

Compliance in spectrometry:- quality assurance of spectrophotometric measurements at NIST

Jack J. Hsia, Thomas C. Larason, and P. Yvonne Barnes

Radiometric Physics Division, National Institute of Standards and Technology,
Gaithersburg, MD 20899, USA

Abstract

This paper documents the compliance in spectrophotometric measurements at NIST to ISO/IEC Guide 25. The areas of implementation include the quality manual, equipment records, calibration methods, computer software documentation, test report format, and complaint handling procedures.

Keywords

Calibration; ISO/IEC Guide 25; Quality assurance; Reflectance; Spectrometry; Spectrophotometry; Transmittance

1. INTRODUCTION

The Radiometric Physics Division at the National Institute of Standards and Technology (NIST) began to implement a calibration quality system [1] in 1993 conforming to the International Organization for Standardization (ISO) / International Electrotechnical Commission (IEC) Guide 25 on general requirements for the competence of calibration and testing laboratories (ISO/IEC Guide 25) [2]. This system covers calibration services for radiation thermometry, spectral radiance and irradiance, photometry, photodetector spectral response, and spectrophotometry. Previous quality procedures varied among the different calibration services. Using the ISO/IEC Guide 25 format will result in a uniform quality system through out the Division's calibration services.

Current spectrophotometric measurement services consist of :

- (1) Fixed fee calibration services which include spectral transmittance of cobalt blue, copper green, carbon yellow, and selenium orange glass filters and wavelength of holmium oxide glass filters.
- (2) At cost calibration services of special tests for regular spectral transmittance from 250 nm to 2500 nm, and regular and diffuse spectral reflectance from 250 nm to 2500 nm.

and

- (3) Standard reference materials for calibrating transmittance, reflectance, and wavelength scales of spectrophotometers.

The Division's quality system structure, policy, and procedures have been established. All five calibration services are being unified with standard formats and procedures. In the following sections, the implementations in six essential areas for the spectrophotometric measurements are described. These six areas are the quality manual, equipment records, calibration methods, computer software documentation, test report format, and complaint handling procedures.

2. QUALITY MANUAL

A quality manual is established following the framework of Section 5.2 of ISO/IEC Guide 25. It describes general aspects such as policy, organization structure, and administrative procedures. This manual also contains specifics as well as reference to the following items in Sections 3 to 7 for spectrophotometric measurements. These specifics include job descriptions of calibration staff, traceability of measurements, measurement scope, handling of test and calibration items, and quality assurance practices.

3. EQUIPMENT RECORDS

Equipment records are established and maintained according to ISO/IEC Guide 25 Section 8.4. These records include reference reflectance and transmittance spectrophotometers and their attachments, peripherals and apparatus. Also included are commercial spectrophotometers used as transfer instruments. These records contain manufacturer, serial number, NIST identification number, and current location. The date of last calibration and due date of next calibration of critical equipment are included in these records.

4. CALIBRATION METHODS

Section 10 of ISO/IEC Guide 25 covers calibration and test methods. The calibration services for transmittance and reflectance are documented in two NIST Special Publications (SP): SP 250-6 on regular spectral transmittance and SP 250-8 on spectral reflectance as well as other publications. These documents describe instrumentation, calibration and measurement procedures and methods, transfer measurements, and expanded uncertainty. The quality manual combines essential parts of these documents with other detailed procedures, instrument upgrades, and current calibration processes.

5. COMPUTER SOFTWARE DOCUMENTATION

Computer software used for instrument control, data acquisition, computing, tabulating, and plotting are documented based on the guidelines of Part 7 in Section 10 of ISO/IEC Guide 25. These software programs are for: (1) reference instruments to measure regular reflectance, diffuse reflectance, 45/0 reflectance factor, bidirectional reflectance distribution function (BRDF), and regular transmittance, and (2) transfer instruments to measure regular reflectance, diffuse reflectance and regular transmittance. These documents consist of computer code, flow chart, defined input and output for the program and subroutines, data location and format, and a user manual.

6. TEST REPORT FORMAT

A unified test report format is established according to ISO/IEC Guide 25 Section 13. This format contains the name of laboratory, description of test items, description of test, test results with tables and/or graphs, expanded uncertainty, name of the calibration staff writing the report, name of the technical manager approving the report, laboratory temperature and relative humidity, calibration date, and NIST test number.

7. COMPLAINT HANDLING PROCEDURES

A set of procedures conforming to ISO/IEC Guide 25 Section 16 for resolving complaints is established. These procedures include a complaint response form and complaint log. The complaint response form contains the date the complaint was received, the name of the customer, the content of the complaint, action taken, the name of the NIST personnel handling the complaint, and the date the complaint was resolved. After a complaint is resolved, the quality manager and technical manager review and initial the complaint response form. A phone log of conversations with customers is maintained by each calibration staff useful for resolving possible complaints.

8. SUMMARY

The Radiometric Physics Division at NIST has started a quality system for all calibration services based on the ISO/IEC Guide 25. This paper described the effort in spectrophotometric measurements. Six areas were focused on first, namely, the quality manual, equipment records, calibration methods, computer software documentation, test report format, and complaint handling procedure. Effort will continue toward full implementation of the ISO/IEC Guide 25.

9. REFERENCES

1. Larason, T. C., The Radiometric Physics Division's Efforts at Building a Quality System Based on ISO/IEC Guide 25, presented at AMMAC Conference, May 1994, published in De La Metrologia, 1994
2. International Organization for Standardization (ISO) / International Electrotechnical Commission (IEC), General Requirements for the Competence of Calibration and Testing Laboratories , Guide 25, 1990