

IR Integrating Sphere Accessory for the Frontier



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1. Introduction

1.1 *The IR Integrating sphere accessory*

The following items are included:

- The IR integrating sphere accessory
- USB stick with an electronic copy of this manual, Calibration file and report, configuration files
- Box with reference mirror
- Funnel

1.2 *Features*

This accessory is designed to fit into the sample compartment of the PerkinElmer Frontier series FTIR spectrometer. The design is optimized by minimizing port sizes and the absence of baffles which are not required in our design. The angle of incidence is 10°. The integrating sphere has a diameter of 100 mm and is coated with diffuse gold optimized for a wavelength region of 2 – 20 µm. The detector is a broadband Liquid N2 cooled MCT detector for maximum sensitivity. The minimum wavelength range is 2 – 18 µm.

1.3 *Warnings and Safety Information*

Liquid Nitrogen

The MCT detector module in this accessory is cooled using liquid nitrogen. Before use, ensure that an appropriate risk assessment has been carried out.

2. Installation

2.1 Installation of PC files

There are a total of four files that must be installed on the PC:

<part number>.ini
<port image identifier>.bmp
<port image identifier>1.bmp
<port image identifier>4.bmp

These files are delivered on the USB stick that is delivered with the accessory.

All four files must be copied into the Spectrum installation sub directory named 'bin'.

Normally the IRwinlab installation CD will default to 'c:\pel_apps' as the installation directory (in which case the target directory will be c:\pel_apps\bin) but it is possible for the user to change the Spectrum installation directory.

There is 1 addition needed for correct operation in the Frontier, you need to copy the bitmap files additionally to C:\Program Files\Common Files\PerkinElmer\BeamPath

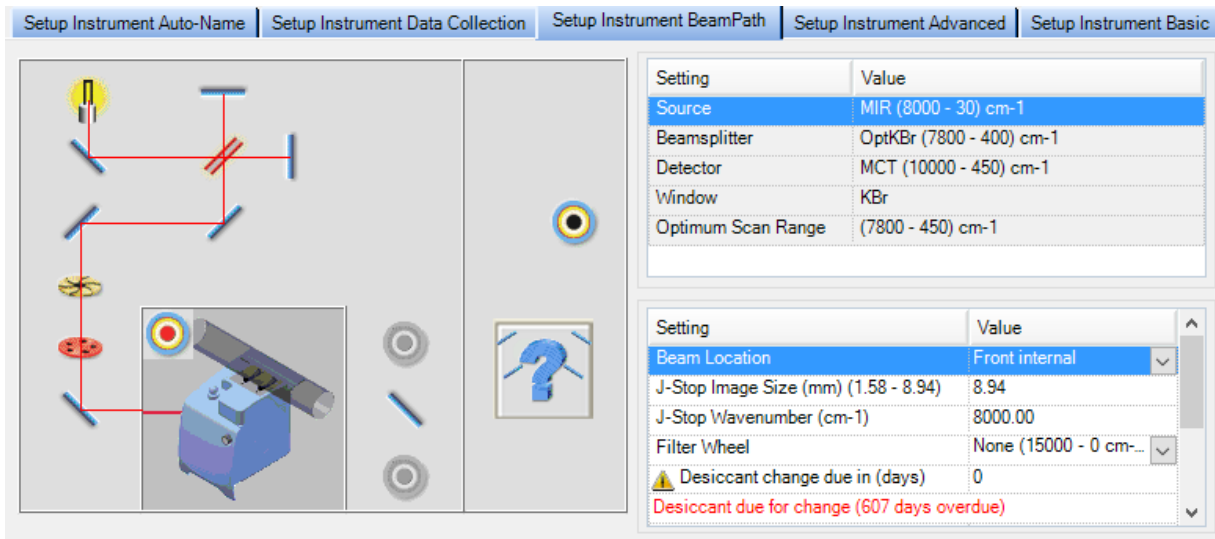
In order to discover the actual directory that the Spectrum One software has been installed into, interrogate the 'pel_apps.ini' file in the c:\windows directory.

This file contains an entry of the following format, indicating the installation directory:

[Data Server]
Installation Directory = c:\pel_apps

2.2 Installation of accessory

When the files have been copied and the accessory has been installed by clicking the accessory in the sample compartment the following picture should be visible when the beam setup in your software has been selected.



Setting	Value
Source	MIR (8000 - 30) cm-1
Beamsplitter	OptKBr (7800 - 400) cm-1
Detector	MCT (10000 - 450) cm-1
Window	KBr
Optimum Scan Range	(7800 - 450) cm-1

Setting	Value
Beam Location	Front internal
J-Stop Image Size (mm) (1.58 - 8.94)	8.94
J-Stop Wavenumber (cm-1)	8000.00
Filter Wheel	None (15000 - 0 cm-1)
Desiccant change due in (days)	0
Desiccant due for change (607 days overdue)	

3. Measurements

3.1 Instrument settings

The recommended instrument settings for optimal performance are given in figures below:

The screenshot shows the 'Setup Instrument Basic' tab of the software interface. The 'Settings' section is configured as follows:

Parameter	Value
Abscissa Units	Wavenumber
Start (cm-1)	5000
Ordinate Units	%T
End (cm-1)	400

The 'Scan Settings' section is configured as follows:

Parameter	Value
Resolution (cm-1)	8
Scan Type	Sample
Data Interval (cm-1)	<input type="checkbox"/>
Accumulations	64 Scans

The 'Accessory' section is set to 'IR Diffuse Reflectance Accessory'.

3.2 Measurement procedure

The THERMES International Round Robin exercise has shown that the main problem affecting accurate reflection measurements with FTIR spectrometers (source drift) can efficiently be corrected using a standardised measurement procedure. This procedure involves multiple measurements according to a specified sequence of reference mirror and sample(s) and correction of measured curves according to a time-weighted average of the reflectance measured for the reference mirrors. The measurement sequence must be completed in its entirety without interruption

The accessory is used in two different positions.

You can easily switch between wall and sample measurement by using the knob on the accessory. By turning the knob left the alignment is on the wall and by rotating the knob to the right the alignment is on the sample.

The following measurement sequence is recommended for measuring a sample:

Measurements	Description
Autozero	Background measurement
M1	Stray measurement 1
M2	Sample +Wall measurement 1 (SW1)
M3	Sample measurement 1 (S1)
M4	Sample +Wall measurement 2 (SW2)
M5	Sample measurement 2 (S2)
M6	Sample +Wall measurement 3 (SW3)
M7	Sample measurement 2 (S3)
M8	Sample +Wall measurement 4 (SW4)
M9	Sample measurement 4 (S4)
M10	Sample +Wall measurement 5 (SW5)
M11	Sample measurement 5 (S5)
M12	Sample +Wall measurement 6 (SW6)
M13	Sample measurement 6 (S6)
M14	Stray measurement 2

The background measurement is always performed with the gold coated reference mirror (Calibrated gold reference 218070 R ∞ 1).

M1 and M14 are performed without any sample and the knob is turned to the sample (right position).

The SW measurements are performed with the sample placed and with the knob turned to the wall position (left position).

The S measurements are performed with the sample placed and with the knob turned to the sample position (right position).

Above mentioned measurement sequence have to be performed on the sample of your choice and on the calibrated gold reference 218070 R ∞ 1.

3.3 Calculation procedure

The reflectance can be calculated according to below formula:

$$R = \left(\frac{(S_{\text{sample, average}} - M_{\text{stray, avg}}) / (SW_{\text{sample, average}} - M_{\text{stray, avg}})}{(S_{\text{ref, average}} - M_{\text{stray, avg}}) / (SW_{\text{ref, average}} - M_{\text{stray, avg}})} \right) R_{\text{Ref}}$$

Where R ref is the calibrated values of your reflectance standard 211061 R_∞ 1 (which have been supplied on the USB supplied with the accessory)

S_{sample, average} is the average of M3,M5,M7,M9,M11,M13 from the measurement sequence on the unknown sample

SW_{sample, average} is the average of M2,M4,M6,M8,M10,M12 from the measurement sequence on the unknown sample

S_{ref, average} is the average of M3,M5,M7,M9,M11,M13 from the measurement sequence on the calibrated reference

SW_{ref, average} is the average of M2,M4,M6,M8,M10,M12 from the measurement sequence on the calibrated reference

M_{stray, average} is the average of M1 and M14 from the corresponding measurement sequence.