

## Design of an automatic apparatus for the calibration of on-line densimeters

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### **Abstract**

The design and the expected performance of a new apparatus suitable to satisfy the traceability requirements in the density field is here presented. The apparatus is mainly intended to measure liquid density range between 500 kg/m<sup>3</sup> and 3000 kg/m<sup>3</sup> and to evaluate the performance of on-line densimeters during the actual use, i.e. the same process liquid under similar flow conditions. Details about the hardware components of the system and the software programs are also given. By using an instrument not specifically selected and calibrated for the intended working conditions, the value of  $u_{instr}(\rho) = 0.20 \text{ kg/m}^3$  is the lowest expected uncertainty of density measurements of fluids. However, higher accuracy can be established for the selected reference instrument by the use of the same fluids employed in the process at the usual operative conditions and whose densities are certified by national metrological institutes (NMIs) or accredited laboratories.

*Keywords:* Calibration, Density, Densimeter.

### **Introduction**

Nowadays the control of the density is the basis of process measurement and control in the chemical and petrochemical industries, environmental technology and other process-oriented industries to increase efficiency, run a safer system, produce higher quality products. Several diversified instruments have long been accepted to measure the density during the process; their calibration, even if carried out in-situ, usually requires meters to be removed from the line to be tested against a reference standard. Often this testing must be conducted by a third party, in off-site location. Although remote calibration would seem to offer higher accuracy, this is not necessarily true because not all the working conditions are taken into account.

A new facility having the possibility to work automatically with several fluids, has been designed by Istituto Nazionale di Ricerca Metrologica (INRIM), Scandura & FEM, and Ente per le Nuove Tecnologie, l'Energia e l'Ambiente (ENEA). It is mainly in response to the need to satisfy the density traceability requirements and to evaluate the performance of on-line densimeters, possibly at

conditions which match their intended use, which is mainly in the range between  $500 \text{ kg/m}^3$  and  $3000 \text{ kg/m}^3$ . The procedure of calibration is based on the comparison method: a fluid flows through the instrument under test, while its density is measured by an appropriate reference instrument [1]. An on-line densimeter of the vibrating tube type, such as the ones commonly in use, has been chosen as the core component of the apparatus.

By the present paper we propose the designed apparatus as a secondary standard to be used in metrology laboratories or to be installed directly in the plant as “density meter prover” (DMP), after its metrological performances have been carefully evaluated to assess its total uncertainty.

At present the design of the apparatus has been completely outlined, as well as the control system and the software for data processing.

This article provides a brief descriptions of the design and presents the expected uncertainty components of the density measurements caused by the DMP only.

## Facility Description

The DMP is designed specifically to calibrate densimeters at their operating conditions in accordance with [2]; it can also be integrated in a plant for measuring the density of fluids. The system is a closed-loop test rig, which is set up in two independent sections or lines: the testing one (in red) and the cleaning one (in blue) (Figure 1).

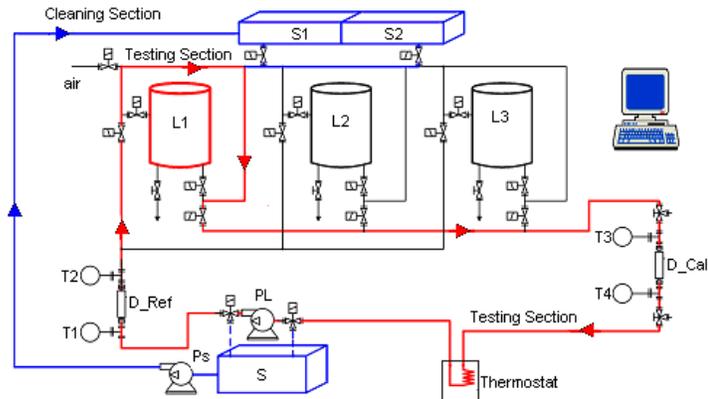


Figure 1. Schematic set-up of the automatic apparatus for the calibration of on-line densimeters.

Air and three testing liquids are used independently as transfer standards whose densities are measured by a reference densimeter of the vibrating tube type ( $D\_Ref$ ) installed on the testing section. In the same line the densimeter to be calibrated ( $D\_Cal$ ) is also inserted. The selected test liquid comes down through a system of three-way valves from one of three upper tanks (L1, L2 or L3) and flows, due to the action of a peristaltic pump (PL), through the test line at constant flow rate.

A chilled-water heat exchanger controlled by a feedback temperature sensor (thermostat) is used to remove from the fluid the heat added by the operation of the pump. The actual liquid temperature is measured by four platinum resistance thermometers (T1, T2 and T3, T4), closely installed upstream and downstream

the input and output ports of the reference densimeter and of the densimeter to be calibrated, respectively.

At the end of each measurement cycle the testing section has to be completely evacuated and dried. For this purpose a suitable solvent is used. The solvent, contained in the upper tank (S1), initially flows (by gravity) in the testing section for “de-greasing” the line. A clean solvent from another tank (S2) is then used for rinsing. This solvent, too, is collected into a lower tank (S), whence a pump (Ps) takes it up into the upper tank S1.

Compressed and dried air is used for drying the testing line each time that the cleaning procedure is over. The state of cleaning is monitored by the density value obtained from the reference densimeter .

## Control System

Although the operator can manually select one of the operating modes, the DMP is usually intended to be operated automatically by means of a personal computer (Pentium 4 or equivalent processor) by running a suitable *LabVIEW*<sup>®</sup> program. The program is designed to give total control of DMP operations, acquire and process the signals from the various measurement devices, as well as to create a database. For this purpose the computer is interfaced to the purposely-built electronic control unit through various communication cards: the 8 RS-232 serial cards (EXSYS EX-41098), the I/O digital one (NI PCI-6518) and the IEEE 488 (NI-488.2) one. On the same control unit are placed the measurement instruments that display the measurement values concerning the period or the frequency of the reference instrument and of the densimeter to be calibrated, as well as fluid temperatures.

Figure 2 shows the scheme of the DMP control system and the related instruments.

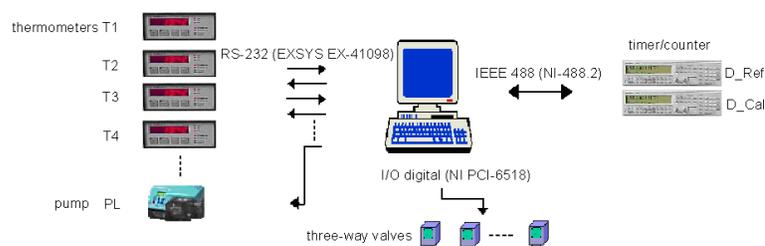


Figure 2. Scheme of the DMP control system and instruments.

## Control Panel

The measurement may be started by running the *LabVIEW*<sup>®</sup> program whose front panel is shown in Figure 3.

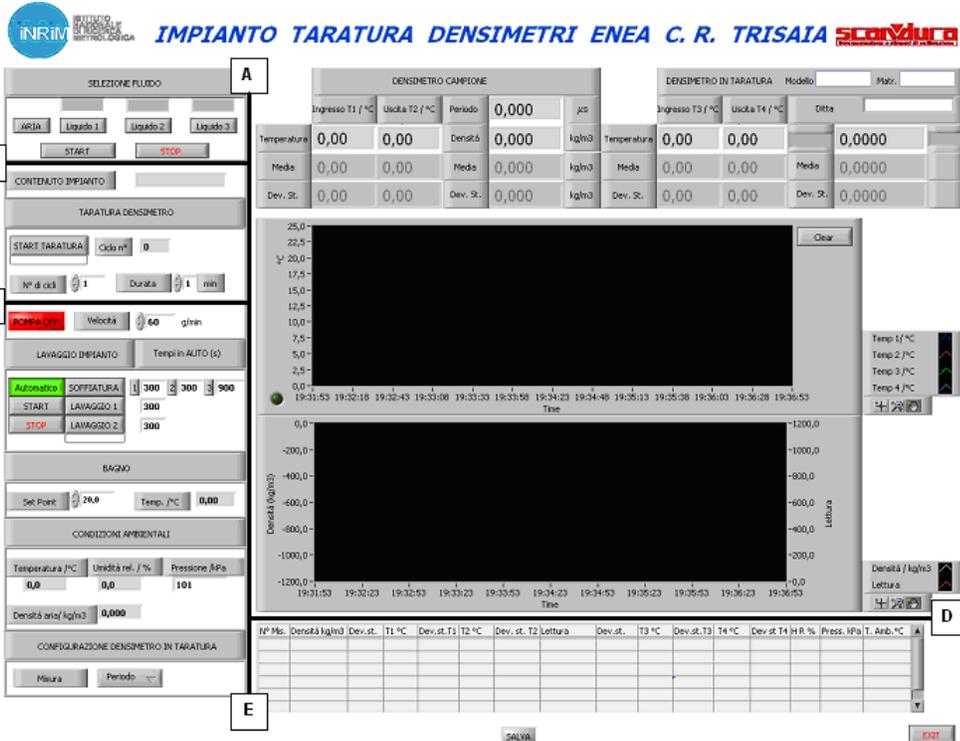


Figure 3. Front panel of the *LabVIEW*<sup>®</sup> program used for DMP setting (AB, BC and CD), for controlling (AD) and for data acquisition (DE).

The front panel is built with controls and indicators which are the interactive input and output terminals of the “virtual instrument” VI, respectively.

The following operations are performed by the front panel:

- Selection of one of the four different fluids, including air, for calibrating (section AB-Fig.3);
- Setting of the number of test repetitions and their duration period (section BC-Fig.3);
- Displaying of the actual reference density value, the period, the temperatures, the instruments readings of the densimeter to be calibrated, and the averages of these quantities with the related standard deviations for both densimeters. In addition four plots of temperature are available to evaluate the temperature stability of the testing fluid and also the plots of the reference density and instruments readings are displayed (section AE-Fig.3);
- Acquisition and storing of the averages and the standard deviations of all quantities measured from the various measurement devices at each test repetition (section DE-Fig.3);
- Performing of the cleaning cycle of the testing line (section CE-Fig.3).

## Data Processing

All data acquired from the various measurement devices are recorded on a spreadsheet by the front panel. The operator is able to open any file by an Excel model created to perform quick and accurate computations to obtain the

coefficients (parameters) of the calibration curve. The following generally accepted mathematical model is used to correlate density to possible different types of electrical output of the densimeter under test:

$$\rho(T, p) = K_0 + K_1 x + K_2 x^2 \quad (1)$$

where:

$\rho(T, p)$  is the density value of the reference fluid measured by the reference densimeter at the temperature  $T$  and pressure  $p$  while it flows in the apparatus,

$K_0$ ,  $K_1$  and  $K_2$  are the instrumental constants of the densimeter under test and  $x$  its output signal: typically they are period  $\tau$ , voltage  $V$  or current  $I$ .

The estimation of the three parameters is based on the application of the weighted least square method (WLS) [3]. The use of  $n$  reference fluids and  $m$  test repetitions allows to have a set of  $n \times m$  equations in three variables ( $K_0$ ,  $K_1$  and  $K_2$ ) for which the best solution of the estimated parameters  $\hat{\beta}$  is

$$\hat{\beta} = (\mathbf{x}^T \psi_p^{-1} \mathbf{x})^{-1} \mathbf{x}^T \psi_p^{-1} \boldsymbol{\rho} \quad (2)$$

with covariance matrix

$$\psi_{\hat{\beta}} = (\mathbf{x}^T \psi_p^{-1} \mathbf{x})^{-1} \quad (3)$$

The terms in the equations (2) and (3) are

$$\hat{\beta} = \begin{bmatrix} K_0 \\ K_1 \\ K_2 \end{bmatrix}, \quad \mathbf{x} = \begin{bmatrix} 1 & x_{11} & x_{11}^2 \\ \vdots & \vdots & \vdots \\ 1 & x_{1m} & x_{1m}^2 \\ \vdots & \vdots & \vdots \\ 1 & x_{n1} & x_{n1}^2 \\ \vdots & \vdots & \vdots \\ 1 & x_{nm} & x_{nm}^2 \end{bmatrix}; \quad \boldsymbol{\rho} = \begin{bmatrix} \rho_1 \\ \vdots \\ \rho_1 \\ \vdots \\ \rho_n \\ \vdots \\ \rho_n \end{bmatrix} \quad (4)$$

and  $\psi_p$  is the covariance matrix of the fluid density that, assuming that all density values are obtained independently, is a diagonal matrix. The diagonal element of such matrix are the reciprocal of  $s_{\rho_i}^2$ , which yield both the uncertainty  $u_{\rho_i}$  of each dependent quantity  $\rho_i$ , and also the contributions of the uncertainty of the independent variable  $x_i$

$$s_{\rho_i}^2 = u_{\rho_i}^2 + \left( \frac{\partial \rho}{\partial x_i} s_{x_i} \right)^2 \quad (5)$$

The term  $u_{\rho_i}$  usually gives a constant contribution to uncertainty of calibration so that the choice of the reference densimeter plays an important role in foretelling its amount.

## The Expected Uncertainty

To maximize the performance of the designed apparatus the most important influence factors should be taken into account in order to minimize the systematic contribution of uncertainty due to the density measurement of the reference fluid.

Density measurement uncertainty is caused by a number of factors, mainly due to physical properties of the fluid, to the reference densimeter and in general to the working conditions.

The fundamental differences between the measurement of gases and liquids are that gases require lower limits of uncertainty due to the lower gas density. In addition, the pressure is more important for gases. Whereas the measurements of liquid densities are mainly performed at constant pressure, mostly just above the atmospheric one, for gas density measurements the variations of pressure can hardly be avoided. Due to these conditions neither a theoretical model for the vibrating tube densimeter equation nor the influences of possible disturbing factors are known [4].

However, our present task is to evaluate the uncertainty in liquid density measurements. Let us consider a typical case where the DMP is mainly designed to work with three different liquids in the range from 500 kg/m<sup>3</sup> to 2000 kg/m<sup>3</sup> at ambient pressure and at reference temperature close to 20 °C with an uncertainty of 0.02 °C.

The density  $\rho$  of many liquids shows (in a narrow zone) a linear dependence on temperature  $T$ , such that

$$\rho(T_{20}) = \rho_T (1 + \alpha(T - T_{20})) \quad (6)$$

In agreement with the ISO Guide [5] the liquid density uncertainty  $u(\rho)$  at the reference temperature can reasonably be given as:

$$u(\rho) = \sqrt{u_{instr}^2(\rho) + \rho_T \alpha u_T^2 + \rho_T (T - T_{20}) u_\alpha^2} \quad (7)$$

where:

$\alpha$  is the density-temperature coefficient,

$u_{instr}(\rho)$  is the component of the liquid density uncertainty measured by the reference densimeter,

$u_T$  is the uncertainty of the temperature measurements, and

$u_\alpha$  is the uncertainty of the density-temperature coefficient of the liquid.

From equation (7), only some factors concerning the reference densimeter, such as its initial calibration uncertainty, temporal stability of calibration, reproducibility and influence of temperature and pressure, can be firstly taken into account to evaluate the expected uncertainty of the liquid density.

For this purpose, a commercially-available densimeter (Solartron 7835) has been used as the reference density standard for the apparatus. This instrument is well established as the more popular industry standard for high accuracy on-line density measurement applications for fiscal metering of crude and refined hydrocarbons or non-aggressive process liquids. The vibrating element is manufactured from Ni-Span-C and all other wetted parts are made in AISI 316L stainless steel. Moreover the instrument is equipped of direct analogue and digital communication outputs suitable to transfer continuous, accurate, instantaneous frequency signal to timer/counter.

Table 1 lists the typical technical specifications for the Solartron 7835 based on factory test procedure. The factory procedure uses three specified fluids: air whose density is derived from BIPM equation [7], a hydrocarbon oil of about  $830 \text{ kg/m}^3$  and a high density fluid of about  $1500 \text{ kg/m}^3$ . Measurements are also made under conditions of changing temperature and pressure to establish their effect on the instrument.

In agreement with the technical specifications shown in the Table 1 and with the working conditions for the DMP stated above, the different sources and magnitudes affecting the uncertainty of the fluid density measured by the reference densimeter are listed in Table 2.

The value of  $u_{instr}(\rho) = 0.20 \text{ kg/m}^3$  is the lowest expected uncertainty of the density measurements of the reference fluid in the DMP using an instrument not specifically calibrated for the intended working conditions. Higher accuracy can be obtained for the selected reference instrument by calibrating it through the use of the same fluids employed in the process at the usual operative conditions, if their densities are certified by national metrological institutes (NMIs) or accredited laboratories.

## Conclusion

The design of a new apparatus suitable to satisfy the traceability requirements in the density measurements, to be used as secondary standard in metrology laboratories and possibly to be directly installed in the plant has been presented. As regards the performances of the designed apparatus, the main uncertainty component is by far the one contributed by the reference densimeter. However, better performances can be obtained by calibrating the reference densimeter through the use of the same fluids employed in the process at the usual operative conditions, if their density has been measured in a way traceable to SI units by national metrological institutes (NMIs) or accredited laboratories.

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