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# Automatic testing facility for determining liquids and solids density; and determining the volume of E1 weights

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**Abstract**

Fundamental aperture for density determination:

In cooperation with Sartorius AG in Goettingen and the Vienna University of Technology, the Austrian Federal Office of Metrology and Surveying (BEV) has developed a test System for hydrostatic weighing and determining the density of liquids and solids, whereby mass comparators are automatically loaded alternately with weights and submerged plummets. With the load alternator for weights and plummets, the density of a liquid on one side and the density of a submerged solid (and therefore its volume) on the other side can be determined hydrostatically via direct comparison with Standard plummets and the applied substitution weights.

Volume comparator:

To determine consecutively the volume of E1 weights and plummets, additional a fully automatic handling system for a 1-kg mass comparators, a volume comparator was developed. Thus, the hydrostatic weighing principle can be applied for serial volume determination on masses from 1 g to 1 kg by direct comparison with a single volume reference (e.g., a silicon sphere). This prinziple is implemented with a newly developed, fully automatic insertion mechanism for both completely submersed weights and substitution weights.

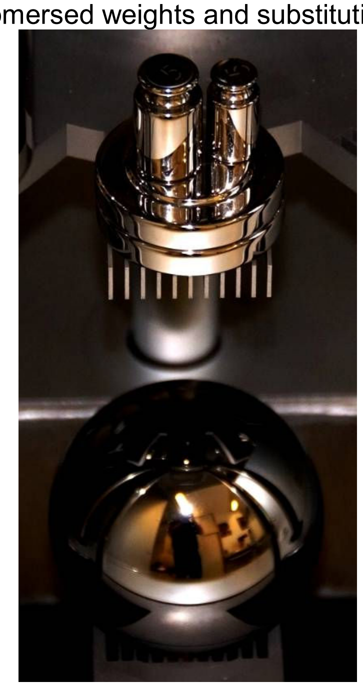

Fig. 1: The new volume-comparator: a Silicium volume standard and the corresponding substitutions weights

Both Systems are used for the dissemination of density and volume by BEV as well as for testing and calibrating of liquids and plummets and determining the volume of weights in connection with the determination of mass scale.

The volume comparator is also used in other metrology institutes and in accredited calibration laboratories.

Fig. 2: The new volume-comparator (left) based on the new fundamental density aperture for solids (right) in the BEV; both are hydrostatic-below-weighing systems from 1g up to 1kg;
Left: in the volume comparator the magazine changer, the suspension system and the magazines for weights, the standard sinker and the test weights can be seen
Right: on the test tower for measuring the density of solids
the cylinder for lifting weights, standard sinker holder and test object holder can be seen here.

## Introduction

In metrological concerns high-precision determination of the density of liquids and solids and therefore their volume is often necessary for further measurements, calculations or for information about material characteristics.

For example, the determination of mass using conventional testing facilities is generally carried out in air under normal conditions. The mass of air, however, causes a systematic error in the procedure. Every solid body is affected by buoyancy in relation to the amount of air it displaces (Archimedes' Law). Air buoyancy makes a 1 kg stainless steel weight appear to be approx. 0.15 g lighter than it is. If the test piece has the same volume as its mass reference, the air buoyancy can be discounted, because they will have the same buoyancy. But if the test piece has a different density the air buoyancy have to be corrected in the calculation for the mass.

## Determination of the Volume of Weights is Necessary for the Correction of Air Buoyancy, and thus is an Essential Capability for Today's Metrology Institutes.

This is why determination of density is indispensable for precise determination of mass, which is essential for metrology institutes, accredited calibration services and similar laboratories. This is reflected in international regulations, such as OIML Recommendation R111 for a 1 kg E1 weight, which allows a mass tolerance of only 0.5 mg. Such precision cannot be attained in the determination of mass without determining the density of the weights and making the necessary correction for buoyancy.

## Initial Situation

Up until 2004, BEV had only a simple test assembly for hydrostatic weighing, in which the density of submerged test objects was calculated in individual weighings in an open thermostat. The temperature measurement, thermal stratification, stability of the mass comparator and the required volume of liquid were all considered insufficient for the standards required in this area. In order to meet the appropriate standards for a national metrology institute, the complete reconfiguration of the test assembly and new construction of a test system were essential.

## Specifications and Objectives

The main objective was to find an option for hydrostatically determining the density of less than one litre of liquid. For this measurement and for the hydrostatic determination of the density of solids, the primary aim was direct comparison of the test object with standard plummets or weights. In order to achieve the greatest possible reproducibility and minimize measurement uncertainty, it was specified that one or more load alternators were to be constructed for weight measurement below the mass comparator.

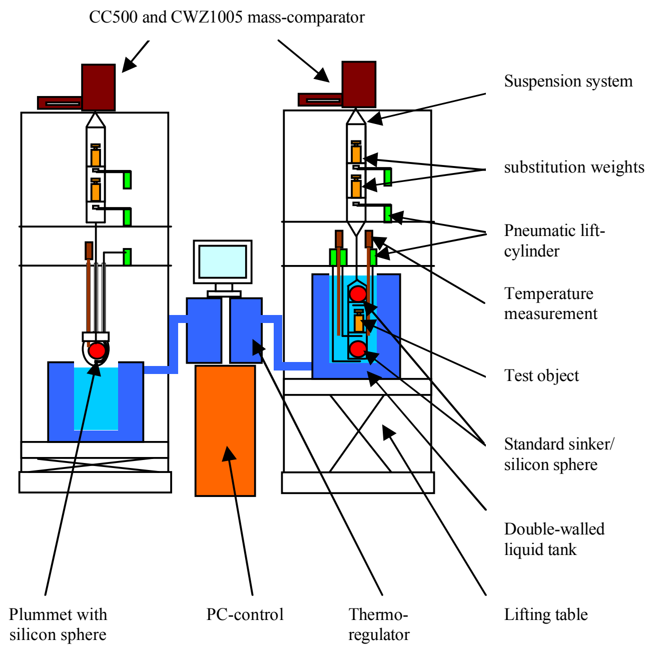


Fig. 2: Left: test station for measuring the density of liquids; right: test station for measuring the density of solids

The entire measurement process up to and including evaluation of the measurement results was to be fully automatized. Particular consideration was given to the use of different mass comparators and to thermal stratification and other influences, such as surface tension, magnetic fields, etc.

Building on years of excellent cooperation, the BEV and the Institute of Production Engineering (IFT) of the Vienna University of Technology, together with Sartorius AG in Goettingen, have developed and built a fully automatic fundamental aperture for the determination of density.

**Concept of the Fundamental Aperture**

The system consists of an insulated, thermally controllable, double-walled liquid tank that can be raised using a lifting table. In the lower position, the test station can be equipped easily with weights, standard plummets and test objects. By raising the liquid tank, the test objects and weights suspended from a mass comparator can be immersed in the liquid and thermostatized. During actual measurement, the individual objects and the applied substitution weights can be raised and lowered using lifting rods that are moved pneumatically. This places and replaces the mass on the suspension system of the mass comparator. System control and data documentation are carried out via a PC.

**Functionality and Procedure of the Fundamental Aperture**

The entire measurement system consists of two separate units (towers), whereby each tower can be considered an independent test assembly. The first tower is used to determine the density of liquids; the second, the density of solids. This second tower can also be used universally for processes such as determining the density of relatively large volumes of liquid or the calibration of hydrometers or immersed objects.

Fig, 4: Test station for measuring the density of solids in CAD

Each tower, consisting of an aluminium frame with movable shelves, has a mass comparator on top. The mass comparator specifications correspond to the range of the test objects to be measured. In addition, each tower has a lifting table with a thermostat reservoir and the appropriate number of pneumatically operated lifting units for the specific application. In order to keep heat-related currents as low as possible, the entire control system, power electronics and thermostat are located outside the enclosures. The measuring tower interiors are also provided with several partition plates in order to minimize convection currents in higher temperature measurement ranges.

Once the test objects and weights and plummet(s) have been positioned in one of the two towers, measurement can begin after thermostatization. Density can be determined in a fully automatic process by running the user-defined program steps of the evaluation software, and is based on the weight values and other relevant parameters that are measured by positioning the test objects and weights at different heights. The result of one measurement series is the calculated density or volume along with an extensive report that documents the individual measurements.

The measurement units used to determine the weight values are based on a modified Sartorius CC500 mass comparator for the liquid density and a modified Sartorius CWZ1005 mass comparators for the solid density. Both were adapted for this specific purpose by Sartorius, whereby the mass comparator for density determination of solids was subject to particular adaptation so that a weight value of up to 1 kg can be measured with a resolution of 10 µg. The liquid temperature is determined via two resistance bridges, each with two 25-ohm platinum resistance sensors set at 0.1 mK. The weights are positioned directly (without pallet) on the suspension system or the lift cylinder rack.

Fig. 5: determination of liquid density: measurement vessel with a bell-shaped chamber holding the silicon sphere before the thermostatized bath is raised

A barred, bell-shaped measuring vessel with a standard- silicon sphere inside a suspension system is the main component used to determine the density of liquids. It has a capacity of 650 ml and, for insulation reasons, is connected to the outer environment only by glass tubes. These tubes are used for temperature measurement, suspending and manipulating the silicon sphere inside and filling/emptying the bell chamber. Individual screw fasteners for the glass tubes have been specially developed for this purpose. In addition, a method had to be found that would ensure the reproducible build-up of the liquid meniscus around the suspension wire of the mass comparator. The temperature range in which density can be determined reaches from 5°C to 60°C. Particular consideration was given to the problems of thermal stratification and condensation.

Fig. 6: Weight holder: lifting cylinder with mass comparator suspension system

**Test Process of the Fundamental Aperture**

The standard weights (the applied substitution weights), standard plummets and test objects are manually positioned in the appropriate areas. To measure the density of a liquid, the plummet with the bell-shaped chamber is closed and filled with the test liquid. The corresponding thermostatized bath is raised, and the system is thermostatized. All necessary basic data and the preselected test routine are feed into the computer in preparation for measurement.

Fig. 7: Weight holder: a couple of substitutions weights is lifted from the mass comparator suspension system

If the system is balanced sufficiently, measurement may begin. Only those measurement series that are determined under optimum conditions are used for final evaluation; here, the standard deviations obtained for the weight values and temperature measurement are compared with predefined tolerance levels. All individual measurements are recorded along with the results for each measurement series, and test/calibration certificates are recorded and printed for the test object measured.

**Measurements Results with the Fundamental Aperture**

Measurement series with already calibrated BEV test weights and plummets have been carried out at different temperatures to validate the system since summer 2005. Excellent results have been achieved in terms of reproducibility and standard deviation in international comparisons. For final validation, international comparative measurements have been taking place on a bilateral basis with the national metrology institutes of Hungary and Germany (MKEH (former OMH) and PTB). The system is already being used for disseminating density and volume at BEV for liquids and solids up to 1 kg, it is used continuously in internal and external tests and calibrations and has been implemented in the BEV quality system.

Fig. 8: System installed at BEV for the fundamental density determination

**Development of a Volume Comparator**

High-precision measurement of the density of solids through comparative measurements with density standards, such as those carried out by the BEV with the fundamental aperture or with similar procedures by national metrology institutes are generally too time-consuming for a large numbers of samples, due to the complicated preparation and installation in measurement cells.

Beginning with September 2005, the information and knowledge collected from the project of the fundamental aperture have been used to formulate the basis of a similar density application concept for serial determination of the volume of weights with a mass ranging from 1 g up to 1 kg.

Once again it was possible to engage the IFT and the Sartorius AG for a competent partnership for the implementation of this project.


Fig. 9: Volume comparator VD1005: prototype at BEV

### Requirements and Objectives

In this application, a load alternator is to be provided to enable to determine the volume of test objects by direct comparison with standard weights and a plummet.

The aim was to enable hydrostatic density determination in accordance with OIML R111 (class E1 for multiple solid bodies or weights). The procedure is based on mass comparison, as opposed to weighing, with specific steps taken to ensure that the temperature in the measurement chamber is as stable as possible. The result is a testing facility that analyzes the density of up to eight weights ranging from 1 g to 1 kg. With this approach, uncertainty of measurement is not related to mechanical factors, and thus depends on ambient conditions and the quality of the measuring instruments.

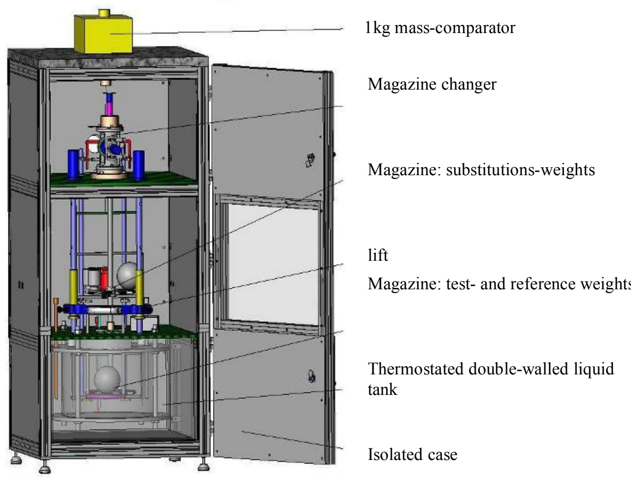


Fig. 10: CAD model of the volume comparator

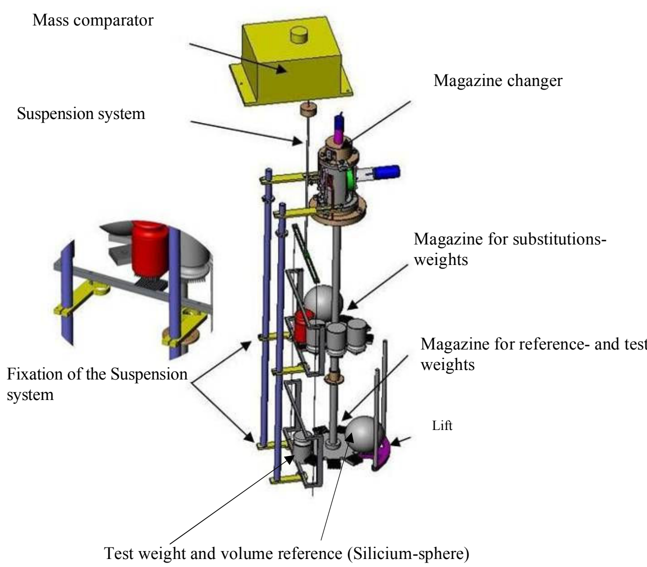


Fig. 11: CAD model of the transport unit

### System Design

The testing facility is constructed in the form of a tower. A double-walled storage container for liquids is installed at the lowest level. The outer layer contains normal water and serves to regulate the temperature; it is supplied and regulated by an external thermostat. Baffle plates provide for a homogenous flow of water around the internal container at all times. A stable temperature is ensured by means of a 40-mm layer of insulation around the entire system and the use of insulated metal inserters. Additionally, the insulation of the individual levels of the testing facility prevents convection currents around the mass comparator. The inner vessel – the measurement cell – has been kept as small as possible in order to use as little as possible of the highly purified measuring liquid and, with the exception of a small entry portal, it is completely surrounded by the liquid in the outer layer. Even regulation of the temperature on all sides makes it possible to reduce the formation of temperature layers to a negligible level. The temperature of the measuring liquid is monitored by two diagonally mounted high-precision sensors (25 ohm standard platinum-resistant thermometers (SPRT) in compliance with ITS 90).

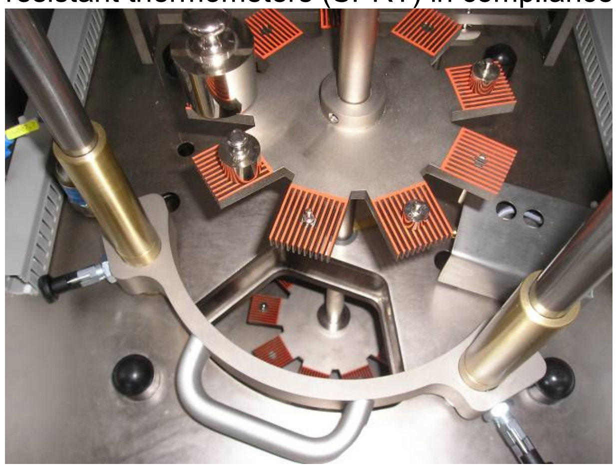

Fig. 12: Magazine for substitution weights, insertion mechanism, and view from above to the magazine in the weighing chamber

The used mass comparator is the well-tried modified Sartorius CWZ1005 with a possible dead load of up to 400g. The maximum load is 1 kg with a resolution of 10 µg.

For both, the substitution weights and the test weights a position below the mass comparator was chosen. This eliminates off-center loading problems on the weigh cell. The specially developed load alternator makes it possible to insert the 1 g to 1 kg weights and density references spheres with no rearrangement required, and to position them precisely in the suspension device of the mass comparator. The insertion mechanism positions the weights on the magazine spaces in the measuring fluid vessel.

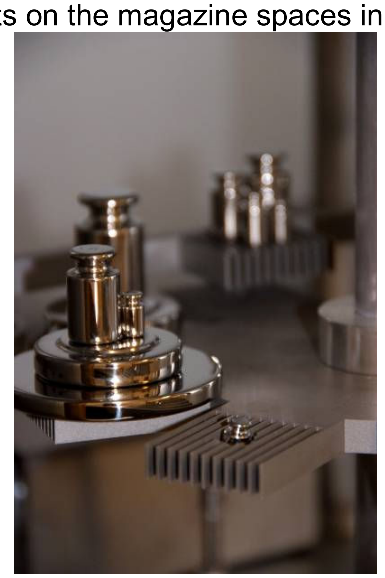

*Fig. 13: weights from 1 g to 1 kg and also spheres can be carried directly*

The entire control system as well as all electronic components are housed in a control cabinet. The system is controlled by a computer, which is also used to evaluate the data. In order to determine environmental parameters the unit has sensors for air pressure, humidity and air temperature. A number of additional sensors are provided in order to exclude errors in the measuring process.

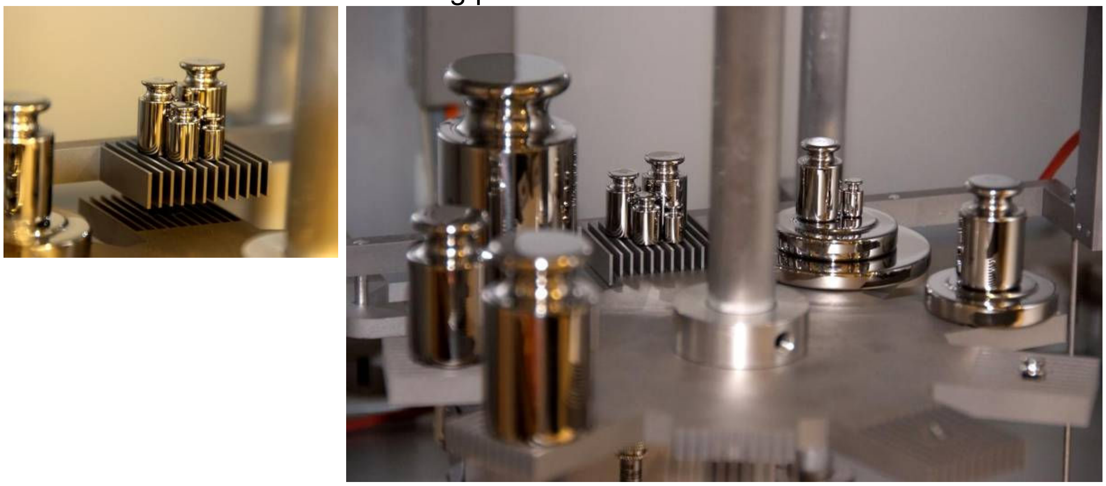

Fig. 13: Magazine for substitution weights: a couple of substitutions weights is lifted on the mass comparator suspension system

**Measurement Procedure**

Unlike other systems, this unit uses a 1 kg mass comparator to compare each test weight directly with only one volume reference. Each measurement run is used to compare the weight value of the test weight with the weight value of the Standard weight. This means, however, that either the test weights must have a reference of their own with a similar mass, or the measured weight of each object must be corrected through reference to a different mass by using substitution weights in air. This unit realizes both methods described above. Thus it is possible to adapt test weights with different masses directly to a volume reference (e.g., a silicon sphere). Although the density of the measurement fluid as a transmitter is constantly monitored, it is of secondary importance due to this mass dissemination capability. It is important, however, to make sure that the density remains constant during the course of a measurement (approximately 2 minutes).

Fig. 14: a substitutions weights is lifted on the mass comparator suspension system; the positions system for the suspension system is opened.

The software's user interface prompts the operator to prepare the measurement inserting the reference weights, the test weights and the appropriate substitution weights whereas a database can be used. This entails use of the weight insertion mechanism; it is important at this point to make sure there are no air bubbles on the weights once they are immersed in the liquid. A total of nine spaces are available for the reference and test weights. Once the sequence and number of cycles have been entered, the measurement can begin.


Fig. 15: Windows surface of the software

**Software**

The software assists the operator in the preparation and execution of the measurements. In addition to the fully automatic measurement program, operators have the option of performing individual steps separately (single-step mode). In addition, the program shows the operator all the measurement data from the sensors in real time as well as the current progress of the measurement.

The result of each measurement is the volume, the density and the mass of each test weight and the standard deviation of the results, with complete documentation of the conditions of measurement and default values. Measurement values and all associated data can be provided as raw value output or on printouts that contain a complete evaluation of the measurement data.

Fig. 16: calibration of a clients volume comparator in the BEV

In validation and testing of the initial system at BEV, in-house comparative measurements of the density of solid bodies – in this case with a sinker (Pyrex sphere; mass approx. 119 g, volume approx. 97 m³) – with the new testing facility showed an uncertainty in volume of less than 0.9 mm³ and in mass of less than 0.07 mg. Comparisons with reference weights at BEV also showed excellent results. The prototype unit is already being used to measure the volume of weights with a mass of 1 g to 1 kg in the scope of mass determination at BEV, for ongoing in-house and external calibrations and it has been implemented in the BEV quality system.

Further units have been built up for other metrology institutes and for accredited calibration laboratories whereas every unit is given an examination by the Physico-Technical Testing Service of the BEV ,

Fig. 17: comparison of a 1 kg, 500 g and 200 g test-weight with a national Volume standard (500 g Silicium sphere Nr. 2) of the BEV