during installation *+/- 3σ*". Some example backgrounds and 3σ limits are:

Observed background	3σ
46 - 50	9.5
41 - 45	9.0
36 - 40	8.5
31 - 35	7.9
26 - 30	7.3
21 - 25	6.7
16 - 20	6.0
11 - 15	5.2
6 - 10	4.2
0 - 5	3.0

E.g. if the background is 21 then 3σ is 6.7 which means the minimum and maximum limits are 14.3 and 27.7 respectively. You can calculate the 3σ from the expression: $3\sigma = 3 * \sqrt{(BGD/t)}$

where BGD is the measured mean background rate in the window and t is the counting time in minutes.

Note: a typical change in efficiency of 2% for ^{3}H over a period of a year may be expected.

Serial port settings

The factory settings of the printer serial port on WinSpectral are as follows:

Baud rate:	4800 PC
Parity:	No
Data bits:	8
Stop bits:	2
Handshake:	DTR i.e. ESN

These settings may be changed by selecting System parameters in the System menu. See the User manual on page 87.

MultiCalc installation

Introduction

If you are planning to use MultiCalc to handle results from Wallac WinSpectral, please follow the installation instructions below in addition to the normal MultiCalc installation procedure.

- do the normal MultiCalc installation (see the MultiCalc Installation module in the Supervisor's manual)

- start up Windows

- with File Manager copy the file c:\1400w\mc\winbeta.c01 to the directory c:\wiacalc\0com

- create the directory c:\wiacalc\0com\winbeta

- create a new program in the Windows StartUp group: click Browse to select c:\1400w\mc\WIA.PIF.

- start MultiCalc by double clicking that icon

- select Level 5 (if your starting level is 3 or less then press F7 LEVELS then F4 Level 4 and then again LEVELS (F7) and Level 5 (F5))

- select the System properties to be AOSPMTIV (follow the sequence: F7 SYSTEM F8 ETC F3 COMM PROT F4 PROPERTY, select the protocol winbeta and then type in the properties)

- go to the MultiCalc main menu by pressing ESC ESC ...

- create a startup macro for MultiCalc as follows:

Press ctrl A
Select MACROS
Select CREATÉ
Enter @ as the macro name
Select level 2
Press Enter as the comment
Press function key F1 (this is the body of the macro and it means that the program will go to COUNTER). Now the macro is complete and should be ended by pressing the ESC key.

To save the macro on disk you now have to exit from MultiCalc. To do so, press ESC once and then press X and select F1 Yes for the question "Exit MultiCalc Yes/No".

- now use the Windows Program Manager and copy the icon WinSpectral to the StartUp group. To do this, drag it from the WinSpectral group to the StartUp group while you hold down the CTRL key.

Changes to Autoexec.bat

Edit the MS-DOS Autoexec.bat file. (Use Windows Notepad or a DOS editor to do this).

Make sure that you have there the command COMRS and that it has the parameter 00000000 i.e. eight zeros. This is to force MultiCalc not to use serial ports at all. Otherwise you will get the question about which should use the COM1 or COM2 port, the Windows application or the DOS application when MultiCalc is started.

While you are editing the Autoexec.bat file, add also there the command WIN to make Windows start automatically after a bootup.

Setting up a protocol in MultiCalc

In WinSpectral, go to System/Options/Directories and give the MultiCalc assay path as: C:\WIACALC\0COM\WINBETA.

From the MultiCalc main menu select F4 PROTOCOLS and create a new protocol. The name should be a short one, e.g. MCASS. Select the protocol type, e.g. RATIO and edit the protocol or use the default settings. Save the protocol.

After you exit from Protocol editing, go to the main menu and select from there F1 COUNTER. MultiCalc is then ready to evaluate results from WinSpectral.

Switch now to the WinSpectral program.

Setting up a protocol in WinSpectral

Create a protocol for any user group and give the protocol the same name as you gave for the assay protocol. WinSpectral allows longer protocol names but use exactly the same name as for the assay protocol, e.g. MCASS.

Set the counting time and the isotopes used. Then go to Output devices and select Filing data. Click the More button and select MultiCalc export. Save these settings.

You are then ready to run the protocol.

WinSpectral will receive data and save it on the hard disk of the PC. As the samples in the assay are being counted, WinSpectral will create a file in the MultiCalc communication protocol directory. MultiCalc will find the file and start evaluating it in about 30 seconds, depending on the speed of your PC and the amount of time you have given for DOS applications.

6. Specifications, Safety and Routine maintenance

Specifications

Description

Wallac 1414-001 WinSpectral, 1414-002 WinSpectral α/β and 1414-003 Guardian are general purpose liquid scintillation counters for counting sample vials from racks.

Physical dimensions

Width 900 mm
Height 700 mm
Depth 600 mm
Weight 218 kg (WinSpectral), 250kg (WinSpectral α/β), 244 kg (Guardian)

Power

Mains voltage selectable 100, 115, 120, 220 and 240 V, +/-10%, 50/60 Hz. Power consumption is 200 VA without the temperature control unit and 300 VA with it.

Standby power supply

Power failure recovery and protection by back-up standby power supply provided with NiCd accumulators. In the case of power failure the status of the counting process will be stored on diskette, thus providing effectively an unlimited standby time for recovery from power failure.

Input/output connections

Serial ASCII interface RS-232C. Two output terminals: port 1 for printer, port 2 for laboratory PC.

Radiation shield

Low specific activity lead around detector assembly and external standard rest position. Weight 103 kg (WinSpectral), 135 kg (WinSpectral α/β), 128 kg (Guardian), thickness at least 48 mm around the measuring chamber.

Operating conditions

Temperature +15 to +35 °C
Humidity max. 75 %.

FlexiRack™ sample changer and conveyor

Sample capacity varies from 336 to 1248 vials, depending on the used rack combination. When using only standard vial racks, 336 samples in 28 racks of 12 samples each. With minivial racks, 720 samples in 40 racks of 18 samples each. With small mini/microvial racks, 1248 samples in 52 racks of 24 samples each.

Rack transport by friction belt drive system, transverse movement by toothed chain driven by electric motors. Sample lifted up to measuring chamber by elevator driven by electric motor, single "dual plate" light shutter system, sample change time 5 s. Rack and sample position movements controlled by electro-optical sensors.

Optional external standard

^{152}Eu 440 kBq (12 µCi) enclosed in a stainless steel capsule.

Optional temperature control unit

Controls sample and instrument +/- 5 °C of ambient.

Detector assembly

Two PM tubes in coincidence. Assembly includes also light emitting diodes for the automatic spectrum stabilizer. Coincidence resolution time is 15 ns.

Racks and modification kits

Sample racks

1410-401 for 20 ml vials, max. vial diameter 28.4 mm, 12 sample positions per rack.
1410-402 for 6 ml vials, max. vial diameter 18.4 mm, 18 sample positions per rack.
1410-403 for 4 ml vials, Skatron or Biovial, max. vial diameter 13.4 mm, 24 sample positions per rack.
Max. acceptable vial height is 68 mm.

Rack modification kits

1410-408 for Eppendorf tubes to be used in 1410-402 racks.
1410-409 for microfuge tubes to be used in 1410-403 racks.

PosIden™ sample identification system

Each rack can be provided with a 1410-407 ID clip for 1450-452 ID labels. The ID clip has two marked areas, the upper for the protocol number and the lower for rack number or special code. This provides the information needed for Good Laboratory Practice.

Electronic hardware

A 16-bit microprocessor controls counting and data reduction. Memory configuration is 64 kB ROM and 1 MB RAM.

Logarithmic A/D converter, energy range 1 - 2000 keV. Dual 1024-channel multichannel analyzer. Built-in dead time correction.

Performance

Efficiency

Counting efficiency, typical:
^{3}H : 68 % (min. 64 %)
^{14}C : 96 % (min. 94 %)

Stability

Count variation less than 0.2 % / 24 h (not including random statistics).

Safety and radioactive materials

The following comments about precautions and safety measures in handling radioactive materials are included as a guide and are not intended to be fully comprehensive. More complete details may be found elsewhere, for example in the booklet SAFE HANDLING OF RADIONUCLIDES, published by the International Atomic Energy Agency, Vienna; this may be recommended as a useful code of practice appropriate to radio-chemical laboratories.

Unless a specially designed radioisotope laboratory is used, limitations should be placed on the amount of active material in the laboratory area depending on toxicity and type of chemical operation. For high toxicity material and wet chemical operations involving the risk of spillage, the IAEA recommend a maximum activity of about 10 μCi.

Personnel should be properly trained in the safe handling of these materials, maximum levels of stored activities should be set, proper records should be kept, and a definite monitoring schedule maintained.

The areas where samples are handled should be kept clean and free of dust. This is most easily accomplished if all surfaces are as smooth as possible and if the minimum number of extra surfaces is introduced into the room. Lastly it is extremely important to store all radioactive materials in a separate room to which access is restricted.

Routine maintenance

Keeping the instrument clean

The conveyor cover should always be closed to prevent dust from getting onto the conveyor and into the electronics below the conveyor. If there is still dust and other dirt on the conveyor then remove the dirt with a dry cloth.

ID labels

Check the labels on the ID clip. Those in bad condition should be replaced by new ones to guarantee correct reading of the labels. When fixing ID labels on the clip, ensure that the area where the label is to be fixed is clean, e.g. there is no perspiration from your fingers on it.

Power supply fan

Check that the cooling fan in the power supply unit is working by listening for its sound.

Cables

Check that the power cable and the cables to the peripherals are tightly connected and that the cables and connectors are not damaged. Any damaged cable should immediately be replaced!

Response mapping - MCA calibration

What is response mapping?

The quench correction system and DOT DPM technique make use of built-in libraries of information derived from a reference counter. Response mapping is the procedure by which a connection is established between the MCA channel values in your actual counter and the reference counter. This allows the libraries in your counter to be customized to fit your counter in the case that there are small differences between it and the reference counter.

This is done in the factory before you receive your counter but mapping must be done again when the measurement electronics is adjusted or repaired or the PM tubes are replaced or the external standard capsule is changed. Mapping can also be done at any time and it is recommended that it should be performed at least once a year as part of routine maintenance.

How to do response mapping

Boot the instrument with the 1414 WinSpectral instrument program disk.

Create or edit a fine tuning protocol, e.g. Finetuning 01. Set the protocol name to "MAPPING". Use the sealed ^3H standard sample. Set the DPM value according to the value printed on top of the standard. Set half-life correction on and set the half-life zero time according to the date printed on top of the standard. Set the counting time to 300 s. Save the protocol.

Load a rack with the sealed ^3H standard sample onto the conveyor in-stream. Other positions in the rack should be left empty.

Start counting the protocol.

To stop the instrument after measurement of the mapping sample put an empty rack on the conveyor after the mapping sample rack.

Results are printed out shown in the example. The plot has the reference counter channel number on the x-axis and the measured real channel number on the y-axis. Two lines are drawn, one is a solid line and the other is dashed (on the raw data printer it is marked with crosses (+)). The solid line represents the reference counter MCA channels and the dashed (crossed) line the measured channels. On the display the reference line is in blue and the actual in red. This line may reside slightly above and below the ideal line. There should be no straight horizontal or vertical section which would indicate missing MCA channels.

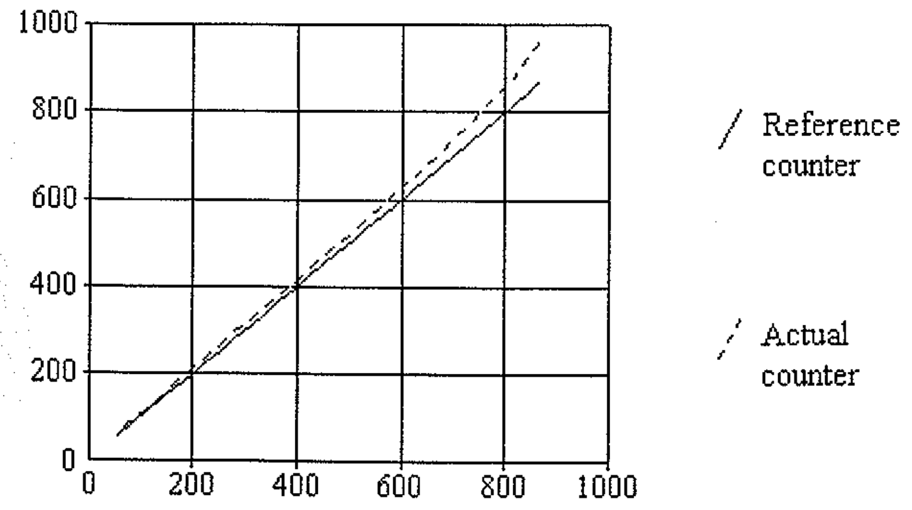
The mapping information is also saved in numerical form in the printer and instrument history file.

When the Ready message is displayed on the Status bar after mapping has been done, it shows that the mapping information has been saved in the EEPROM memory. Then switch the instrument off and on again to reboot it.

```
PROTOCOL :  1 MAPPING
DATE     : 1994/08/11
TIME     : 10:25
ID       : P01AS002

Quench standardization
Wallac 1414 WinSpectral DSA version 1.0
Counting mode            : DPM
Quench index             : SQP(E)
Isotope(s)               : H3
Protocol name            : MAPPING
Counting time            : 300
Activity(DPM)            : 202400
2sigma%                  : 0.01
Minimum cpm              :0.00 Checking time:10
Vial type                :Clear
Liquid system            :HiSafe
Advanced modes           :Halflife
Half life zero time      :
Zero time of H3          :1994/08/01 ,12:00:00
Output to printer        :
POS,CTIME,CPM1,CPMer1,EFF1,SQPE,SQPer
Additions to printer     : Listing
Spectrum                 :Beta

Quench standard samples:
Pos Ctime H3_CPM H3_CPMer H3_Eff%   SQPE SQPer
  1    60 2018.0      0.0    1.00 977.87  0.04
```

```
EEPROM LISTING

LRC = 200  A0 = 110  A1 = 0
TEMP =    25.0
CONV STEPS = 114

Mapping data

Channels: WLIB    TRUE  TRUE-WLIB
           0.0     0.0        0.0
          48.9    58.4        9.5
          95.0    99.2        4.2
         140.4   142.6        2.1
         159.3   160.6        1.3
         177.4   178.0        0.6
         195.7   195.8        0.1
         214.8   214.3       -0.5
         682.9   695.2       12.3
         746.1   768.1       22.1
         798.6   834.1       35.5
         886.0   977.9       91.9
         894.7   993.6       98.9
        1000.0  1000.0        0.0

Total activity:
H3201800.0DPM3.363kBq
```

Mapping



7. Index

Index