

Article

Traceable Calibration of Process Oscillation Type Density Meters: Corrosion Induced Drift Correction

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Abstract

Oscillation type (vibrating tube) density meters are widely used for quality control and custody transfer in petrochemical processes. In service, corrosion and wear of the tube wall change the effective mass of the resonator. The measured density drifts, and the drift accumulates with time. A calibration methodology for process oscillation type density meters is used, traceable to the national primary density standard of the National Institute of Metrology (NIM) of China. The analytical relationship between the oscillation period and the liquid density was derived and extended with temperature and pressure correction terms, and a complete calibration system was established to reproduce pressure and temperature conditions. For three meters of two types, in service from lightly to severely corrosive duty, out of date coefficients produced a density error of 3.35 kg/m³ at the density of water, where the internal corrosion of the tube has its greatest effect. After a full coefficient calibration the residual at the reference liquids fell to 0.01–0.10 kg/m³, with an expanded uncertainty of 0.1 kg/m³ ($k = 2$). The correction model and the traceable procedure therefore restore the required accuracy. Periodic, traceable full coefficient calibration is essential to correct corrosion induced drift, and preliminary full coefficient calibration intervals of 6 to 36 months follow from those instruments according to service severity.

Keywords: oscillation-type density meter; vibrating tube; calibration; corrosion induced drift; traceability

1. Introduction

On-line liquid densitometers are widely used in the petrochemical industry. They serve quality control, pipeline monitoring and trade settlement [1,2]. For example, the Colonial Pipeline, one of the world's most famous refined product pipelines, crosses 13 states in the USA, with a total line length of about 8.6×10^3 km, an annual transmission capacity of 9.3×10^{10} kg and 118 grades of refined product. Its design pressure is 15 MPa [2]. In China, the Lanzhou-Chongqing product pipeline is about 1.25×10^3 km long, with an annual throughput of 5.8×10^9 kg, a design pressure of 10 MPa and about ten product grades. Because many grades are transported in the same line, the oil density must be monitored in real time so that the mixed interface can be distinguished and the flow can be switched to the correct tank or off take station for the appropriate grade. The measured density is also combined with volumetric flow meters to obtain the mass for trade settlement. Accurate density measurement matters in all of them. A small error, accumulated over a long period, leads to considerable economic loss [3].

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Density is one of the most fundamental thermophysical properties of liquids and, for a process stream, a direct indicator of composition, concentration and quality. On-line density measurement now reaches well beyond pipelines: the chemical, petrochemical, food and beverage, pharmaceutical and environmental sectors all rely on it. There it underpins mass balance, blending and batching control, product quality assurance and the monitoring of transport and storage [4,5]. The energy transition has widened that space further. Metering liquefied carbon dioxide in carbon capture and storage (CCS) networks, or liquefied natural gas and hydrogen carriers, requires reliable density data at cryogenic temperatures and high pressures [6,7]. In each case the measured density is combined with volumetric metering to obtain the mass flow, so the metrological performance of the densitometer directly determines the accuracy of the fiscal or custody transfer quantity [3,7]. That reliance has stimulated two opposite developments. One is physically based calibration models that remain valid over wide ranges of temperature and pressure [8,9]. The other is miniaturized architectures built on micro electromechanical resonators [5].

Common on-line vibrating tube densitometers adopt either a straight tube or a U tube configuration, and their measurement accuracy can be as high as 0.1 kg/m^3 [10,11]. Physically based calibration models extend the valid range of a single instrument to pressures of tens of megapascals and to temperatures from cryogenic values to several hundred kelvin [6,8,9]. At the same time, the same resonant principle has been miniaturized into micro electromechanical (MEMS) microchannel resonators with piezoelectric or piezoresistive readout and revisited for both glass and metallic tubes, so that the family of vibrating tube sensors has become more diverse [5]. This versatility, together with the absence of moving parts and a continuous real time output, is why the vibrating tube densitometer has become the de facto standard for in-line density measurement.

International standards and guidelines, such as ISO 15212-2 [12] and the SIM Guidelines on the calibration of oscillation type density meters [13], describe how calibration laboratories can perform routine calibration of on-line liquid density meters, and prescribe the traceable reference materials to which such calibrations must be linked [14,15]. The effect of static pressure on the calibration of vibrating tube density meters is well documented [3,16]. One limitation remains, in spite of this maturity. The accuracy demonstrated at calibration is not necessarily retained in service. In most process applications the fluid is corrosive or carries deposits. The inner wall of the resonant tube is then progressively eroded or coated. The tube wall contributes directly to the effective vibrating mass. A gradual change of the wall therefore alters the resonance period for a given liquid and shifts the relationship between the measured period and the density. The result is a slow, systematic drift. It accumulates with time, and the original factory coefficients do not capture it. The present work quantifies that drift. For the heavily corroded meter studied in Section 3.3 the density error produced by the nameplate coefficients reached 3.35 kg/m^3 at the density of water nine years after the nameplate calibration, an average of about 0.4 kg/m^3 per year. Although such in-service drift is recognized [4,11], it is rarely quantified for process instruments. The corresponding requirement for periodic, traceable recalibration is seldom expressed in metrological terms. The drift is also coupled to the temperature and pressure responses of the sensor [17,18]. A simple offset correction is not enough. The correction model itself must be redetermined.

The present study addresses these challenges through the calibration of process meters returned from service. The procedure used here and the standard apparatus follow established practice. The indication error of the meter under test is determined against liquids of known density, as in the factory calibration performed by the manufacturer of these instruments [19] and as the guidelines and the specifications prescribe [12,13,20]. The correction coefficients are then obtained by least squares fitting.

2. Materials and Methods

The on-line density meter operates on the resonant measurement principle of a double end fixed (fixed-fixed) structure.

2.1. Theoretical Analysis

A three-dimensional model of a long straight tube resonator with fixed constraints at both ends is shown in Figure 1. The displacement of any point on a long straight pipe at a given moment can be expressed as $y(x,t)$. In practice, $y(x,t)$ consists of 3 parts: axial, radial and circumferential. However, when the ratio of the effective length of the straight pipe to the outer diameter is large enough (>10), the axial and circumferential displacements can be ignored. The long straight pipe can then be equated to a one-dimensional model, as shown in Figure 2.

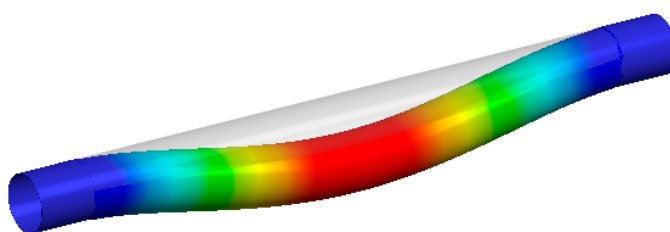


Figure 1. Three-dimensional model of a long straight tube. The colours show the computed vibration amplitude of the fundamental bending mode, from least (blue) to greatest (red).

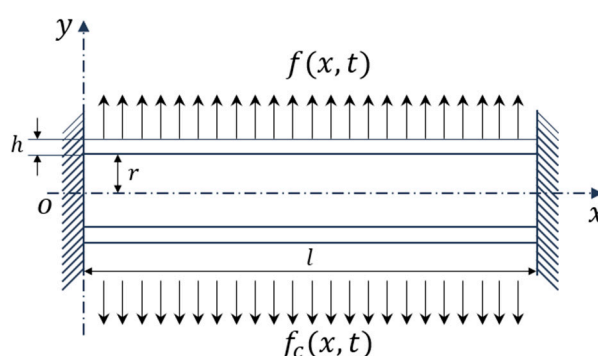


Figure 2. One-dimensional force diagram of a long straight tube.

2.2. Mathematical Modelling

We take the micrometric element of the one-dimensional model shown in Figure 2 and analyse the forces acting on it. Applying Newton's second law and the law of conservation of moments [21,22] then gives the equations of motion and of rotation of the harmonic oscillator. These are shown as (1) and (2), with higher-order terms neglected.

$$f(x,t) - f_c(x,t) - \frac{\partial Q(x,t)}{\partial x} dx = m dx \cdot \frac{\partial^2 y(x,t)}{\partial t^2} \quad (1)$$

$$\frac{\partial M(x,t)}{\partial x} dx = Q(x,t) \quad (2)$$

where $Q(x,t)$ and $M(x,t)$ are the shear force and bending moment applied to the microelement, respectively; $f(x,t)$ is the uniformly distributed excitation load and $f_c(x,t)$ is the uniformly distributed viscous damping load; and m and dx are the mass and the length of the microelement. Because the viscous damping load is proportional to the velocity of the motion, the proportionality factor is called the viscous damping coefficient c .

According to elastic mechanics, the bending moment is equal to the product of the curvature and the flexural stiffness, which in turn can be approximated as a spatial second-order derivative of the deflection $y(x,t)$:

$$M(x,t) = EI \frac{\partial^2 y(x,t)}{\partial x^2} \quad (3)$$

where EI is the product of the modulus of elasticity and the cross-sectional moment of inertia of the resonator.

The control equations for the vibration of the long straight tube resonator can be obtained by substituting (3) into (2) and combining it with (1), as shown in (4) and (5). Where (5) represents the spatial boundary conditions with fixed constraints at both ends.

$$m \frac{\partial^2 y(x,t)}{\partial t^2} + c \frac{\partial y(x,t)}{\partial t} + EI \frac{\partial^4 y(x,t)}{\partial x^4} = f(x,t) \quad (4)$$

$$y(x,t)|_{x=0,l} = 0, \frac{\partial y(x,t)}{\partial x} \Big|_{x=0,l} = 0 \quad (5)$$

where l is the effective length of the long straight pipe.

2.3. Solution

To obtain the density-measurement sensitivity of the resonant tube, the analytical expression for its natural frequency must be solved. Because the natural frequency is an inherent property of the resonator, it is unaffected by the applied load; the natural frequency information is therefore contained in the transient (homogeneous) solution of (4) subject to (5), which can be obtained by the method of separation of variables. From the basic theory of mechanical vibration [21,22], the vibration of a continuum has the synchronous-motion property, so the displacement solution $y(x,t)$ can be decomposed into a spatial part $Y(x)$ and a temporal part $Z(t)$:

$$y(x,t) = Y(x) \cdot Z(t) \quad (6)$$

where: $Y(x)$ is the general shape of vibration, $Z(t)$ is the vibration law.

Substituting (6) into the homogeneous problem for (4) and applying the method of separation of variables transforms the difficult partial differential equation into two ordinary differential equations that are easier to solve:

$$\begin{cases} EI \cdot \frac{d^4 Y(x)}{dx^4} - m\omega^2 Y(x) = 0 \\ \frac{d^2 Z(t)}{dt^2} + 2\xi\omega \cdot \frac{dZ(t)}{dt} + \omega^2 Z(t) = 0 \end{cases} \quad (7)$$

$$Y(x,t)|_{x=0,l} = 0, \frac{\partial Y(x,t)}{\partial x} \Big|_{x=0,l} = 0 \quad (8)$$

where: ω^2 is positive constant; ξ is the ratio of the viscous damping coefficient c to its critical value, known as the damping ratio.

The homogeneous solutions of (4) and (5) can be obtained by analysing the eigenvalues of the two ordinary differential equations in (7):

$$\begin{aligned} \bar{y}(x,t) = \sum_i \frac{\sin a_i l - \sinh a_i l}{\cos a_i l - \cosh a_i l} \times [(\cosh a_i x - \cos a_i x) - (\sinh a_i x - \sin a_i x)] \\ \times A_i e^{-\xi_i \omega_i t} \sin \left(\sqrt{1 - \xi_i^2} \omega_i t - \varphi_i \right) \end{aligned} \quad (9)$$

where a_i and φ_i are determined by the initial conditions, and a_i is the eigenvalue (frequency parameter) of the spatial problem defined in (10):

$$a_i = \sqrt[4]{\frac{m\omega^2}{EI}} \quad (10)$$

Owing to the spatial boundary conditions, only specific values of a_i make the mode shape function a nontrivial solution. For the boundary conditions with fixed constraints at both ends, a_i must satisfy the transcendental equation shown in (11), whose first five positive roots are 4.730, 7.853, 10.996, 14.137 and 17.279:

$$\cos a_i l \cdot \cosh a_i l = 1 \quad (11)$$

Equation (9) shows that, under certain initial conditions, the motion of the long straight tube resonator behaves as a linearly weighted sum of the vibration modes of each order vibrating at their corresponding damped vibration frequencies. Combining (10) and substituting the expressions for the mass per unit length m and the cross-sectional moment of inertia I , an analytical equation for the intrinsic frequency of the long straight tube resonator can be obtained:

$$f_i = \frac{(a_i l)^2 \sqrt{E} \sqrt{(r+h)^4 - r^4}}{4\pi l^2 [(2rh + h^2)\rho_s + r^2\rho_f]^{0.5}} \quad (12)$$

where ρ_s and ρ_f are the material density and the internal liquid density of the long straight tube, and r and h are its inner radius and wall thickness.

If f_{ivac} is the i order natural frequency of the resonant tube in vacuum, f_{iflu} is the i order natural frequency of the resonant tube in the measured liquid filling. Obviously, f_{iflu} is constant when the structure size of the resonant tube is fixed. Then according to (12), there can be:

$$\frac{f_{ivac}^2}{f_{iflu}^2} = \frac{\frac{(a_i l)^4 E [(r+h)^4 - r^4]}{16\pi^2 l^4 (2rh + h^2)\rho_s}}{\frac{(a_i l)^4 E [(r+h)^4 - r^4]}{16\pi^2 l^4 [(2rh + h^2)\rho_s + r^2\rho_f]}} = 1 + \frac{r^2}{(2rh + h^2)\rho_s} \rho_f \quad (13)$$

After simplification, we can get:

$$\rho_f = G_2 \cdot T_{iflu}^2 - G_0 \quad (14)$$

where: $G_2 = f_{ivac}^2 (2rh + h^2)\rho_s r^{-2}$, $G_0 = (2rh + h^2)\rho_s r^{-2}$, $T_{iflu} = f_{iflu}^{-1}$ which represents the i -th order natural period of the resonant tube filled with the measured liquid. Equation (14) shows that the density quantity can be measured by measuring the natural period of the resonant tube under the measured liquid filling. The two constants G_2 and G_0 are geometric properties of the resonator, and the symbols are used here to distinguish them from the fitted coefficients K_0 , K_1 and K_2 of the correction model (15).

In practice, most instrument manufacturers provide a density model with temperature and pressure correction coefficients, to be used with the temperature and pressure transmitters on site, and additional temperature and pressure correction terms are therefore required:

$$\rho_T = K_0 + K_1 T + K_2 T^2 \quad (15)$$

$$\rho_t = \rho_T [1 + K_{18}(t - 293.15)] + K_{19}(t - 293.15) \quad (16)$$

$$\rho_P = \rho_t [1 + K_{20}P] + K_{21}P \quad (17)$$

where:

$$K_{20} = K_{20A} + K_{20B}P \quad (18)$$

$$K_{21} = K_{21A} + K_{21B}P \quad (19)$$

In Equations (15)–(19), T is the measured natural period, t is the liquid temperature and P is the static pressure. K_0 , K_1 and K_2 are the density period coefficients; K_{18} and K_{19} are the temperature-correction coefficients; and K_{20A} , K_{20B} , K_{21A} and K_{21B} are the pressure correction coefficients. All coefficients are obtained from a multipoint calibration at controlled temperature and pressure followed by a least squares fit, so that the model reproduces the reference density over the whole operating range. Because the density of the calibration liquid depends on both temperature and pressure, the reference densities were obtained with a laboratory oscillation type density meter and certified reference liquids. The coefficients are identified from a least squares fit to four reference liquids covering 750–1000 kg/m³. The residual of that fit, and the uncertainty it propagates to the indicated density, are quantified in Section 3.1.

2.4. Density-Measurement Sensitivity and Quality Factor of the Resonator

The density indication is obtained from the measured oscillation period through Equation (14). The quality of the result is then governed by two resonator characteristics: the density-measurement sensitivity, which sets the achievable resolution, and the quality factor, which sets the stability of the measured period. The sensitivity S is defined as the change in the natural frequency produced by a unit change in the liquid density, $S = \partial f / \partial \rho$, and the period and the frequency are related by $\tau = 1/f$, so that the two measures are linked by $S = -(1/\tau^2) \cdot \partial \tau / \partial \rho$. Differentiating Equation (14) shows that S is not constant. It is largest for low density liquids and falls as the density rises, so that the period to density coefficient $\partial \rho / \partial \tau$ of the calibration model grows with density. For the meters studied here $\partial \rho / \partial \tau$ is about 3 kg·m⁻³·us⁻¹ near 1000 kg/m³, which is the value used in the uncertainty budget of Section 3.1.

The second index is the quality factor, $Q = f/\Delta f$, where Δf is the half power bandwidth; physically it is 2π times the ratio of the energy stored in the resonator to the energy dissipated per cycle. In a feedback driven density meter it is decisive, because the controller locks the excitation to the natural frequency. A resonator of high quality factor has a steep phase characteristic and a narrow bandwidth, so a given phase error corresponds to a smaller frequency error, and hence to a smaller density error [10,11]. Both indices depend on the geometry of the tube, on its material and on the mode order.

2.5. Calibration System

2.5.1. Traceability System

As shown in Figure 3, the NIM primary standard achieves the highest level, with a relative standard uncertainty of 2.0×10^{-7} , with which the absolute density is determined by laser interferometry. The density scale is then transferred to the solid density national standard (relative uncertainty 4.0×10^{-6}). By the hydrostatic weighing method the solid-density capability is transferred to pure liquids, establishing national liquid density reference materials (relative uncertainty 2.0×10^{-5}) that are used to calibrate both laboratory and process oscillation type density meters.

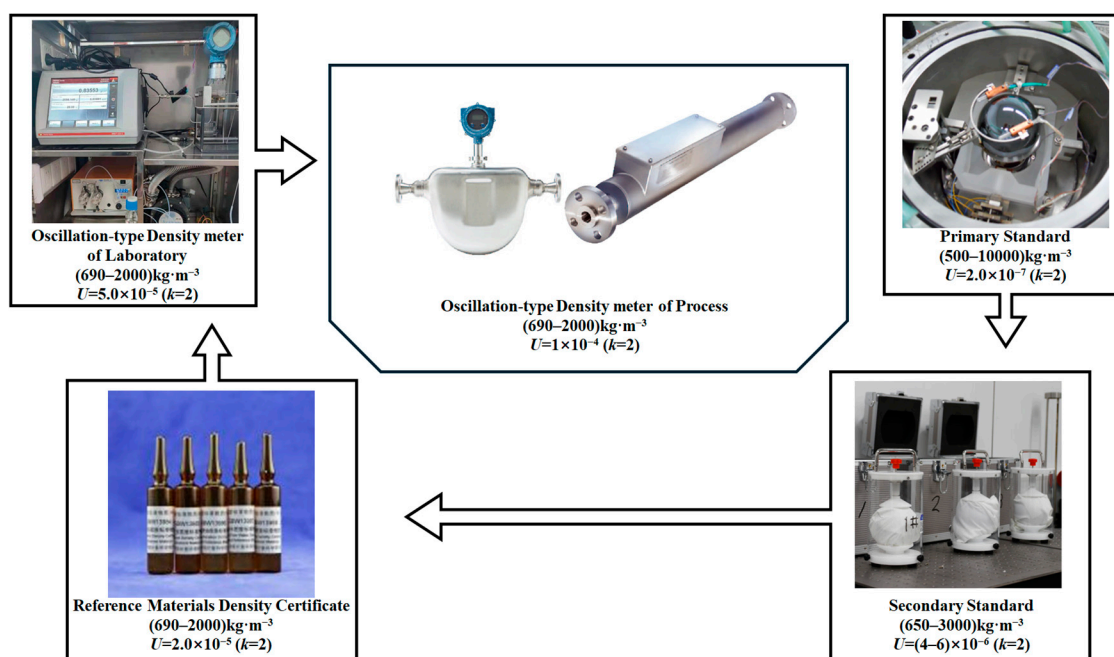


Figure 3. Density transfer system in NIM.

2.5.2. Calibration Apparatus and Procedure

The complete calibration system is shown schematically in Figure 4. It is composed of six main parts: the operating console, the standard device, the measurement chamber, the pressure loading system, the primary thermostatic system and the air system. Take the air circulation path as an example. Air is compressed by an air compressor and stored in a gas tank, then passes through an air dryer and the primary thermostatic system before an air circulation pump delivers it to the measurement chamber. A temperature sensor installed inside the measurement chamber provides real time feedback to the chamber temperature control system. Once the temperature is stable within 0.02 K, the data acquisition system records the density, temperature and pressure for further processing.

In the measurement chamber, the red vessel is filled with tridecane and the blue vessel with pure water, and the operation and measurement procedure for the two liquids are identical. Pressurization is achieved by a servo hydraulic loading system: after the temperature inside the chamber has stabilized, the circulation is stopped and the pipeline valves are closed, so that the different pressure points can be loaded automatically. The primary standard of the set consists of a liquid-density reference material together with a temperature and pressure controllable vibrating tube density measurement system, which supplies the traceable reference density for the calibration.

Two operations are referred to as calibration in this paper and are distinguished as follows. An indication error calibration determines the indication error of the meter against liquids of known density with the factory coefficients retained; this is the operation permitted by JJF 2164-2024 [20]. A full coefficient calibration redetermines the complete coefficient set K_0 to K_{21} from the accuracy, temperature and pressure tests by least squares, and it is this operation that restores the factory accuracy of a meter whose tube has degraded.



Figure 4. Calibration system in NIM. The red dashed box marks the density meter under test.

Figure 5 shows the calibration procedure. The calibrations followed the verification regulation JJG 370-2019 [23], since superseded by the calibration specification JJF 2164-2024 [20]. The influence of product density, pressure and temperature on the performance of process densitometers, and the practical requirements of in-service calibration, are examined in [3]. They normally comprise an accuracy test and a temperature test. A least-squares fit to the data then gives the accuracy and temperature correction coefficients K_0 , K_1 , K_2 , K_{18} and K_{19} , defined as in the model above. A static pressure test is then performed on the meter to obtain the pressure correction coefficients K_{20} and K_{21} . Residual bubbles must be fully expelled from the sensor. The liquid is therefore admitted from the lower port and pumped through the tube cavity until it reaches the upper liquid outlet at the flange, where the vent valve above the meter is closed. The three way valve below is then switched so that the liquid inlet is connected to the pressure calibrator. To further eliminate dissolved gas, the pressure in the tube is raised to above 2.0 MPa by the pressure calibrator and then gradually reduced to 0.5 MPa. This pressurization depressurization cycle is repeated three to five times to remove all the internal gas. The pressure is then increased from 0 to 2.5 MPa in 0.5 MPa steps. When the pressure calibrator reading is stable, the pressure, temperature and vibration period of the density meter are recorded at that state. The pressure correction coefficients K_{20} and K_{21} are then obtained by the least squares method. Figure 6 shows one of the meters under test, together with endoscope images of the inner wall of the resonant tubes of several meters of the same type. They range from a well maintained sensor to a heavily corroded one. In recent years we noticed the indication drifting during calibration, so we also examined the tube bore with an endoscope, although this is not required by the calibration procedure. That examination is qualitative, showing the corrosion but not quantifying the change in wall thickness or in the effective mass. The tubes shown come from instruments of different serial numbers.

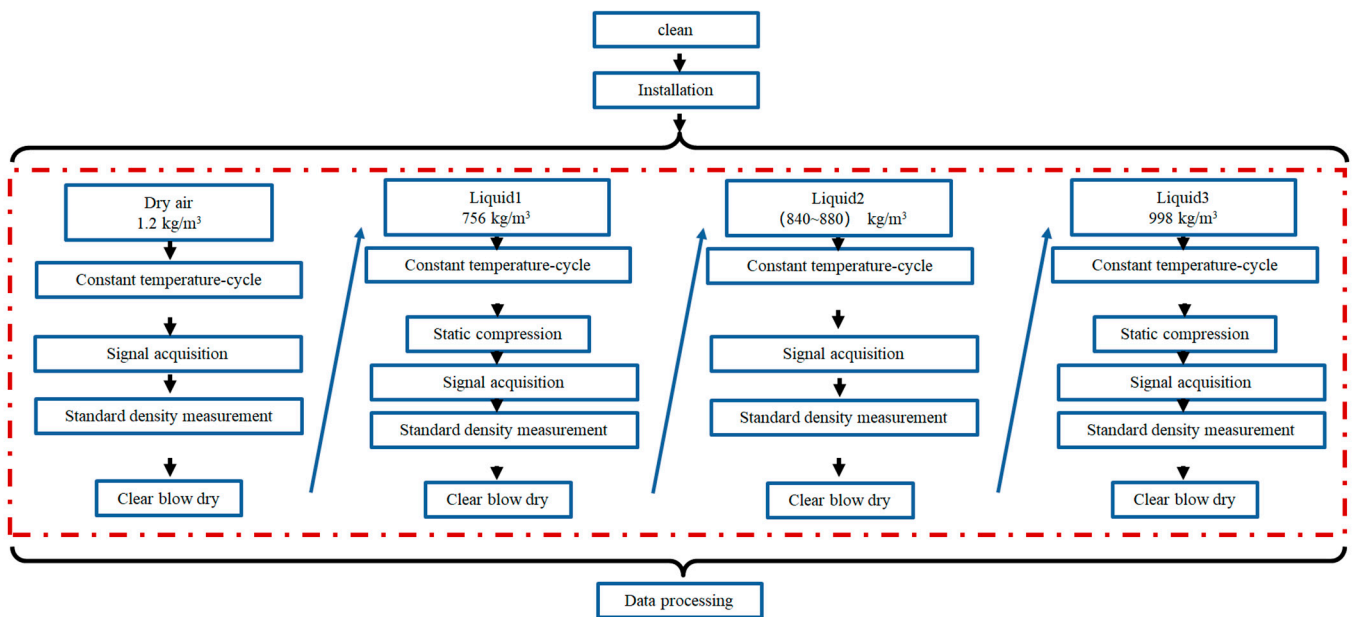


Figure 5. Calibration process. The red dash-dot frame groups the steps carried out inside the calibration system.

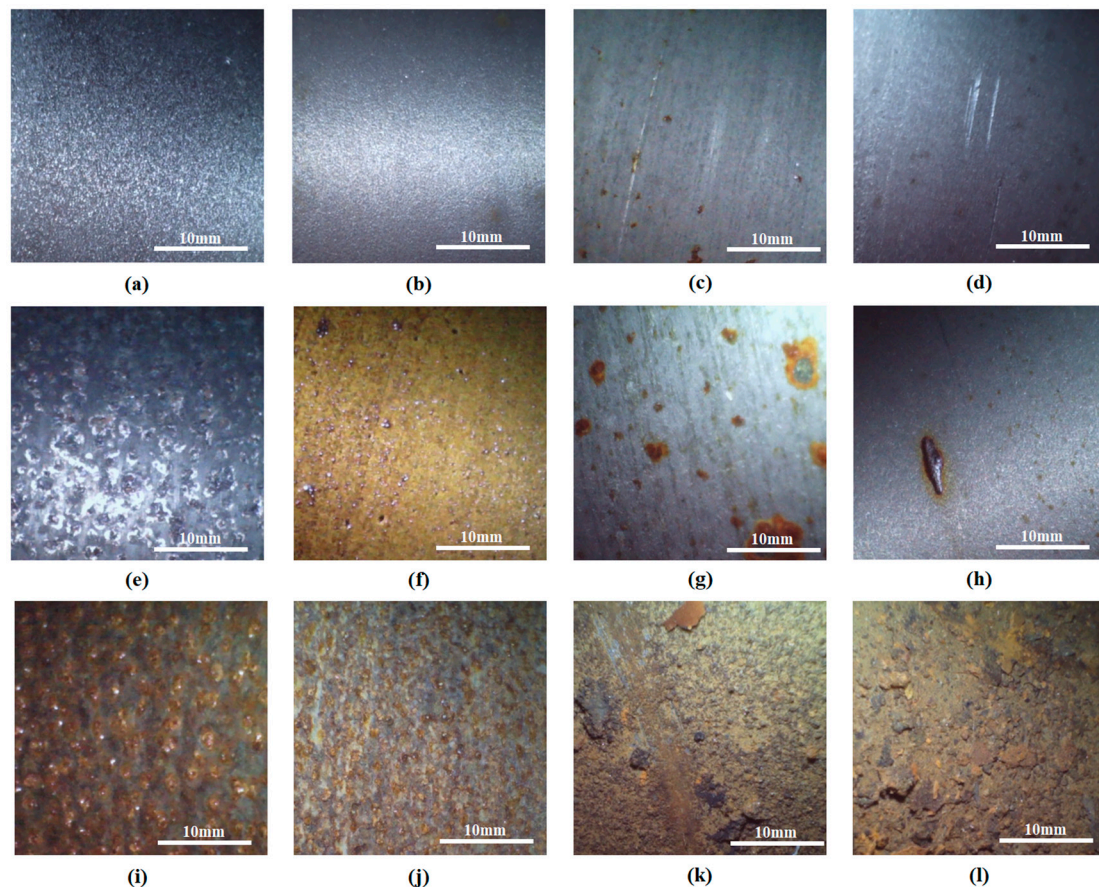


Figure 6. Oscillation type density meter and inner wall of the resonant tube: (a,b) normal; (c,d) scratch; (e,f) deposition; (g,h) rust; (i–l) corrosion.

All three meters under test have an internal bore of DN25. The resonant tube of the 7835 type meter is made of Ni-Span-C, a nickel iron chromium alloy with a stable elastic modulus.

Its best case accuracy is $\pm 0.15 \text{ kg/m}^3$ over a nominal range of 300–1100 kg/m^3 , with a repeatability of $\pm 0.02 \text{ kg/m}^3$. The two CDM type meters are compact density meters that obtain the density from the resonance of a U tube [24]. They share a nominal range of 0–3000 kg/m^3 and a repeatability of $\pm 0.02 \text{ kg/m}^3$. They differ in wetted material and in specification. The CDM100P is the C22 nickel alloy version, with a best case accuracy of $\pm 0.1 \text{ kg/m}^3$, and the CDM100M is the 316L stainless steel version, with a best case accuracy of $\pm 0.2 \text{ kg/m}^3$ [24]. The three instruments therefore carry different specifications. The comparisons in Section 3 use the specification of the instrument concerned. The instruments resonate at very different frequencies. With pure water in the tube at 293.15 K, the 7835 type meter resonates at about 700 Hz, a period of about 1.4 ms. The CDM type meters resonate at about 270 Hz, a period of about 3.7 ms.

The calibration matrix is the set of liquid, temperature and pressure states at which the meter is compared with the reference. The accuracy coefficients K_0 , K_1 and K_2 are identified at $293.15 \pm 0.02 \text{ K}$ and atmospheric pressure from four reference liquids: ultrapure water, tridecane and two ethanol water mixtures, spanning 750–1000 kg/m^3 . Their reference densities are measured with a density meter calibrated against the certified reference materials, with a resolution of 0.001 kg/m^3 and an uncertainty of 0.050 kg/m^3 ($k = 2$). The temperature coefficients K_{18} and K_{19} are then identified from the fourteen measurements of the temperature test. The pressure coefficients K_{20} and K_{21} are identified from static loading at six pressure steps from 0 to 25 bar in 5 bar increments. The resonant period is read with a frequency counter and the liquid temperature at the inlet and the outlet of the meter with calibrated sensors.

A point is accepted when the liquid temperature has been stable within $\pm 0.02 \text{ K}$. The mean of the inlet and outlet thermometer readings of the meter under test must also differ from the corresponding mean of the reference device by less than 0.02 K. Ten repeated readings are recorded at each calibration point. The density coefficients, the temperature coefficients and the pressure coefficients are determined in that order by least squares.

3. Results

3.1. Uncertainty of the Calibrated Density

The calibration result is expressed as the indication error of the meter under test, $E = \rho_{\text{ind}} - \rho_{\text{ref}}$, where ρ_{ind} is the mean density indicated by the process meter and ρ_{ref} is the mean reference density of the liquid. Five contributions are mutually independent: the reference density (u_{ref}), the propagation of the least squares fit that determines the coefficients (u_{fit}), the resolution of the indication (u_{res}), the liquid temperature (u_t) and the repeatability of the meter (u_s). Two further contributions, the resonant period (u_r) and the static pressure (u_p), are negligible and are quantified below for completeness. The budget is built following the Guide to the Expression of Uncertainty in Measurement [25].

The reference liquids are ultrapure water and tridecane together with two ethanol water mixtures that cover the intervening range, and their densities are measured with a laboratory oscillating tube density meter. That instrument is verified against the certified reference materials GBW13987 (ultrapure water) and GBW13985 (tridecane), which are traceable to the NIM liquid density scale through hydrostatic weighing (Section 2.5.1), so that the working liquid densities are obtained from a continuous traceability chain. The expanded uncertainty of the reference density is 0.050 kg/m^3 ($k = 2$), so that $u_{\text{ref}} = 0.025 \text{ kg/m}^3$; this term alone dominates the budget.

Because the density follows from $\rho = K_0 + K_1 T + K_2 T^2$, the uncertainty of the fit that determines K_0 , K_1 and K_2 propagates into the result. Every reference density in the fit is measured with the same laboratory density meter, so the reference density standard uncertainty $u_{\text{ref}} = 0.025 \text{ kg/m}^3$ established above applies at all four points. The covariance of the fit gives $u_{\text{fit}} = 0.025 \text{ kg/m}^3$ at the calibration points, which is the largest value over the

calibrated range and the one listed in Table 1. Section 3.2 states how that contribution grows outside the calibrated range.

Table 1. Uncertainty budget of the calibrated density at the calibration point.

Source	Standard Uncertainty	Sensitivity Coefficient	Contribution (kg/m ³)
Reference density, u_{ref}	0.025 kg/m ³	1	0.025
Least-squares fit, u_{fit}	0.025 kg/m ³	-	0.025
Indication resolution, u_{res}	0.1 kg/m ³	$1/(2\sqrt{3})$	0.029
Liquid temperature, u_t	0.01 K	$0.21 \text{ kg}\cdot\text{m}^{-3}\cdot\text{K}^{-1}$	0.002
Repeatability, u_s	-	1	0.002
Resonant period, u_T	1×10^{-4} us	$3 \text{ kg}\cdot\text{m}^{-3}\cdot\text{us}^{-1}$	<0.001
Static pressure, u_P	0.0009 MPa	$<0.1 \text{ kg}\cdot\text{m}^{-3}\cdot\text{MPa}^{-1}$	<0.001
Combined standard uncertainty, u_c	-	-	0.045
Expanded uncertainty, U ($k = 2$)	-	-	0.1

The resolution of the indication contributes $u_{\text{res}} = \delta/(2\sqrt{3})$; with $\delta = 0.1 \text{ kg/m}^3$ this gives $u_{\text{res}} = 0.029 \text{ kg/m}^3$. The manufacturer specifies this instrument to 0.1 kg/m^3 at its best case point.

The liquid temperature is measured at the inlet and at the outlet of the meter with sensors whose expanded uncertainty is 0.02 K ($k = 2$); near 293.15 K the density of pure water changes by $0.21 \text{ kg}\cdot\text{m}^{-3}\cdot\text{K}^{-1}$, so that $u_t = 0.0021 \text{ kg/m}^3$. The repeatability of the meter contributes $u_s = 0.002 \text{ kg/m}^3$. The period is read with a frequency counter whose expanded uncertainty of $2 \times 10^{-7} \text{ Hz}$ ($k = 2$) corresponds to $1 \times 10^{-4} \text{ us}$; with the density sensitivity of about 3 kg/m^3 per us established in Section 2.4 this gives $u_T = 0.0003 \text{ kg/m}^3$. The static-pressure test uses a 0.05-class pressure calibrator over 0–3 MPa, giving $u_P < 0.0001 \text{ kg/m}^3$.

Combining the independent contributions gives $u_c = (u_{\text{ref}}^2 + u_{\text{fit}}^2 + u_{\text{res}}^2 + u_t^2 + u_s^2 + u_T^2 + u_P^2)^{1/2} = 0.045 \text{ kg/m}^3$, and hence $U = k \times u_c = 0.09 \text{ kg/m}^3$, quoted as 0.1 kg/m^3 ($k = 2$) at the resolution of the indication. Table 1 collects the budget. The result lies inside the 0.15 kg/m^3 accuracy declared for this instrument. It equals the best case figure of 0.1 kg/m^3 at the resolution of the indication, which confirms that the full coefficient calibration of a corroded meter restores the accuracy required for process monitoring and custody transfer. The budget is evaluated at the reference temperature and pressure of the accuracy test, so that the value of Table 1 applies at those conditions. Over the wider range of the temperature test, about 286 to 320 K, the scatter of the temperature fit is larger, 0.05 to 0.13 kg/m^3 .

3.2. Validity of the Calibrated Coefficients over the Full Operating Range

The coefficients identified from four reference liquids are strictly valid over the calibrated range of $750\text{--}1000 \text{ kg/m}^3$, whereas the meters are specified over a wider interval, so it is necessary to state how the correction affects the result outside that range. Outside the reference liquids the correction is an extrapolation of the fitted polynomial rather than a measurement, and the uncertainty it carries grows with the distance from the calibrated range. The contribution of the fit rises from 0.025 kg/m^3 at the calibration points to about 0.055 kg/m^3 at 700 kg/m^3 and about 0.17 kg/m^3 at 1200 kg/m^3 . Outside the range the dominant uncertainty is therefore the extrapolation itself rather than any individual measurement. The calibration described here covers only the middle part of that interval, $750\text{--}1000 \text{ kg/m}^3$, and it is over that narrower interval that the coefficients obtained here are valid.

3.3. Corrosion Induced Drift and Recalibration

A 7835 type process oscillation type density meter was used as the meter under test. Table 2 gives the coefficients of its nameplate set and of the three full coefficient calibrations of 2021, 2023 and 2024.

Table 2. Coefficients of the 7835 type meter.

Coefficients	Unit	Original Factory (2015)	2021	2023	2024
K_0	$\text{kg}\cdot\text{m}^{-3}$	-1.29441×10^3	-1.43867×10^3	-1.20001×10^3	-1.56425×10^3
K_1	$\text{kg}\cdot\text{m}^{-3}\cdot\mu\text{s}^{-1}$	-3.01497×10^{-1}	-9.06438×10^{-2}	-4.31847×10^{-1}	8.54369×10^{-2}
K_2	$\text{kg}\cdot\text{m}^{-3}\cdot\mu\text{s}^{-2}$	1.3525×10^{-3}	1.27400×10^{-3}	1.39593×10^{-3}	1.21217×10^{-3}
K_{18}	K^{-1}	-2.30548×10^{-5}	-1.373300×10^{-4}	-1.360204×10^{-4}	-2.559478×10^{-4}
K_{19}	$\text{kg}\cdot\text{m}^{-3}\cdot\text{K}^{-1}$	1.0038×10^{-2}	1.309164×10^{-1}	1.269514×10^{-1}	2.487909×10^{-1}
K_{20A}	bar^{-1}	-2.32404×10^{-4}	1.39625×10^{-5}	-7.42796×10^{-5}	-5.34558×10^{-5}
K_{20B}	bar^{-2}	1.3288×10^{-6}	-3.27253×10^{-6}	-1.06335×10^{-6}	-3.57340×10^{-6}
K_{21A}	$\text{kg}\cdot\text{m}^{-3}\cdot\text{bar}^{-1}$	2.0151×10^{-1}	3.03958×10^{-2}	1.07002×10^{-1}	7.68132×10^{-2}
K_{21B}	$\text{kg}\cdot\text{m}^{-3}\cdot\text{bar}^{-2}$	-3.18032×10^{-3}	1.43680×10^{-4}	-1.56243×10^{-3}	1.12630×10^{-3}

A CDM100P and a CDM100M process oscillation type density meter were used as further meters under test. Table 3 gives the coefficients of both, from their nameplate sets and from the intervening full coefficient calibrations.

Table 3. Coefficients of the CDM100P and CDM100M meters: (a) the CDM100P meter and (b) the CDM100M meter.

(a) CDM100P Meter						
Coefficients	Unit	Original Factory (2017)	2021	2022	2023	2025
K_0	$\text{kg}\cdot\text{m}^{-3}$	-3.47370×10^3	-3.47477×10^3	-3.47455×10^3	-3.47522×10^3	-3.47297×10^3
K_1	$\text{kg}\cdot\text{m}^{-3}\cdot\mu\text{s}^{-1}$	0	0	0	0	0
K_2	$\text{kg}\cdot\text{m}^{-3}\cdot\mu\text{s}^{-2}$	3.19642×10^{-4}	3.19717×10^{-4}	3.19703×10^{-4}	3.19744×10^{-4}	3.19581×10^{-4}
K_{18}	K^{-1}	-2.99197×10^{-4}	-3.20953×10^{-4}	-3.27884×10^{-4}	-3.04803×10^{-4}	-2.63165×10^{-4}
K_{19}	$\text{kg}\cdot\text{m}^{-3}\cdot\text{K}^{-1}$	-9.36254×10^{-1}	-9.29210×10^{-1}	-9.21243×10^{-1}	-9.45005×10^{-1}	-9.80402×10^{-1}
K_{21A}	$\text{kg}\cdot\text{m}^{-3}\cdot\text{bar}^{-1}$	9.86657×10^{-2}	1.09650×10^{-1}	1.10834×10^{-1}	1.06208×10^{-1}	1.04049×10^{-1}
(b) CDM100M Meter						
Coefficients	Unit	Original Factory	2017	2019	2021	2023
K_0	$\text{kg}\cdot\text{m}^{-3}$	-3.46550×10^3	-3.46535×10^3	-3.46382×10^3	-3.46761×10^3	-3.46872×10^3
K_1	$\text{kg}\cdot\text{m}^{-3}\cdot\mu\text{s}^{-1}$	0	0	0	0	0
K_2	$\text{kg}\cdot\text{m}^{-3}\cdot\mu\text{s}^{-2}$	3.11081×10^{-4}	3.11117×10^{-4}	3.10994×10^{-4}	3.11241×10^{-4}	3.11291×10^{-4}
K_{18}	K^{-1}	-2.99677×10^{-4}	-4.62854×10^{-4}	-2.65635×10^{-4}	-3.26518×10^{-4}	-3.01191×10^{-4}
K_{19}	$\text{kg}\cdot\text{m}^{-3}\cdot\text{K}^{-1}$	-9.34349×10^{-1}	-7.64668×10^{-1}	-9.77025×10^{-1}	-9.17495×10^{-1}	-9.45364×10^{-1}
K_{21A}	$\text{kg}\cdot\text{m}^{-3}\cdot\text{bar}^{-1}$	9.33063×10^{-2}	9.49553×10^{-2}	7.11316×10^{-2}	9.05720×10^{-2}	1.02936×10^{-1}

In Table 3 the coefficient K_1 is zero for both CDM type meters in every campaign. This reflects the density model form used by the manufacturer of those instruments, in which the density is expressed as a quadratic function of the period without a linear term, so that only K_0 and K_2 are identified by the fit. K_1 is not a physical quantity that happens to vanish, and its zero value therefore carries no metrological meaning.

Figure 7 shows the density error that results when the coefficients of an earlier calibration are retained for the 7835 type meter. The severe internal corrosion changed the mass of the resonant tube substantially and therefore caused large deviations in the

measured density. With the original factory coefficients the density error reached 3.35 kg/m^3 for water, far exceeding the declared accuracy of 0.15 kg/m^3 . The meter received a full-coefficient calibration in 2021, in 2023 and in 2024. At the four reference liquids the residual of the recalibrated coefficients was at most 0.052 kg/m^3 in 2021, 0.013 kg/m^3 in 2023 and 0.010 kg/m^3 in 2024. Each full coefficient calibration therefore restored agreement with the traceable reference densities to well within the 0.15 kg/m^3 specification of the instrument. This confirms that the correction model in (15)–(19) and the recalibration procedure effectively restore the accuracy of a corroded meter.

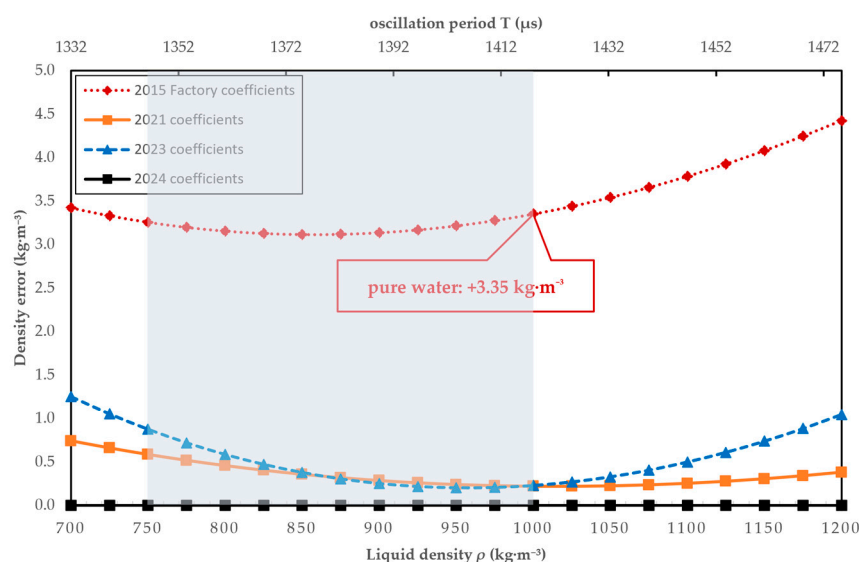


Figure 7. Density error produced by out-of-date calibration coefficients for a 7835-type process oscillation density meter. The shaded band marks the calibrated range; outside it the curves are an extrapolation of the fitted model.

Figure 8 shows the corresponding analysis for the CDM100P meter, for which the most recent calibration (2025) is taken as the baseline. Although this meter has also been in service for several years, its drift is much smaller than that of the corroded meter. At 293.15 K and 0 MPa the worst error over 700–1200 kg/m^3 is only 0.20 kg/m^3 (2022 coefficient set), the remaining sets giving 0.15 kg/m^3 (2017 factory), 0.19 kg/m^3 (2021) and 0.13 kg/m^3 (2023). These values stay within about twice the 0.1 kg/m^3 specification, that is, an order of magnitude better than the 7835-type meter. The reason is that the density coefficients of this meter are almost unchanged, so the two meters illustrate the same degradation mechanism at very different magnitudes.

A CDM100M meter, calibrated on several occasions between 2017 and 2023, provides an intermediate case; its coefficients are given in Table 3b. Over the calibrated range an out of date coefficient set produces a density error of 1.01 kg/m^3 for the 2017 set, 0.88 kg/m^3 for the 2019 set and 0.43 kg/m^3 for the 2021 set. This lies between the 0.20 kg/m^3 of the lightly corroded CDM100P meter and the 3.35 kg/m^3 of the severely corroded 7835 type meter, so the three instruments span the range of degradation met in service.

For the two CDM type meters the corresponding residual at the reference liquids at 293.15 K was at most 0.050 kg/m^3 over the four campaigns of the first meter and at most 0.102 kg/m^3 over the three campaigns of the second. In every campaign of all three meters this residual is of the same order as the calibration uncertainty established in Section 3.1. These residuals are those of the accuracy fit at the four reference liquids, and they are not an independent check of the meter in service. Such a check is available from the campaigns themselves, in which a coefficient set identified in one campaign is applied to the reference

liquids measured in another campaign. Over all such pairs the out of sample error is 0.06 to 0.92 kg/m³, against at most 0.102 kg/m³ for the largest residual of any fit; the per point data are given in Tables S2–S4 of the Supplementary Material.

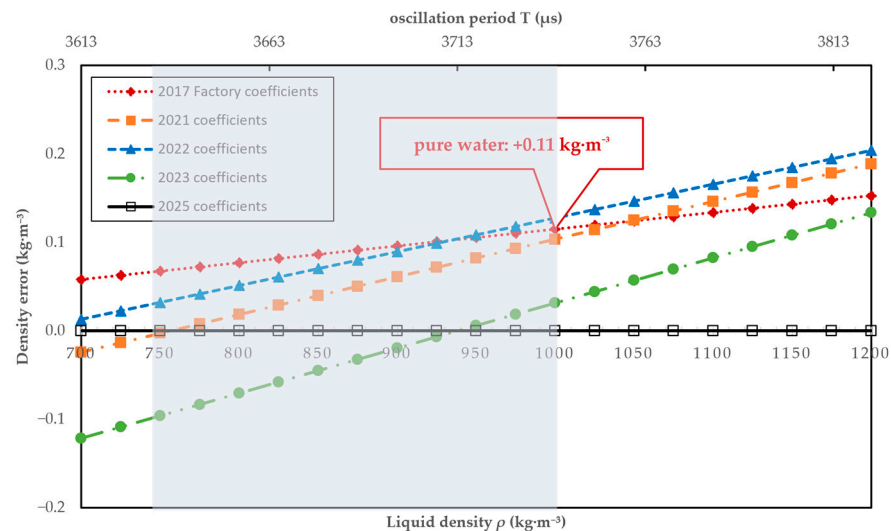


Figure 8. Density error produced by out-of-date calibration coefficients for a CDM100P process oscillation density meter. The shaded band marks the calibrated range; outside it the curves are an extrapolation of the fitted model.

The manufacturer of the 7835 type meter quotes a long term stability of 0.15 kg/m³ per year for that instrument, together with residual temperature and pressure effects after correction of 0.005 kg·m⁻³·K⁻¹ and 0.003 kg·m⁻³·bar⁻¹ [26]. The same manual notes that the air check it provides highlights long term drift, corrosion and deposition [26]. The drift measured here for the heavily corroded instrument is much larger. Between the 2021 and the 2023 calibration the older set produced an error of 0.52 kg/m³ at 700 kg/m³, and between the 2023 and the 2024 calibration the older set produced 1.25 kg/m³. Over the calibrated range the corresponding annual drift is 0.15 to 0.88 kg/m³ per year, up to six times the figure quoted by the manufacturer. The degradation therefore exceeds the stability on which the manufacturer specification rests, which is the quantitative reason why an interval derived from that specification alone is not adequate in corrosive service. In the other respects the coefficients obtained here agree with the manufacturer error model. The nameplate coefficients imply a pressure sensitivity of $\rho K_{20A} + K_{21A} = -0.0030$ kg·m⁻³·bar⁻¹, and the 2024 coefficients imply +0.0023 kg·m⁻³·bar⁻¹. Both agree with the corrected pressure effect of 0.003 kg·m⁻³·bar⁻¹ quoted by the manufacturer. The temperature sensitivity of the nameplate coefficients, 0.0130 kg·m⁻³·K⁻¹, is likewise consistent with the uncorrected temperature effect of 0.02 kg·m⁻³·K⁻¹.

4. Discussion

In these experiments the 7835 type meter had suffered very severe internal corrosion, the CDM100P meter only light corrosion, and the CDM100M meter an intermediate degree of degradation. For the severely corroded meter each recalibration reproduces the reference densities at the calibration points to within 0.1 kg/m³. A coefficient set three years old nevertheless produces an error of 0.74 kg/m³ at 700 kg/m³, and a set one year old produces 1.25 kg/m³. For the lightly corroded CDM100P meter the same error stays within 0.20 kg/m³, and for the intermediate CDM100M meter within 1.01 kg/m³, even for sets that are several years old. A likely reason is that corrosion of a heavily damaged tube is neither

uniform nor stable. It is localised and continues to change in service, so the shift it produces in the resonance period depends on the density of the liquid. A single set of coefficients determined from a few reference liquids cannot absorb such a shift. The annual recalibration interval recommended here is therefore a compromise. It is adequate for a lightly corroded meter such as the CDM100P instrument, but for a severely corroded meter it bounds the error over the plotted interval only marginally. A shorter interval, or replacement of the meter, should then be considered.

A comparison of the coefficients determined in the 2023 and 2024 campaigns, one year apart, shows how quickly the instrument degrades. Over that interval the density coefficient K_2 changed by about -13% (Figure 7). These trends are consistent with the progressive corrosion of the inner wall, which increases the effective vibrating mass of the resonator and shifts its natural frequency. For a meter in this condition an interval of six months to one year is therefore the longest that can be justified, whereas a longer interval may be acceptable in clean, non-corrosive service (Table 4). For future work we plan to extend this study in two directions. The first is the influence of liquid viscosity on the measured density, which matters for high viscosity products. The second is a drift prediction model based on operating history and process conditions, so that recalibration can be scheduled on condition rather than at a fixed interval.

The drift measured here cannot come from the reference side, because a change there would shift all three instruments in the same way, whereas it is ordered by the state of each tube. The reading chain is bounded in the same way: its contributions are all below 0.001 kg/m^3 in Table 1.

The industrial question that follows is how long the full coefficient calibration interval should be. An indication error calibration of the kind permitted by JIF 2164-2024 [20] answers it with two observables: the indication error measured at a few key points, which gives the present error, and the drift rate between two consecutive such calibrations. Table 4 sets out the preliminary guidance those two observables support. The instruments studied here are specified to between 0.1 and 0.2 kg/m^3 , and the manufacturer quotes 0.15 kg/m^3 per year as the long term stability of this meter type.

Table 4. Preliminary guidance for the full coefficient calibration interval based on the three instruments tested.

Service Severity	Indication Error at the Key Points (kg/m^3)	Drift rate Between Two Consecutive Indication Error Calibrations (kg/m^3 per year)	Suggested Full Coefficient Calibration Interval and Action
Clean, non corrosive duty	$\leq$ declared accuracy	$\leq$ manufacturer long term stability (0.15)	24–36 months
Lightly corrosive duty	$\leq$ double the declared accuracy	0.02–0.13	12 months
Intermediate duty	0.4–1.0	0.12–0.22	12 months; bring forward once the error exceeds 0.5
Severely corrosive duty	>0.5	0.15–0.88 (the higher figure over the most recent year)	6 months; replace the meter if a full coefficient calibration can no longer bring the residual at the reference liquids within the calibration uncertainty
Unknown or variable duty	not available	not available	12 months for the first two intervals, then set from the measured drift rate

Severity is defined by the two observables, not by the instrument model. The thresholds and the intervals come from three DN25 instruments: a straight tube (0.15 kg/m^3 ,

severely corroded) and two U-tubes (0.1 kg/m^3 and 0.2 kg/m^3 , lightly and intermediately corroded). They are preliminary guidance for instruments of these types.

5. Conclusions

This study applied a metrologically traceable full coefficient calibration to process oscillation type density meters returned from corrosive service, and used it to quantify, correct and bound the corrosion-induced drift that is the main threat to in-service accuracy. The analytical model of the straight tube resonator links the measured oscillation period to the liquid density. Adding temperature and pressure correction terms extends it across the whole operating range. All the coefficients are obtained from a single multi point least squares fit against traceable reference liquids. A complete calibration system, traceable to the NIM national primary density standard, was established to reproduce the pressure and temperature conditions of the process meter. For a heavily corroded 7835 type meter, the original factory coefficients were in error by 3.35 kg/m^3 at the density of water, far outside the declared best case accuracy of 0.15 kg/m^3 , whereas after recalibration the error fell well inside it. The results confirm the central conclusion: the accuracy of a process vibrating-tube densitometer in corrosive service is not maintained by the factory calibration, and periodic traceable full coefficient calibration is essential. For the conditions studied an annual full coefficient calibration restores the accuracy required for process monitoring, custody transfer and fiscal metering, while more aggressive fluids warrant a shorter interval.

The framework established here applies wherever density is used as a process variable. It resolves product interfaces in a multi product pipeline, converts volumetric into mass flow in a carbon capture and storage transport network [7], and controls concentration in a chemical or food process [4,5]. The present work has three limitations. The three meters studied span a wide range of degradation, but they belong to only two instrument types and were operated in a few industrial duties. The coefficients are identified over $750\text{--}1000 \text{ kg/m}^3$, whereas the meters are specified over a wider interval, and the influence of liquid viscosity is not accounted for. Future work will incorporate viscosity dependent correction and a drift prediction model that allows recalibration to be scheduled on condition rather than at a fixed interval. The results are of direct value to both audiences. For the academic reader they provide a quantified, traceable account of how in-service degradation of a resonant sensor propagates into its calibration coefficients, together with the uncertainty of that account. For the industrial reader they provide the evidence and criteria by which the recalibration interval of a process density meter can be set, documented and defended.

The three instruments studied here are 25 mm straight tube and U tube resonators, and the drift rates and thresholds above were measured on them. The mechanism is not specific to them: in any vibrating-tube resonator the tube wall contributes to the effective vibrating mass, so degradation shifts the period density relation in a way that no offset can absorb and only reidentification of the coefficient set can correct. The decision rule of Table 4 is general in the same sense, since it uses only the indication error and the drift rate that an in-service calibration delivers, whereas the intervals listed there are preliminary guidance from three instruments.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/app162110572/s1>, Table S1, the coefficient sets of the three instruments; Tables S2–S4, every measurement point of every campaign; Table S5, the inputs of the uncertainty budget; and Tables S6 and S7, the data behind Figures 7 and 8.

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