

The modification and re-assessment of the NPL 1 kg solid density apparatus

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ABSTRACT

The apparatus used by the Mass and Density Section of NPL for the density determination of solid artefacts in the range 200 g to 1 kg by hydrostatic weighing has been modified to improve mechanical performance and versatility. This report describes the changes made and the subsequent re-assessment of the apparatus.

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1. INTRODUCTION

The NPL 1 kg solid density apparatus is used for the routine high accuracy density determination of artefacts with a nominal mass of between 200 g and 1 kg. It consists essentially of a Mettler H315 balance mounted over a temperature controlled water bath. A glass vessel is placed within the bath and filled with de-ionised and distilled water. The artefact to be tested is supported by a cradle which can be suspended directly from the balance so that it is totally immersed in the water.

The basic procedure is to weigh the artefact in air to determine its true mass and then in pure water to determine its apparent mass. By using Archimedes principle and applying various corrections, the volume and hence the density of the artefact can then be calculated. A more detailed explanation, including a description of the apparatus in its original configuration, is contained in NPL Report MOT3 [1].

Since it was installed in the early 1980s the apparatus has been used extensively for the density measurement of mass standards belonging to NPL and external customers. It has proved able to give consistent and reliable results with a relative uncertainty approaching 1 part in 10^5 . However, in recent years a number of operational shortcomings have developed which have impaired its efficiency and safety.

2. MODIFICATION OF APPARATUS

2. PROBLEMS ENCOUNTERED

All the shortcomings were centred around the operation of the weight exchanger. In the original design this consisted of an inner cradle suspended from the balance, and a motor operated outer cradle which could lift an artefact off of the inner cradle. The problems could be summarised as follows:

- A marked tendency for the inner cradle to rotate axially on the suspension, which frequently caused difficulty during the artefact exchange operation.
- Limited provision for coping with different size artefacts. Several non-adjustable pairs of inner and outer cradles had to be used to accommodate different diameter artefacts.
- Lack of confidence about the security of the artefact during exchange. As a safety precaution a silicone rubber mat was placed at the bottom of the vessel, resulting in the possibility of water contamination.

DESIGN REQUIREMENTS

To eliminate these problems it was necessary to totally re-design the weight exchanger and, at the same time, take the opportunity to incorporate some new features which would prove beneficial in the future. The main requirements were as follows:

- The capacity to accommodate 100 g to 1 kg stainless steel or platinum-iridium mass standards of integral design. In addition the potential to accommodate spherical artefacts with nominal diameters of up to 100 mm.
- The ability to accommodate two artefacts of the same nominal mass, thus allowing the potential for traceability through solid density reference standards.
- All materials subject to immersion to be chemically inert to prevent contamination of the water.
- The artefacts to be securely supported at all times therefore eliminating the need for a silicone mat.
- The weight exchange mechanism to be operated by stepper motors to give semi-automatic control, with the potential for incorporating full automation at a later date.

3. DESIGN OF THE NEW WEIGHT EXCHANGER

In April 1998 Dušan Trochta, a metrologist from the Slovak Institute of Metrology based in Bratislava started work in the Mass and Density Section on temporary contract to NPL as a SEMAT¹ secondee. Mr Trochta was familiar with hydrostatic weighing techniques and had experience in the design and manufacture of related equipment. Whilst at NPL his main task was the re-design of the weight exchanger.

FEATURES OF THE NEW DESIGN

The new weight exchanger was designed to replace the existing one with the minimum of modification to the rest of the apparatus. A general view of the modified apparatus is shown in Figure 1. The principle features are a totally re-designed inner cradle, and the replacement of the outer

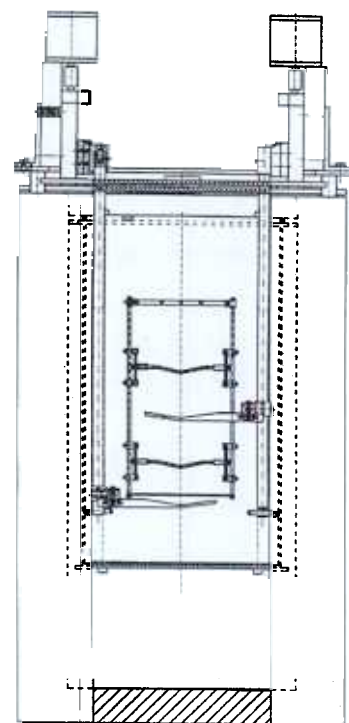


Figure 1. General view of weight exchanger.

¹ Secondment in European Measurement and Testing. A Department of Trade and Industry funded scheme operated on its behalf by NPL.

cradle by two pairs of lifting arms operated by stepper motors.

A new top plate supports the two motors, each of which can drive a rod vertically up and down within the water bath (Figure 2).

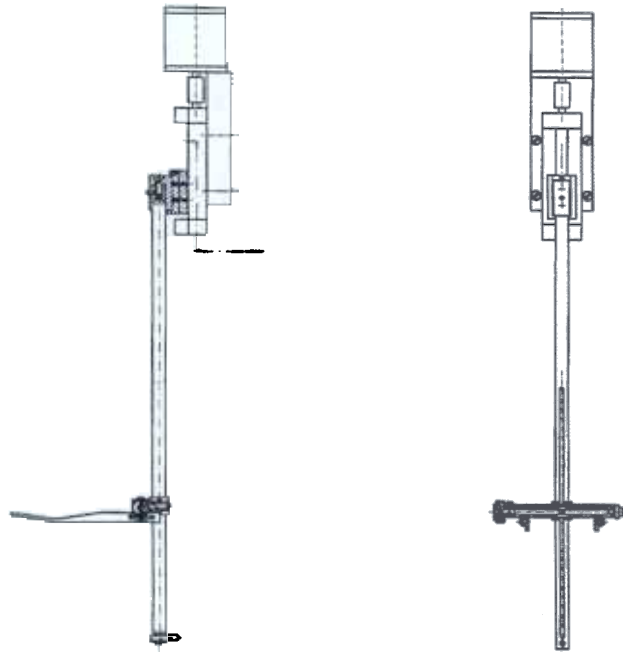


Figure 2. Stepper motor and lifting arm assembly.

Fitted to each rod is a pair of arms which can be adjusted for separation by means of a knurled knob to enable a range of different sized artefacts to be accommodated (Figure 3). The depth of immersion of the arms in the water can also be adjusted by altering their location on the rod.

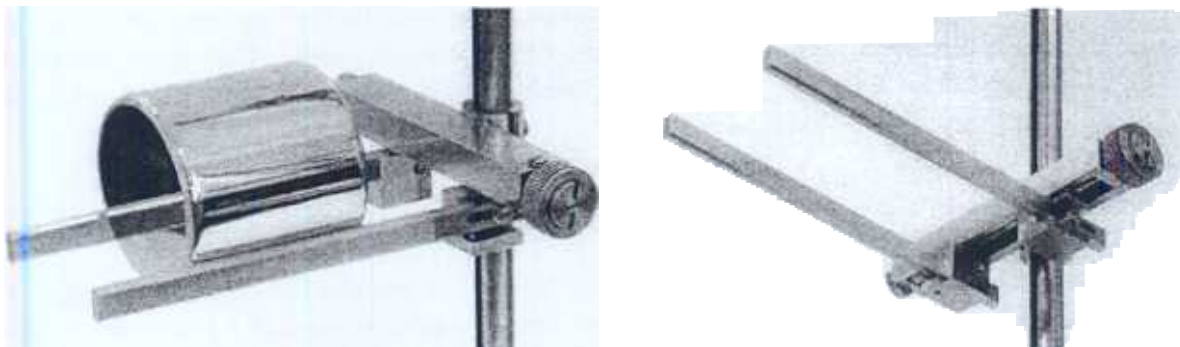


Figure 3. Lifting arms. The photograph on the left shows weight 'A1' in position.

The cradle (shown in more detail in Figure 4) allows two different weights to be suspended from the balance in turn. It can be adjusted to suit different sized weights and also allows the vertical separation between them to be altered. The straight rods which support the artefacts can be replaced by V-shaped ones to locate spherical artefacts positively and securely.

3.2 INITIAL ASSESSMENT

After assembly, the following problems were encountered:

- The lifting arms lacked rigidity in the horizontal plane with a tendency for them to splay apart, particularly when loaded with a 1 kg artefact.
- It was difficult to maintain the horizontal position of the arms, since each was only secured at one end by a 3 mm diameter machine screw. It also proved difficult to adjust them to a truly horizontal position, which is desirable to prevent the artefact 'walking' along the arms during continued exchanges.
- The arms were designed with a shallow V-shape on their upper surface to provide a positive location for spherical artefacts. However, this made it difficult to maintain a cylindrical artefact in a horizontal position during the exchange process.

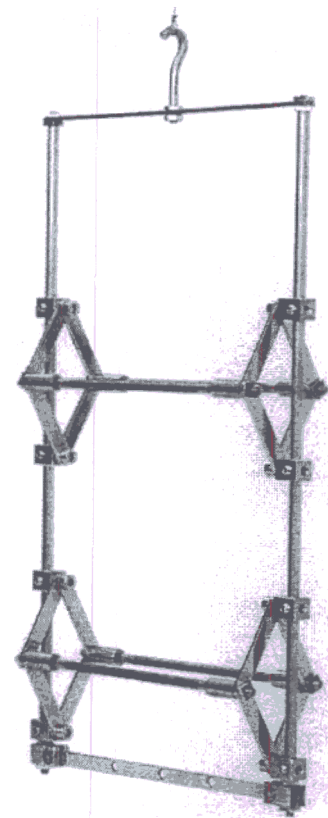


Figure 4. Cradle.

3.3 REMEDIAL MODIFICATIONS

To overcome these problems the moving nuts supporting the arms were re-designed with a longer engagement length (15 mm instead of 6.5 mm) and were individually machined to be a good sliding fit within the guides. Provision was also made for adjusting the arms to an accurate horizontal position by means of a set screw in each of the moving nuts operating on the underside of the arms. New arms were made with a straight profile. They were designed to be interchangeable with the original ones thus allowing them to be re-fitted when it becomes necessary to use a spherical artefact.

3.4 MODIFICATIONS TO BALANCE AND SUSPENSION

The nominal capacity of the Mettler H315 balance is 1 kg. In practice the cradle is included in the weighing configuration. The mass of the new cradle was approximately 110 g more than the original one. To compensate for this difference, all of the brass components of the pan assembly and suspension system were re-made in aluminium alloy and the beams of the suspension system were reduced in section and milled with transverse slots. All the hooks and the suspension rod were re-designed to reduce the axial rotation of the cradle while still permitting easy alignment with the lifting arms.

A further gain was achieved by attaching a disc of stainless steel weighing approximately 60 g to the rear of the balance beam. With a mechanical advantage of 1.7:1, this allowed an additional mass of up to about 100 g to be applied to the balance.

3.5 PROTECTIVE SLEEVING

To avoid the risk of damage due to the artefact coming into direct contact with stainless steel, protective sleeving is fitted to the cradle rods and lifting arms. It is essential that the sleeving should not contaminate the water, and that it should be resistant to acetone which is used for cleaning the apparatus. Preliminary tests were therefore carried out on two types of heat shrink sleeving:

- A transparent, modified polyvinylidene flouride sleeving.
- An adhesive lined, white, irradiated polyolefin sleeving.

Short lengths of each type were heat shrunk onto pieces of dural rod and immersed in water and in acetone at room temperature. In water, neither type of sleeving showed any obvious physical effect after 7 days immersion. In acetone, the polyvinylidene flouride sleeving expanded after twenty minutes or so to approximately its previous (unshrunk) size. There was no obvious effect on the polyolefin sleeving except that the adhesive at each end showed some evidence of softening. Each type was also immersed in de-ionised and distilled water for 7 days. The polyvinylidene flouride sleeving had no effect on the conductivity of the water, but the polyolefin sleeving increased the conductivity from 2 to 6 μS .

Owing to the possibility of the adhesive lining of the polyolefin sleeving affecting the purity of the water over a long period, and because of the difficulty of physically removing it (if replacement became necessary), it was decided to use the polyvinylidene flouride sleeving. However, it was evident that contact with acetone during cleaning of the apparatus would have to be kept to a minimum because of its effect on the sleeving.

4. ASSESSMENT

4 BALANCE

The Mettler H315 is a single-pan, two-knife-edge, constant load balance with a resolution of 0.10 mg. Following the modifications described previously, the balance was assessed for linearity and sensitivity of the optical scale, and repeatability at zero and maximum load. Sensitivity of the optical scale and repeatability proved satisfactory. There was a small systematic error of the optical scale amounting to about 0.5 mg over its full deflection of 1 g. Since, in practice, comparison weighings are made with the balance poised to within a maximum of 20 mg, this error was not considered significant for the purpose for which the balance is to be used.

4.2 MECHANICAL OPERATION OF THE WEIGHT EXCHANGER

Mechanical operation was checked with the rig fully assembled and the vessel filled with water. A cylindrical weight of nominal mass 1 kg was used for test purposes. The modified suspension assembly allowed the cradle to be easily aligned axially with the lifting arms and any slight propensity to swing was quickly damped out by the water. Weight exchange in both directions took place in a smooth and controlled manner. The lifting arms supported the weight securely. Two or three exchanges were enough to centre the weight in the cradle with no further tendency for it to 'walk'.

4.3 HYDROSTATIC WEIGHING

An NPL 1 kg stainless steel standard weight (identified as A1) was used to assess the performance of the apparatus for its purpose of solid density measurement. This standard was chosen because its history was well known and it had been used in two intercomparisons. Prior to hydrostatic weighing the true mass of this weight was determined to an uncertainty appropriate to OIML Class E₁.

The hydrostatic weighing procedure used was essentially as described in NPL Report MOT 3 [1]. Since water density increases with depth, and because of the possibility of temperature stability differences between the upper and lower positions on the cradle, assessments were made with the weight in both positions. Six measurement runs were made with the weight supported in the lower position on the cradle, then the whole procedure was repeated with the weight in the upper position. To assess the reproducibility of the apparatus, a further six runs were made with the weight transferred again to the lower position on the cradle.

Water temperature was recorded at the beginning and end of each stage of the weighing scheme using a platinum resistance thermometer in close proximity to the weight.

5. RESULTS

Tables A1.1 to A1.3 given in Appendix 1 show all the readings recorded throughout each measurement run together with the calculated densities. The water density was calculated using the thermal dilatation formula given by Patterson and Morris [2]. Corrections for saturation by air [3], depth of immersion and ambient atmospheric pressure [4], and isotopic content of the water² were then applied.

The final values, together with the estimated uncertainty (based on a standard uncertainty multiplied by a coverage factor $k=2$), are summarised in Table 1.

² A sample of tap water from NPL was sent to BIPM in 1986 for analysis of its isotopic content.

Date of calibration	Weight position	Density at 20° C kg m ⁻³	Estimated uncertainty ± kg m ⁻³
9-12 Feb 1999	Lower (1st determination)	7 889.85	0.14
2-5 Mar 1999	Upper	7 889.87	0.14
13-14 May 1999	Lower (2nd determination)	7 889.85	0.14

Table 1. Summary of results of hydrostatic density determination of weight 'A1'

Comparison with density values previously obtained for this weight are shown in Table 2. The original calculations were made using different formulae to determine the density of water. The reported values have been corrected on a consistent basis with the values given in Table 1.

Reference	Date of calibration	Density at 20° C kg m ⁻³	Estimated uncertainty ± kg m ⁻³
Bi-lateral Comparison [1] NPL value	1994-5	7 889.85	0.12
CCM Density Intercomparison [5] Mean value	1986	7 889.86	0.03*

Table 2. Summary of previous values obtained for the density of weight 'A1'

*Standard deviation (combined uncertainties not quoted in report [5])

An ABABA weighing scheme was used for each measurement run. To check the stability of the water temperature at each cradle position, the measured temperature differences between the start and finish of each run were compared for all the determinations made (Table 3).

Weight position	Temp. difference for each run mK						Mean mK	σ_{n-1} mK
	1	2	3	4	5	6		
Lower (1st determination)	8	7	4	2	8	3	5.3	2.7
Upper	13	13	9	7	0	8	8.3	4.8
Lower (2nd determination)	2	8	10	8	8	11	7.8	3.1

Table 3. Summary of measured temperature differences between the beginning and end of each run

6. CONCLUSIONS

The quantitative assessment using the 1 kg weight 'A1' has proved very satisfactory with excellent agreement between the new values and those previously determined. Insignificant temperature variations between the upper and lower positions in the cradle have no effect on the density determination.

Ease of use, efficiency and adaptability have been considerably improved by the modifications to the apparatus. All objectives of the design requirements have been achieved except that there has not yet been an opportunity to test the operation of the apparatus using spherical artefacts.

ACKNOWLEDGEMENTS

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APPENDIX 1

MEASUREMENTS AND RESULTS

Mass: 999.557 704 g											
X coefficient: $-0.370\ 5\ \text{kg m}^{-3}\ ^\circ\text{C}^{-1}$											
Cubic coefficient: $-4.70\text{E-}5\ ^\circ\text{C}^{-1}$											
Run	Weight in water g	Air density kg m^{-3}	Corrected weight in water g	Ambient pressure mbar	Water temp $^\circ\text{C}$	Water density kg m^{-3}	Corr for Bignell kg m^{-3}	Corr for ambient kg m^{-3}	1- Corr W-in-W Mass	Density kg m^{-3}	Density at 20°C kg m^{-3}
1	873.199 800	1.184 4	873.070 523	1005.83	18.643	998.476 2	998.473 5	998.473 2	0.126 543 15	7 890.377	7 889.874
	873.199 780	1.183 4	873.070 612	1005.89	18.644	998.476 0	998.473 3	998.473 0	0.126 543 06	7 890.381	7 889.879
2	873.199 220	1.184 7	873.069 910	1005.94	18.628	998.479 0	998.476 4	998.476 0	0.126 543 76	7 890.361	7 889.853
	873.199 310	1.183 7	873.070 109	1005.99	18.631	998.478 5	998.475 8	998.475 5	0.126 543 56	7 890.369	7 889.862
3	873.230 270	1.204 5	873.098 794	1021.14	20.161	998.172 3	998.169 8	998.170 2	0.126 514 87	7 889.746	7 889.806
	873.230 110	1.203 5	873.098 743	1021.16	20.164	998.171 7	998.169 2	998.169 6	0.126 514 92	7 889.738	7 889.799
4	873.232 000	1.207 3	873.100 218	1021.66	20.168	998.170 8	998.168 4	998.168 8	0.126 513 44	7 889.824	7 889.886
	873.231 300	1.206 0	873.099 660	1021.66	20.168	998.170 8	998.168 4	998.168 8	0.126 514 00	7 889.789	7 889.851
5	873.230 670	1.207 1	873.098 910	1021.84	20.149	998.174 8	998.172 3	998.172 8	0.126 514 75	7 889.774	7 889.829
	873.230 520	1.205 6	873.098 924	1021.83	20.154	998.173 8	998.171 3	998.171 7	0.126 514 74	7 889.766	7 889.823
6	873.266 910	1.209 7	873.134 861	1026.82	21.781	997.821 4	997.819 1	997.819 8	0.126 478 78	7 889.226	7 889.886
	873.266 910	1.208 4	873.135 003	1026.11	21.782	997.821 2	997.818 9	997.819 5	0.126 478 64	7 889.233	7 889.893
7	873.267 000	1.212 5	873.134 645	1026.35	21.782	997.821 2	997.818 9	997.819 5	0.126 479 00	7 889.211	7 889.871
	873.265 930	1.212 3	873.133 597	1026.26	21.754	997.827 4	997.825 1	997.825 8	0.126 480 05	7 889.195	7 889.845
Mean density at 20°C : $7\ 889.854\ \text{kg m}^{-3}$										Standard deviation: $0.029\ \text{kg m}^{-3}$	

Table A1.1. Measurements and results of hydrostatic weighing
Lower cradle position (1st determination)

Mass: 999.557 704 g											
X coefficient: $-0.354\ 8\ \text{kg m}^{-3}\ ^\circ\text{C}^{-1}$											
Cubic coefficient: $-4.50\text{E-}5\ ^\circ\text{C}^{-1}$											
Run	Weight in water g	Air density kg m^{-3}	Corrected weight in water g	Ambient pressure mbar	Water temp $^\circ\text{C}$	Water density kg m^{-3}	Corr for Bignell kg m^{-3}	Corr for ambient kg m^{-3}	1- Corr W-in-W Mass	Density kg m^{-3}	Density at 20°C kg m^{-3}
1	873.199 050	1.183 0	873.069 926	1003.84	18.608	998.482 9	998.480 2	998.479 8	0.126 543 75	7 890.392	7 889.898
	873.198 930	1.181 0	873.070 024	1003.79	18.612	998.482 1	998.479 5	998.479 0	0.126 543 65	7 890.392	7 889.899
2	873.197 440	1.177 3	873.068 938	997.35	18.585	998.487 3	998.484 6	998.483 8	0.126 544 74	7 890.363	7 889.860
	873.197 730	1.175 2	873.069 457	997.28	18.592	998.485 9	998.483 3	998.482 5	0.126 544 22	7 890.384	7 889.885
3	873.225 700	1.162 1	873.098 853	985.90	20.131	998.178 5	998.176 1	998.174 7	0.126 514 81	7 889.786	7 889.832
	873.226 250	1.161 2	873.099 501	985.93	20.137	998.177 3	998.174 8	998.173 5	0.126 514 16	7 889.816	7 889.865
4	873.227 170	1.162 8	873.100 246	986.71	20.152	998.174 2	998.171 7	998.170 4	0.126 513 41	7 889.838	7 889.892
	873.226 800	1.161 9	873.099 975	986.81	20.154	998.173 8	998.171 3	998.170 0	0.126 513 69	7 889.818	7 889.873
5	873.257 350	1.170 6	873.129 571	993.51	21.489	997.886 6	997.884 3	997.883 3	0.126 484 08	7 889.399	7 889.927
	873.256 820	1.170 0	873.129 106	993.57	21.490	997.886 4	997.884 1	997.883 1	0.126 484 54	7 889.368	7 889.897
6	873.256 170	1.175 3	873.127 878	995.55	21.469	997.891 1	997.888 7	997.887 8	0.126 485 77	7 889.329	7 889.850
	873.256 310	1.174 2	873.128 138	995.62	21.473	997.890 2	997.887 8	997.887 0	0.126 485 51	7 889.338	7 889.861
Mean density at 20°C : $7\ 889.878\ \text{kg m}^{-3}$										Standard deviation: $0.025\ \text{kg m}^{-3}$	

Table A1.2. Measurements and results of hydrostatic weighing
Upper cradle position

Mass: 999.557 704 g			X coefficient: -0.362 6 kg m ⁻³ °C ⁻¹			Cubic coefficient: -4.60E-5 °C ⁻¹					
Run	Weight in water g	Air density kg m ⁻³	Corrected weight in water g	Ambient pressure mbar	Water temp °C	Water density kg m ⁻³	Corr for Bignell kg m ⁻³	Corr for ambient kg m ⁻³	1- Corr W-in-W Mass	Density kg m ⁻³	Density at 20°C kg m ⁻³
1	873.231 720	1.190 5	873.101 772	1 008.90	20.226	998.158 8	998.156 3	998.156 1	0.126 511 89	7 889.820	7 889.902
	873.231 370	1.188 6	873.101 630	1 008.75	20.228	998.158 3	998.155 9	998.155 6	0.126 512 03	7 889.808	7 889.891
2	873.231 100	1.189 2	873.101 294	1 007.67	20.229	998.158 1	998.155 7	998.155 4	0.126 512 37	7 889.785	7 889.868
	873.230 920	1.186 7	873.101 387	1 007.59	20.230	998.157 9	998.155 4	998.155 2	0.126 512 27	7 889.789	7 889.873
3	873.229 880	1.188 3	873.100 173	1 009.07	20.229	998.158 1	998.155 7	998.155 4	0.126 513 49	7 889.716	7 889.799
	873.230 130	1.186 4	873.100 630	1 009.06	20.237	998.156 5	998.154 0	998.153 8	0.126 513 03	7 889.731	7 889.817
4	873.231 080	1.190 4	873.101 143	1 008.86	20.226	998.158 8	998.156 3	998.156 1	0.126 512 52	7 889.781	7 889.863
	873.231 370	1.188 7	873.101 619	1 008.90	20.229	998.158 1	998.155 7	998.155 4	0.126 512 04	7 889.806	7 889.889
5	873.230 930	1.191 1	873.100 917	1 008.72	20.224	998.159 2	998.156 7	998.156 5	0.126 512 74	7 889.770	7 889.852
	873.230 850	1.188 5	873.101 121	1 008.80	20.229	998.158 1	998.155 7	998.155 4	0.126 512 54	7 889.775	7 889.858
6	873.231 510	1.189 1	873.101 715	1 009.55	20.247	998.154 4	998.151 9	998.151 7	0.126 511 94	7 889.782	7 889.872
	873.230 950	1.187 0	873.101 384	1 009.69	20.254	998.152 9	998.150 4	998.150 3	0.126 512 28	7 889.750	7 889.842
Mean density at 20°C: 7 889.861 kg m ⁻³						Standard deviation: 0.028 kg m ⁻³					

Table A1.3. Measurements and results of hydrostatic weighing
Lower cradle position (2nd determination)