

DENSITY CALIBRATIONS AT MIKES

Heikki Kajastie, Jorma Manninen and Kari Riski
Centre for Metrology and Accreditation (MIKES)

ABSTRACT

The density measurement instruments at MIKES will be described. Also a construction of a instrument for weights 1 g – 200 g will be presented. The main test results will be given.

1. INTRODUCTION

In weight calibrations the air buoyancy correction is one of the main sources of uncertainty. In order to determine this correction the density of the weight must be known with required accuracy. The required accuracy depends on the relative uncertainty of the mass calibration. In practise the density of small weights ($m < 5$ g) can be estimated from the specifications or composition of the weight material. For OIML weights general requirements for density determination are given in OIML R111 [1]. Also methods for density determination have been described extensively in Annex B (Test procedures for weights) of the Ref. 1.

At MIKES there are several instruments for the determination of density of solid objects. For weights with masses between 1 g and 2 kg the hydrostatic method in which the weight is immersed in water and the mass of the weight in water is compared with reference weights in air.

For weights with masses between 5 kg and 50 kg the pycnometric method is used. Two pycnometers one for 5 kg and 10 kg weights and one for 20 kg and 50 kg weights are in use.

At MIKES a range of density calibrations (Table 1.) with hydrostatic and pycnometric methods is 1 g – 50 kg. Traceability of density measurements at MIKES is presented in Table 2.

| Density calibrations | | |
|----------------------|----------------------------|--------------------------------|
| Mass | Density | Uncertainty (k=2) |
| 1 g – 2 kg | 0,1 – 255 cm ³ | 0,0003 – 0,008 cm ³ |
| 2 kg – 50 kg | 250 – 6500 cm ³ | 0,3 – 2 cm ³ |

Table 1. A range of density calibrations with uncertainties.

The density of water is calculated from water density tables [3]. Distilled water is used. The conductivity of water is typically 1 μ S/cm.

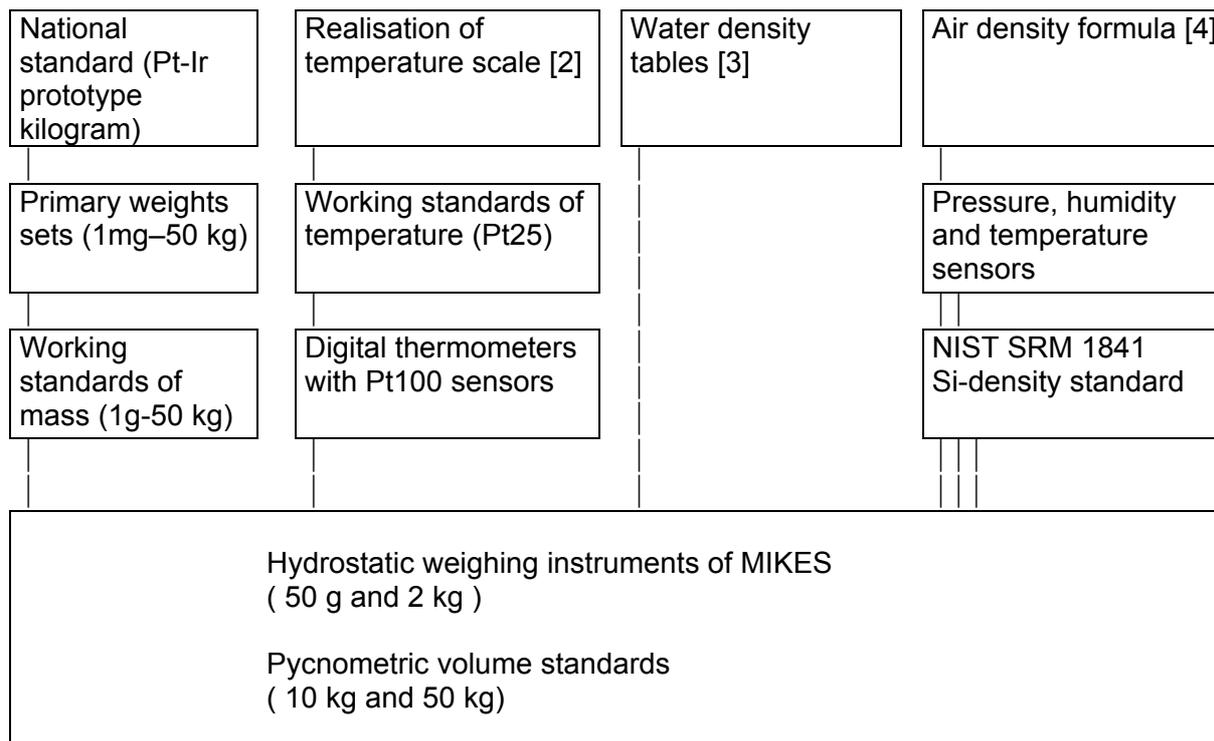


Table 2. Traceability of density measurements at MIKES.

2. THE PYCNOMETRIC METHOD

The pycnometric method is for the determination of density of weights 5 kg – 50 kg. In the Ref. 1 testing routine for the determination of density of weights are described. In the Ref. 1 the method is called method D.

In the method three weighing with a container of stable volume are performed; weighing when empty, result mass of the container, weighing when filled with distilled water, result volume of the container and weighing when the weight is inside the container and it is filled with distilled water, results volume of water and the volume of the weight.

MIKES has two pycnometers. The small container (about 2,8 litres) is a glass bottle with two main parts (Fig. 1). The parts have flat ground surfaces for sealing. At the top there is a cone seal for the filling tube and for the adjustment tube. The adjustment tube has an engraved ring up to which the water is filled. A small amount of silicon grease is used for sealing.

The large container (about 8,6 litres) consists of a glass bell and a glass plate. The bell has a flat ground lower surface for sealing. At the top there is a cone seal for the filling tube and for the adjustment tube. The adjustment tube has an engraved ring up to which the water is filled. A small amount of silicon grease is used for sealing.

Special attention has to be paid to reach thermal equilibrium, to remove air bubbles and to adjust the water level. Also the change of water level during the measurement has to be monitored. In the measurement a 50 kg mass comparator ($d=10$ mg) with a weight handler is utilised.



Fig. 1 Pycnometer for 20 kg and 50 kg weights.

To determine the volume of the container, the water filled container is weighed against reference weights. The density of air and water are determined from measured water temperature, air temperature, air pressure and humidity. Standard formulas for their densities are applied.

3. HYDROSTATIC WEIGHING

For weights with masses between 1 g and 2 kg the standard hydrostatic comparison method (method A) in which the weight is immersed in water and the mass of the weight in water is compared with reference weights in air. Two different instruments are in use and a third, new instrument is under testing (see the next chapter). Instruments have weight handlers which operate in water.

An instrument "1" is for weights 10 g – 2 kg and an instrument "2" is for weights 100 mg – 50 g. The new apparatus is for weights 1 g – 200 g and small spheres.

The instrument "1" consists of a glass vessel and a manual weight handler (Fig. 2) with two weight position. The volume at the vessel is about 20 litres. The balance is above the vessel. The weighing pan is inside the water and it is connected to the balance with the wire. The thin platinum (dia 0,4 mm) wire or the tantalum (dia 0,25 mm) wire minimise the effect of surface tension. Balances Mettler AT1004 or Sartorius LC 5100 MC is used.



Fig. 2 Hydrostatic weighing of 10 g – 2 kg weight.

The instrument “2” (Fig. 3) has a small glass vessel. The volume of the vessel is about 0,4 litres. The glass vessel is placed inside of the weight chamber of Sartorius MC210S. Also here a wire is used to transmit a force through the surface of water. The system has a small weight handler whose upper surface is moving. The up-down movement is controlled manually.

In hydrostatic weighings special attention has be paid to reduce temperature gradients, to remove air bubbles and to minimise the effect of surface tension.



Fig. 3 Density measurements for small weights.

4. NEW APPARATUS FOR HYDROSTATIC WEIGHING

New equipment (Fig. 4) for hydrostatic weighing for weight 1 g – 200 g has been developed at MIKES. Test measurements has been done and results are given below.

The weighing pan and the lifting knob of the weight handler has been shaped for weights 1 g – 200 g. The shaping also enables lifting of spheres with a maximum diameter of about 35 mm.

The handler is used by a step motor which is driven by a programmable controller. A bush of a button activates the up-down motion of the weighing pan.

Main parts of the mechanical construction of the vessel are a bottom part of stainless steel and a cylindrical part which is made of glass. The heavy bottom part enables a smooth operation of the weight handler. The glass part is attached to the bottom part by silicone glue. At the bottom of the stainless steel vessel there is an outlet for water.

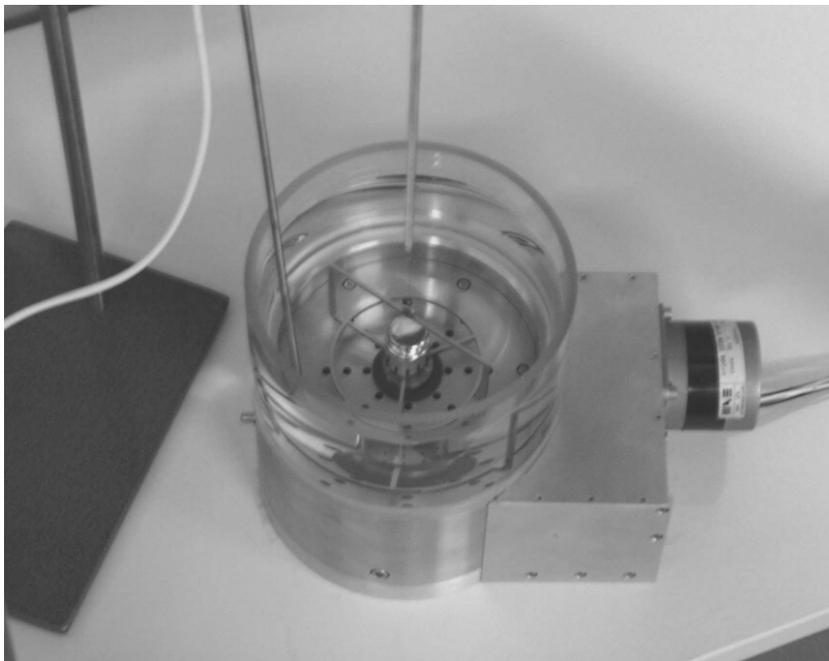


Fig. 4 The new apparatus for weights 1 g – 200 g.

The lifting knob is used by an eccentric wheel. The shaft of the wheel is rotated by the step motor. The shaft goes through the stainless steel vessel and it is sealed by four O-rings.

Between the shaft and the step motor is a $\frac{1}{4}$ -gear made by two plastic tooth wheels.

5. TEST RESULTS

The density measurement instruments have been tested in several ways. Density comparisons with different instruments have been made. Test measurements with a silicon crystal of known density have also been made. There are also interlaboratory comparison results. [5]

The pycnometric method has been compared with dimensional measurements of a 10 kg weight. The profile of the 10 kg weight (class E₂) was measured with a coordinate measuring machine (CMM). From this profile the volume was calculated. The results agreed within the uncertainties calculated.

The new density measurement instrument has been tested with a Si –cylindrical, a Si-sphere and OIML-shape weights. Test results are shown in Table 3.

| Date | Test weight | Density (kg/m ³) | Uncertainty (k=2) (kg/m ³) | Previous meas. (kg/m ³) | Uncertainty (k=2) (kg/m ³) |
|----------|------------------|------------------------------|----------------------------------------|-------------------------------------|----------------------------------------|
| 29-05-02 | S3 (cylindrical) | 2329,27 | 0,08 | 2329,31 | 0,10 |
| 29-05-02 | SiB (sphere) | 2329,38 | 0,10 | 2329,39 | 0,10 |
| 30-05-02 | P107 5 g | 7955,8 | 6,2 | 7955,5 | 3,8 |
| 31-05-02 | P22 100 g | 7958,33 | 0,30 | 7958,61 | 0,30 |

Table 3. Measurements results.

SUMMARY

The uncertainty of density determination is sufficient for the calibration of OIML class E₁ weights. The relative uncertainty of density determination for the standard hydrostatic method is at its best 10⁻⁵. For pycnometric method it is about 10⁻⁴.

REFERENCES

- [1] OIML R111: "Weights of classes E₁, E₂, F₁, F₂, M₁, M₂, M₃", OIML, 1994.
- [2] H. Preston-Thomas, "The International Temperature Scale of 1990 (ITS-90)", Metrologia, 1990, 27, 3-10
- [3] Tanaka M., Girard G., Davis R., Peuto A., Bignell N., "Recommended table for the density of water between 0 °C and 40 °C based on recent experimental reports", Metrologia, 2001, 38, 301-309.
- [4] Davis R.S., "Equation for the Determination of the Density of Moist Air" Metrologia, 1992, 29, 67-70.
- [5] K. Riski, "Mass and volume comparisons at MIKES", Julkaisu J04/2000, MIKES

Heikki Kajastie, Jorma Manninen and Kari Riski
 Centre for Metrology and Accreditation (MIKES), P.O. Box 239, FIN-00181 Helsinki, Finland
 Telephone: +358-9-616761
 E-mail: heikki.kajastie@mikes.fi