Importance of Calibration Laboratories in In-situ Test Methods

Nico Parasie[1#], *Gerry* Sinjorgo[1], *Leon* Vrielink[1], and *Joek* Peuchen[1]

[1]*Fugro, Prismastraat 4, 2631 RT Nootdorp, The Netherlands*
[#]*Corresponding author: n.parasie@fugro.com*

ABSTRACT

Geotechnical in-situ test methods provide valuable data for asset development, operation, and decommissioning. For confidence in test results, industry typically requires calibration and verification of in-situ test sensors to be conducted in a calibration laboratory. A calibration laboratory typically operates according to ISO/IEC 17025 (2017) ‘general requirements for the competence of testing and calibration laboratories’, or similar standard. This paper summarises observations from practice, with focus on the following challenges (1) evaluation of measurement uncertainty of key parameter values for which no standardised methods or verification materials are available, (2) validation of test methods with no backup from a formal standard published by a standardisation organisation and (3) field test site and interlaboratory comparisons within a competitive industry setting. Specific examples are presented for the (1) estimation of calibration uncertainty for sleeve friction of a subtraction-type cone penetrometer and (2) method validation for thermal conductivity of soil derived from in-situ heat flow measurements.

Keywords: calibration, verification, validation, uncertainty, subtraction cone penetrometer, thermal conductivity

1. Introduction

Geotechnical in-situ test methods provide valuable data for asset development, operation, and decommissioning. For confidence in test results, industry typically requires calibration and verification of in-situ test sensors to be conducted in a calibration laboratory.

This paper summarises observations from the perspective of calibration laboratories providing calibration and verification certificates for in-situ test sensors. It is assumed that a calibration laboratory operates according to ISO/IEC 17025 (2017) ‘general requirements for the competence of testing and calibration laboratories’ (hereafter referenced as ISO/IEC 17025), or similar standard. Laboratory accreditation (e.g. Peuchen et al. 2018) is not covered.

The provision of calibration certificates and verification certificates is relatively straightforward where methods and procedures are standardised or specified. By exception, this is the case for one particular type of cone penetrometer that can be calibrated and verified according to specified methods given in ISO 22476-1 (2022). The common setting is that a calibration laboratory has to select or develop an appropriate method. In this paper, the focus is on the following challenges related to the common setting: (1) evaluation of measurement uncertainty of key parameter values for which no standardised methods or verification materials are available, (2) validation of test methods with no backup from a formal standard published by a standardisation organisation and (3) interlaboratory and test site comparisons within a competitive industry setting.

This paper uses metrological terms as defined in ISO/IEC Guide 99 (2007), i.e.:

- *Calibration*: operation that, under specified conditions, in a first step, establishes a relation between the quantity values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and, in a second step, uses this information to establish a relation for obtaining a measurement result from an indication
- *Measurand*: measured parameter, quantity
- *Measurement uncertainty*: non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used
- *Verification*: provision of objective evidence that a given item fulfils specified requirements
- *Validation*: verification, where the specified requirements are adequate for an intended use.

2. In-situ test sensor calibration and guidance by standards

Table 1 presents an overview of common in-situ test methods that include sensors for key measurements, such as length, force, torque, temperature and pressure. The standard penetration test (ISO 22476-3:2005 and ASTM 1586:2018) is thus excluded. Table 1 also includes references to international standards. The column ‘Standard’ lists primary test standards per test method, i.e. excludes any normative references to other standards included in the primary standards.

The column ‘CL’ (calibration laboratory) of Table 1 indicates where a primary standard includes mandatory requirements for a calibration laboratory (marked by √).

This setting applies to some ISO standards, all of which refer normatively to ISO/IEC 17025. None of the other primary standards include mandatory requirements for a calibration laboratory. Note that this paper distinguishes between requirements for calibration and requirements for a calibration laboratory.

Calibration requirements can be comprehensive. For example, 82 mandatory calibration requirements are included in Annex B of ISO 22476-1 (2022). The reference to ISO/IEC 17025 adds another 168 mandatory generic requirements, independent of a specific in-situ test method. These numbers can be compared to Annex A.1 of ASTM D5778 (2020) with 89 mandatory calibration requirements and Annex C.3 of ISO 22476-9 (2020) with 3 mandatory calibration requirements. These numbers are indicative, as a single requirement can cover an extensive bullet list of specific activities for the calibration laboratory.

Table 1. Common in-situ test methods

In-situ Test	Standard	CL*
Cone penetration test (CPT)	ISO 22476-1 (2022) ISO 19901-8 (2023) ASTM D5778 (2020)	√ √ -
Seismic velocity test with seismic CPT	ISO 19901-8 (2023) ASTM D7400 (2019)	√ -
Field vane test	ISO 22476-9 (2020) ISO 19901-8 (2023) ASTM D2573 (2018)	- √ -
Pressuremeter tests:		
Prebored	ISO 22476-5 (2023)	-
Prebored	ISO 19901-8 (2023)	√
Prebored	ASTM D4719 (2020)	-
Full displacement	ISO 22476-8 (2018)	-
Flat plate dilatometer test	ISO 19901-8 (2023) ASTM D6635-01 (2001)	√ -

***CL** = calibration laboratory

3. Development of appropriate methods of calibration

3.1. General

ISO/IEC 17025 provides guidance and mandatory requirements for development of appropriate methods of calibration. For this setting, Clause 7.2.1.4 of ISO/IEC 17025 recommends: 'Methods published either in international, regional or national standards, or by reputable technical organizations, or in relevant scientific texts or journals, or as specified by the manufacturer of the equipment are recommended. Laboratory-developed or modified methods can also be used.' Furthermore, Clause 7.2.1.1 of ISO/IEC 17025 provides mandatory requirements for validation: 'The laboratory shall validate non-standard methods, laboratory-developed methods and standard methods used outside their intended scope or otherwise modified.' This clause also describes six techniques for method validation, of which examples from Fugro's calibration laboratory are included in sections 3.1, 3.2 and 3.3.

A calibration laboratory should provide calibration of sensors incorporated in in-situ test probes. This can be mandatory, as shown in Table 1.

In some cases, the measurand of a sensor is direct, i.e. is for the intended measurand. An example would be a pressure sensor used for 'pore pressure u_2' according to ISO 22476-1 (2022).

In many cases, the measurand of a sensor is indirect. An example would be a temperature sensor used for deriving parameter values for 'thermal conductivity k'. Calibration would thus apply to temperature T. The default setting would be that a calibration laboratory would provide no calibration results for derived parameters.

A calibration laboratory can be commissioned to provide support for validation of test results derived from 'indirect' in-situ test sensors. An example is included in Section 3.3 for k: evaluation of bias and precision using reference materials.

3.2. Example - sleeve friction with subtraction cone penetrometer

Cone penetrometers of the subtraction type are widely used and specifically covered by the three standards for cone penetration tests (CPTs) presented in Table 1. For example, ISO 22476-1 (2022) considers cone penetrometers of Type I, cone resistance and sleeve friction load cells in compression, and Type II, subtraction type cone penetrometer (Figure 1).

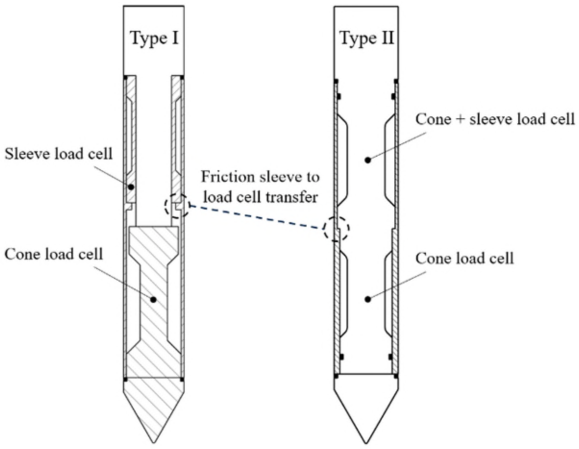

Figure 1. Type I (left) and Type II (right) Cone Penetrometers.

ISO 22476-1 (2022) provides a detailed method for calibration of sleeve friction f_s for Type I cone penetrometers. Notably, calculation of calibration uncertainties is according to Formula (B.5) of ISO 22476-1:2022, which covers dimensional (geometry) uncertainties (u_{dim}) and calibration uncertainties (u_c).

For Type II, calibration uncertainty 'shall be determined according to a formula similar to Formula (B.5), if all of the following applies: - calibration of f_s for a subtraction type cone penetrometer; - F_r is applied to the cone and thus indirectly to the force sensor for combined axial force on the cone and friction sleeve', where F_r is the reference axial force applied during calibration. A key element of Formula (B.5) is the determination of combined standard uncertainty for calibration (u_c):

$$u_c = \sqrt{\sum_{i=1}^{8} u_i^2} \tag{1}$$

where:

- u_1 is the standard uncertainty associated with F_r
- u_2 to u_7 are the standard uncertainties associated with axial forces applied to the force sensor (load cell) for f_s, particularly reproducibility, repeatability, resolution, zero drift, interpolation and reversibility
- u_8 is the standard uncertainty associated with apparent load transfer from the (force) sensor for cone resistance q_c to the force sensor for f_s, and vice versa.

The above requirements for Type II imply a 'non-standard method' as described by ISO/IEC 17025, for which method validation is required. The example below describes the method selected by Fugro's calibration laboratory. It can be seen that Techniques a), b), c) and f) of Table 2 apply.

Table 2. Summary of techniques for method validation (ISO/IEC 17025)

ID	Description
a)	Calibration or evaluation of bias and precision using reference standards or reference materials
b)	Systematic assessment of factors influencing results
c)	Testing of method robustness by variation of controlled parameters
d)	Comparison of results with other validated methods
e)	Interlaboratory comparisons
f)	Evaluation of measurement uncertainty of results based on theoretical principles and practical experience

The following 'formula similar to Formula (B.5)' was selected, based on Peuchen and Terwindt (2014):

$$u_{c,fs} = \sqrt{\left(\sqrt{\sum_{i=1}^{8} u_{i,qc+fs}^2}\right)^2 + \left(\sqrt{\sum_{i=1}^{8} u_{i,qc}^2}\right)^2} \tag{2}$$

The subscript $_{qc+fs}$ refers to the combined axial force on the cone and friction sleeve F_{qc+fs}. The subscript $_{qc}$ refers to the axial force on the cone F_{qc}.

Values of $u_{c,fs}$ are calculated for selected pairs of F_r, giving combined data sets for $u_{c,qc+fs}$ and $u_{c,qc}$. Specifically, values of $u_{c,fs}$ are taken as the higher of $u_{c,fs}$ values for $((F_{qc+fs} - F_{qc})/A_s)/(F_{qc}/A_c) = 0.06$ and $((F_{qc+fs} - F_{qc})/A_s)/(F_{qc}/A_c) = 0.01$, where A_s is the surface area of the friction sleeve and A_c is the cross-sectional projected area of the cone. The values of 0.06 and 0.01 broadly correspond with CPT friction ratios $R_f = 6$ % and $R_f = 1$ %.

Values of $u_{1,qc+fs}$ and $u_{1,qc}$ are calculated as specified for u_1 according to ISO 22476-1 (2022).

Values of $u_{2,qc+fs}$ to $u_{7,qc+fs}$ are the standard uncertainties associated with the recorded force F_{qc+fs}. These values are calculated as specified for u_2 to u_7 in Equation 1. Values of $u_{2,qc}$ to $u_{7,qc}$ for axial force on the cone (F_{qc}) are also calculated as for u_2 to u_7 in Equation 1.

The selected approach for $u_{8,qc+fs}$ considers uncertainty estimates based on database records of special testing with axial forces concurrently applied to the cone and the friction sleeve, at multiple ratios of F_{qc}/F_{qc+fs}, including $F_{qc}/F_{qc+fs} = 1$ (default calibration records) and $F_{qc}/F_{qc+fs} = 0$ (zero axial force on cone). The estimates are according to a Type B evaluation (ISO/IEC Guide 98-3:2008), i.e. obtained by scientific judgement based on all of the available information on the possible variability of input parameters. This approach considers both F_{qc+fs} and F_{qc}, allowing values of u_8 in Equation 2 to be selected.

Figure 2 presents example results of special testing for $F_{qc}/F_{qc+fs} = 1$ and for $F_{qc}/F_{qc+fs} = 0$. The presented results are for a cone penetrometer with $A_c = 1000$ mm^2. Values for the case $F_{qc}/F_{qc+fs} = 1$ are presented in red. Values for the case $F_{qc}/F_{qc+fs} = 0$ are presented in black. Solid lines show values of apparent cone resistance $q_{c;a}$ (right vertical axis) and dashed lines show values for apparent sleeve friction $f_{s;a}$ (left vertical axis). The bottom graph ($F_r = 0$ to 2 kN) is a zoom of the top graph ($F_r = 0$ to 50 kN). The vertical axes include negative intercepts, as recorded values for $q_{c;a}$ and $f_{s;a}$ can be lower than zero. Although not visible, values for $f_{s;a}$ differ slightly from zero: ~ 0.07 % of F_r/A_s for $F_{qc}/F_{qc+fs} = 1$ and similarly for $q_{c;a}$: ~ 0.10 % of F_r/A_c for $F_{qc}/F_{qc+fs} = 0$.

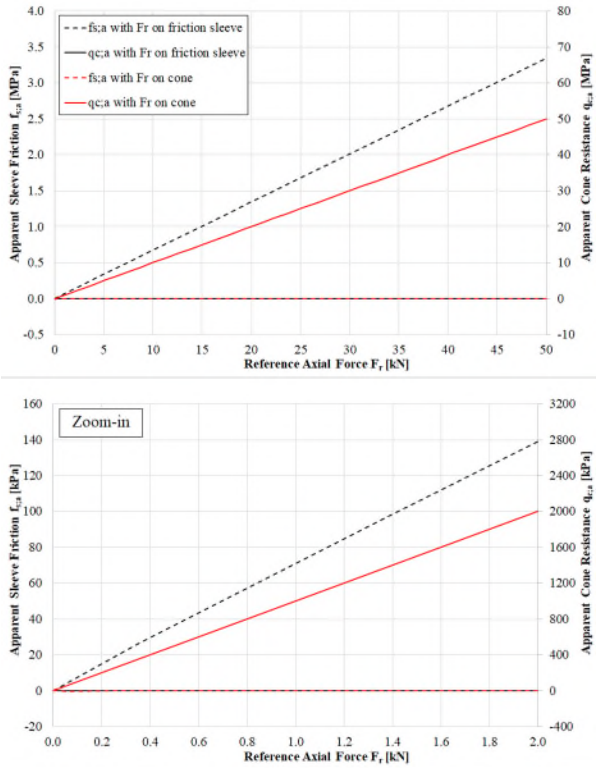

Figure 2. Example of calibration results for special testing for Type II cone penetrometer.

3.3. Example - validation for thermal conductivity of soil derived from in-situ heat flow measurements

The example presented here is about validation of a non-standard method: CPT with heat flow test (HF-CPT). The test method provides values of thermal conductivity (k). The HF test procedure requires interruption of push-in penetration, with time-based recording of temperature T and applied power P to a thermal source. Particularly, the apparatus includes a heat flow add-on module above a (CPT) cone penetrometer.

The Fugro HF module (Vrielink et al. 2023) presented in Figure 3 has a geometry of a short hollow cylinder, mounted in a recess of the shaft of the cone penetrometer or the push rod. The external surface of the HF module is flush with the external surface of sections above and below the HF module. The upper and lower parts of the HF module consist of solid thermal insulation material, each part with a height of 25 mm. The central part of the HF module is 94 mm long and has a length to diameter ratio (L/d) of 2.14. The central part consists of (1) a composite metal cylinder (high thermal conductivity) including a cylindrical thermal source and temperature sensors at mid-height points and (2) thermal insulation between the composite metal cylinder and the push rod.

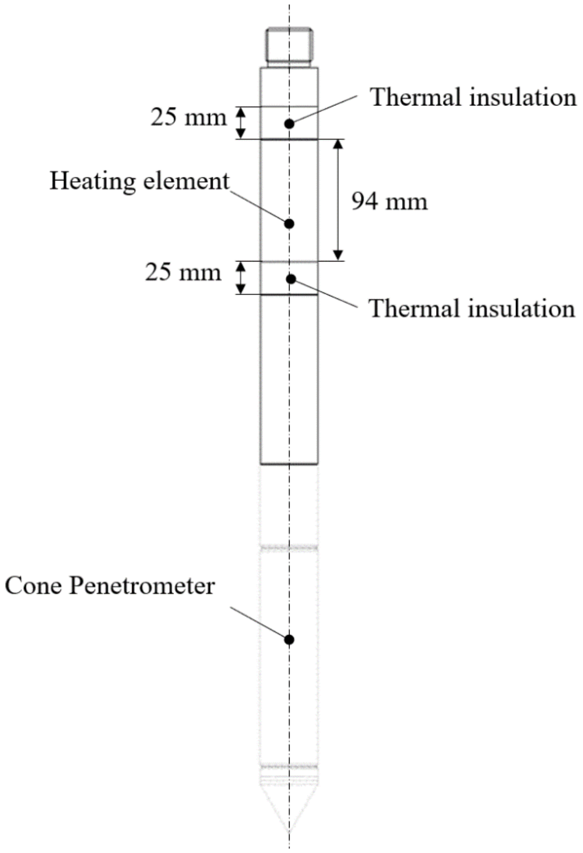

Figure 3. Fugro HF Module

Calibration of the sensors for T and P can be performed according to standard methods. This would achieve compliance with ISO/IEC 17025.

Values for k are obtained by an advanced interpretation method that combines CPT results and HF testing. To the knowledge of the authors, no standard method for calibration of k is available. The following example presents method validation (not calibration) for k using the sensors for T and P calibrated according to ISO/IEC 17025. Method validation for k is performed in Fugro's calibration laboratory using Techniques a), b), c), d) and f) of Table 2. The example presented here is about use of an agar-water mixture and a ceramic mould as reference materials, i.e. Technique a).

The use of an agar-water mixture is described by ASTM D5334 (2022) as a possible 'calibration standard' (reference material) for (laboratory) thermal needle probes. An agar-water mixture can be regarded as a solid with a jelly-like consistency. It may include air. Comments from practice are as follows.

- The thermal conductivity of an agar-water mixture is in the order of k = 0.6 W/(m·K) at 20 °C and atmospheric pressure, which can be in the range of unsaturated soils and is below the general range of k values for water-saturated clays, sands and soft rock.
- The use of a solid mitigates issues with free convection that can cause errors in measurements for thermal conductivity. Free convection issues are typical for liquids.
- Agar-water mixtures can show time-dependent changes in k. For reasonable accuracy, it can be necessary to acquire values of k that are pertinent to the time of each validation activity. Here, use

can be made of e.g. transient plane source testing (ISO22476-1:2022).

The ceramic mould is used for verification and checks of specific HF modules, covering a heating test with a minimum duration of ten minutes. The mould is made from a manufactured ceramic with a specified reference value of $k = 3.1$ W/(m.K). A thermal paste provides thermal connection between the HF module and the mould. The thermal conductivity of the paste is close to that of the ceramic. Comments from practice are as follows.

- The thermal conductivity of the ceramic is within the general range of k values for water-saturated clays, sands and soft rock
- The ceramic shows no significant time-dependent changes in k. The specified thermal conductivity of the ceramic was checked for multiple locations of the material by transient plane source testing (ISO22476-1:2022). Agreement with the specified reference value was found for all locations
- The minimum geometry of the mould was assessed by finite element simulation using COMSOL Multiphysics' Heat Transfer Module (COMSOL, 2018). Model application included heating of the HF module, the thermal paste and the mould.

4. Interlaboratory comparisons and test site comparisons

Reference force load cells and reference pressure sensors to calibrate in-situ test devices are sometimes exchanged between different calibration laboratories and national measurement institutes as part of an interlaboratory comparison programme. The authors are however not aware of any historic interlaboratory comparisons for laboratory validation of standard and non-standard methods for the in-situ test sensors itself. Comments are as follows.

- Standard methods for calibration are available only for a few types of CPT sensors
- Interlaboratory comparisons would be complex for non-standard methods that differ per calibration laboratory
- Intellectual property barriers can apply to non-standard, laboratory-developed methods
- Accreditation of a calibration laboratory can be achieved by interlaboratory comparisons for sensors that are not specific to a particular in-situ test.

Test site comparisons typically involve two or more operators deploying their in-situ test systems at a particular site with relatively uniform ground conditions. Comments are as follows.

- Test site comparisons are valuable, expensive and uncommon. Fugro participates in test site comparisons at a frequency in the order of a few years
- Some test site comparisons showed a particular in-situ test system providing results labelled as 'consistent outliers'. In some cases, this situation can be traced to inappropriate or inadequate method validation, with no involvement of a calibration laboratory as defined in Section 1
- Historically, the evaluation of the comparisons typically included 'the provision of calibration certificates', with no specific evaluation of validation methods used by the providers of the calibration certificates.

5. Conclusion

Calibration laboratories operating according to ISO/IEC 17025 or equivalent are important for geodata value of in-situ testing.

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References

ASTM International, "ASTM D1586/D1586M-18e1 Standard test method for standard penetration test (spt) and split-barrel sampling of soils", ASTM International. West Conshohocken, 2018.

ASTM International, "ASTM D2573/D2573M-18 Standard test method for field vane shear test in saturated fine-grained soils", ASTM International. West Conshohocken, 2018.

ASTM International, "ASTM D4719-20 Standard test methods for prebored pressuremeter testing in soils", ASTM International. West Conshohocken, 2020.

ASTM International, "ASTM D5334-22ae1 Standard test method for determination of thermal conductivity of soil and soft rock by thermal needle probe procedure", ASTM International. West Conshohocken, 2022.

ASTM International, "ASTM D5778-20 Standard test method for electronic friction cone and piezocone penetration testing of soils", ASTM International. West Conshohocken, 2020.

ASTM International, "ASTM D6635-01 Standard test method for performing the flat plate dilatometer", ASTM International. West Conshohocken, 2001.

ASTM International, "ASTM D7400-D7400M-19 Standard test methods for downhole seismic testing", ASTM International. West Conshohocken, 2019.

International Organization for Standardization, "ISO/IEC 17025:2017 General requirements for the competence of testing and calibration laboratories", International Organization for Standardization, Geneva, 2017

COMSOL. (2018). Introduction To Heat Transfer Module. COMSOL Documentation.

International Organization for Standardization, "ISO 19901-8:2023 Oil and gas industries including lower carbon energy — offshore structures — Part 8: Marine soil investigations", International Organization for Standardization, Geneva, 2023.

International Organization for Standardization, "ISO 22476-1:2022 Geotechnical investigation and testing - field testing - Part 1: Electrical cone and piezocone penetration test", International Organization for Standardization, Geneva, 2022

International Organization for Standardization, “ISO 22476-3:2005 Geotechnical investigation and testing - field testing - Part 3: Standard penetration test”, International Organization for Standardization, Geneva, 2005.

International Organization for Standardization, “ISO 22476-5:2023 Geotechnical investigation and testing - field testing: Part 5: Prebored pressuremeter test”, International Organization for Standardization, Geneva, 2023

International Organization for Standardization, “ISO 22476-8:2018 Geotechnical investigation and testing - field testing: Part 8: Full displacement pressuremeter test”, International Organization for Standardization, Geneva, 2018.

International Organization for Standardization, “ISO 22476-9:2020 Geotechnical investigation and testing - Field testing - Part 9: Field vane test (FVT and FVT-F)”, International Organization for Standardization, Geneva, 2020.

International Organization for Standardization, “ISO/IEC Guide 98-3:2008 Uncertainty of measurement - Part 3: Guide to the expression of uncertainty in measurement (GUM:1995). International Organization for Standardization , Geneva: ISO.

Ocłoń, P. (2021). The effect of soil thermal conductivity and cable ampacity on the thermal performance and material costs of underground transmission line. Energy, 231, 120803. https://doi.org/10.1016/j.energy.2021.120803.

Peuchen, J. and Terwindt, “Introduction to CPT accuracy”. In: 3rd International symposium on cone penetration testing CPT14: May 12-14 (2014) - Las Vegas, Nevada.

Vrielink, L., Parasie, N., Peuchen, L.J., Daniilidis, A. and Vardon, P.J., "Development of the heat flow cone penetration test (HF-CPT)". In: Symposium on Energy Geotechnics: accelerating the energy transition 3-5 October 2023 at Delft University of Technology. TU Delft Open, https://doi.org/10.59490/seg23.2023.606.