CEN

WORKSHOP

AGREEMENT

CWA 18134

September 2024

ICS 17.180.30

English version

Raman instruments twinning protocol

This European Standard was corrected and reissued by the CEN-CENELEC Management Centre on 11 September 2024.

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Foreword

This CEN Workshop Agreement (CWA 18134:2024) has been developed in accordance with the CEN-CENELEC Guide 29 "CEN/CENELEC Workshop Agreements – A rapid prototyping to standardization" and with the relevant provisions of CEN/CENELEC Internal Regulations - Part 2. It was approved by a Workshop of representatives of interested parties on 2024-07-11, the constitution of which was supported by CEN following the public call for participation made on 2023-12-20. However, this CEN Workshop Agreement does not necessarily include all relevant stakeholders.

The final text of this CEN Workshop Agreement was provided to CEN for publication on 2024-07-17.

Results incorporated in this CWA received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 952921.

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Introduction

Raman spectroscopy is increasingly popular in different industries to characterize advanced materials at different stages, since it is a relatively easy technique to implement that offers a rapid characterization of the specific chemical fingerprint of each material. The growth in the use of this technique has led to the development in recent decades of new and different Raman instruments, ranging from portable to high resolution equipment [1][2]. As a consequence, there is a great interest in the standardization and harmonization of Raman to increase interoperability in terms of comparability, reproducibility and reliability of the data obtained in different fields.

The first step to make data comparable is to have calibration protocols that allow obtaining valid and corrected Raman spectra in each Raman instrument. Nowadays, there are different studies and standards that indicate how to perform these calibrations, both in the x-axis of the Raman shift and in the y-axis of the relative intensity [3]. However, despite having the Raman instruments calibrated in the same way, equal Raman spectra that can be compared are not obtained, since these corrections do not allow obtaining the same Raman intensity counts and, therefore, do not fully harmonize the Raman spectra.

Despite the great interest in being able to compare Raman spectra in terms of Raman intensity, there are currently no harmonization standards or studies with protocols to obtain equal Raman spectra. The main reason is that Raman intensity depends on a large number of factors and parameters of the Raman instrument, such as the optical components (i.e. objectives, gratings, mirrors…), the laser wavelength, or the quantum efficiency of the detector [4][5]. In addition to all these instrumental factors, the Raman intensity also depends on the Raman cross-section of the measured material. Calculating the contribution of all these factors to the total Raman intensity is not feasible due to the great difficulty in controlling each element that influences the Raman signal. For this reason, a new protocol has been developed that allows two different Raman instruments (previously calibrated) to be twinned, and from there to harmonize their Raman spectra in terms of Raman intensity. The protocol uses a reference sample that allows extracting quantitative information from Raman spectra.

The concept of Raman twinning in the protocol involves the calculation of an experimental constant that includes in a single value all the differences between all the experimental variables of two Raman instruments. To do this, it is necessary to have a homogeneous reference sample that always has a fixed Raman cross-section and to obtain the linearity of the Raman intensity with the laser power of each instrument.

This Raman twinning protocol will allow progress in the use of this technique since the same Raman spectra can be obtained despite having measured in different instruments, achieving solutions in the interoperability and harmonization of the data between different Raman instruments, applications and industries.

1 Scope

This CEN Workshop Agreement (CWA) provides a procedure for twinning Raman instruments using a test sample to harmonize their Raman spectra in terms of intensity.

This twinning protocol allows to correlate different Raman instruments to obtain equal Raman spectra in terms of Raman intensity, improving comparability, reproducibility and reliability. It is intended to be applied by end-users of Raman spectroscopy instruments, Raman manufacturers or users of Raman data.

The twinning protocol is applicable to any kind of Raman instrument (non-confocal and confocal) within the boundaries described in Section 6.1. The protocol has been developed using Raman instruments using 532 nm and 785 laser sources. Prior to use, this protocol requires that the Raman instrument or the acquired test sample data has already undergone a full calibration on x and y-axis. This twinning protocol has been tested after applying the calibration protocol described in CWA 18133:2024. The protocol may have applications beyond the stated limits, such as other previous calibration protocols or systems using different excitation sources, but its effectiveness is not confirmed.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

CWA 18133:2024, *Raman instruments calibration and verification protocols*

3 Terminology

Where used, this document follows the definitions outlines in ISO 18115 series. For the purpose of this document, the following terms, definitions, symbols and abbreviations apply.

3.1 Terms and definitions

3.1.1
calibrated instrument
Raman instrument that has followed a previous calibration protocol that allows obtaining Raman spectra corrected in both Raman shift and relative intensity

3.1.2
test material
material that exhibits homogeneity and stability concerning specific properties and has been validated as suitable for its intended application within a measurement procedure

3.1.3
reference Raman instrument
Raman instrument that serves as a reference to twin the second Raman instrument (instrument to be twinned)

3.1.4
instrument to be twinned
Raman instrument that wants to be paired with the reference Raman instrument to harmonise Raman spectra in intensity