

ISO 16140-4:2020 (E)

Microbiology of the food chain — Method validation — Part 4: Protocol for method validation in a single laboratory

Contents

	Foreword
	Introduction
1	Scope
2	Normative references
3	Terms and definitions
4	General principles of the single-laboratory detection or quantification method validation
4.1	General
4.2	Principles of the factorial approach
4.3	Principles of the conventional approach
5	Technical protocol for validation — Factorial approach
5.1	Qualitative methods
5.1.1	Single-laboratory method validation study against a reference method
5.1.1.1	General considerations
5.1.1.2	Factorial, orthogonal method comparison study
5.1.1.2.1	Selection of samples
5.1.1.2.2	Selection of method factors
5.1.1.2.3	Experimental design
5.1.1.3	Calculation and interpretation for sensitivity
5.1.1.4	Calculation and interpretation of the RLOD
5.1.1.5	Inclusivity/exclusivity study
5.1.2	Single-laboratory method validation study without a reference method
5.1.2.1	General considerations
5.1.2.2	Factorial study
5.1.2.3	Calculation and interpretation for sensitivity
5.1.2.4	Calculation and interpretation of LOD ₅₀
5.1.2.5	Inclusivity/exclusivity study
5.2	Quantitative methods
5.2.1	Single-laboratory method validation study against a reference method
5.2.1.1	General considerations
5.2.1.2	Selection of samples
5.2.1.3	Selection of method factors
5.2.1.4	Experimental design
5.2.1.5	Relative trueness
5.2.1.6	Accuracy profile
5.2.1.7	In-house precision (in-house repeatability and in-house reproducibility)
5.2.1.8	Inclusivity/exclusivity study
5.2.2	Single-laboratory method validation study without a reference method
5.2.2.1	General considerations
5.2.2.2	Selection of samples
5.2.2.3	Selection of method factors
5.2.2.4	Experimental design
5.2.2.5	Relative trueness
5.2.2.6	Accuracy profile
5.2.2.7	In-house precision (in-house repeatability and in-house reproducibility)
5.2.2.8	Inclusivity/exclusivity study
6	Technical protocol for validation — Conventional approach

- 6.1 Qualitative methods
 - 6.1.1 Single-laboratory method validation study against a reference method
 - 6.1.1.1 General
 - 6.1.1.2 Sensitivity study
 - 6.1.1.3 RLOD study
 - 6.1.1.4 Inclusivity/exclusivity study
 - 6.1.2 Single-laboratory method validation study without a reference method
 - 6.1.2.1 General
 - 6.1.2.2 Specificity
 - 6.1.2.3 LOD50 study
 - 6.1.2.4 Calculation and interpretation for sensitivity
 - 6.1.2.5 Inclusivity/exclusivity study
- 6.2 Quantitative methods
 - 6.2.1 Single-laboratory method validation study against a reference method
 - 6.2.1.1 General
 - 6.2.1.2 Relative trueness study
 - 6.2.1.3 Accuracy profile study
 - 6.2.1.4 Limit of quantification study
 - 6.2.1.5 In-house precision study
 - 6.2.1.6 Inclusivity/exclusivity study
 - 6.2.2 Single-laboratory method validation study without a reference method
 - 6.2.2.1 General
 - 6.2.2.2 Relative trueness study
 - 6.2.2.3 Accuracy profile study
 - 6.2.2.4 Limit of quantification study
 - 6.2.2.5 In-house precision study
 - 6.2.2.6 Inclusivity/exclusivity study
- 7 Summary of acceptability limits
- Annex A (informative) List of factors and factor levels for factorial method validation
- Annex B (informative) Calculation of in-house reproducibility for qualitative methods based on the LOD50 study described in 6.1.2.3
- Annex C (informative) Example of a factorial single-laboratory method validation study for a quantitative method against a reference method
 - C.1 General
 - C.2 Study design
 - C.3 Calculations and summary of data
 - C.3.1 Summary of the results
 - C.3.2 Relative trueness
 - C.3.3 Accuracy profile
 - C.3.4 Precision data
 - C.3.5 Interpretation
- Annex D (informative) Example of a factorial single-laboratory method validation study for a qualitative method against a reference method
- Annex E (informative) Example of a factorial single-laboratory method validation study for the variability of the LOD50 for a qualitative method without a reference method
- Annex F (informative) Determination of precision if the artificially contaminated samples are unstable
 - F.1 General
 - F.2 Adjustment of measurement values by using a linear trend
 - F.3 Adjustment of measurement values by using a reference method
- Annex G (informative) Protocol for single-laboratory validation of alternative methods for microbiological confirmation and typing procedures