

General European OMCL Network (GEON)

QUALITY MANAGEMENT DOCUMENT

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QUALIFICATION OF EQUIPMENT

QUALIFICATION OF IR SPECTROPHOTOMETERS

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**ANNEX 4 OF THE OMCL NETWORK GUIDELINE
“QUALIFICATION OF EQUIPMENT”**

QUALIFICATION OF IR SPECTROPHOTOMETERS

Note: Mandatory requirements in this annex are defined using the terms “shall” or “must”. The use of “should” indicates a recommendation. For these parts of the text other appropriately justified approaches are acceptable. The term “can” indicates a possibility or an example with non-binding character.

1. INTRODUCTION

The present document is the 4th Annex of the core document “Qualification of Equipment”, and it shall be used in combination with it when planning, performing and documenting the qualification process of Infrared (IR) spectrophotometers.

The core document contains the Introduction and general forms for Levels I (Selection of instruments and suppliers) and II (Installation and release for use) of qualification, which are common to all types of instruments.

The present Annex 4 contains a general introduction and requirements for IR spectrophotometers. Level III (Periodic and motivated instrument calibration/checks) and IV (In-use instrument checks) qualifications must be carried out as an ISO 17025 requirement.

Requirements and (if applicable) corresponding typical acceptance limits given in bold should be applied; however other appropriately justified approaches are acceptable.

Exemplary procedures provided in this document have non-binding character. They can be helpful when carrying out the required qualification. Nevertheless, it is left to the professional judgement and background experience of each OMCL to decide on the most relevant procedures to be undertaken in order to provide evidence that their IR spectrophotometers are working properly and are suitable for their intended use.

If the qualification of equipment is done by the manufacturer or an external service provider, it is the responsibility of the OMCL to make sure that this is in line with the requirements set out in this guideline.

TABLE I

Level III. Periodic and motivated instrument checks

Requirements and related typical acceptance limits are indicated in bold

Parameter to be checked	Typical acceptance limits*	
Accuracy of wavenumber scale*	Refer to Ph. Eur. Chapter 2.2.24. "Control of equipment performance"	
Spectral Resolution*	Refer to Ph. Eur. Chapter 2.2.24. "Control of equipment performance"	
Detector energy ratio	Limit to be set based on OMCL experience/service provider's instructions	
Signal-to-Noise ratio	Limit to be set based on OMCL experience/service provider's instructions	
Zero test	Limit to be set based on OMCL experience/service provider's instructions	
Contamination check (only for ATR instruments)	Wavenumber (cm ⁻¹)	Upper limit (A)
	3100.0 – 2800.0	0.1
	1800.0 – 1600.0	0.1
	1400.0 – 1100.0	0.2
Throughput check (only for ATR instruments)	T_{min} = 80 % (n=3 wavenumbers) (The lower limit of the transmittance for the 3 wavenumbers must be 80 %)	

TABLE II

Level IV. In-use instrument checks

Examples of requirements for IR spectrophotometers

Parameter to be checked/Typical acceptance limits
System suitability check: according to Ph. Eur. Chapter 2.2.24. "Control of equipment performance" or specific Monographs or MAH dossier or validated in-house method

2. LEVEL III: examples of periodic and motivated instrument checks

This section 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 OMCLs as possible approaches to perform 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's instructions.

2.1 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}

Energy ratio test specifications vary for each spectrometer configuration.

2.2 SIGNAL-TO-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)

Noise level test specifications vary for each spectrometer configuration.

2.3 ZERO TEST

Method:

When using a polystyrene film of approximately 35 μm in thickness as standard at wavelengths 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} , a negative value may be observed. The objective of the test is to evaluate whether, despite the fact that there is almost complete absorption, energy is still detectable. Non-valid results are an indication of non-linear behaviour of the detector and the electronic system.

2.4 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, a background spectrum should be recorded and compared with the one generated during the instrument qualification or provided by the supplier.

2.5 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 wavenumbers e.g. 4000 , 2600 and 1000 cm^{-1} . The background spectrum should be compared with the one generated during the instrument qualification or provided by the supplier.

3. LEVEL IV: examples of in-use instrument checks

This section 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 OMCLs as possible approaches to perform 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's instructions.

3.1 SYSTEM SUITABILITY TEST OF THE METHOD

Method:

This test should be performed according to the Ph. Eur. 2.2.24. "Control of equipment performance", the MAH dossier or a suitably validated in-house method.

Note: regeneration or replacement of the desiccant should be done if the system suitability test fails (e.g. by drying it for 8-12h at 250°C, then flushing with N₂).

REFERENCES

(For all references, the latest version applies)

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