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EXAMPLE / CALIBRATION of WEIGHT MASSES

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```
clear
close all
```

CALIBRATION of WEIGHT MASSES

Problem specified by Jaroslav ZUDA / Czech Metrology Institute (CMI) E-MAIL: [jzuda@cmi.cz](mailto:jzuda@cmi.cz) Related EMPIR Project: Improvement of the realisation of the mass scale Short Name: RealMass, Project Number: 19RPT02

The fundamental challenge faced by any primary laboratory is ensuring the accurate calibration of weights. While the procedure for routine calibration of weights of lower accuracy classes is well-documented in OIML R111, procedures for calibration at the highest level are not as extensively covered. This issue is addressed by the RealMass project, which aims to develop such procedures.

Calibration at the highest level often requires the use of the split method for weight units. This procedure involves deriving individual weights from one or more reference weights using a measurement system with known weight values. Typically, these reference weights are prototypes made of a Pt and Ir alloy, although some institutes may use stainless steel weights. The weight is then calculated using the least squares method.

A significant challenge in accurate weight measurement is accounting for buoyancy forces. To address this, it is necessary to determine the density of the surrounding medium, which affects either the density or the volume of the weights. Additionally, the temperature of the environment must be considered since the volume of the weights changes with temperature.

There are various methods for determining the volume of weights, such as measuring the apparent weight in a liquid or comparing the apparent masses with a known volume body in the liquid. In recent years, there has been progress in measuring weights in a vacuum, particularly in light of the new definition of the unit of weight. However, measuring in a vacuum presents challenges due to surface phenomena on the weights. Therefore, it is often more practical to measure at low pressure, approximately corresponding to 50% atmospheric pressure, where surface changes are negligible.

Measurements in air and at low pressure can effectively determine both the volume and weight of weights. Comparisons indicate that this method yields results comparable to determining volume in a liquid. Consequently, there is interest in whether low-pressure measurement can be utilized for current weight and volume determination using the split method for weight units.

Load data

```
load EXAMPLE_MassCalibration.mat
```

Function of implicate restrictions on the model parameters

```
% function fun = funMassCalibration(mu,beta)
% % funMassCalibration - defines the necessary restrictions on the EIV model
% % parameters for the calibration of weight masses.
% %
% % funMassCalibration is an anonymous function that takes arguments mu and
% % beta and returns fun, a q-dimensional column vector of constraints.
% % Ideally, these constraints should equal zero. In this context, mu can be
% % specified as either an m-dimensional column vector or as a cell array
% % with one column vector {mu}, while beta is a p-dimensional vector. The
% % output fun from funMassCalibration(mu, beta) represents the q
% % constraints on the model parameters mu and beta. If the model
% % restrictions are satisfied, fun = funMassCalibration(mu, beta) = 0.
% %
% % Variable related to the vector parameter mu:
% %
% % mu(1) = X1 = X1R_1 % comparison measurement: M1 - MR in environment 1
% % mu(2) = X2 = X2R_1 % comparison measurement: M2 - MR in environment 1
% % mu(3) = X3 = X21_1 % comparison measurement: M2 - M1 in environment 1
% % mu(4) = X4 = X1R_2 % comparison measurement: M1 - MR in environment 2
% % mu(5) = X5 = X2R_2 % comparison measurement: M2 - MR in environment 2
% % mu(6) = X6 = X21_2 % comparison measurement: M2 - M1 in environment 2
% % mu(7) = X7 = X1R_3 % comparison measurement: M1 - MR in environment 3
% % mu(8) = X8 = X2R_3 % comparison measurement: M2 - MR in environment 3
% % mu(9) = X9 = X21_3 % comparison measurement: M2 - M1 in environment 3
% % mu(10) = dMR      % difference from nominal of the reference weight
% % mu(11) = VR       % volume of the reference weight
% % mu(12) = G        % gravitational ratio
```

```

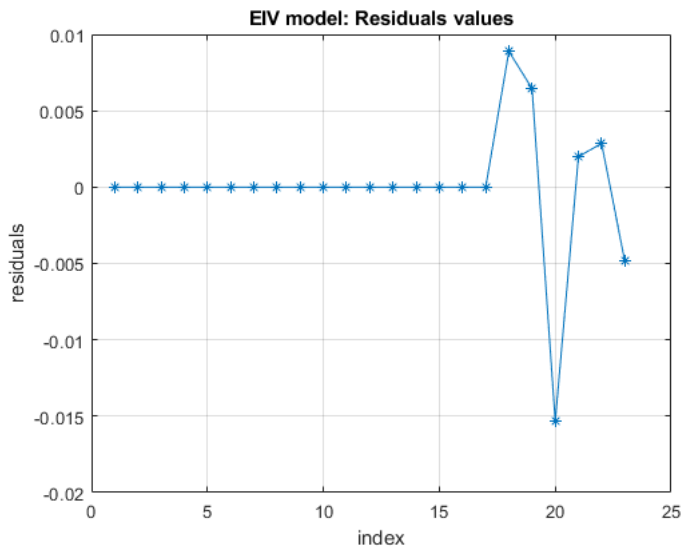
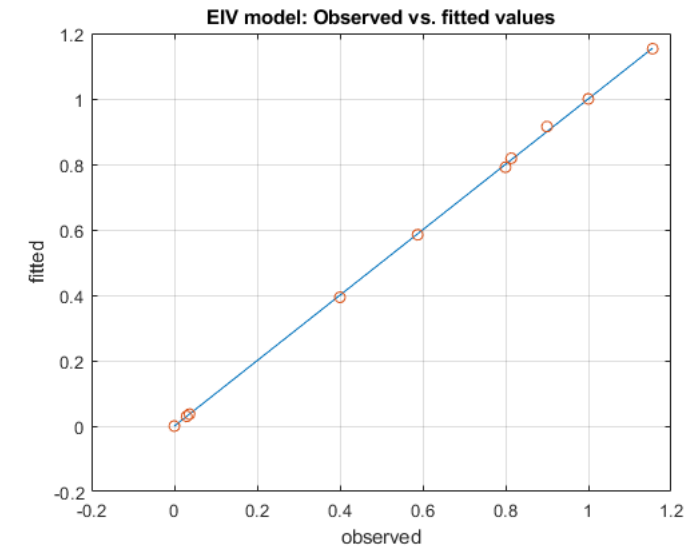
%% mu(13) = h1 = hR      % center of gravity of the reference weight
%% mu(14) = h2 = h1      % center of gravity of the measured weight 1
%% mu(15) = h3 = h2      % center of gravity of the measured weight 2
%% mu(16) = K             % adjustment constant
%% mu(17) = alpha         % coefficient of thermal expansion
%% mu(18) = dT1 = dT_1    % temperature difference T_1 - 20 in environment 1
%% mu(19) = dT2 = dT_2    % temperature difference T_2 - 20 in environment 2
%% mu(20) = dT3 = dT_3    % temperature difference T_3 - 20 in environment 3
%% mu(21) = rho1 = rho_1  % density of air in environment 1
%% mu(22) = rho2 = rho_2  % density of air in environment 2
%% mu(23) = rho3 = rho_3  % density of air in environment 3
%%
%% Variable related to the vector parameter beta:
%%
%% beta(1) = dM1          % difference from nominal of the measured weight 1
%% beta(2) = dM2          % difference from nominal of the measured weight 2
%% beta(3) = V1           % volume of the measured weight 1
%% beta(4) = V2           % volume of the measured weight 2
%%
%% EXAMPLE
%% data = [-9.7550e-08 -9.0350e-08 6.3200e-09 -4.7849e-07 9.4810e-08 ...
%%         3.8381e-07 -3.2604e-07 -9.3040e-08 2.3331e-07 6.4100e-07 ...
%%         1.2544e-04 3.0000e-07 3.6500e-02 3.6500e-02 3.0000e-02 ...
%%         9.9986e-01 4.8000e-05 8.0000e-01 4.0000e-01 9.0000e-01 ...
%%         1.1552e+00 5.8770e-01 8.1380e-01]';
%% ux = [ 5.0000e-10 3.0000e-10 2.0000e-10 3.0000e-10 3.0000e-10 ...
%%        3.0000e-10 2.0000e-10 3.0000e-10 2.0000e-10 4.0000e-08 ...
%%        1.0000e-09 3.0000e-08 5.0000e-04 5.0000e-04 5.0000e-04 ...
%%        5.0000e-06 2.0000e-06 1.0000e-01 1.0000e-01 1.0000e-01 ...
%%        5.0000e-04 5.0000e-04 5.0000e-04]';
%% U = diag(ux.^2);
%% fun = @(mu,beta) funMassCalibration(mu,beta)
%% mu0 = data;
%% beta0 = [mu0(10) mu0(11) mu0(10) mu0(11)]';
%% clear options
%% options.method = 'oeffpils2';
%% options.criterion = 'function';
%% options.numDiffMethod = 'LynnesMoller';
%% options.q = 9;
%% options.tol = 1e-15;
%% result = OEFFPIL(data,U,fun,mu0,beta0,options);
%%
%% Viktor Witkovsky (witkovsky@savba.sk)
%% Ver.: 17-Feb-2024 13:16:08
%%
%% CHECK THE INPUTS AND OUTPUTS
%%
%% if iscell(mu)
%%     mu = mu{1};
%% end
%%
%% Set the number of constraints
q = 9;
fun = zeros(q,1);
%%
%% Constraints based on equation (8) in [Zuda (2023)] / Environment 1
%%
fun(1) = mu(1)*mu(16) - ((1+beta(1))*(1-mu(12)*mu(14))) ...
        + ((1+mu(10))*(1-mu(12)*mu(13))) ...
        + (mu(21)*(1+mu(17)*mu(18))*(beta(3)-mu(11)));
%%
fun(2) = mu(2)*mu(16) - ((1+beta(2))*(1-mu(12)*mu(15))) ...
        + ((1+mu(10))*(1-mu(12)*mu(13))) ...
        + (mu(21)*(1+mu(17)*mu(18))*(beta(4)-mu(11)));
%%
fun(3) = mu(3)*mu(16) - ((1+beta(2))*(1-mu(12)*mu(15))) ...
        + ((1+beta(1))*(1-mu(12)*mu(14))) ...
        + (mu(21)*(1+mu(17)*mu(18))*(beta(4)-beta(3)));
%%
%% Constraints based on equation (8) in [Zuda (2023)] / Environment 2
%%
fun(4) = mu(4)*mu(16) - ((1+beta(1))*(1-mu(12)*mu(14))) ...
        + ((1+mu(10))*(1-mu(12)*mu(13))) ...
        + (mu(22)*(1+mu(17)*mu(19))*(beta(3)-mu(11)));
%%
fun(5) = mu(5)*mu(16) - ((1+beta(2))*(1-mu(12)*mu(15))) ...
        + ((1+mu(10))*(1-mu(12)*mu(13))) ...
        + (mu(22)*(1+mu(17)*mu(19))*(beta(4)-mu(11)));
%%
fun(6) = mu(6)*mu(16) - ((1+beta(2))*(1-mu(12)*mu(15))) ...
        + ((1+beta(1))*(1-mu(12)*mu(14))) ...
        + (mu(22)*(1+mu(17)*mu(19))*(beta(4)-beta(3)));
%%
%% Constraints based on equation (8) in [Zuda (2023)] / Environment 3
%%
fun(7) = mu(7)*mu(16) - ((1+beta(1))*(1-mu(12)*mu(14))) ...
        + ((1+mu(10))*(1-mu(12)*mu(13))) ...
        + (mu(23)*(1+mu(17)*mu(20))*(beta(3)-mu(11)));
%%
fun(8) = mu(8)*mu(16) - ((1+beta(2))*(1-mu(12)*mu(15))) ...
        + ((1+mu(10))*(1-mu(12)*mu(13))) ...
        + (mu(23)*(1+mu(17)*mu(20))*(beta(4)-mu(11)));
%%

```

### EXAMPLE

### OEFPIL fit

OEFPIL ESTIMATION METHOD = oefpils2									
fun = @(mu,beta)funMassCalibration(mu,beta)									
n	m	p	q	ITERATIONS	CRITERION	FUNCCRIT	FUNCCRIT_LIN	wRSS	RSS
1	23	4	9	5	1.90495529755996e-16	1.90495529755996e-16	2.26741453357779e-19	208156.886721661	0.00039134490526936
ESTIMATE				STD	FACTOR	LOWER		UPPER	PVAL
beta_1				-1.1279918056735e-07	4.00058100245652e-08	1.95996398454005	-1.91209127387849e-07	-3.43892337468504e-08	0.00480880457263945
beta_2				7.54278541973189e-07	4.00048732661016e-08	1.95996398454005	6.7587043116554e-07	8.32686652780837e-07	2.690302297137e-79
beta_3				0.000124891615012868	1.22208267535257e-09	1.95996398454005	0.000124889219774838	0.000124894010250898	0
beta_4				0.000125642839434949	1.18168967316397e-09	1.95996398454005	0.000125640523365748	0.000125645155504149	0



## RESULTS

```

mufit = result.mu;

% Estimated (fitted) differences from nominal weight 1kg
dMRfit_51699 = mufit(10);      % Reference weight: 51699
dM1fit_15N   = result.beta(1); % Measured weight: 15N
dM2fit_15963 = result.beta(2); % Measured weight: 15936

dmass = [ dMRfit_51699; dM1fit_15N; dM2fit_15963];
massNominal = 1;
mass = massNominal + dmass;

% Standard uncertainty of the weight
u_MRfit_51699 = result.umu(10); % Uncertainty of the reference weight: 51699
u_M1fit_15N   = result.ubeta(1); % Uncertainty of the measured weight: 15N
u_M2fit_15963 = result.ubeta(2); % Uncertainty of the measured weight: 15936

u_mass = [ u_MRfit_51699; u_M1fit_15N; u_M2fit_15963];

% Estimated (fitted) volumes of the weights
VRfit_51699 = mufit(11);      % Volume of the reference weight: 51699
V1fit_15N   = result.beta(3); % Volume of the measured weight: 15N
V2fit_15963 = result.beta(4); % Volume of the measured weight: 15936

volume = [ VRfit_51699; V1fit_15N; V2fit_15963];

% Standard uncertainty of the volumes
u_VRfit_51699 = result.umu(11); % Uncertainty of the volume of the reference weight: 51699
u_V1fit_15N   = result.ubeta(3); % Uncertainty of the volume of the measured weight: 15N
u_V2fit_15963 = result.ubeta(4); % Uncertainty of the volume of the measured weight: 15936

u_volume = [ u_VRfit_51699; u_V1fit_15N; u_V2fit_15963];

```

## Best Estimates by OEFPII

```

BestEstimates = [mass;volume];

disp(table(BestEstimates))

```

```
% BestEstimates
%
%
% 1.000000641
% 0.999999887200819
% 1.00000075427854
% 0.00012544
% 0.000124891615012868
% 0.000125642839434949
```

BestEstimates
1.000000641
0.999999887200819
1.00000075427854
0.00012544
0.000124891615012868
0.000125642839434949

### Uncertainty (covariance) matrix of the OEFPIIL Best Estimates

Estimated parameters of interest (M\_R, M\_1, M\_2, V\_R, V\_1, V\_2):

```
id_BestEstimates = [10 24 25 11 26 27];
U_BestEstimates = result.Umubeta(id_BestEstimates,id_BestEstimates);

disp(U_BestEstimates)

% U_BestEstimates =
%
% 1.0e-14 *
%
% 0.1599999999999415 0.159999999999991 0.159999999687991 0.000116400000000 0.000116400000000 0.000116400000000
% 0.159999999999991 0.160046483572160 0.160017224314753 0.000116400000000 0.000156928900609 0.000137367876887
% 0.159999999687991 0.160017224314753 0.160038988503685 0.000116399999773 0.000137232205722 0.000149667266298
% 0.000116400000000 0.000116400000000 0.000116399999773 0.000100000000000 0.000100000000000 0.000100000000000
% 0.000116400000000 0.000156928900609 0.000137232205722 0.000100000000000 0.000149348606540 0.000126375525396
% 0.000116400000000 0.000137367876887 0.000149667266298 0.000100000000000 0.000126375525396 0.000139639048366
```

1.0e-14 *
Columns 1 through 3
0.1599999999999415 0.159999999999991 0.159999999687991
0.159999999999991 0.160046483572160 0.160017224314753
0.159999999687991 0.160017224314753 0.160038988503685
0.000116400000000 0.000116400000000 0.000116399999773
0.000116400000000 0.000156928900609 0.000137232205722
0.000116400000000 0.000137367876887 0.000149667266298
Columns 4 through 6
0.000116400000000 0.000116400000000 0.000116400000000
0.000116400000000 0.000156928900609 0.000137367876887
0.000116399999773 0.000137232205722 0.000149667266298
0.000100000000000 0.000100000000000 0.000100000000000
0.000100000000000 0.000149348606540 0.000126375525396
0.000100000000000 0.000126375525396 0.000139639048366

### Standard uncertainties of the OEFPIIL Best Estimates

Estimated parameters of interest (M\_R, M\_1, M\_2, V\_R, V\_1, V\_2):

```
u_BestEstimates = sqrt(diag(U_BestEstimates));

disp(table(u_BestEstimates))

% u_BestEstimates
%
%
% 3.9999999999269e-08
% 4.00058100245652e-08
% 4.00048732661016e-08
% 1e-09
% 1.22208267535257e-09
% 1.18168967316397e-09
```

u_BestEstimates
3.9999999999269e-08
4.00058100245652e-08



```
TABLE_volume.LOWER = volume - coverageFactor*u_volume;
TABLE_volume.UPPER = volume + coverageFactor*u_volume;
TABLE_volume.PVAL = 2*normcdf(-abs((0.000125-volume)./u_volume));
TABLE_volume.Properties.RowNames = {'V_R (51699)' 'V_1 (15N)' 'V_2 (15963)'};
TABLE_volume.Properties.VariableNames = {'ESTIMATE [m^3]' 'STD [m^3]' 'FACTOR' 'LOWER BOUND [m^3]' 'UPPER BOUND [m^3]' 'PVAL [H0: Volume = 125 cm^3]'};

disp(TABLE_volume)
```

%	ESTIMATE [m^3]	STD [m^3]	FACTOR	LOWER BOUND [m^3]	UPPER BOUND [m^3]	PVAL [H0: Volume = 125 cm^3]
%						
%						
% V_R (51699)	0.00012544	1e-09	1.95996398454005	0.000125438040036015	0.000125441959963985	0
% V_1 (15N)	0.000124891615012868	1.22208267535257e-09	1.95996398454005	0.000124889219774838	0.000124894010250898	0
% V_2 (15963)	0.000125642839434949	1.18168967316397e-09	1.95996398454005	0.000125640523365748	0.000125645155504149	0

	ESTIMATE [m^3]	STD [m^3]	FACTOR	LOWER BOUND [m^3]	UPPER BOUND [m^3]	PVAL [H0: Volume = 125 cm^3]
V_R (51699)	0.00012544	1e-09	1.95996398454005	0.000125438040036015	0.000125441959963985	0
V_1 (15N)	0.000124891615012868	1.22208267535257e-09	1.95996398454005	0.000124889219774838	0.000124894010250898	0
V_2 (15963)	0.000125642839434949	1.18168967316397e-09	1.95996398454005	0.000125640523365748	0.000125645155504149	0

TABLE / Estimated parameters / OEFPIIL

```
p = length(BestEstimates);
TABLE_OEFPIIL = table;
TABLE_OEFPIIL.Properties.Description = 'Estimated Mass and Volume of the Weights by OEFPIIL';
TABLE_OEFPIIL.ESTIMATE = BestEstimates;
TABLE_OEFPIIL.STD = u_BestEstimates;
TABLE_OEFPIIL.FACTOR = coverageFactor*ones(size(BestEstimates));
TABLE_OEFPIIL.LOWER = BestEstimates - coverageFactor*u_BestEstimates;
TABLE_OEFPIIL.UPPER = BestEstimates + coverageFactor*u_BestEstimates;
TABLE_OEFPIIL.Properties.RowNames = {'M_R (51699)' 'M_1 (15N)' 'M_2 (15963)' 'V_R (51699)' 'V_1 (15N)' 'V_2 (15963)'};
TABLE_OEFPIIL.Properties.VariableNames = {'ESTIMATE [kg | m^3]' 'STD [kg | m^3]' 'FACTOR' 'LOWER BOUND [kg | m^3]' 'UPPER BOUND [kg | m^3]'};

disp(TABLE_OEFPIIL)
```

%	ESTIMATE [kg   m^3]	STD [kg   m^3]	FACTOR	LOWER BOUND [kg   m^3]	UPPER BOUND [kg   m^3]
%					
%					
% M_R (51699)	1.000000641	3.99999999999269e-08	1.95996398454005	1.00000056260144	1.00000071939856
% M_1 (15N)	0.999999887200819	4.00058100245652e-08	1.95996398454005	0.999999808790873	0.999999965610766
% M_2 (15963)	1.00000075427854	4.00048732661016e-08	1.95996398454005	1.00000067587043	1.00000083268665
% V_R (51699)	0.00012544	1e-09	1.95996398454005	0.000125438040036015	0.000125441959963985
% V_1 (15N)	0.000124891615012868	1.22208267535257e-09	1.95996398454005	0.000124889219774838	0.000124894010250898
% V_2 (15963)	0.000125642839434949	1.18168967316397e-09	1.95996398454005	0.000125640523365748	0.000125645155504149

	ESTIMATE [kg   m^3]	STD [kg   m^3]	FACTOR	LOWER BOUND [kg   m^3]	UPPER BOUND [kg   m^3]
M_R (51699)	1.000000641	3.99999999999269e-08	1.95996398454005	1.00000056260144	1.00000071939856
M_1 (15N)	0.999999887200819	4.00058100245652e-08	1.95996398454005	0.999999808790873	0.999999965610766
M_2 (15963)	1.00000075427854	4.00048732661016e-08	1.95996398454005	1.00000067587043	1.00000083268665
V_R (51699)	0.00012544	1e-09	1.95996398454005	0.000125438040036015	0.000125441959963985
V_1 (15N)	0.000124891615012868	1.22208267535257e-09	1.95996398454005	0.000124889219774838	0.000124894010250898
V_2 (15963)	0.000125642839434949	1.18168967316397e-09	1.95996398454005	0.000125640523365748	0.000125645155504149

Save the results

save RESULTS\_MassCalibration.mat