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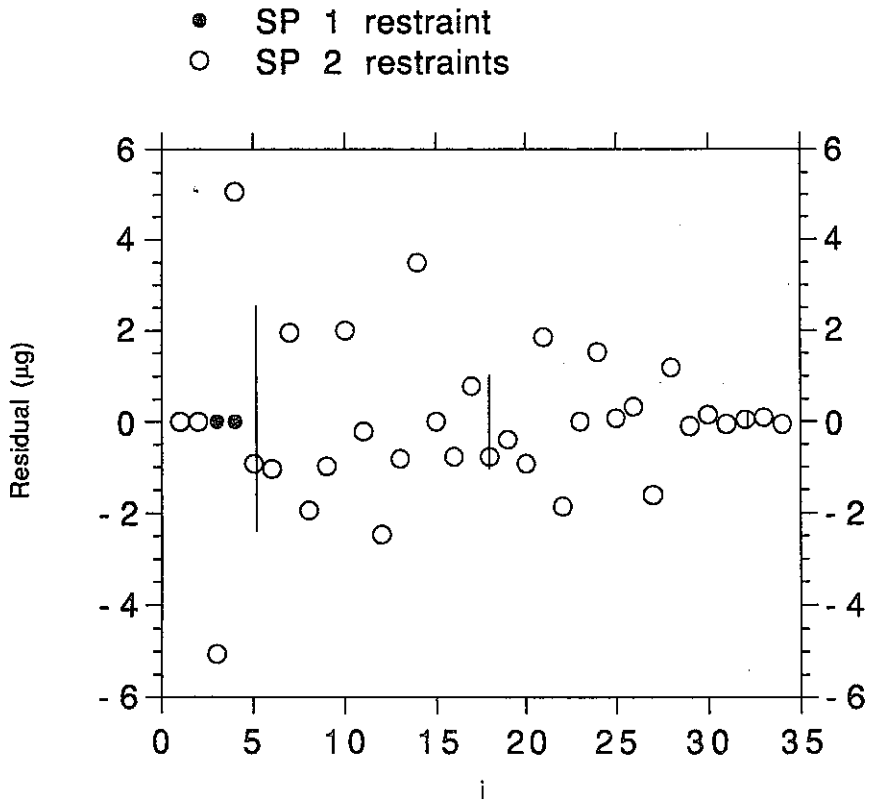
Calibration of Submultiples of the Kilogram: Weighted Least Squares Analysis of Data from Weight Set NPL W43

EUROMET project 231



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Abstract

Calibration of submultiples of the kilogram: Weighted least-squares analysis of data from Weight Set NPL W43. EUROMET project 231

Mass standards which are submultiples of the kilogram may be calibrated through a subdivision procedure employing a variety of balances which may have different measurement performance. Data from such a subdivision, performed recently at the National Physical Laboratory, Teddington, United Kingdom, in the form of untreated weighing results, have been chosen for analysis by the participants in a EUROMET project (231) on uncertainties in mass determination. This report concerns the analysis performed by the Swedish National Testing and Research Institute (SP). As such it represents an extension of subdivision procedures at the SP to include (non-orthogonal) subdivision over an extended mass range (200 g to 1 g).

Key words: Submultiple of kilogram, Sweden, Mass Standard, Traceability; Euromet.

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Introduction

Mass standards which are submultiples of the kilogram may be calibrated through a subdivision procedure employing a variety of balances which may have different measurement performance.

At the SP, when subdivisions of the kilogram have been performed [Ivarsson *et al* 1989], it has been found practicable to arrange the weighings so that complete orthonormal [Grabe 1978, Chapman 1991] subdivision designs may be performed, divided up in equal variance and complete blocks for each balance or set of balances with similar weighing performance.

Data from a subdivision extending over weighings involving balances of various performance, performed recently at the National Physical Laboratory, Teddington, United Kingdom [Lewis and Davidson 1990,1992], in the form of untreated weighing results, have been chosen for analysis by the participants in a EUROMET project (231) on uncertainties in mass determination.

This report concerns the analysis performed by the Swedish National Testing and Research Institute (SP) as its contribution to the EUROMET project on measurement uncertainty. At the same time it represents an extension of subdivision procedures at the SP to include (non-orthogonal) subdivision over an extended mass range (200 g to 1 g).

0 Summary of subdivision theory

0.1 Calibration equations

A set of calibration equations, relating linearly the observed differences in mass, d_i, between a number, M, of combinations of N weights to the required corrections, c_j, to the nominal masses of the weights, can be written in matrix form as:

$$\mathbf{A \cdot C = D} \qquad (0.1)$$

where $\mathbf{A} = \{a_{ij}\}$ is a matrix of dimension M x N, the so-called **design matrix ;**

$\mathbf{C} = \{c_j\}$ is a column matrix with dimension N x 1 --the **correction matrix;**

and $\mathbf{D} = \{d_i\}$ is a column matrix with dimension M x 1 -- the **deflection matrix.**

An exact solution, $\mathbf{C} = \mathbf{A}^{-1} \cdot \mathbf{D}$, of eqn. (0.1) is not obtainable in most cases because of measurement uncertainty. The best fit may be sought with application of the least-squares method.

0.2 Restraints

It is easily demonstrated that the calibration equations are not of full rank, as is the property of all designs where only differences are measured. In mass calibration, one has access to one of more standards whose correction can be taken as known and these can be used as restraints in order to solve the calibration equations uniquely. Usually one starts with a single head weight or with a unique summation of a number of standards [Cameron *et al* 1977]. In most cases a single restraint is sufficient. The use of more than one restraint and dealing with restraints subject to "random error" has been discussed elsewhere [Zelen 1962, Goldman and Zelen 1964, Bich 1992].

In this work, the restraint in the form of calibrated values for one or more of the masses

$$c_r = d_r \qquad (0.2)$$

is introduced by adding equation (0.2) to equation (0.1). Because the value of the restraint is taken as fixed, its value is given infinite weight in the weighting matrix, W (see below).

0.3 Uncertainties

The measured deflections have associated uncertainties, described by a variance-covariance matrix **V(D)**. An initial estimate of these uncertainties is given by the NPL on the basis of the previous, long-term performance of their three balances used in the subdivision. Because the three balances have different variances σ^2_i, it is necessary to weight the system of calibration equations (0.1). This is done conventionally by introducing a weighting matrix **W**, which is assumed here to be a (MxM) diagonal matrix with

$$W_{ii} = (s^2_{norm}/\sigma^2_i) \; ; \; W_{ij} = 0 \; (i \neq j). \tag{0.3}$$

$$(\sigma^2)^{-1} = \frac{\mathbf{W}}{s^2_{norm}}$$

where $\sigma^2 = \mathbf{V(D)}$.

The term s^2_{norm} can be considered as an arbitrary factor whose purpose is simply to make the weighting matrix dimensionless. As will be seen, this term cancels out of both the least-squares estimate **C'** of the corrections as well as from the variance-covariance matrix **V(C')** of these estimates [Hunt 1981, pg. 17].

In this work : $s^2_{norm} = \Sigma\, s^2$ of the different balances.

0.4 Least-squares estimate and its variance

It can be shown that [Hunt 1981] the least-squares estimate of the corrections

$$\mathbf{C'_W = (A^T \bullet W \bullet A)^{-1} \bullet A^T \bullet W \bullet D} \tag{0.4}$$

and the associated variances in the least-squares estimate of the corrections

$$\mathbf{V(C'_W)} = \langle \mathbf{(C'_W - C_W).(C'_W - C_W)^T} \rangle \tag{0.5}$$

$$= \mathbf{(A_W^T \bullet A_W)^{-1} \bullet A_W^T \bullet V(D_W) \bullet A_W \bullet (A_W^T \bullet A_W)^{-1}}$$

where $\mathbf{A_W = W^{-1/2} \bullet A}$ and $\mathbf{D_W = W^{-1/2} \bullet D}$.

Hunt [1981, appendix iii] proves that $\mathbf{C'_W}$ has a minimum variance in the class of linear unbiased estimators if

$$\mathbf{V(D_W) = \sigma^2 I} \tag{0.6}$$

where σ^2 is a positive number.

In this case equation (0.5) simplifies to:

$$\mathbf{V(C'_w)} = \sigma^2 (\mathbf{A^T \cdot W \cdot A})^{-1} \qquad (0.7)$$

From eq (0.3), if $\sigma^2 = s^2_{norm}$, that is, is set equal to the normalisation factor in the weighting matrix, then this arbitrary factor cancels out both from $\mathbf{V(C'_w)}$

$$\mathbf{V(C'_w)} = (\mathbf{A^T}.(\sigma^2)^{-1}.\mathbf{A})^{-1} \qquad (0.8)$$

as well as from $\mathbf{C'_w}$, equation (0.4).

Another (unbiased) estimator of σ^2 is shown by Hunt [1981, appendix (iv)] to be the weighted group variance of the least-squares fit to the subdivision data:

$$\sigma'^2 = \frac{1}{(k-j)} (\mathbf{D_w} - \mathbf{A_w C'_w})^T \cdot (\mathbf{D_w} - \mathbf{A_w C'_w}) \qquad (0.9)$$

PTB, for instance, calculates σ^2 in this way [Schwartz 1992].
This different choice of σ^2 does not affect the least-squares estimate, $\mathbf{C'_w}$, equation (0.4). However, it does lead to a different estimate of the variance in the estimator in equation (0.7), since PTB at the same time as using an *a posteriori* value for σ^2 , uses a weighting matrix, **W**, based on the *a priori* balance variances used above (equ. (0.3)).

In the present work, this problem is tackled differently by calculating the variance estimates entirely *a posteriori* from the group variance of the fit separately for each of the three subdivision section associated with a different balance, as described in the next section. A recursive procedure is then applied to obtain a self-consistent fit to the actual measurements (section 3.2.2). Paule and Mandel [1989] have considered this approach more generally.

0.5 Weighings of equal variance

In those cases where all weighings are performed on a single balance and where one can assume that all weighings are statistically independent of each other and have a common variance σ^2 associated with the process then

$\mathbf{V(D)} = \sigma^2\ \mathbf{I}$, where **I** is the unit matrix, and

$$\mathbf{V(C')} = \sigma^2 (\mathbf{A^T}.\mathbf{A})^{-1} \qquad (0.10)$$

and the least-squares solution for the correction matrix is

$$\mathbf{C'} = (\mathbf{A^T}.\mathbf{A})^{-1}.\mathbf{A^T}.\mathbf{D} \qquad (0.11)$$

where $\mathbf{A^T}$ is the transpose of the design matrix, and $(\mathbf{A^T}.\mathbf{A})^{-1}$ is the inverse of the non-singular matrix $(\mathbf{A^T}.\mathbf{A})$.

In considering estimates of the uncertainties and inaccuracies in the corrections, an initial choice may be based on the square of the long run value of the standard deviation for the balance [Cameron *et al* 1977] (section 0.3). A second choice is to set the common variance σ^2 equal to the group variance of the residuals (observed minus fitted deflections) of the fit as given by :

$$g_v = \frac{1}{(k-j)} \sum_i R_i^2 \qquad (0.12)$$

where k is the total number of comparisons; j is the number of independent unknowns (corrections); and R_i is the residual for the i^{th} comparison [Cameron *et al* 1977].

No allowance is made here for the covariances in the solutions for the corrections, as given by the off-diagonal elements of **V(C')** which are non-zero because of the choice of a non-orthogonal design matrix.

It is usually possible with weighings of equal variance to choose an orthogonal design **A**, which leads to the minimum variance in the most efficient way [Grabe 1978, Hunt 1981, Chapman 1991, Ivarsson *et al* 1989].

It can also be noted that orthogonality is lost generally when weighting the design. It may be however that an underlying orthogonal design may still lead to the most efficient solution [Morris 1993].

1 Analysis of NPL weighing data [Lewis & Davidson 1990, 1992]

1.1 Weighing deflections

A FORTRAN 77 program was written (NPL.FOR in Appendix A.1) which read a data file containing all the weighing data, ordered by protocol number. An example of a data file is given in Appendix A.4, while a example of printout from a run of the program is given in Appendix A.2. A datafile containing all the results of running NPL.FOR on the protocols 1 to 32 was created from the program, and is given in Appendix A.3.

1.2 Air buoyancy corrections

The air density is calculated with the usual CIPM-recommended formula [Giacomo 1982] in a subroutine of program NPL.FOR, as a function of air pressure, temperature and humidity.

The volumes of the masses, including the rider, are used to compensate the weighing result for air buoyancy (*buoy* in NPL.FOR).

1.3 Equilibrium position calculation from turning points

1.3.1 Stanton balances

The centre of swing of the balance is calculated from the five turning points with the formula :

centre of swing rest point,

$$y(i) = \frac{1}{12}.(2y_1 + 3y_2 + 2y_3 + 3y_4 + 2y_5) \qquad (1.1)$$

1.3.2 Mettler balance

The rest points are given.

1.4 Deflections

Each deflection of the balance is converted to mass values, d_i, by multiplying the corresponding differences in rest point by the quoted balance sensitivity.

2 Least-squares calculation of mass corrections from deflections

A BASIC program, MATRIX.FOR, given in Appendix B.1, was written to perform a least-squares calculation of the mass corrections from the observed deflections and also calculate the associated uncertainties. In Appendix B.2 is given an example of the printout from a run of the program for all of the protocols 1 to 32, and yields values for the corrections to the masses using as standards the masses 200 g and 200'g. The design matrix A is given in Appendix B.3. The weighting matrix, W, used, and given in Appendix B.4, is based on equation (0.3) and the NPL-quoted standard deviations of the balances. A second fit is then performed with a weighting matrix based on the actual weighings at hand.

2.1 Restraints, residuals and goodness of fit

The true masses of 200 and 200', i.e.

$$c(200) = 1140{,}8\ \mu g \text{ and } c(200') = 1154{,}7\ \mu g$$

can be used as restraining values as described in section (0.2). Two least-squares fits have been performed here, with one restraint (200g) and with two restraints (200g and 200'g).

In order to judge which gives the better fit, the (unweighted) residuals of the fit are examined, as shown in figure 1 below.

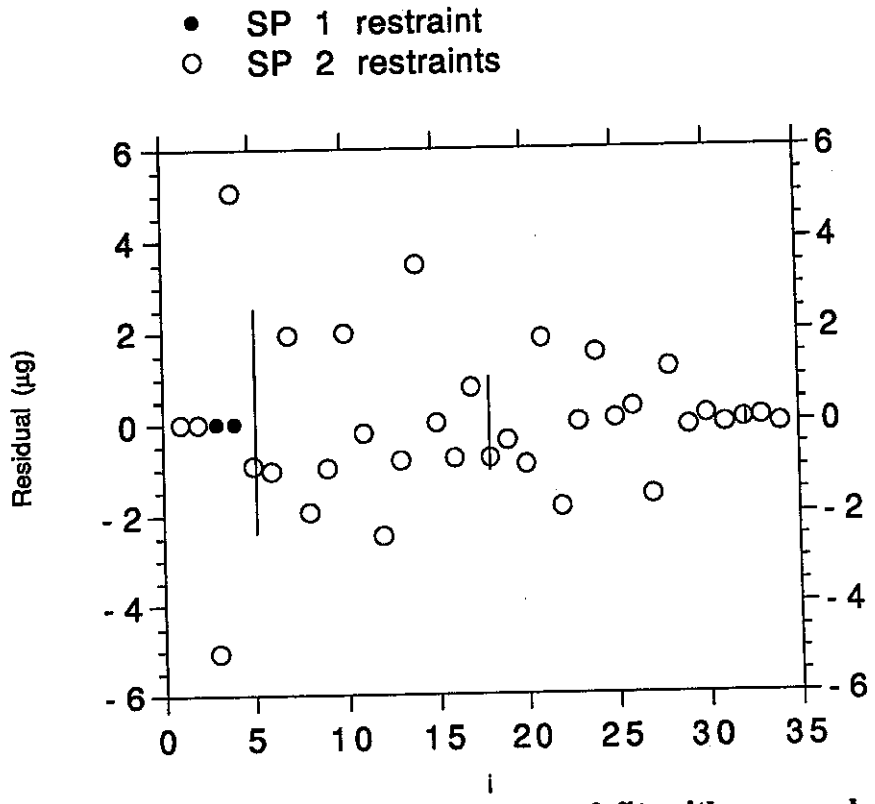

Figure 1 Plot of (unweighted) residuals of fit with one and two restraints

Also drawn in the figure are the bars indicating the quoted standard devition of each of the three balances used in the subdivision.

From figure 1, it is evident that the only effect of choosing two restraints instead of one is to antisymmetrically perturb the next two residuals in the subdivision fit:

With one restraint the weighing combinations With two restraints

200 = 100 + 100' residual d_2 = 0,0 µg residual d_3 = - 5,1 µg
200' = 100 + 100' residual d_3 = 0,0 µg residual d_4 = + 5,1 µg

One obtains the mass corrections listed in Appendix B.2.

To see explicitly the effect of this on the corrections:

With one restraint:

$c(200g) = 0$
$c(200g') = c(200g) - (5/6)d_2 + (5/6)d_3$
$c(100g) = (1/2)^* c(200g) - (5/12)d_2 + (1/6)d_4 + (1/6)d_5 + (1/12)d_6 - (1/12)d_7$

$c(100g') = (1/2)^* c(200g) - (5/12)d_2 - (1/6)d_4 - (1/6)d_5 - (1/12)d_6 + (1/12)d_7$
...
etc

(2.1)

Here d_2 and d_3 are very small.

With two restraints:

$c(200g) = 0$
$c(200g') = 0$
$c(100g) = (1/2)^* c(200g) - (25/12)d_3 - (25/12)d_4 + (1/6)d_5 + (1/6)d_6 + (1/12)d_7 - (1/12)d_8$
$c(100g') = (1/2)^* c(200g) - (25/12)d_3 - (25/12)d_4 - (1/6)d_5 - (1/6)d_6 - (1/12)d_7 + (1/12)d_8$
...
etc

(2.2)

Here $d_3 = - d_4$, and thus the perturbed residuals do not contribute.

The goodness of fit can be judged from the scatter of the residuals to be better with one restraint.

It should be noted that the results of the two weighings involving the two 200g weights (and assumed to be the standards for the purpose of this exercise) show poor agreement with the values assigned to these weights. This is because the data used here are only a part of the much larger set upon which their values are based [Davidson and Lewis 1992].

3 Discussion of uncertainties

In general, the correction c_j, for the j^{th} weight is :

$$c_j = \alpha_j c_r + \frac{1}{n_j} S_j - z_j - \rho_a \cdot (V_j - V_r)$$

where $\alpha_j = \frac{M_j}{M_r}$, is the quotient of the sum, M_j, of the nominal masses of the unknown weight of volume V_j and M_r, the nominal mass of the reference weight(s) of volume V_r; z_j is the rider correction; ρ_a, the air density; and where n_j is the number of deflections, d_i, in the sum

$$S_j = \Sigma_i \, a_{ij} \, d_i \tag{3.2}$$

The variance is

$$\sigma^2 (c_j) = \alpha_j{}^2 \sigma^2 (c_r) + V(S_j) + \sigma^2 (s) + \sigma^2 (z_j) \tag{3.3}$$

$$+M_j^2 [V(\rho_a)(\frac{1}{\rho_j} - \frac{1}{\rho_r})^2 + \rho_a^2 (\frac{V(\rho_r)}{\rho_r^4} + \frac{V(\rho_j)}{\rho_j^4})]$$

where σ^2 (s) is the variance associated with the balance sensitivity; and $V(\rho)$ is the variance in density ρ [Chapman 1991].

3.1 Variance of standards

Each standard has associated uncertainties quoted by the NPL as type A and type B [CIPM, Giacomo 1981] values, respectively 4,3 µg and 6,2 µg. These values are said by the NPL to correspond to a 95% confidence level where t=2,090. (Students factor for 20 degrees of freedom.)

According to the CIPM recommendation:

$$\sigma (c_r) = \sqrt{[(4,3/2.09)^2 + (6,2/2.09)^2]} = 3,61 \text{ µg}; \quad k=1$$

For each correction, the variance contribution from the standard(s) is propagated through the subdivision as $\alpha_j{}^2 \sigma^2 (c_r)$, as given in eq (3.3).

3.2 Weighing process variance

3.2.1 Prior balance performance

A first estimate of the weighing process uncertainties can be made from the long term, prior balance performance, as discussed in connection with equation (0.8):

$$V(S) = V(C'_W) = \sigma^2 (A^T.W.A)^{-1} \qquad (3.4)$$

where $\sigma^2 = s^2_{norm} = \Sigma\, s^2$ of the different balances, as given by the NPL on the basis of the long term balance performance. As a numerical example:

$V(100g) = 7{,}29\ (\mu g)^2 \times 0{,}292 = 2{,}13\ (\mu g)^2$

so that $\sqrt{V(100g)} = 1{,}46\ \mu g$.

This value will always be obtained with the given balance standard deviations provided the weighting matrix is normalised with $\sigma^2 = s^2_{norm}$.

PTB [Schwartz 1992] on the other hand chose s^2_{norm} to equal the variance of the first balance in the weighting matrix **W**, but then chose a "σ^2" outside the brackets on the RHS of eq. (3.4) to be the weighted group variance as given by eq (0.9). The value $V(100) = 8{,}35(\mu g)^2 \times 0{,}35 = 2{,}92\ (\mu g)^2 \Rightarrow \sqrt{V(100g)} = 1{,}71\ \mu g$.

3.2.2 Variance of fit and fit recursion

A second choice for σ^2 in equation (3.4) is that based on the group variance of the fit. The procedure adopted in the present work is to calculate σ^2 *a posteriori* for each section of the subdivision associated with a different balance in turn, based on equation (0.12) where $\sigma^2 = g_v$,

$$g_v = \frac{1}{(k-j)} \sum_i R_i^2$$

The group standard deviations of the three balances were found to be as given in the first row of the table below, whereas the quoted NPL balance standard deviations are given in the second row.

Table 1. Standard deviations of the balances

Mettler Me22	Stanton 20g	Stanton 200g
0,38 µg	1,88 µg	3,1 µg
0,2 µg	1,0 µg	2,5 µg

In order to use the current balance performance information, it is necessary to rerun the least-squares fitting with a new weighting matrix, using the new standard deviation estimates. Again, the choice of normalisation factor $\sigma^2 = s^2_{norm}$ is arbitrary. It is observed that this does not significantly change the least-squares estimate (maximum change of 0,1 µg on the estimate for the 10g weights) , thus indicating satisfactory convergence. The estimated uncertainty does however change for each weight. For example,

$V(100g) = 13{,}288(\mu g)^2 \times 0{,}139 = 1{,}85\ (\mu g)^2$

so that $\sqrt{V(100g)} = 1{,}35\ \mu g$.

The weighing process uncertainties given in Table 2 under the column $\sqrt{V(Sj)}$ are based on these converged, recursive fit values.

3.2.3 Individual weighing performance

A third possibility is to use the standard deviation observed on each weighing together with the usual formula for the propagation of errors.

In general $S_j = \Sigma_i\, a_{ij}\, d_i$ so that $V(S_j) = \Sigma_i\, (a_{ij})^2\, V(d_i)$

From eq (2.1), as an example,

$c(100g) = (1/2)^*\, c(200g) - (5/12)d_2 + (1/6)d_4 + (1/6)d_5 + (1/12)d_6 - (1/12)d_7$

so that

$V(100g) = (1/4) * V(200) + 0{,}42 * V(d_2) + 0{,}03 * V(d_4) + 0{,}03 * V(d_5) + 0{,}0069 * V(d_6) + 0{,}0069 * V(d_7)$.

Too few weighings were performed in order to estimate $V(d_i)$ for each individual weighing. If we instead take the respective balance performance (as given in Table 1), then one obtains

$V(100g) = 0{,}49 * (2{,}5)^2 = 3{,}06\ \mu g^2 \Rightarrow \sqrt{V(100g)} = 1{,}75\ \mu g$, long-term
$V(100g) = 0{,}49 * (3{,}1)^2 = 4{,}71\ \mu g^2 \Rightarrow \sqrt{V(100g)} = 2{,}18\ \mu g$, fit variance

3.3 Sensitivity weight variance

The variance associated with the uncertainty in weighing the sensitivity weight used for balance calibration is given by :

$$\sigma^2(s) = \frac{v(\delta)}{n_j} \cdot \frac{MS(d)}{\delta^2} \tag{3.4}$$

where $v(\delta)$ is the variance in the mass, δ, of the sensitivity weight; and MS(d) is the mean-square sum of the deflections, d_i:

$$MS(d) = \frac{1}{n_j} \cdot \sum_i d_i^2 \tag{3.5}$$

From the quoted uncertainty in the sensitivity we obtain, for example, for the Stanton HP40 balance:

$$\frac{v(\delta)}{\delta^2} = \left(\frac{0,005}{0,628}\right)^2 \tag{3.6}$$

Equ (3.4) is evaluated for the various balances (a relative sensitivity uncertainty (2s) of 0,0006 is assumed for the Mettler balance), and for each balance the standard deviation from sensitivity variance was found to be:

Mettler Me22	Stanton 20g	Stanton 200g
.01 µg	.30 µg	.23 µg

Summing the variances obtained for the balances, one obtains an overall sensitivity standard deviation for the subdivision of .38 µg

3.4 Rider variance

For those weighings performed with a rider.

The rider correction to each weighing

$$z_i = (p_z/div).m_z$$

where p_z is the rider position, m_z the rider mass, and div the number of divisions on the rider bar.

The associated variance in the rider correction is thus

$$\sigma^2(z_j) = \sigma^2(p_z).(m_z/div)^2 + \sigma^2(m_z).(p_z/div)^2$$

As given,

$m_z = 1020\ \mu g$, div = 20 bar units

$\sigma^2(p_z) = (0{,}001)^2$ (bar units)^2 and $\sigma^2(m_z) = (1{,}0/2)^2\ (\mu g)^2$.

The largest rider correction for the Stanton 200g balance is $p_z = 3{,}0$ bar units (no 31); the corresponding variance is

$$\sigma^2(z_j) = 351 \times 10^{-4}\ (\mu g)^2$$

$$\sigma(z_j) = 0{,}19\ \mu g$$

The corresponding value for the Stanton 20g balance is $\sigma(z_j) = 0{,}08\ \mu g$.

3.5 Air buoyancy variance, $\sigma^2(buoy)$

The given values for the weight, j, for example, 100g and reference, r, 200 g, are (at k=2):

$\rho_a = 1{,}185\ kg.m^{-3}$ $\sigma(\rho_a) = 1\ g.m^{-3}$ (assumed here)

$\rho_j = 7827\ kg.m^{-3}$; $\sigma(\rho_j) = 2\ kg.m^{-3}$

$\rho_r = 7853\ kg.m^{-3}$; $\sigma(\rho_r) = 2\ kg.m^{-3}$

Inserting values for the variance in air density and the variances in the weight volumes into the last terms on the RHS of equation (3.3), one obtains for these contributions, respectively:

Air density : 3,0 μg Weight volumes: 7,9 μg, all at k=1.

Uncertainties in the air density determination may be dominated by calibration uncertainties in the various measuring instruments employed, in which case a large part of their contributions will be correlated in the same way for all weighings in the subdivision.

Secondly, the uncertainty quoted in the reference mass may already include air buoyancy uncertainties, in which case one has to be careful not to calculate the same terms twice.

3.6 Numerical examples

Explicit expressions for $\sigma(c_j)$, the combined uncertainty in mass correction c_i as given by equation (3.3), may be found in the BASIC program 'MATRIX' reproduced in Appendix B.1. Some examples of numerical values of the various uncertainties (where the numbers in parentheses indicate the appropriate coverage factor, k, or corresponding confidence limit factor, in units of standard deviation):

		2nd iter.	1st iter.				
	$\alpha_j\sigma(c_r)$	$\sqrt{V(Sj)}$	$\sqrt{V(Sj)}$	$\sigma(s)$	σ(buoy)	$\sigma(z)$	$\sigma(c_j)$
Weight	(μg)	(μg)	(μg)	(μg)	(μg)	(μg)	(μg)
200g	3,60	–	–	–	–	–	7,20
200'g	3,60(1)	3,25 (1)	3,49(1)	0,23(1)	0	0,19(1)	9,73(2)
100g	1,80	1,36	1,46	0,23	0	0,19	4,55
100'g	1,80	1,36	1,46	0,23	0	0,19	4,55
50g	0,90	1,20	1,28	0,23	0	0,19	3,05
50'g	0,90	1,20	1,28	0,23	0	0,19	3,05
20g	0,36	0,78	0,74	0,30	0	0,14	1,84
20'g	0,36	0,78	0,74	0,30	0	0,14	1,84
10g	0,18	0,65	0,53	0,30	0	0,14	1,50
10'g	0,18	0,65	0,53	0,30	0	0,14	1,50
5g	0,09	0,68	0,51	0,30	0	0,08	1,52
5'g	0,09	0,68	0,51	0,30	0	0,08	1,52
2g	0,04	0,37	0,26	0,15	0	0	0,81
2'g	0,04	0,37	0,26	0,15	0	0	0,81
1g	0,02	0,22	0,16	0,01	0	0	0,45
1'g	0,02	0,22	0,16	0,01	0	0	0,45

Table 2. Numerical examples of various uncertainty contributions

(one fixed restraint, 200g)

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Appendix A.1

Fortran 77 program for calculation of deflections

```
c Name:     NPL.FOR
c Description: Calculates NPL weighings A:B:B:A
c Author: 910415 Leslie Pendrill
c Changed: 920312 LRP
c*************************************************************

      Program NPL

      Dimension x(100,100),y(100),z(100)

*************************************************************
c Assign input data file

      OPEN (Unit=10, File='[leslie.neddel.data]NPL5050.dat',
     1       Status='Old')

c READ DATAFILE

c INPUT facts in weighings

      Read(10,*) TT
      Write(6,*) 'Weighing No', TT

c Nominal masses (g)

      Read(10,*) Sleft
      Read(10,*) Sright1
      Read(10,*) Sright2
      Read(10,*) Sright3

c Volumes of masses (cm3)

      Read(10,*) vleft
      Read(10,*) vright

c Volume difference of weights in m3

      V= (vright - vleft)*1E-6

c Balance sensitivity (µg/unit)

      Read(k,*) BS

c Rider mass (µg)

      Read(k,*) zm
```

```
c Rider density (kg/m3)

      zd = 8E3

c Input air parameter values

      read(k,*) TRYCK,temp,humid
      humid = humid/1E2

      DO 10 i=1,4
           DO 5 j=1,5
                Read(k,*) x(i,j)
 5         continue

c rest point calculation (units)

      y(i) = (2*x(i,1) + 3*x(i,2) + 2*x(i,3) + 3*x(i,4) + 2*x(i,5))/12E0

c rider position (div)

      Read(k,*) z(i)

 10   continue

c Rider volume (m3)

      zv =(((z(2)-z(1)) + (z(3)-z(4)))/4E0)*zm*(1E-9)/(1E1*zd)

c CO2 concentration

      coc=.0004

c Air buoyancy (µg)

      CALL densi(TRYCK,temp,humid,coc,dens)

      buoy = (V+zv)*dens*1E9

c*************************************************************
c Mean B - A (µg)
      d = -(((y(2) - y(1)) + (y(3) - y(4)))/4E0)*BS
     dd = d - (((z(2)- z(1)) + (z(3) - z(4)))/4E0)*zm/1E1


c RESULTS

      write(6,*) 'Air density=',dens,'kg/m3'
      write(6,*) 'Mean weight diff=',dd,'µg'
      write(6,*) 'Air compensated=',dd - buoy,'µg'
     write(50,*) TT, dens, dd, dd - buoy

 15  continue
```

```
      stop
      end

      SUBROUTINE densi(TRYCK,temp,humid,coc,dens)
c*************************************************************
c
c Beskrivning: Beräkning av luftens tryck och densitet
c Författare: Leslie Pendrill 870421
c Ändrat: 910813
c
c*************************************************************

      Implicit Double Precision (A-H,O-Z)

      DOUBLE PRECISION press,humid,psv1,f1,Xv
     1 ,y1,y2,y3,ZZ,TRYCK,temp,tempT,coc,dens,A,B,C

c*************************************************************

c air pressure in Pa
      press=TRYCK*100
c Absolute temperatures
      tempT=temp + 273.15
c Water vapour
      A=1.2811805*1.D-5
      B=-1.9509874*1.D-2
      C=34.04926034D0
      D=-6.3536311*1.D3
c Enhancement factors
      f1=1.00062 + press*(3.14*(1.D-8)) +
     1 (temp**2)*(5.6*(1.D-7))
c Saturated vapour pressures in Pa
      psv1=DEXP(A*(tempT**2) + B*tempT + C + D/tempT)

c Xv
      Xv= humid*f1*psv1/press
c compressibility of air
      y1=1.62419*(1.D-6) - (2.8969*(1.D-8))*temp
     1 + (1.088*(1.D-10))*(temp**2)
      y2=Xv*(5.757*(1.D-6) - (2.589*(1.D-8))*temp)
      y3=(Xv**2)*(1.9297*(1.D-4) - (2.285*(1.D-6))*temp)
      ZZ= 1- press/tempT*(y1+y2+y3) + ((press/tempT)**2)
     1    *(1.73*(1.D-11) - (Xv**2)*1.034*(1.D-8))

c AIR DENSITY in kg.m-3
      dens=press/(ZZ*tempT)*(1-.378*Xv)*(3.48353
     1 + 1.44*(coc-.0004))*(1.D-3)

      return
      end
```

Appendix A.2

Example of printout from a run of program NPL.FOR

```
$ run npl
Weighing No  7.000000
Air density=  1.192671   kg/m3
Mean weight diff= -75.96875   µg
Air compensated= -75.98083   µg

FORTRAN STOP
```

Appendix A.3

Output of NPL.FOR for all weighing protocols

Prot no.	Air dens (kg/m3)	Diff (µg)	Corr diff (µg)
1	1,183246	181,77	81,21
2	1,185886	185,76	84,99
3	1,185886	105,81	105,82
4	1,185634	105,91	105,93
5	1,185071	45,87	70,77
6	1,184702	-55,14	-30,26
7	1,184038	29,60	29,60
8	1,181504	26,63	26,63
9	1,172263	0,46	0,46
10	1,169217	-13,51	-13,51
11	1,171721	13,46	13,46
12	1,169875	-31,87	-31,87
13	1,199120	-1,56	-1,56
14	1,196229	-0,79	-0,79
15	1,194567	14,14	14,14
16	1,212538	17,25	17,25
17	1,209900	12,80	12,80
18	1,199579	13,33	13,33
19	1,198566	7,50	7,50
20	1,197790	-1,21	-1,21
21	1,195709	2,18	2,18
22	1,194817	0,66	0,66
23	1,193707	-72,69	-72,70
24	1,186748	-65,76	-65,77
25	1,185980	-61,66	-61,67
26	1,192671	-75,97	-75,98
27	1,180961	-7,00	-7,00
28	1,180961	-7,25	-7,25
29	1,180747	-48,35	-48,35
30	1,180534	-41,35	-41,35
31	1,180534	9,25	9,25
32	1,180534	9,40	9,40

Appendix A.4

Example of weighing data file for protocol 28

28	- *Protocol no.*
100	- *Nominal mass LH (g)*
50	- *Nominal mass RH1 (g)*
50	- *Nominal mass RH2 (g)*
0	- *Nominal mass RH3 (g)*
12.778	- *Volume LH (cm^3)*
12.757	- *Volume RH (cm^3)*
0.620	- *Sensitivity (µg/unit)*
1022.8	- *Rider value (µg)*
1010.95	- *Air pressure (mb)*
23.0	- *Temperature (°C)*
36	- *Humidity (%)*
4752	
5433	
4762	- *Turning points*
5423	
4772	
1.2	- *Rider position*
4523	
5588	
4541	
5563	
4557	
0.6	
4607	
5498	
4622	
5488	
4637	
0.6	
4837	
5393	
4846	
5388	
4856	
1.2	

Appendix B.1

BASIC program MATRIX.BAS for calculating the least-squares solution for the mass corrections and related uncertainties from the deflections

```
OPTION SIZE = REAL DOUBLE

Print " Name: MATRIX.BAS                    "
Print " PROGRAM MATRIX CALCULATION OF MASS DIFFERENCES"
Print " Created: 1991 04 26   Author: LESLIE PENDRILL"
Print " Changed: 1993 02 26 LRP"


PRINT " DESIGN MATRIX DIMENSION? INPUT M(ROWS) &
N(COLUMNS)"
M = 33
N = 16

DIM A(M,N), AT(N,M), T(N,N), D(N,N), C(N,M)
DIM DM(M,1), S(N,1), R(M,1), VF(N,1), W(M,M)
DIM TW(N,N), DW(N,N), CW(N,M), SW(N,1), WA(M,N), WDM(M,1)

print "design.dat"
OPEN 'DESIGN200.DAT' for input as file #1

PRINT "INPUT DESIGN MATRIX"
MAT INPUT #1, A(M,N)
CLOSE #1

PRINT "__DESIGN_____________________________________________________"

MAT AT = TRN(A)

MAT T = AT*A
PRINT "__ATA_____________________________________________________"

print "weighting matrix WEIGHT.DAT"
OPEN 'WEIGHT200.DAT' for input as file #1

PRINT "INPUT WEIGHTING MATRIX"
MAT INPUT #1, W(M,M)
CLOSE #1

MAT WA = W*A

MAT TW = AT*WA
```

```
MAT D = INV(T)
PRINT "__ATA-1_____"
PRINT "MAT PRINT D;"
MAT DW = INV(TW)
PRINT "Variance-covariance matrix - weighted"
MAT PRINT DW;

MAT C = D*AT
PRINT "__C___Unweighted_____"


MAT CW = DW*AT
PRINT "__C___Weighted_____"
MAT PRINT CW,

print "data DATANPL.DAT"
OPEN 'DIFNPL200.DAT' for input as file #1

Print "Input mass differences (ug)"
MAT INPUT #1, DM(M,1)
CLOSE #1


print
PRINT  "calculate least-squares solution (ug)"
Print "unweighted"
MAT S=C*DM

print

Print "weighted"

MAT WDM = W*DM
MAT SW=CW*WDM

print
MAT PRINT SW,
Print

Print " UNCERTAINTY CALCULATIONS"

Print "Weighing process calculation"
Print "s2norm (μg)2"
gv = 7.3

for j=1 to N
VF(j,1) = gv*DW(j,j)
next j

MAT PRINT VF;
```

```
Print "Group variance"
Print "Residuals of fit"

MAT R = A*S
MAT R = R - DM

gv1 = 0
Print "Stanton 200g"
for k=2 to 13
 gv1 = gv1 + R(k,1)**2
next k
gv1 = (1/(12-9))*gv1

print  Sqr(gv1)

gv2 = 0
Print "Stanton 20g"
for k=14 to 27
 gv2 = gv2 + R(k,1)**2
next k
gv2 = (1/(14-9))*gv2

print Sqr(gv2)

gv3 = 0
Print "Mettler Me22"
for k=28 to 33
 gv3 = gv3 + R(k,1)**2
next k
gv3 = (1/(6-3))*gv3

print Sqr(gv3)

Print "Mean square deflection (μg)Σ"
Print "Stanton 200"

msd=0
for j=2 to 13
msd=msd+(dm(j,1))**2
next j
 vd = (0.0025/0.628)**2
msd1 = msd*vd/14
print sqr(msd1)

msd=0
Print "Stanton 20g"
for j=14 to 27
msd=msd+(dm(j,1))**2
next j
 vd = (0.001/0.125)**2
msd2 = msd*vd/14
print sqr(msd2)
```

```
 msd=0
Print "Mettler Me22"
 for j=28 to 33
 msd=msd+(dm(j,1))**2
next j
 vd = (0.0003)**2
msd3=msd*vd/6
print sqr(msd3)

Print "Variance in sensitivity"
msd = msd1 + msd2 + msd3
print msd; sqr(msd)

end
```

Appendix B.2

Example of printout from run of program MATRIX.FOR for the weighing protocols 32 to 1

$ run matrix
Name: MATRIX.BAS
PROGRAM MATRIX CALCULATION OF MASS DIFFERENCES
Created: 1991 04 26 Author: LESLIE PENDRILL
Changed: 1991 09 06 LRP
DESIGN MATRIX DIMENSION? INPUT M(ROWS) & N(COLUMNS)
design.dat
INPUT DESIGN MATRIX
__DESIGN_____
__ATA_____
weighting matrix WEIGHT.DAT
INPUT WEIGHTING MATRIX
__ATA-1_____
Variance-covariance matrix - weighted
__C___Unweighted_____
__C___Weighted_____
data DATANPL.DAT
Input mass differences (ug)

calculate least-squares solution (ug)

One restraint, 200g, weighted $\Sigma i\ \sigma_i^2$

c_j (2) (μg)	c_j (1) (μg)	Mass (g)
1140.8	1140.8	*200*
1144.58	1144.58	*200'*
582.247	582.247	*100*
477.343	477.343	*100'*
269.086	269.086	*50*
240.459	240.459	*50'*
107.413	107.413	*20*
108.975	108.975	*20'*
52.5696	52.454	*10*
39.9294	40.045	*10'*
22.6406	22.6406	*5*
20.4639	20.4639	*5'*
23.7129	23.7129	*2*
30.8129	30.8129	*2'*
40.7319	40.7317	*1*
31.381	31.3812	*1'*

Variance-covariance matrix - unweighted - one restraint

1 1 .5 .5 .25 .25 .1 .1 .05 .05 .025
.025 .01 .01 .005 .005

1 3 1 1 .5 .5 .2 .2 .1 .1 .05 .05
.02 .02 .01 .01

.5 1 .6 .4 .25 .25 .1 .1 .05 .05 .025
.025 .01 .01 .005 .005

.5 1 .4 .6 .25 .25 .1 .1 .05 .05 .025
.025 .01 .01 .005 .005

.25 .5 .25 .25 .333333 .166667 .1 .1 .05 .05 .025
.025 .01 .01 .005 .005

.25 .5 .25 .25 .166667 .333333 .1 .1 .05 .05 .025
.025 .01 .01 .005 .005

.1 .2 .1 .1 .1 .1 .2 0 0 0 0 0 0 0 0 0

.1 .2 .1 .1 .1 .1 0 .2 0 0 0 0 0 0 0 0

.05 .1 .05 .05 .05 .05 0 0 .171429 .285714E-01 .05 .05
.02 .02 .01 .01

.05 .1 .05 .05 .05 .05 0 0 .285714E-01 .171429 .05 .05
.02 .02 .01 .01

.025 .05 .025 .025 .025 .025 0 0 .05 .05 .233333
.666667E-01 .06 .06 .03 .03

.025 .05 .025 .025 .025 .025 0 0 .05 .05 .666667E-01 .233333
.06 .06 .03 .03

.01 .02 .01 .01 .01 .01 0 0 .02 .02 .06 .06 .184
-.016 -.008 -.008

.01 .02 .01 .01 .01 .01 0 0 .02 .02 .06 .06 -.016
.184 -.008 -.008

.005 .01 .005 .005 .005 .005 0 0 .01 .01 .03 .03 -.008
-.008 .179333 .126667E-01

.005 .01 .005 .005 .005 .005 0 0 .01 .01 .03 .03 -.008
-.008 .126667E-01 .179333

Variance-covariance matrix - weighted - one restraint

.0001 .0001 .00005 .00005 .000025 .000025 .00001 .00001 .000005 .000005 .25E-05 .25E-05 .000001 .000001 .5E-06 .5E-06

.0001 1.66677 .416717 .416717 .208358 .208358 .833433E-01 .833433E-01 .416717E-01 .416717E-01 .208358E-01 .208358E-01 .833433E-02 .833433E-02 .416717E-02 .416717E-02

.00005 .416717 .291692 .125025 .104179 .104179 .416717E-01 .416717E-01 .208358E-01 .208358E-01 .104179E-01 .104179E-01 .416717E-02 .416717E-02 .208358E-02 .208358E-02

.00005 .416717 .125025 .291692 .104179 .104179 .416717E-01 .416717E-01 .208358E-01 .208358E-01 .104179E-01 .104179E-01 .416717E-02 .416717E-02 .208358E-02 .208358E-02

.000025 .208358 .104179 .104179 .225701 .868118E-01 .625025E-01 .625025E-01 .312512E-01 .312512E-01 .156256E-01 .156256E-01 .625025E-02 .625025E-02 .312512E-02 .312512E-02

.000025 .208358 .104179 .104179 .868118E-01 .225701 .625025E-01 .625025E-01 .312512E-01 .312512E-01 .156256E-01 .156256E-01 .625025E-02 .625025E-02 .312512E-02 .312512E-02

.00001 .833433E-01 .416717E-01 .416717E-01 .625025E-01 .625025E-01 .747727E-01 .473754E-01 .236877E-01 .236877E-01 .118439E-01 .118439E-01 .473754E-02 .473754E-02 .236877E-02 .236877E-02

.00001 .833433E-01 .416717E-01 .416717E-01 .625025E-01 .625025E-01 .473754E-01 .747727E-01 .236877E-01 .236877E-01 .118439E-01 .118439E-01 .473754E-02 .473754E-02 .236877E-02 .236877E-02

.000005 .416717E-01 .208358E-01 .208358E-01 .312512E-01 .312512E-01 .236877E-01 .236877E-01 .038396 .012689 .127712E-01 .127712E-01 .51085E-02 .51085E-02 .255425E-02 .255425E-02

.000005 .416717E-01 .208358E-01 .208358E-01 .312512E-01 .312512E-01 .236877E-01 .236877E-01 .012689 .038396 .127712E-01 .127712E-01 .51085E-02 .51085E-02 .255425E-02 .255425E-02

.25E-05 .208358E-01 .104179E-01 .104179E-01 .156256E-01 .156256E-01 .118439E-01 .118439E-01 .127712E-01 .127712E-01 .349244E-01 .120934E-01 .940356E-02 .940356E-02 .470178E-02 .470178E-02

.25E-05 .208358E-01 .104179E-01 .104179E-01 .156256E-01 .156256E-01 .118439E-01 .118439E-01 .127712E-01 .127712E-01 .120934E-01 .349244E-01 .940356E-02 .940356E-02 .470178E-02 .470178E-02

.000001 .833433E-02 .416717E-02 .416717E-02 .625025E-02 .625025E-02 .473754E-02 .473754E-02 .51085E-02 .51085E-02 .940356E-02 .940356E-02 .989913E-02 .880204E-02 .440102E-02 .440102E-02

.000001 .833433E-02 .416717E-02 .416717E-02 .625025E-02 .625025E-02 .473754E-02

.473754E-02 .51085E-02 .51085E-02 .940356E-02 .940356E-02 .880204E-02 .989913E-02
.440102E-02 .440102E-02

.5E-06 .416717E-02 .208358E-02 .208358E-02 .312512E-02 .312512E-02 .236877E-02
.236877E-02 .255425E-02 .255425E-02 .470178E-02 .470178E-02 .440102E-02 .440102E-02
.342128E-02 .207683E-02

.5E-06 .416717E-02 .208358E-02 .208358E-02 .312512E-02 .312512E-02 .236877E-02
.236877E-02 .255425E-02 .255425E-02 .470178E-02 .470178E-02 .440102E-02 .440102E-02
.207683E-02 .342128E-02

UNCERTAINTY CALCULATIONS
Weighing process calculation
s2norm (μg)2

2 nd interation	1 st iteration
.00073	.00073
10.5804	12.1674
1.85163	2.12935
1.85163	2.12935
1.43271	1.64761
1.43271	1.64761
.603276	.545841
.603276	.545841
.417188	.28029
.417188	.28029
.46645	.254948
.46645	.254948
.135898	.722637E-01
.135898	.722637E-01
.476208E-01	.249753E-01
.476208E-01	.249753E-01

Group variance
Residuals of fit
Stanton 200g
3,1 μg
Stanton 20g
1,88 μg
Mettler Me22
0,38 μg

Mean square deflection (μg)Σ
Stanton 200
.226009
Stanton 20g
.303067
Mettler Me22
.805267E-02
Variance in sensitivity
.142994 .378146

Appendix B.3

Design matrix with one restraint, 200g

1,0,0,0,0,0,0,0,0,0,0,0,0,0,0,0,
1,0,-1,-1,0,0,0,0,0,0,0,0,0,0,0,
0,1,-1,-1,0,0,0,0,0,0,0,0,0,0,0,
0,0,1,-1,0,0,0,0,0,0,0,0,0,0,0,
0,0,1,-1,0,0,0,0,0,0,0,0,0,0,0,
0,0,1,0,-1,-1,0,0,0,0,0,0,0,0,0,
0,0,0,1,-1,-1,0,0,0,0,0,0,0,0,0,
0,0,0,0,1,-1,0,0,0,0,0,0,0,0,0,
0,0,0,0,1,-1,0,0,0,0,0,0,0,0,0,
0,0,0,0,1,0,-1,-1,-1,0,0,0,0,0,0,
0,0,0,0,0,1,-1,-1,0,-1,0,0,0,0,0,
0,0,0,0,1,0,-1,-1,0,-1,0,0,0,0,0,
0,0,0,0,0,1,-1,-1,-1,0,0,0,0,0,0,
0,0,0,0,0,0,1,-1,0,0,0,0,0,0,0,
0,0,0,0,0,0,1,-1,0,0,0,0,0,0,0,
0,0,0,0,0,0,1,0,-1,-1,0,0,0,0;0,0,
0,0,0,0,0,0,0,1,-1,-1,0,0,0,0,0,
0,0,0,0,0,0,0,0,1,-1,0,0,0,0,0,
0,0,0,0,0,0,0,0,1,-1,0,0,0,0,0,
0,0,0,0,0,0,0,0,1,0,-1,-1,0,0,0,
0,0,0,0,0,0,0,0,0,1,-1,-1,0,0,0,
0,0,0,0,0,0,0,0,0,1,-1,0,0,0,0,
0,0,0,0,0,0,0,0,0,0,1,0,-1,-1,-1,0,
0,0,0,0,0,0,0,0,0,0,0,1,-1,-1,0,-1,
0,0,0,0,0,0,0,0,0,0,1,0,-1,-1,0,-1,
0,0,0,0,0,0,0,0,0,0,0,1,-1,-1,-1,0,
0,0,0,0,0,0,0,0,0,0,0,0,1,-1,0,0,
0,0,0,0,0,0,0,0,0,0,0,0,1,-1,0,0,
0,0,0,0,0,0,0,0,0,0,0,0,1,0,-1,-1,
0,0,0,0,0,0,0,0,0,0,0,0,0,1,-1,-1,
0,0,0,0,0,0,0,0,0,0,0,0,0,0,1,-1,
0,0,0,0,0,0,0,0,0,0,0,0,0,0,1,-1,

Buffer: DESIGN.DAT

Appendix B.4

Weighting matrix with one restraint, 200g.

$ type weight200.dat

1	10000	10000
2 - 13	1,2	1,38
14 - 27	7,3	3.76
28 - 33	182,3	92.28