The Assignment of Uncertainties to Mass Standards at NPL

I Severn

March 2004

The Assignment of Uncertainties to Mass Standards at NPL

I Severn

ABSTRACT

The National Physical Laboratory has undertaken a project to investigate the uncertainties associated with the calibration of high precision mass standards. As a result of this work it has proved possible to significantly improve the best measurement capabilities of the Mass and Density Standards Section.

ISSN 1473-2734

National Physical Laboratory
Teddington, Middlesex, United Kingdom, TW11 0LW

Approved on behalf of the Managing Director, NPL,
by Dr Roy Preston, authorised by Head of NPL Materials Centre

CONTENTS

1. INTRODUCTION

The Mass and Density Standards Section of the National Physical Laboratory (NPL) is responsible for the realisation and dissemination of the mass scale within the United Kingdom. Part of this task involves the calibration of NPL's mass standards used to realise the scale and those of other organisations requiring mass calibrations with low uncertainties. Historically the uncertainty in the calibration of a weight for third party customers has been quoted as a fraction of the tolerance specified by the International Organisation for Legal Metrology (OIML) for particular classes of weights [1] but this represents an uncertainty that may be achieved relatively easily at NPL. This report describes the model that is used for the calculated uncertainties achieved at NPL. The analysis has been carried out in accordance with the 'Guide to the Expression of Uncertainty in Measurement' [2].

2. THE WEIGHING PROCESS

Mass calibrations at NPL are carried out by comparing test-weight(s) with a minimum of two standards. When considering the comparison of weights on a conventional mass basis the relationship between the test-weight (W_X) and the standard (W_S), both given in g, may be written as:-

$$W_x = W_s + (V_x - V_s)(\rho_A - 1.2\times10^{-3}\,\mathrm{gcm}^{-3}) + S\times R + W_{MW} \quad (1)$$

where

V_X and V_S are the volumes of the test-weight and standard respectively (in cm^3)
ρ_A is the air density (in g cm^{-3}) at the time of the measurement
S is the comparator sensitivity (in g per display unit)
R is the difference between the comparator reading when the test-weight and the standard are applied (in display units)
W_{MW} is the conventional mass of any make-weights used in the calibration (in g)

It should be noted that there are additional factors that influence the weighing result such as the difference between the heights of the centres of gravity of the weights under comparison and the thermal expansion of the weights. However, these are generally insignificant and will be discussed in Section 3.2.7.

The uncertainty [U(Wx)] associated with the comparison of weights is given by:

$$U(W_x)^2 = \left(\frac{\partial W_x}{\partial W_s}\right)^2 U(W_s)^2 + \left(\frac{\partial W_x}{\partial V_x}\right)^2 U(V_x)^2 + \left(\frac{\partial W_x}{\partial V_s}\right)^2 U(V_s)^2 + \left(\frac{\partial W_x}{\partial \rho_A}\right)^2 U(\rho_A)^2 + \left(\frac{\partial W_x}{\partial S}\right)^2 U(S)^2 + \left(\frac{\partial W_x}{\partial R}\right)^2 U(R)^2 + \left(\frac{\partial W_x}{\partial W_{MW}}\right) U(W_{MW})^2 \quad (2)$$

which may be written as:

$$U(W_x)^2 = U(W_s)^2 + (\rho_A - 1.2)^2 U(V_x)^2 + (1.2 - \rho_A)^2 U(V_s)^2 + (V_x - V_s)^2 U(\rho_A)^2 + R^2 U(S)^2 + S^2 U(R)^2 + U(W_{MW})^2 \quad (3)$$

The practical origin of each of the uncertainty components above, and their magnitude is discussed in Section 3.

3. UNCERTAINTY CONTRIBUTIONS

The uncertainty budget may be split into Type A and Type B contributions. In general Type A uncertainties are random effects related to the measurement of a quantity while Type B uncertainties generally relate to systematic influences on the measurement process. Throughout this Section all uncertainties are quoted with a coverage factor k=1.

3.1 TYPE A UNCERTAINTY

3.1.1 Weighing Scheme Uncertainty (U(R))

The weighing scheme uncertainty is an indication of the repeatability of the measurements on a weight. A single weight being calibrated at NPL will be subject to between four and six comparisons with standards. It is probable that these comparisons will not all assign exactly the same value to the test-weight. The sample standard deviation of the measurements (σ_{n-1}) gives an indication of how well the comparisons agree and may be calculated from the following formula:-

$$\sigma_{n-1} = \sqrt{\frac{\sum_{i=1}^{n}(\bar{x}^2 - x_i^2)}{n-1}} \quad (4)$$

where $\bar{x}$ = mean of all measurements
x_i = result of measurement i
n = number of readings

and the standard uncertainty of the measurements (U_R) is given by:-

$$U_R = \frac{\sigma_{n-1}}{\sqrt{n}} \quad (5)$$

At NPL, it is more usual for a weight be calibrated as part of a set of weights by a process known as sub-division [3]. In this process two or more standards, normally of a nominal value 1 kg, are compared with various combinations of test-weights in order to assign a value to each test-weight. A simple example of the sub-division process is the comparison of two 500 g test-weights with each other prior to comparing the pair of them with a 1 kg standard. This would allow the mass of each of the two 500 g test-weights to be calculated. Similarly two 1 kg standards could be compared with a 2 kg test-weight in order to assign it a value. The most frequent use of this sub-division process at NPL is the calibration of weight sets from 20 kg to 1 mg using two 1 kg standards. Since 1981, NPL has used a piece of software that performs the calculations for the sub-division process using a least squares analysis and outputs the value of each of the test-weights and the associated weighing scheme uncertainty. The calculation of these quantities is described by Hunt [4].

3.2 TYPE B UNCERTAINTIES

3.2.1 Uncertainty in Mass Standards ($U(W_S)$)

There is an uncertainty associated with the mass standards used in the calibration of test-weights. A minimum of two standards are used in all mass calibrations undertaken at NPL and so when using this composite standard (ie a group of weights that are used to make up the standard) the uncertainties associated with each of the weights are added arithmetically to calculate the total uncertainty of the standard.

Where two or more standard weights of the same mass are used to calibrate a single weight, again of the same mass, and there is a difference between the uncertainties associated with the standards, the largest figure has been used.

When sub-division has been used in a calibration, the standards will have a different nominal value to most of the test-weights. In this case the uncertainty contribution due to the uncertainty in the standards for a particular test-weight is given by the following:-

$$U_s(W_S) = U_s \frac{M_T}{M_S} \qquad (6)$$

where

$U_S(W_S)$= Uncertainty in the value of the standard
M_S = Nominal mass of standard
M_T = Nominal mass of test-weight

It should be noted that the standard itself, unless it is Kilogram 18 (the United Kingdom's National Standard of Mass which has its uncertainty assigned by the Bureau International des Poids et Mesures (BIPM)), has been assigned an uncertainty based on the methods described in this Report. The uncertainties of all NPL's mass standards are traceable back to the uncertainty assigned to Kilogram 18 by the BIPM.

3.2.2 Uncertainty in Air Density Measurement ($U(\rho_A)$)

In order to measure the air buoyancy effect experienced by a weight being calibrated it is necessary to determine the density of the air in which a weighing is being performed. At NPL the air density is normally calculated from measurements of temperature, air pressure, dew-point and carbon dioxide concentration using the empirical equation recommended by the Comité International des Poids et Mesures (CIPM) [5],[6]. The uncertainty in the air density is multiplied by the volume difference between the mass standard and the test-weight in order to give an equivalent mass uncertainty.

Typically, in the Mass and Density Standards Section at NPL, the total uncertainty in the determination of air density is calculated to be 0.000 9 kg m^{-3} for routine measurements involving a manually operated mass balance and 0.000 16 kg m^{-3} for measurements performed on an automatic mass comparator. The contributions to these values are outlined below.

3.2.2(a) Air Temperature

When one of NPL's automatic mass comparators is used to compare weights, the air temperature is measured by a pair of platinum resistance thermometers (PRTs) located inside the comparator's case. Electronic thermistors are used in the manually operated comparators.

The calibration uncertainty of the PRT is 0.0025 °C, but when additional contributions such as the change in calibration constants between calibrations and thermal gradients across the balance case are considered, the uncertainty is estimated to be 0.01 °C.

The uncertainty in the calibration of the thermistor sensors is 0.015 °C, but, when the contributions discussed in the previous paragraph are considered, the uncertainty is estimated to be to 0.02 °C.

3.2.2(b) Air Pressure

The air pressure in the mass laboratory is measured using Druck vibrating element barometers. The calibration uncertainty for these instruments is 0.05 mbar. Repeated calibrations of the instruments in use within the Mass and Density Section have found these devices to be stable to within this uncertainty and therefore an uncertainty of 0.05 mbar is assigned to the measurement.

3.2.2(c) Dew-point

Humidity in the mass laboratory is measured using a chilled mirror dew-point meter. The quoted calibration uncertainty is only 0.02 °C on these instruments but when the changes in instrument behaviour between calibrations and the potential variations in the dew-point at various locations in the laboratory are considered this quantity is assigned an uncertainty of 0.25 °C.

3.2.2(d) Carbon Dioxide Concentration

The carbon dioxide in the mass laboratory is not routinely measured. A value of 600 parts per million (ppm) with an uncertainty of 50 ppm is assumed for this quantity. These figures are based on historical measurements in the laboratory.

3.2.2(e) The Air Density Equation

There is an uncertainty associated with the empirical equation used in the calculation of air density [5]. This contributes 0.000 12 kg m^{-3} to the uncertainty budget.

3.2.3 Uncertainty in the volume of the Test-Weight ($U(V_x)$)

The upthrust experienced by a weight, i.e. air buoyancy, is dependent on the weight's volume, which is determined by measurement of its mass and density. There is therefore an uncertainty in the determination of the density [7] of a weight which leads to an uncertainty in the calculated upthrust. The volume of a weight (V) is linked to the mass (m) and density (ρ) by the following expression:

$$V = \frac{m}{\rho}$$

and the uncertainty in the volume (U(V)) is linked to that in the density (U(ρ)) and the mass U(m) by the following expression:

$$U(V)^2 = \left(\frac{\partial V}{\partial \rho}\right)^2 U(\rho)^2 + \left(\frac{\partial V}{\partial \rho}\right)^2 U(m)^2$$

The partial derivatives may be evaluated to give:

$$U(V)^2 = \left(\frac{m}{\rho^2}\right)^2 U(\rho)^2 + \left(\frac{1}{\rho}\right)^2 U(m)^2$$

In all cases when this component is significant the second term will be insignificant relative to the first. For example in the case of a 1 kg stainless steel weight of density 8 050 kg m^{-3}, mass uncertainty 15 µg and density uncertainty 0.02 kg m^{-3} the volume uncertainty (in cm^3) is given by:

$$U(V)^2 = \left(\frac{1000}{8.05^2}\right)^2 \times 20^2 + \left(\frac{1}{8.05}\right)^2$$

3.2.3(a) Conventional Mass Calibration

The weighing model described in Section 2 is based on the calibration of weights on a conventional mass basis [8]. In this case the uncertainty in the volume of the test-weight is multiplied by the difference in the air density at the time of measurement from the conventional value of 1.2 kg m^{-3}. At NPL the air density lies in the range 1.14 kg m^{-3}to 1.22 kg m^{-3}, so giving a maximum difference of 0.06 kg m^{-3} from 1.2 kg m^{-3}. The uncertainty in the volume of the test-weight is therefore multiplied by 0.06 kg m^{-3} to obtain a mass equivalent uncertainty for this contribution.

3.2.3(b) Mass Calibration

If a test-weight is being calibrated on a mass basis rather than a conventional mass basis the weighing equation (1) is modified to become:

$$W_x = W_s + (V_x - V_s)\rho_A + S \times R + W_{MW} \qquad (7)$$

and the expression for the uncertainty in the measurement becomes:

$$\begin{aligned} U(W_x)^2 = U(W_s)^2 + \rho_A{}^2 U(V_x)^2 + \rho_A{}^2 U(V_s)^2 + (V_x - V_S)^2 U(\rho_A)^2 \\ + R^2 U(S)^2 + S^2 U(R)^2 + U(W_{MW})^2 \end{aligned} \qquad (8)$$

Comparison of the above equation with Equation (3) indicates that the only difference between the uncertainty models for mass and conventional mass calibrations relates to the coefficients of the volume uncertainty for both the test-weight and the standard. In the case of a mass basis calibration it is necessary to multiply the volume uncertainty by the air density at the time of calibration. For the purpose of this uncertainty estimate NPL assumes the air density to be 1.2 kg m^{-3}, a typical value for air density at NPL, to give a mass equivalent uncertainty for this contribution.

3.2.4 Uncertainty in the volume of Standards

The uncertainty contribution due to the volume of the standards is similar to that due to the uncertainty in the volume of the test-weight. The only difference comes for the case of calibration by sub-division where this contribution must be scaled according to the relative mass of the standard and the test-weight. This procedure is analogous to that described in Section 3.2.1 for the uncertainty in the mass value of the standards and is described by:

$$U_x(V_X) = U(V_s)\frac{M_x}{M_s} \qquad (9)$$

where

- $U_x(V_x)$ = the volume uncertainty influencing the test-weight
- $U(V_s)$ = the standard's volume uncertainty
- M_x = nominal mass value of the test-weight
- M_s = nominal mass value of the standard

3.2.5 Uncertainty due to the Make-Weights ($U(W_{MW})$)

When two weights are compared there is, unless they are of identical conventional mass, some reliance on the linearity and repeatability of the measurement scale of the mass comparator. The greater the difference between the conventional mass values of the two weights the more importance is placed on the comparator's measurement scale. Such differences, and the resulting problems, may be minimised by the use of additional weights to equate the conventional masses of the loads under comparison. There is an uncertainty in the values of these make-weights that will contribute to the overall uncertainty in the value of the weights under calibration. When significant, the buoyancy correction associated with the make-weights is applied and there is an associated uncertainty (if necessary the uncertainty in the volumes of the make-weights are considered along with those of the weights under comparison).

3.2.6 Comparator Uncertainties (U(S))

In principle there are three significant contributions to the mass comparator uncertainty budget, but in practice these are often be taken into account by other contributions.

3.2.6(a) Comparator Sensitivity Error (U(S))

The mass difference between two weights under comparison is usually indicated by an electronic scale on the comparator in use. Often there is a linear error in this scale which must be taken into account in the uncertainty budget. However, the problem may be minimised by matching the apparent mass of the weights under comparison using make-weights (see Section 3.2.5). The error in comparator sensitivity is multiplied by the difference between the weights under comparison to give the mass error for that particular load difference.

3.2.6(b) Weight Exchanger Anomalies

Some mass comparators have automatic mechanisms that allow for the automatic comparison of several weights. It is possible for these mechanisms to influence the results of a comparison such that the measured mass difference is dependent on the relative location of the weights on the exchanger. It is possible to eliminate this effect by making comparisons with the weights in many different locations on the exchanger. Where multiple weighings of this type are not carried out a component for this is included in the uncertainty budget.

3.2.6(c) Repeatability

This component takes into account the repeatability of the comparator used to make the measurements. For a particular comparator this is determined as part of its regular assessment by making ten repeated comparisons of a pair of weights. If, during the calibration process, several measurements are made on a weight (as is the case for sub-division weighing) it is not necessary to include this component, as it is taken into account by the uncertainty in the weighing scheme.

3.2.7 Weight Stability

The uncertainty in the stability of a weight (U_{stab}) must be included in the uncertainty budget. The most straightforward way to do this is to consider the change in its value between successive calibrations (Δm). This has a rectangular distribution which means that the contribution to the uncertainty budget is given by:-

$$U_{Stab} = \frac{\Delta m}{\sqrt{3}} \qquad (10)$$

This contribution only remains valid for a time up to the interval between the two calibrations that were considered in the calculation of Δm.

Obviously it is only possible to estimate this contribution with knowledge of how the weight has been used between calibrations (for example if a weight is not used between the two calibrations used to calculate Δm this figure may be artificially small and would not be valid for a similar timescale when the weight is in regular use). For this reason the NPL Mass and Density Standards Section only includes this parameter when calculating uncertainties for its own standards. In the case of weights calibrated for third parties it is left to them to add this quantity into their uncertainty budget.

3.2.8 Other Sources of Uncertainty

There are other sources that may potentially contribute to the uncertainty of a mass standard, but in general these contributions are small relative to those already discussed.

3.2.8(a) Thermal Expansion

When calibrating weights to the very best uncertainties it is necessary to correct their volumes for thermal expansion. If this correction is made in the correct manner the uncertainty from this source is insignificant (indeed, when comparing two weights of similar material it is often not necessary to make this correction at all).

3.2.8(b) Magnetism

For high quality mass standards such as those normally calibrated at NPL this is not a problem, but it is potentially a major source of error in the calibration of lower class mass standards.

3.2.8(c) Gravity

It is necessary to make a correction due to variation in the Earth's gravitational field with height if two weights with different centre of gravity heights are being compared to extremely high precision. If this correction is made, the uncertainty associated with it (arising from uncertainty in the gravitational gradient and the height of the centre of gravity of the weights) is insignificant. It is unusual to make a gravitational correction in any of NPL's mass work apart from that involving its best mass standards.

4. EXAMPLE CALCULATIONS

This section contains two worked examples of the uncertainty budget for various weights calibrated by the NPL Mass and Density Standards Section.

4.1.1 1 kg Stainless Steel Weight Versus a 1 kg Pt-Ir Mass Standard

This example is typical of the calibration of one of NPL's primary 1 kg stainless steel standards using secondary platinum-iridium standards.

Quantity	**Uncertainty**		**Contribution to Uncertainty Budget (µg)**	
			Conventional Mass	**Mass**
Weighing scheme	1.4	µg	1.4	1.4
Mass standard	5	µg	5	5
Air Density				
Temperature	0.02	°C	6.4	6.4
Pressure	0.05	mbar	4.7	4.7
Dew-point	0.25	°C	5.3	5.3
CO_2 Concentration	50	ppm	1.9	1.9
CIPM Equation	0.000 12	kg m^{-3}	9.3	9.3
Test-weight volume	0.001	cm^3	0.1	1.2
Standard volume	0.004	cm^3	0.3	4.8
Make-weights	0.001	µg	1	1
Comparator	0	µg	0	0
TOTAL (*k*=1)			**14.5**	**15.3**
TOTAL (*k*=2)			**29.0**	**30.6**

It is worth noting the larger uncertainty from the volume uncertainties when calculating the mass uncertainty on a true mass basis.

4.1.2 A 200 g weight versus 1 kg standard (both stainless steel)

This example relates to the calibration of a 200 g weight of density 7 960 kg m^{-3} with a 1 kg standard of density 8 050 kg m^{-3}. This represents a typical case for the calibration of a 200 g weight by sub-division.

Quantity	**Uncertainty**		**Contribution to Uncertainty Budget (µg)**	
			Conventional Mass	**Mass**
Weighing scheme	1.2	µg	1.2	1.2
Mass standard	15	µg	3	3
Air Density				
Temperature	0.02	°C	0.03	0.03
Pressure	0.05	mbar	0.02	0.02
Dew-point	0.25	°C	0.03	0.03
CO_2 Concentration	50	ppm	0.01	0.01
CIPM Equation	0.000 12	kg m^{-3}	0.04	0.04
Test-weight volume	0.0003	cm^3	0.01	0.36
Standard volume	0.001	cm^3	0.02	0.24
Make-weights	0	µg	0	0
Comparator	0	µg	0	0
TOTAL (*k*=1)			**3.23**	**3.26**
TOTAL (*k*=2)			**6.46**	**6.52**

5. SUMMARY OF NPL'S BEST MEASUREMENT CAPABILITY

The figures in the table below relate to conventional mass calibrations carried out on stainless steel weights. The calculated best capability reflects NPL's UKAS accreditation for conventional mass measurement.

Nominal Value	Calculated Best Capability	Unit	Previous Estimate	Unit
500 kg	0.65	g	N/A	
250 kg	0.25	g	N/A	
100 kg	80	mg	N/A	
50 kg	12	mg	N/A	
20 kg	1.4	mg	5	mg
10 kg	0.4	mg	2	mg
5 kg	0.2	mg	1	mg
2 kg	0.07	mg	0.4	mg
1 kg	0.03	mg	0.03	mg
500 g	0.015	mg	0.1	mg
200 g	0.007	mg	0.04	mg
100 g	0.004	mg	0.025	mg
50 g	0.003	mg	0.02	mg
20 g	0.0015	mg	0.008	mg
10 g	0.001	mg	0.006	mg
5 g	0.0006	mg	0.005	mg
2 g	0.0003	mg	0.003	mg
1 g	0.0002	mg	0.002	mg
500 mg	0.0002	mg	0.002	mg
200 mg	0.0002	mg	0.002	mg
100 mg	0.0002	mg	0.001	mg
50 mg	0.0002	mg	0.001	mg
20 mg	0.0002	mg	0.001	mg
10 mg	0.0002	mg	0.001	mg
5 mg	0.0002	mg	0.001	mg
2 mg	0.0002	mg	0.001	mg
1 mg	0.0002	mg	0.001	mg

Table 1: NPL's Best Measurement Capability. All uncertainties at a coverage factor k=2

6. CONCLUSION

The calculation of the mass calibration uncertainty budgets has enabled NPL to quote significantly better uncertainties than before. Typically the improvement is by a factor of four or five. Whilst it is not anticipated that these better uncertainties will be required by a large number of third party customers, they will be of use to the laboratories calibrating weights to OIML Classes E_1 and E_2. However, an important issue that must not be forgotten is the fact that the figures represent the uncertainty at the time of measurement and do not include any component for the drift in the value of the weights. There is a clear indication to this effect on all certificates issued by NPL and customers are encouraged to carry out a rigorous analysis of the stability of their weights.

7. REFERENCES

[1] OIML International Recommendation R111, Weights of Classes E_1, E_2, F_1, F_2, M_1, M_2, M_3
[2] Vocabulary of metrology, Part 3, Guide to the expression of uncertainty in measurement, PD6461:Part 3: 1995,
[3] Lewis, S.L. and Davidson S., NPL Report MOM 96, 'Calibration of Mass Standards by Subdivision of the kilogram', 1990
[4] Hunt, R.A., NPL Report MOM 50, The Assignment of Values to a Set of Weights, 1981
[5] Giacomo, Metrologia, **18**, 33-40, 1982
[6] Davis, R.S., Metrologia, **29**, 67-70, 1992
[7] Downes, S.P and Elandaloussi, S., NPL Report MOT 3, 'A Comparison of Hydrostatic Weighing Methods used to Determine the Density of Solid Artefacts', 1997
[8] OIML International Recommendation R33, Conventional Value of the Result of Weighing in Air, 1979