

# BUREAU INTERNATIONAL DES POIDS ET MESURES



## INTERCOMPARISON OF SMALL MASS METROLOGY

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## TABLE OF CONTENTS

1. Summary

Table 1. Participating laboratories

2. Introduction

3. Organisation of the work

Table 2. Organisation of the mass intercomparison

4. Samples

5. Results

Table 3. Calibration round

Table 4. Adopted values of the samples

Table 5. Intercomparison

6. Discussion

7. Conclusions

8. Recommendations

9. References

Appendix - Remarks concerning the BIPM measurements

Table A. Results of the intercomparison (BIPM)

## INTERCOMPARISON OF SMALL MASS METROLOGY

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### 1. Summary

A comparative experiment was organized to establish the lower limit of inaccuracy obtainable by radioactivity metrology laboratories in weighing drops.

This lower limit was supposed to be set by mass metrology on solid (metal) samples, where effects relative to drop formation, evaporation, etc., are eliminated.

Professional mass metrology laboratories are able to work at  $\pm 1 \mu\text{g}$  on metal samples in the 20 to 100-mg range.

The lowest inaccuracy obtained by radioactivity metrology laboratories, when weighing the same samples on Mettler M5 balances, was about  $6 \mu\text{g}$ . To obtain this performance, substitution weighing must be applied, or the balance must be calibrated. Careful and regular check on the calibrations of the reference weights and the balance is also a strict necessity.

Uncalibrated balances yielded errors up to  $17 \mu\text{g}$ . Considerable errors (up to  $60 \mu\text{g}$ ) are reported when reading the optical scale in the range above 10 mg. Actual drop mass determinations will have still poorer accuracies.

TABLE 1Participating Laboratories

BIPM	Bureau International des Poids et Mesures, Sèvres, France.
CBNM	Central Bureau for Nuclear Measurements, Geel, Belgium.
ISN	Institut des Sciences Nucléaires Boris Kidrič, Vinča-Beograd, Yugoslavia .
LMRI	Laboratoire de Métrologie des Rayonnements Ionisants, C.E.A., Saclay, France .
NBS	National Bureau of Standards, Washington D. C., USA .
PTB	Physikalisch-Technische Bundesanstalt, Braunschweig, Germany.

## 2. Introduction

In the standardization of radioactive solutions a commonly used method is to deposit a drop of the solution onto a substrate. The drop is then allowed to dry, leaving the radioactive substance behind.

The activity of the residue is then counted accurately. Knowing the initial drop mass, the specific activity of the solution can be determined.

When a counting equipment is to be calibrated, such a standardized solution can be used in the same way. By determining the initial drop mass the activity on the substrate can be computed.

The best accuracy obtainable in drop mass determination is found when weighing metal samples as a substitute for the drops. Effects due to evaporation, splashing, electrostatic charging of pycnometers, buoyancy, etc., are then negligible. The remaining errors may be ascribed to mass metrology only.

The limits of a meaningful interpretation of the results obtained from intercomparisons of radionuclides<sup>(1)</sup> are set by systematic errors. Attempts at separating the various sources of errors have not always been successful. In particular, a report by Y. Le Gallic<sup>(2)</sup> has drawn the attention of radioactivity laboratories to weighing errors as a possible serious limitation of accuracy. For the balance (Mettler, type M5) normally used in international comparisons, this report states the following minimum errors:

- |   |                       |
|---|-----------------------|
| - random error (repeatability):                       | 5 $\mu\text{g}$       |
| - systematic errors in readings of the optical scale; |                       |
| calibration:  | 8 $\mu\text{g}$       |
| non-linearity:  | 5 $\mu\text{g}$       |
| - systematic errors in weighing samples $> 20$ mg:    | 8 to 15 $\mu\text{g}$ |
| (for a single dial weight)                            |                       |

Thus, according to this author, a total error of at least 18  $\mu\text{g}$  must be considered as a favourable case. He reports errors of 30 to 50  $\mu\text{g}$  in weighings of a 20 mg sample and concludes that weighing errors are one of the most important obstacles to an improvement of the accuracy in radioactivity

metrology. Therefore, an intercomparison of small masses should precede any other intercomparison of activity measurements.

These findings were discussed at the meeting<sup>(3)</sup> of Section II of the Comité Consultatif pour les Etalons de Mesure des Rayonnements Ionisants, and a working party on "Problems in Microweighing" was created.

Starting from the actual needs for accuracy in standardization of radionuclides, this working party discussed<sup>(4)</sup> technical problems in drop weighing and balance testing. Finally, it decided to organize a limited comparison of small masses in order to find out what level of accuracy may be expected in actual practice and how such measurements might be improved.

### 3. Organization of the work

The intercomparison should be representative of the normal practice of drop weighing.

A large-scale action would demand too much work by the mass metrology laboratories involved. Therefore, six laboratories using Mettler M5 balances were invited to take part (see Table 1). The choice of this balance type was justified by the fact that, out of 25 participants in the 1967  $^{60}\text{Co}$  comparison<sup>(5)</sup> organized by the BIPM, 14 used Mettler balances, mostly of the M5 type.

The six participating laboratories each received a set of three samples with nominal masses of 20, 50 and 100 mg. These were to be weighed under conditions normal for drop weighing.

In order to detect deviations from the "true" values, mass metrology laboratories had to determine the real masses of the samples with the best accuracy they could achieve.

Therefore, the sets were calibrated by one of the mass metrology laboratories (CBNM) before they were sent to the participants, and upon return.

Two of the six sets were also calibrated (before and after) by two other mass metrology laboratories, viz. BIPM and PTB.

For each of the three masses four different weighing methods were proposed:

	1. <u>Without tare</u>	2. <u>With a tare</u> of appropriate mass*
[20 mg]	1a) using the optical scale only 1b) using the 10-mg dial weight and the optical scale	2a) using dial weights 2b) substitution method
[50 mg] and [100 mg]	1a) using dial weights 1b) substitution method	2a) using dial weights 1b) substitution method

\* simulating a half-filled pycnometer, i.e. 3 to 4 g.

The participants were requested to use at least one method "with tare" and one "without tare", and to carry out their weighings as they normally do. However, each participant had to use both methods 1a) and 1b) for the 20-mg sample. Buoyancy corrections could be neglected in most cases. The results of the pre-standardizations were not communicated before the end of the intercomparison.

The organization schedule is shown in Table 2. A set of forms with precise instructions was delivered to each participant.

General information was asked from the participants on the type, age, maintenance and installation of the balance, on the availability of reference weights and on checks of randomness and sensitivity.

The forms distributed provided detailed schemes for each of the 12 methods which explained the sequence of manipulations and what information should be supplied.

With each method, each sample had to be weighed ten times. The operator had to indicate details about the tare, the time he spent in front of the balance before and between the weighings and the readings of pressure, temperature and relative humidity. For each weighing the zero readings and dial positions, the charge on the pan, the corrections applied and the results had to be given. Finally, the arithmetic mean and its standard error were to be calculated.

TABLE 2Organisation of the mass intercomparison

	Laboratories	Sets of weights (Nos)
Pre-calibrations (mass metrology laboratories)	CBNM 1	1 2 3 4 5 6
	BIPM 1	1 2
	PTB 1	1 2
	CBNM 2	1 2
Mass comparison by radioactivity metrology labora- tories	LMRI	1
	BIPM	2
	CBNM	3
	ISN	4
	NBS	5
	PTB	6
Post-calibrations (mass metrology laboratories)	BIPM 2	1 2
	PTB 2	1 2
	CBNM 3	1 2 3 4 5 6

The BIPM, PTB and CBNM initials marked with a number are the mass metrology laboratories of the institutes.

Those without number are the radioactivity metrology laboratories of the same institutes and are different and independent from the former laboratories (different personnel, balances, reference weights, weighing rooms).

The results obtained by the participating laboratories were sent to BIPM.



#### 4. Samples

The samples were made by CBNM. The material is Vachromium, a 80/20 Ni-Cr alloy, with a density of  $8.3447 \text{ g.cm}^{-3}$  at  $20^\circ \text{ C}$ .

They have the form of pieces of wire :

100 mg with a diameter of 1 mm, length about 15 mm;

50 mg, 0.8 mm diam., length = 12 mm

20 mg, 0.5 mm diam., length = 12 mm

The surface of each had a very smooth finish.

The weights were adjusted with fine abrasive paper. All samples received a mass which was below the nominal value, so that optical scale readings would be necessary.

Each set was packed in a stainless steel box into which two discs, made of synthetic ivory, were fitted. One of the discs had 3 recesses to house the 3 samples.

#### 5. Results

##### 5.1. Calibrations

All sets were calibrated by CBNM. Set Nos 1 and 2 were also certified by BIPM and PTB to check CBNM's calibrations.

The values are reported in table 3, which presents the corrections to the nominal values.

All 3 laboratories based the measurements on their own standard kilogramme.

Apart from the 100 mg sample of set No 1, which suffered an unexplained loss of about  $7 \mu\text{g}$ , all other results agree within  $1 \mu\text{g}$ . In table 4 the adopted reference values are presented. The pre-calibration figures are the CBNM 2 values for set Nos 1 and 2, and the CBNM 1 values for the other sets. For the post-calibration figures the CBNM 3 values were taken.

TABLE 3

Calibration round

	Corrections to the nominal values (in $\mu\text{g}$ )							
		Pre-calibrations				Post-calibrations		
Set No	Nom. mass (mg)	CBNM 1	BIPM 1	PTB 1	CBNM 2	BIPM 2	PTB 2	CBNM 3
1	20	- 71	- 71	- 71	- 70	- 71	- 71	- 71
	50	- 551	- 550	- 552	- 551	- 550	- 551	- 552
	100	- 293	- 299	- 300	- 300	- 299	- 299	- 301
2	20	- 180	- 180	- 180	- 180	- 180	- 180	- 180
	50	- 534	- 533	- 535	- 535	- 533	- 534	- 535
	100	- 299	- 298	- 298	- 297	- 297	- 297	- 298

TABLE 4

		Adopted reference values of the samples - Masses in mg					
Set No	Nom. mass	1	2	3	4	5	6
Pre-calibration	20	19.930	19.820	19.935	19.872	19.873	19.819
Post-calibration		19.929	19.820	19.935	19.871	19.873	19.819
Adopted value		19.930	19.820	19.935	19.872	19.873	19.819
Pre-calibration	50	49.449	49.465	49.581	49.441	49.488	49.510
Post-calibration		49.448	49.465	49.580	49.440	49.488	49.509
Adopted value		49.448	49.465	49.580	49.440	49.488	49.510
Pre-calibration	100	99.700	99.703	99.307	99.633	99.508	99.386
Post-calibration		99.699	99.702	99.298	99.629	99.508	99.387
Adopted value		99.700	99.702	99.298*	99.629*	99.508	99.386

\* these values were adopted as they agree best with the values reported by the radioactivity metrology laboratories.

### 5.2. Intercomparison

Table 5 summarizes the information and data supplied by the participating laboratories.

The results of the mass determinations are given as the differences between the reported masses and the reference values as given in table 4.

Wherever possible, both raw data and data corrected for dial weights and optical scale readings are indicated.

The meaning of the terms in the second column (lower half of the table) is as follows :

20 mg sample, without tare :

opt. scale only : 1. zero reading close to zero of scale

2. with sample on : reading near to the end of the scale.

opt. sc. + 10 mg: 1. zero reading

2. reading near to 10-mg point on scale.

All other operations :

dial weights : 1. zero reading

2. reading on dials and scale

substitution : comparing readings for sample and for set of reference weights.

TABLE 5 - INTERCOMPARISON

Data = reported values minus adopted values ( in  $\mu\text{g}$  )  
 ( ) = uncorrected data

Legend : X = yes - = no or not measured

- 1) air cond. not running during measurements
- 2) only one reference weight (100 mg) available
- 3) time spent in front of the balance, before weighing is started (min.).

LABORATORY			LMRI			BIPM		CBNM		ISN		NBS			PTB		
SET No.			1			2		3		4		5			6		
Balance	year of purchase		1970	70	66	61		63		62		63		72		66	
	maintenance interval (months)		6	6	6	12		12		12		12		12		12	
air conditioning			X	X	X	(X) 1)		X		X		X		X		X	
dial weights calibrated			-	-	-	X		X		-		X		X		X	
reference weights available			2)	2)	2)	X		X		-		X		X		X	
samples cleaned			X	X	X	X		-		-		-		-		-	
check of randomness			X	X	X	X		-		-		-		-		-	
sensitivity calibrated			X	X	X	X		X		-		X		X		X	
time(min) spent	before 3) weighings		30			60		30		15		30			60		
	between		1.5			3 to 4		3 to 4		4		1.5 to 3			3 to 5		
20 mg	without tare	opt. scale only	(+2)	(+4)	(-5)	(+6)	-6	(+33)	(+60)	(-12)	-1	(-14)	-3	(-3)	+1		
		opt. scale +10 mg	(-2)	(+1)	(-6)	(+5)	-3	(+10)	+7	(-4)	(-1)	-3	(-9)	+4	(-3)	+1	
	with tare	dial weights	(-1)	(-2)	(-4)	(+5)	-3	(+1)	-1	(-1)				(0)	+2		
		substitution	-	-	-		+11		+3		-	-1		+1	-		
50 mg	without tare	dial weights	(-2)	(-4)	(-15)	(+10)	-5	(+6)	+4	(-11)				(-5)	-1		
		substitution	-	-	-		+12		-6		-	+4		+3	-		
	with tare	dial weights	(0)	(+2)	(-3)	(+9)	-6	(+2)	0	(+2)				(-4)	0		
		substitution	-	-	-		+11		+4		-	+2		+3	-		
100 mg	without tare	dial weights	(-1)	(+5)	(-4)	(+10)	-3	(+5)	-4	(-17)				(+3)	+4		
		substitution	+3	+1	0		+7		-4		-	+3		+2	-		
	with tare	dial weights	(-3)	(+1)	(-6)	(+10)	-3	(+2)	+1	(+2)				(-2)	+6		
		substitution	-	-	-		+6		0		-	-1		0	-		

## 6. Discussion

### 6.1. Set No 1

Three different balances (selected among seven because of the quality of the optical scale) were used by two different operators. Apart from one reference weight of about 100 mg (date of calibration 1970), no reference weights were available.

Substitution weighing on the 100 mg sample was only applied without tare.

The balances were not calibrated.

Air conditioning was available and running. All temperatures reported remained between 19.5 °C and 21 °C. During individual operations the reported temperatures are equal.

The standard error\* on the results varies from 0.18 to 0.4 µg. Zero was reset each time.

### 6.2. Set No 2

A very complete programme was followed : a calibrated balance was available, but also substitution weighing was applied.

Air conditioning was available, but it was switched off during measurement.

All operations took place during three successive days, during which barometric pressure, temperature and relative humidity did not change. The linearity of the optical scale was checked with great care.

The balance was purchased in 1961.

Application of the correction on the optical scale reading for the 20 mg weight does not improve considerably the result :

(+ 6) into -6. An explanation might be that the balance sensitivity was not checked with each determination, but that the values obtained some time ago were used.

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\* Standard error =  $\left[ \sum_{i=1}^n (x_i - \bar{x})^2 / (n(n-1)) \right]^{1/2}$

Rectification, such that the 20-mg point would be correct, would yield an excellent set of data.

It is remarkable that the errors are smaller when using the calibrated balance weights than with substitution weighing. The calibration of the balance dates from October 1971 whereas the reference weights were calibrated in 1965. An explanation might be that the reference weights changed in the mean time.\* A bias of roughly + 10  $\mu$ g is suggested by the results. Placing a tare weight on the balance does not have a significant influence on the results. Zero was reset each time. Corrections on the dial weights were applied. The standard error on the results ranges from 0.2 to 0.5  $\mu$ g.

### 6.3. Set No 3

The balance was obtained in 1963.

Air conditioning was running during the weighings. Temperatures between 19.5 and 21 °C and relative humidities of 46 to 59 % are reported. Variations are not stated.

Reference weights were available (calibration in 1972).

Although the balance had been calibrated in 1972, the laboratory reported that the optical scale appeared not to be reliable, probably due to damage to the knife edges. Apart from a sensitivity check no corrections on the optical scale reading were applied. Both direct weighing and substitution weighing were reported. The scheme was worked through in a period of two weeks. The zero points were always read (not adjusted). Zero drifted considerably, even within one series of 10 readings (up to 13  $\mu$ g). The uncorrected optical scale reading on the 20 mg-sample shows an error of + 33  $\mu$ g.

When determining the 50-mg samples by substitution weighing also readings below zero were taken. This might explain the difference in error between the weighing with, and without, tare.

\* see Appendix

The 100-mg sample showed a loss of 9  $\mu\text{g}$  (see table 3). As it is impossible to detect at which stage this occurred, the CBNM 3 value was adopted, this value being closer to the reported value.

Standard errors vary from 0.2 to 0.4  $\mu\text{g}$ .

#### 6.4. Set No 4

The balance was bought in 1962.

Because of the absence of calibrated weights, the dial weights were not calibrated and substitution weighing could not be used.

The optical scale was not checked on linearity.

All measurements took place on one day. The temperature was close to 23.5 °C and varied by less than 0.5 K during the weighings.

Relative humidities of 58 to 66 % have been reported.

No change in barometric pressure was observed. Zero readings were taken, not adjusted. The readings drifted considerably; in one series there was a drift of 14  $\mu\text{g}$ . The optical scale shows an error of + 60  $\mu\text{g}$  at the end of the scale and -4  $\mu\text{g}$  at the 10-mg point, suggesting considerable non-linearity.

The 100-mg sample showed a loss of 4  $\mu\text{g}$ . Here again, the last CBNM value was adopted as the most probable value at the time the sample was measured by the participant.

Standard deviations (not standard errors) were reported.

Division by  $\sqrt{10}$  produces standard errors ranging from 0.8 to 2.1  $\mu\text{g}$ .

#### 6.5. Set No 5

Two balances (1963 and 1972) were used by 2 operators.

The air conditioning is of high quality. Temperature deviations remained within about 1.0 K. Relative humidity was low, i.e. between 42 and 47 %.



Reference weights were available, calibrated in 1966 and 1972. The balances had been calibrated in 1972. But substitution weighing was the only method applied, apart from the first two operations with the 20-mg samples. For one balance the optical scale was calibrated with a standard (reference weight) of 20-mg at full-scale, and at the 10-mg point with the 10-mg dial on. For the older balance, the scale length between mg divisions was measured using tares of approximately equal mass plus a 1-mg weight. Distances are obtained in arbitrary units and the sum normalized with a 10-mg calibrated weight. The measurements took place on several days in a total period of about two months. Standard errors ranged from 0.3 to 1.1  $\mu\text{g}$

The figures seem to hint at a slight positive bias. The substitution weighings on two different balances never show a difference of more than 1  $\mu\text{g}$ .

#### 6.6. Set No 6

A balance of 1966. Air conditioning running.

The balance had been calibrated in 1972.

No reference weights have been used, apart from a 10-mg and a 20-mg standard for calibration of the scale. Substitution weighing was not applied.

The operations cover a period of less than one week. No zero setting; zero reading was taken. The balance seems to be very stable. Only in the case of the 50-mg sample a total drift of 8  $\mu\text{g}$  was observed. Repetition of the series showed the same effect. In both cases the drift was very regular. The difference of the two results was only 1  $\mu\text{g}$ .

The data suggest a positive bias of a few  $\mu\text{g}$ .

Standard errors are between 0.2 and 0.7  $\mu\text{g}$ .

## 7. Conclusions

### 7.1. Calibrations

See table 3.

Mass metrology is able to obtain an accuracy of  $1 \mu\text{g}$  in the range of 20 to 100-mg.

It is an essential fact that the three calibrating laboratories worked independently. The only correlation was that the laboratories trace back their own kilogramme standards to the international standard at the BIPM.

For the calibration of the reference weights also other balances than the Mettler M5 have been used. The laboratories made a special effort to procure themselves with freshly calibrated reference weights, and it may be assumed that the calibrations were of the best they could perform.

Therefore, the agreement within  $1 \mu\text{g}$  may not be considered to be typical for normal use of M5 balances.

The unexplained mass change of the 100-mg sample of set No 1 is sufficient warning that even under optimum conditions mass instabilities cannot always be avoided.

Whether imperfections of the surface finish or manipulation effects are at the origin of the change could not be ascertained. But, the point is that incidents of this nature can happen without being noticed.

### 7.2. Intercomparison

See table 5,

When maximum errors are quoted, the three highest values are mentioned. This is done to avoid too pessimistic conclusions on single outliers.

7.2.1. Reading of the second half of the uncalibrated optical scale ("20 mg, opt. scale only").

Errors of 60, 33 and  $14\ \mu\text{g}$  were found. This agrees with the results obtained on 20-mg samples, as reported by Le Gallic<sup>(2)</sup>.

7.2.2. Uncorrected data

In all other determinations the first half of the optical scale was used. Although adjustment errors of the dial weights interfere with optical scale deviations it seems reasonable to conclude that errors of the optical scale are less in the lower half than above the 10-mg reading. Uncalibrated balances show errors up to 17, 15 and  $10\ \mu\text{g}$ . The view expressed in (2) that total errors of at least  $18\ \mu\text{g}$  must still be considered as favourable cases, seems far too pessimistic. Likewise, as far as the accuracy of dial weights and the random errors are concerned, the present results are in fair agreement with the specifications given by the manufacturer of the balance. However, the accuracy of optical scale readings ( $2\ \mu\text{g}$ ) is not compatible with our findings.

7.2.3. Calibrated balances

Corrected data for "20 mg, opt. scale + 10 mg" and all "dial weights" values show errors up to 7, 6 and  $5\ \mu\text{g}$ .

The error of  $7\ \mu\text{g}$  is found in set No 3. The laboratory reported the balance not to be reliable, see 6.3. Apart from this outlier an inaccuracy of about  $6\ \mu\text{g}$  may be assumed. The conclusions of (2) do not apply to calibrated balances.

7.2.4. Substitution weighing

Top values of 12, 11 and  $7\ \mu\text{g}$  are found (Set No 2). Sets No 3 and 5 show 6, 4 and  $3\ \mu\text{g}$  error.

If it is supposed that the calibration of the reference weights in the case of set No 2 had a bias (see 6.2), an inaccuracy of about  $6\ \mu\text{g}$  seems to follow from the limited amount of information.

#### 7.2.5. Reproducibility

The reproducibility within one series of measurements is, in general, very good. Standard errors of about  $0.3\mu\text{g}$  are frequently reported. Le Gallic gives a random error of  $5\mu\text{g}$  which must be compared to  $3 \times \sqrt{10}$  times the mean error of a single measurement, and which agrees with the present results. It is evident that these (purely statistical) errors are not indicative of overall accuracy.

7.2.6. Balance condition. Environment. Cleaning of samples. A clear correlation between the age of the balance and its quality cannot be established.

A maintenance frequency of once in 12 months is the general rule and is accepted as sufficient. Air conditioning was installed in all cases. The profit of switching off the air conditioning in the case of set No 2 is not apparent from the data, but the laboratory has reasons which are based upon more extensive information than is treated here.

Balance sensitivity was calibrated in all cases except one. In general, the samples remained stable during the intercomparison, as is shown in Table 3. Two samples changed significantly: set No 3, 100-mg sample lost  $9\mu\text{g}$ ; set No 4, 100-mg sample lost  $4\mu\text{g}$ .

Some participants report to have cleaned the samples, others have not. Cleaning with solvents would have shifted the masses. As this was not the case (sets No 3 and 4 were not cleaned, see Table 4), it must be concluded that removal of dust with a brush was understood by some laboratories to be cleaning.

#### 7.2.7. General conclusion

Uncalibrated balances show errors exceeding  $10\mu\text{g}$ . This is not surprising in view of the manufacturer's specification of the adjustment of the dial weights. Calibration of the optical scale improves the accuracy, in particular for readings on the second half of the scale.

Calibration of the optical scale and the dial weights results in an accuracy of about  $6\mu\text{g}$  for direct weighing. Substitution weighing has the same accuracy of about  $6\mu\text{g}$ .

The last two conclusions are conditional (see 7.2.3. and 7.2.4.) and apply only to weighing of metal samples.

In actual determinations of the masses of drops several additional factors will impair the accuracy: drop formation, evaporation, manipulations of pycnometers, less regular time interval between readings, etc. The determination of lower limits of systematic errors in drop weighings strictly demands a careful choice of appropriate mass samples and very reliable calibrations before and after the intercomparison. Since this condition was not fulfilled in the experiments reported by Le Gallic<sup>(2)</sup>, we feel that the present results give a more realistic picture of the performance of Mettler M5 balances.

## 8. Recommandations

8.1. A set of reference weights should be available whether substitution weighing or direct reading is applied. The calibration of the set must be checked regularly.

This requires a more ready access to professional mass metrology services.

8.2. In order to achieve better accuracies in real drop mass determinations the whole procedure of drop formation, deposition and weighing techniques should be studied systematically. A recommendation concerning the best weighing technique will appear as a subsequent report.

8.3. Improvement of the accuracy may also be expected upon the introduction of more advanced balance designs.

"Electronic" balances keep the balance in constant position. Interpolation between dial positions is made with electrical measurement, which is easier to linearize and can be kept under easier control. Reproducibilities are, in general, better. If this is at the expense of the maximum load it may be acceptable for the application of drop source preparation.

## 9. References

- (1) Rapport de la réunion du Groupe de travail pour la mesure des radionucléides, 22-24 novembre 1967, Rapport BIPM (Janvier 1969).
- (2) Y. Le Gallic, La micropesée, source importante d'erreur en métrologie d'activité, Rapport CEA-R-4169 (1971).
- (3) Comité Consultatif pour les Etalons de Mesure des Rayonnements Ionisants, Section II - Mesure des radionucléides, première réunion - 1970, B.I.P.M., Sèvres.
- (4) Report on the first meeting of the Working party b, "Problems in Microweighing" (November 1971), BIPM report.

AppendixRemarks concerning the BIPM measurements (Set No 2)

The results obtained by the BIPM radioactivity metrology laboratory should be completed by the following information which was not yet available when the form of the intercomparison was filled in and which, therefore, is not contained in the report.

The dial weights were calibrated in October 1971 with a set of standard weights (Ni 5) calibrated in 1965. The complete transformation of the BIPM mass metrology laboratory in 1971/72 made it impossible to carry out a new calibration at that period. Thus the BIPM results obtained in May-June 1972 are based on the values determined in 1965.

In February 1973 the standard set Ni 5 was recalibrated and used in a new calibration of the balance M5. All the results of the intercomparison have been adjusted to the new values and are presented in Table A along with the ones given in Table 4.

The new results strongly support the view that the observed changes in the Ni 5 set took place before the intercomparison. They illustrate the importance of frequent checks of the calibration, if the highest accuracy is sought.

Table A  
Results of the intercomparison (BIPM)

Sample (mg)	Tare (g)	Substitution method	Uncorrected	Calibr. 1965	Calibr. 1973
20	-	-	+6	-6	-6
		(x)	+5	-3	-4
	3	-	+5	-3	+1
		x		+11	+2
50	-	-	+10	-5	-1
		x		+12	+6
	3	-	+9	-6	-2
		x		+11	+5
100	-	-	+10	-3	-2
		x		+7	+4
	3	-	+10	-3	-2
		x		+6	+3