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QUALIFICATION OF EQUIPMENT

ANNEX 4: QUALIFICATION OF IR SPECTROPHOTOMETERS

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Concerned Network	GEON

ANNEX 4 OF THE OMCL NETWORK GUIDELINE
"QUALIFICATION OF EQUIPMENT"

QUALIFICATION OF IR SPECTROPHOTOMETERS

Introduction

The present document is the fourth Annex of the core document "Qualification of Equipment", and it should be used in combination with it when planning, performing and documenting the IR spectrophotometer qualification process.

The core document contains the Introduction and general forms for Level I and II of qualification, which are common to all type of instruments.
For FTIR spectrometers, an example has been added to give instrument-specific proposals that may be used in combination with the general requirements presented in the core document "Qualification of Equipment", when drawing up a Level I checklist.

The present annex contains instrument-related recommendations on parameters to be checked at Level III and IV of qualification and the corresponding typical acceptance limits, as well as practical examples on the methodology that can be used to carry out these checks.

TABLE I

Level I. Selection of instruments and suppliers

Example of check-list (non-exhaustive)

Manufacturer:

Provider/Distributor:

Name of instrument and type:

Attribute (This list may be adapted if necessary)	**Specifications**	**Benefits (Instrument/ supplier)**	**Assessment**	
			Pass	**Fails**
Spectrophotometer				
Detector range	The optical bench shall include a DTGS detector with a frequency range of 7400 to 350 cm^{-1}.			
It shall include a compressed air interferometer.				
The instrument shall come with an air-cooled standard infrared source.				
Spectral resolution	The instrument shall have a spectral resolution not exceeding 1.0 cm^{-1}			
Wave number accuracy	Wave number accuracy shall be better than ± 0.01 cm^{-1}			
The interferometer shall have at least four basic velocity levels; software shall permit the selection of a greater number of velocities between the basic levels.				
The mirror's greatest velocity shall allow a speed of at least five sweeps per second.				

Attribute (This list may be adapted if necessary)	**Specifications**	**Benefits (Instrument/ supplier)**	**Assessment**	
			Pass	**Fails**
The laser and infrared beams must be coaxial to enable rapid, easy alignment of the system, depending on the samples or accessories.				
The instrument shall have a beam splitter with a range of 7400 to 350 cm^{-1}.				
The optical bench shall have a main experimentation module with a device to allow purging with nitrogen.				
At purchase, the main experimentation module shall be designed so it can receive 13 and 5 mm potassium bromide pellets.				

Notes:

- This check-list, containing examples of technical attributes that can be taken into account in the selection of an instrument and supplier, can be used in combination with the general check-list presented in Level I in the core document "Qualification of Equipment".

- For Table II (Level II of Equipment Qualification: Installation and release for use) please refer to the core document.

TABLE III

Level III. Periodic and motivated instrument checks

Examples of requirements for IR spectrophotometers

Parameter to be checked	Typical tolerance limits
1. Wave-number scale	See Annex I
2. Detector energy ratio	See manufacturer's specifications
3. Signal/Noise ratio	See manufacturer's specifications
4. Resolution	See Annex I
5. Zero test	See manufacturer's specifications
6. Contamination check (only for ATR instruments)	See Annex I
7. Throughput check (only for ATR instruments)	80 %
8. Spectral library	See Annex I

TABLE IV

Level IV. In-use instrument checks

Examples of requirements for IR spectrophotometers

Parameter to be checked	Typical tolerance limits
1. System suitability check	According to Ph. Eur. or MAH dossier or validated in-house method

ANNEX I

Level III. Periodic and motivated instrument checks

This Annex contains practical examples of tests and their associated tolerance limits for several parameters related to the performance of an IR spectrophotometer.
These examples can be considered by the OMCLs as possible approaches to perform the Level III of the equipment qualification process: "Periodic and motivated instrument checks".

Note: If available and judged appropriate, the use of the automatic internal calibration function of the instrument is encouraged. Please refer to the manufacturer instructions.

1. WAVE-NUMBER SCALE

Method and Limits:
The wave-number scale may be verified by recording the spectrum of a polystyrene film, which has transmission minima (absorption maxima) at the wave numbers (in cm^{-1}) shown in the table below:

Transmission minima (cm^{-1})	Acceptable tolerance (cm^{-1})	
	Monochromator instruments	Fourier-transform instruments
3060.0	± 1.5	± 1.0
2849.5	± 2.0	± 1.0
1942.9	± 1.5	± 1.0
1601.2	± 1.0	± 1.0
1583.0	± 1.0	± 1.0
1154.5	± 1.0	± 1.0
1028.3	± 1.0	± 1.0

2. DETECTOR ENERGY RATIO

Method:
Record the minimum energy ratio value for at least one of the following measurement points and compare it to the vendor's specifications:

- Energy at 3990 cm^{-1} / energy at 2000 cm^{-1}
- Energy at 4000 cm^{-1} / energy at 2000 cm^{-1}
- Energy at 3400 cm^{-1} / energy at 1300 cm^{-1}
- Energy at 2000 cm^{-1} / energy at 1000 cm^{-1}

Limits:
Energy ratio test specifications vary for each spectrometer configuration. Refer to the manufacturer's specification.

3. SIGNAL/NOISE RATIO

Method:
Record the maximum noise level for each of the following regions:

Peak-to-peak noise between:
4050 cm^{-1} and 3950 cm^{-1}
2050 cm^{-1} and 1950 cm^{-1}
1050 cm^{-1} and 950 cm^{-1}
550 cm^{-1} and 450 cm^{-1}
(systems with DTGS detector only)

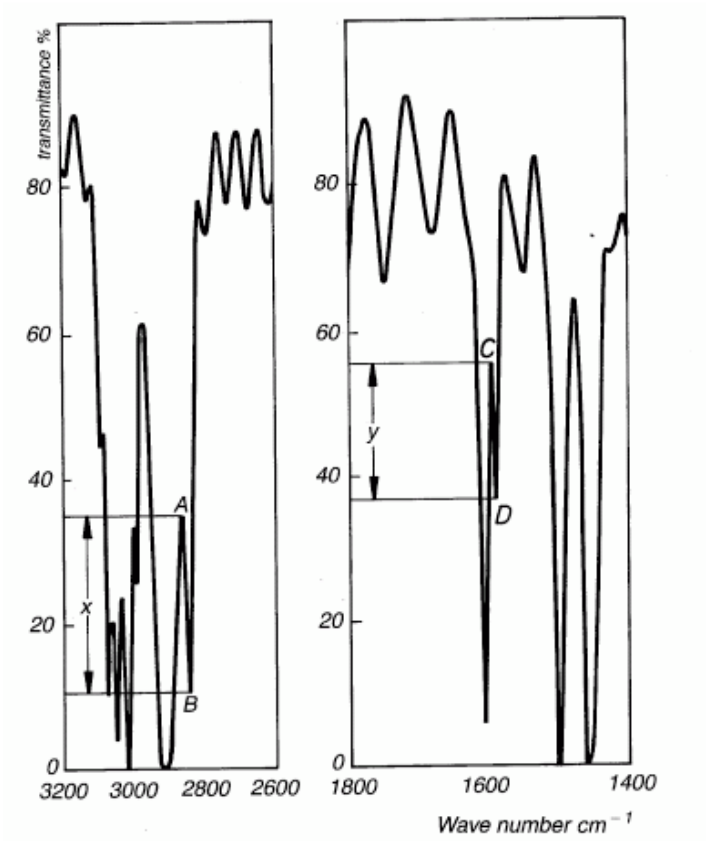
RMS (root mean square) noise between:
4050 cm^{-1} and 3950 cm^{-1}
2050 cm^{-1} and 1950 cm^{-1}
1050 cm^{-1} and 950 cm^{-1}
550 cm^{-1} and 450 cm^{-1}
(systems with DTGS detector only)

Limits (% T):
Noise level test specifications vary for each spectrometer configuration. Refer to the manufacturer's specifications.

4. RESOLUTION

Materials:
Certified polystyrene film of approximately 35 μm in thickness.

Method:
- For instruments having a monochromator, record the spectrum of the polystyrene film.
- For Fourier-transform instruments, use suitable instrument resolution with the appropriate apodisation prescribed by the manufacturer. The resolution is checked by suitable means, for example by recording the spectrum of a polystyrene film approximately 35 μm in thickness.



Typical spectrum of polystyrene used to verify the resolution performance

Limits:

Monochromator instruments:

- Difference x between the percentage transmittance at the transmission maximum A at 2870 cm-1 (3.48 µm) and that at the transmission minimum B at 2849.5 cm-1 (3.51 µm) > 18.
- Difference y between the percentage transmittance at the transmission maximum C at 1589 cm-1 (6.29 µm) and that at the transmission minimum D at 1583 cm-1 (6.32 µm) > 10.

Fourier-transform instruments:

- Difference between the absorbances at the absorption minimum at 2870 cm-1 and the absorption maximum at 2849.5 cm-1 > 0.33.
- Difference between the absorbances at the absorption minimum at 1589 cm-1 and the absorption maximum at 1583 cm-1 > 0.08.

5. ZERO TEST

Method:
When using a polystyrene film of approximately 35 μm in thickness as standard at the wavelength of 2925 cm-1 and 700 cm^{-1}, almost complete absorption of the irradiated energy can be observed. With this test, the remaining transmission is measured. As the maximum absorption can be observed at 700 cm^{-1} negative values may be observed. The objective of the test is to evaluate if, despite the fact that there is almost complete absorption, energy is still detectable.
Non-valid results are an indication of a non-linear behaviour of the detector and the electronic system.

Limits (%T):
See manufacturer's specification.

6. CONTAMINATION TEST (only for Attenuated Total Reflection (ATR) instruments)

Note: if an automated system is available, this test can be run more frequently or it can be transferred to Level IV, to be run before each analysis.

Method:
This test checks the presence of peaks that signal a contamination problem.
Use the automated function of the instrument (if available) to perform this test. If not available, record a background spectrum.

Limits:

Wave-number (cm-1)	Upper limit (A)
3100.0 – 2800.0	0.1
1800.0 – 1600.0	0.1
1400.0 – 1100.0	0.2

7. THROUGHPUT CHECK (only for Attenuated Total Reflection (ATR) instruments)

Note: if an automated system is available, this test can be run more frequently or it can be transferred to Level IV, to be run before each analysis.

Method:
This test checks for an unexpected reduction of the transmittance. An instrument specific automated test can be used.
A background spectrum is recorded and the transmittance is measured at 3 wave numbers e.g. 4000, 2600 and 1000 cm^{-1}.

Limits:
The lower limit of the transmittance for the 3 wave numbers must be 80 %.

8. SPECTRAL LIBRARY

It is recommended to record the spectrum of a newly acquired polystyrene film and to save it in a spectra library.

Electronic libraries for checking the identity of unknown substances should be examined from time to time by using selected examples of well-known reference substances.

Requirements:
The substances are unambiguously identified by the electronic library search.

ANNEX II

Level IV. In-use instrument checks

This Annex contains practical examples of tests and their associated tolerance limits for several parameters related to the performance of an IR spectrophotometer.
These examples can be considered by the OMCLs as possible approaches to perform the Level IV of the equipment qualification process: "In-use instrument checks".

Note: If available and judged appropriate, the use of the automatic internal calibration function of the instrument is encouraged. Please refer to the manufacturer instructions.

1. SYSTEM SUITABILITY TEST OF THE METHOD

Method:
This test should be performed according to Ph. Eur., the MAH dossier or a suitably validated in-house method.

Note: regeneration or replacement of the desiccant should be done in case of failure of the system suitability test (e.g. by drying it for 8-12h at 250°C, then flushing with N_2).

REFERENCES

(For all references, the latest version applies)

1) Ph Eur. 2.2.24, Absorption spectrophotometry, Infrared.