

NPL REPORT

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**An investigation into the
effect of marking mass
standards of OIML class E1**

J Berry and S P Downes

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An investigation into the effect of marking mass standards of OIML class E1

J Berry and S P Downes
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ABSTRACT

The marking of weights in mass laboratories is widely used as a means of identifying a particular weight and hence differentiating between similar weights. This report assesses the effect of a variety of weight marking processes on the mass of a weight and sample materials against the tolerance limits set by the OIML recommendation No 111 for class E1 weights. In addition, the mass stabilities of weights marked with a laser technique were assessed over an eight-month period following their marking. The results obtained demonstrate that die stamping, laser marking and acid etching can be used on E1 class weights. Unacceptably large mass losses occurred from spark erosion and as such this process is not recommended for weight marking.

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1. INTRODUCTION

This report summarises an evaluation of the effects on the masses of weights and sample materials following the application of various marking techniques, including those in common usage by mass standards laboratories. The Organisation International de Métrologie Légale (OIML) recommendation No 111 [1] sets tolerances for the mass values of weights of different classes (e.g. for OIML class E₁ weights, the tolerance values are ± 0.5 mg at one kilogram and ± 0.030 mg at one gram). It is therefore crucial that any process used to mark a weight does not take its mass outside of the stated range for its particular class and additionally the effect of marking should not affect the subsequent stability of the weight. The marking of weights in mass laboratories is widely used as a means of identifying a particular weight and hence differentiating between similar weights. This report aims to provide recommendations on suitable marking processes that can be used on OIML class E₁ weights.

2. DESCRIPTION OF MARKING PROCESSES

2.1 Laser marking

This approach uses a laser to locally anneal the surface of the weight, the effect of which can be seen by eye. Providing the laser intensity is not too high the annealing process should not remove material from the surface, and therefore should not change the mass of a weight.

2.2 Die stamping

A metal stamp is used to produce an indentation in the surface of a weight. In theory, material should not be added or removed from the weight, but rather distributed around the point where the indentation is made. The depth of the indent may affect the long-term stability of the weight from the accumulation of contaminants.

2.3 Spark Erosion

Spark erosion employs an electric arc to erode material from a surface. As such, one would expect material to be removed from a weight and cause its mass to be reduced.

2.4 Acid Etching

A solution of hydrochloric HCl and selenious H_2SeO_3 acid is delivered to a surface by the application of an acid etch pen. The reaction between the solution and a weight's surface causes material to be removed by an etching process.

3. SAMPLES USED FOR MARKING

3.1 Coupons

Stainless steel sheet pieces were cut into squares with an edge length of 15 mm to test the following methods of weight marking:

- Die stamping
- Spark erosion
- Acid etching

Additional test pieces, which were left unmarked, were used to monitor the changes in mass of the samples.

3.2 Circular discs and 1 kg OIML mass standards

Eight OIML shaped stainless steel kilograms from set NPLW 55 and set NPLW 56, of E₁ class surface finish, were sent to the South Yorkshire Trading Standards Unit (SYTSU) for laser marking.

4. TESTING PROCEDURES

4.1 Laser marking

Prior to being laser marked, all eight of the OIML shaped stainless steel kilograms were weighed against two NPL standard kilograms. The eight kilograms were then

taken to SYTSU for laser marking. The device used was a Fobalas Nd:YAG laser, supplied by Alltec GmbH. Each weight was marked with its Set identification number (either a 55 or 56), plus an extra letter(s) denoting its designation within the set (i.e. D, DD or TD). The plain weights within the set (i.e. no extra letter) were solely labelled with the set identification number. The length of each marked letter and numeral was approximately 5 mm, with a line thickness of 1 mm. Figure 1 shows an image of a weight that has been laser marked with the identification '55TD'.



Figure 1: The top of an OIML shape 1 kg stainless steel weight with its identification '55TD' marked with a laser

Following the return of the laser marked kilograms, their mass differences relative to the two NPL standards were re-determined. The value of the mass difference post-marking less the mass difference pre-marking gave the mass change due to laser marking. These values are reported in Table 1.

4.1.1 Monitoring the medium-term mass stability

In addition to establishing the mass change due to marking, the mass values of the four weights that comprise Set 55 were determined on four separate occasions post-laser marking. The intervals were at four days, 2 months, 4 months and 8 months after being marked. The aim of these extra weighings was to check for any subsequent changes in the mass values of the marked weights.

4.2 Die stamping

Two different indenters were used; one that produces a small dot with a diameter of approximately 0.25 mm, and a second that produces an “X” shape which is approximately 4 mm in size. Both indenters are shown in Figure 2. The tip of each indenter is manufactured from hardened, high carbon steel. Prior to marking, the test coupons were weighed against NPL mass standards and both indenter tips were cleaned with acetone.

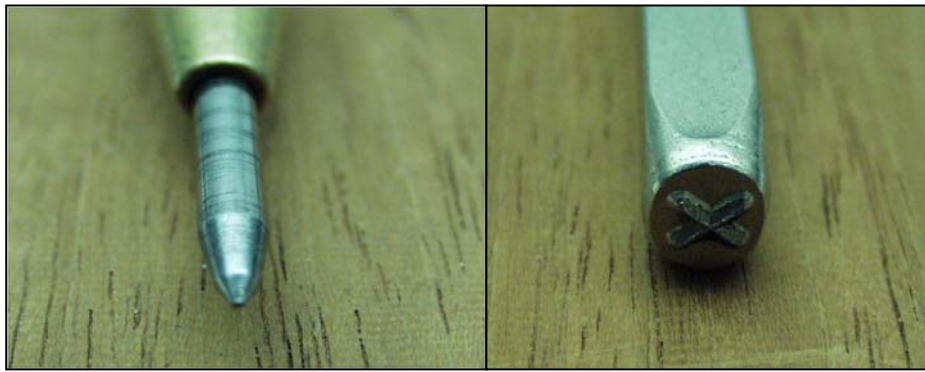


Figure 2: High carbon steel indenters; dot (left) and “X” shape (right)

The dot indenter was then used to stamp a single dot in the centre of four test pieces, and two dots on a further three test pieces. The X shaped indenter was used to stamp the surface of four test pieces. Following their marking, each coupon was gently rubbed with chamois leather to remove any traces of residual contamination left from stamping process. The test pieces were then left for a period of twenty-four hours prior to being re-weighed.



Figure 3: Stainless steel coupons stamped with a single dot (left), double dot (centre) and an X (right)

4.3 Spark erosion

In the same manner as the die stamping procedure, each test piece was weighed against NPL mass standards before and after being marked. The clamps, and adjacent surfaces of the spark-eroding machine, were all cleaned with acetone prior to marking the test pieces. A total of four coupons were marked. Once clamped in position, each coupon was spark eroded with a line of approximately 8 mm in length, an example of which is shown by Figure 4. The test pieces were then gently rubbed with a chamois leather to remove any loose material arising from the spark erosion process.

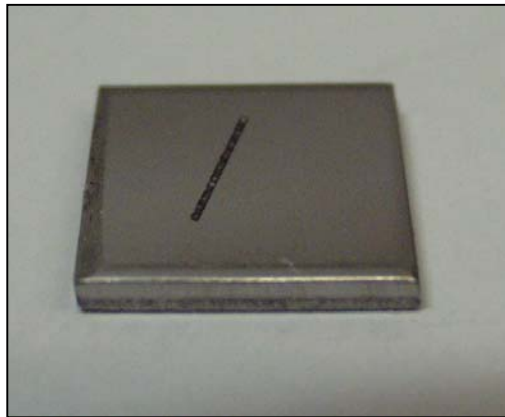


Figure 4: A spark eroded stainless steel test coupon

4.4 Acid etching

Four coupons were cleaned with acetone and left for a period of 24 hours prior to comparison (using a Sartorius C5S comparator) with an unmarked coupon. An acid etch pen was used to etch a line with an approximate length of 10 mm, as shown in Figure 5.



Figure 5: An acid etched stainless steel test coupon

Prior to re-weighing, the coupons were left for a period of at least 24 hours to allow any residual reaction between the acid and stainless steel to diminish. The acid etched coupons were then re-weighed against the control coupon.

5. RESULTS

5.1 Initial mass change due to laser marking

The results for the change in mass of weight Sets 55 and 56 are given in Table 1. The mass of each weight was compared against two NPL standard kilograms (NPLW 61DD & 61TD) both prior to and post-laser marking, and its change in mass evaluated. The reproducibility of the measurements is around 3 μg .

Identification	Mass change relative to 61 DD (μg)	Mass change relative to 61TD (μg)	Average mass change (μg)
55	+0.8	+3.6	+2.2
55D	+27.0	+26.4	+26.7
55DD	+33.3	+33.0	+33.2
55TD	+11.0	+14.0	+12.5
Average mass change for Set 55 = +18.6 μg (σ_{n-1} = 14 μg)			
56	+10.7	+8.6	+9.6
56D	+7.7	+5.0	+6.4
56DD	+8.3	+7.1	+7.7
56TD	+7.4	+7.5	+7.4
Average mass change for Set 56 = +7.8 μg (σ_{n-1} = 1.3 μg)			

Table 1: Mass changes in weights due to laser marking

There is a relatively large variation in the mass change for each weight from Set 55 due to the laser marking process. The average mass change of Set 55 was found to be +18.6 μg with a standard deviation of 14 μg . All the weights gained mass, which may have resulted from the oxidation of their surfaces. The laser marking process uses the beam energy to locally heat a surface thereby providing thermal energy to anneal the material. However, the heat generated may be sufficient to cause further oxidation of

the surface, thereby increasing the mass of each weight. The variation in mass change is indicative of the repeatability of the laser marking process and may vary depending on surface effects (e.g. uniformity of the surface finish or material composition) or the ability to reproducibly focus the laser beam on each surface.

The weights of Set 56 all showed a similar mass increase post-laser marking. The average mass gain in Set 56 was equal to 7.8 μg with a standard deviation of 1.3 μg (a factor of 10 lower than obtained for Set 55). Interestingly, the number of letters etched on each weight did not seem to affect the magnitude of the mass change; if the mass gain were due solely to surface oxidation one would expect the two effects to be correlated and the mass gain to increase with the number of letters marked on a surface. However, the observed change in mass for Set 56 is relatively small and the increases may instead be due to an additional contamination of the weights as they were transported to SYTSU and back to NPL. Further work is therefore necessary to establish the causes of the mass increases seen for each weight set but this is beyond the scope of this current investigation.

5.2 Mass stability of weights post-laser marking

The mass stabilities of the weights that form Set 55 were monitored with respect to the reference weights 61DD and 61TD over an eight-month period following their laser marking. This was done to determine whether laser marking produces any instabilities in a weight's surface that impact its mass over a longer-term. To make this quantification it is necessary to establish the mass stability between the reference weights 61DD and 61TD over the same period of time. The variation of the mass difference between 61DD and 61TD has therefore been evaluated and the results displayed by Figure 6.

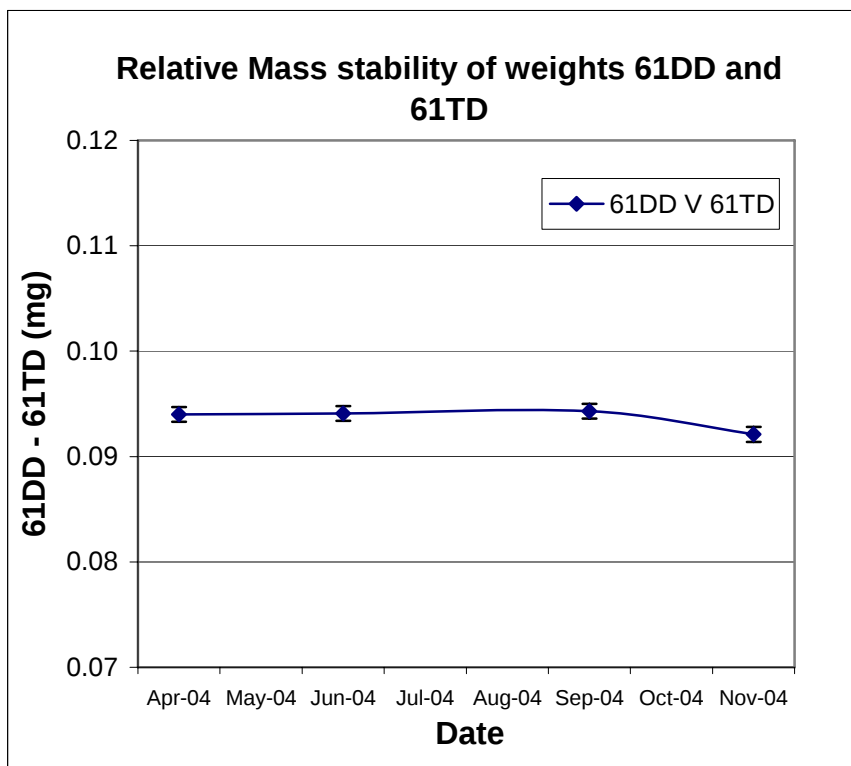


Figure 6: Mass stability of reference weights 61DD & 61TD. The maximum variation within the eight month period was 2.2 μg

The maximum variation in the mass difference between the two reference standards was 2.2 μg and was obtained from the pre-marked value (0.095 3 mg) and the final measured value (0.092 1 mg) made in November after the eight months had elapsed. Figure 7 through to Figure 10 display the mass differences for the weights comprising set NPLW 55 over the same time period. Note the scales of the ordinates are identical for all the plots (i.e. they cover a range of 0.05 mg) to make the visual comparison between the weighing results easier.

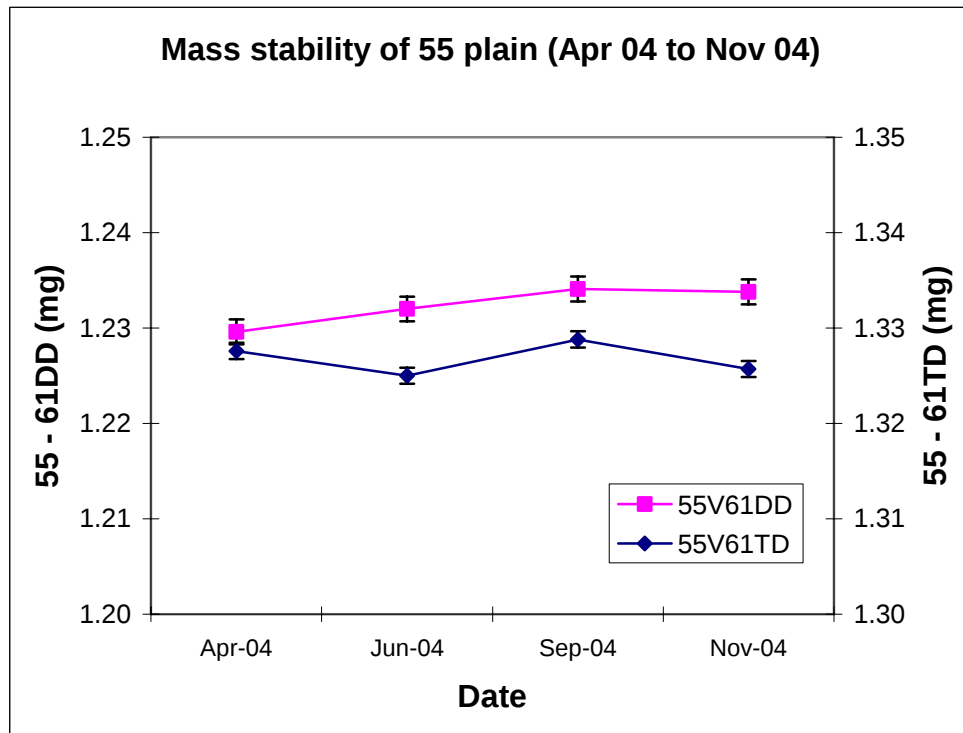


Figure 7: Mass stability of 55 plain monitored with respect to 61DD and 61TD

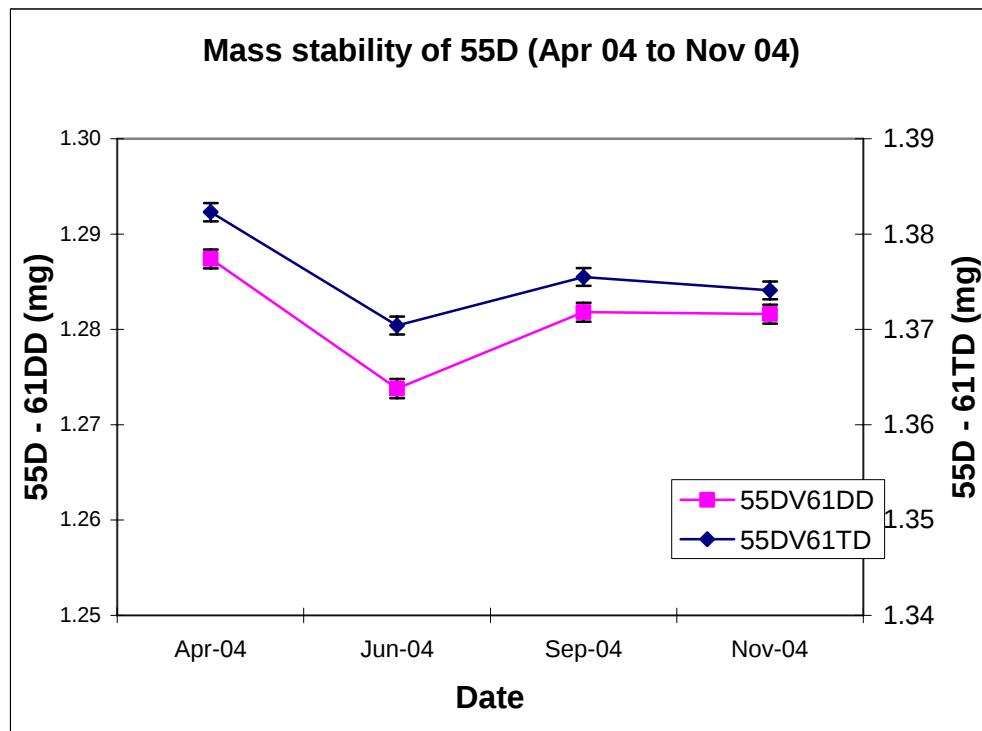


Figure 8: Mass stability of 55D monitored with respect to 61DD and 61TD

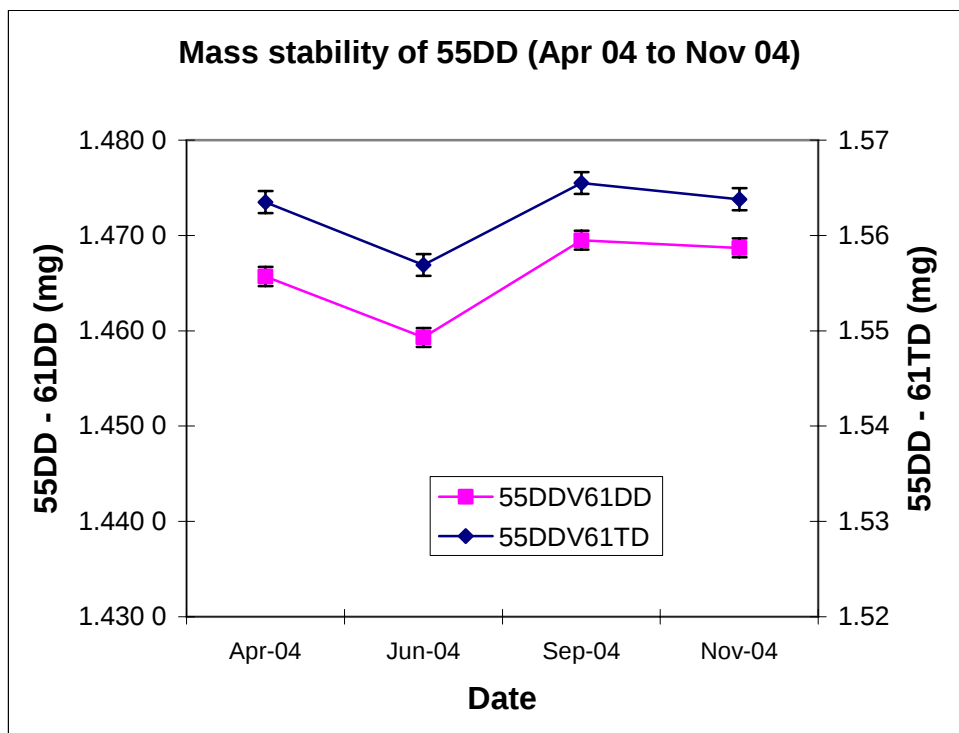


Figure 9: Mass stability of 55 DD monitored with respect to 61DD and 61TD

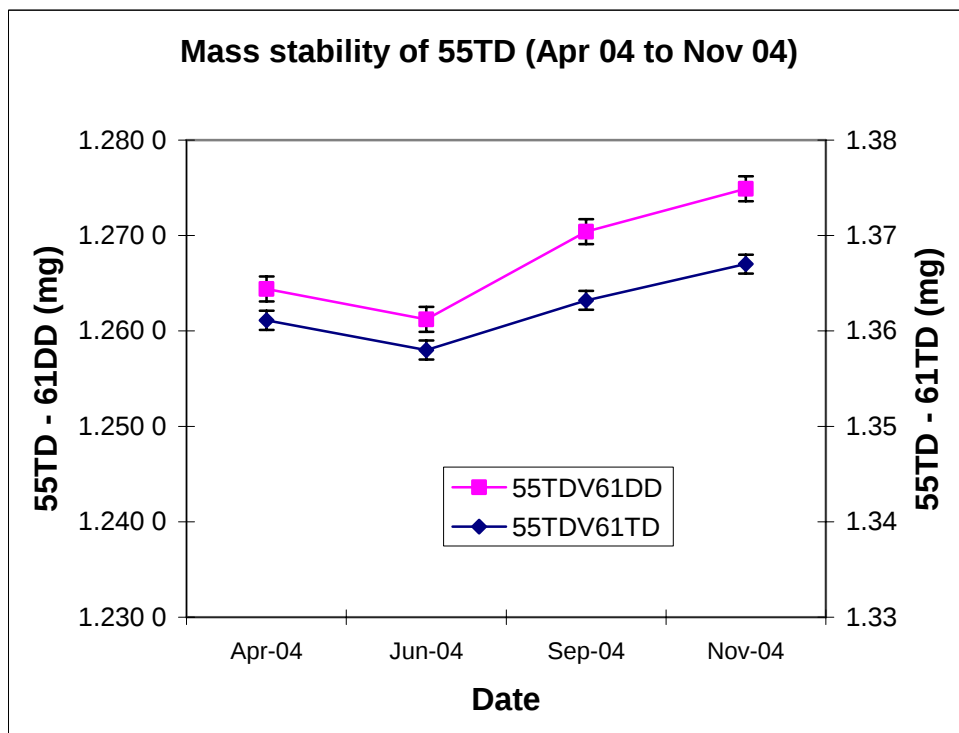


Figure 10: Mass stability of 55TD monitored with respect to 61DD and 61TD

There does not appear to be a particular bias in the drift in mass of the weights over the eight-month period following laser marking. Rather the weights in Set 55 appear

to be less stable in relation to the control weights. However, past history of this weight set would indicate that this behaviour is not uncommon. Also, the overall mass change in any single weight from April to November 2004 does not exceed 20 μg and is small compared with the uncertainty required for the calibration of an OIML E₁ class weight of nominal mass 1 kg, which is $\pm 200 \mu\text{g}$ ($k = 2$).

5.4 Die stamping and spark erosion

The results from the die stamping and spark erosion tests are summarised by Table 2. The masses of all the test pieces were compared with an NPL standard before and after marking. In this manner, the mass change (shown in the final column of Table 2) due to the marking process could be evaluated. The samples identified as P1, P2, P3 and P4 were left unmarked and were compared in the same manner with the NPL standard to determine the reproducibility of the weighing process.

5.4.1 Die stamping

The single dot, double dot and “X” stamped test pieces showed no significant change in mass compared with the four control samples. This result was anticipated as the stamping process should not add or remove material from the weight. Instead, material in the area of impaction will be redistributed. Despite more material being displaced for the double dot test pieces, there was no indication of any change in mass difference and therefore the application of multiple dots on a weights surface should not affect its mass value. Due to the larger contact area between the “X” shaped stamp and the test piece, even more material was displaced than with either the single or double dot stamps. Again, no significant change in mass was detected for the four “X” stamped test pieces and the contact area of the die used does not appear to have an effect on the mass value of a test piece.

5.4.2 Spark erosion

The spark erosion process produced large mass losses in the four stainless steel test pieces. This result was anticipated as the process erodes material from a surface. There was also a large variation in the mass lost by each test piece, which demonstrates the relatively poor reproducibility of the spark erosion process.

Sample	Identification	Mass Pre-marking (g)	Mass Post-marking (g)	Mass Change (μg)
Control	P1	3.622 261 7	3.622 259 5	-2.2
Control	P2	3.620 832 7	3.620 831 5	-1.2
Control	P3	3.669 206 7	3.669 205 5	-1.2
Control	P4	3.559 989 7	3.559 986 4	-3.3
		Average Mass Change		-2.0
		Standard Deviation		1.0
Single dot	D1	3.536 177 4	3.536 174 9	-2.5
Single dot	D2	3.441 450 3	3.441 448 9	-1.4
Single dot	D3	3.520 600 6	3.520 598 6	-2.0
Single dot	D4	3.415 422 2	3.415 420 6	-1.6
		Average Mass Change		-1.9
		Standard Deviation		0.5
Double dot	DD1	3.259 144 0	3.259 142 7	-1.3
Double dot	DD2	3.635 824 0	3.635 822 0	-2.0
Double dot	DD3	3.411 682 3	3.411 681 7	-0.6
		Average Mass Change		-1.3
		Standard Deviation		0.7
X stamped	X1	3.598 607 2	3.598 602 9	-4.3
X stamped	X2	3.522 320 5	3.522 321 2	+0.7
X stamped	X3	3.419 849 0	3.419 847 3	-1.7
X stamped	X4	3.813 741 1	3.813 741 3	+0.2
		Average Mass Change		-1.3
		Standard Deviation		2.3
Spark Erosion	E1	3.443 468 9	3.443 370 9	-98.0
Spark Erosion	E2	3.733 229 7	3.733 051 6	-178.1
Spark Erosion	E3	3.458 388 1	3.458 170 9	-217.2
Spark Erosion	E4	3.662 366 3	3.662 155 7	-210.6
		Average Mass Change		-176.0
		Standard Deviation		54.7

Table 2: Mass changes in test pieces due to die stamping and spark erosion

5.5 Acid etching

The acid etching weighing results are summarised by Table 3. In common with laser marking, the masses of the test pieces were seen to increase after acid etching. The average mass increase of the four test pieces was +18.4 μg , with a standard deviation of 5.8 μg . This level of reproducibility is similar to that obtained when laser marking Set 56. The reproducibility of the acid etching process might be improved by automation; in this test, the manual application by pen made it difficult to control the quantity of acid applied and the length of line etched.

Sample Identification	Mass Pre-marking (mg)	Mass Post-marking (mg)	Mass Change (μg)
A1	-23.803 4	-23.784 6	+18.8
A2	-18.545 6	-18.521 4	+24.2
A3	-39.376 0	-39.365 7	+10.3
A4	-44.565 7	-44.545 3	+20.4
	Average Mass Change (μg)		+18.4
	Standard Deviation (μg)		+5.8

Table 3: Mass changes in test pieces due to acid etching process

6. CONCLUSIONS

6.1 Laser marking

Laser marking was found to increase the mass of all the weights studied in this investigation. The largest mass gain was +33 μg and was obtained for the weight identified as 55DD. This mass increase, however, is small compared with the stated tolerance of an E₁ class kilogram, which is 1 kg $\pm$ 0.5 mg. It is worth noting, however, that a mass gain of this magnitude would exceed the tolerance of an E₁ class 10 gram weight ($\pm$ 0.020 mg) and as such a smaller font size should be considered for E₁ class weights of nominal value equal to or less than 100 g. The mass stabilities of four weights were subsequently monitored over an eight-month period after being laser

marked, and showed no significant change in their mass values. As such, laser marking is an appropriate method of identifying a weight that conforms to the OIML class E₁.

6.2 Die stamping

The application of dot and X shaped indenters had no measurable effect on the mass values of the stainless steel test pieces. Die stamping is therefore a cheap and effective method by which identifying marks may be applied to an E₁ class mass standard.

6.3 Spark erosion

Spark eroding identifying marks on the surface of a stainless steel test pieces resulted in significant loss of material. This loss could easily result in the mass value of an E₁ class weight being taken outside its tolerance range and therefore spark erosion is not recommended as a method for marking E₁ weights.

6.4 Acid etching

Acid etching was found to cause a measurable mass gain but, in common with laser marking, the increase is small compared with the E₁ tolerance for 1 kg weights. Due to the manual nature of the application process, it proved difficult to control the quantity and location of the applied acid. It would therefore be preferable to utilise die stamping or laser marking, but as a cheap and simple method of marking E₁ weights acid etching is an acceptable process.

6.5 Overall conclusion

The results obtained demonstrate that die stamping, laser marking and acid etching can be used on E₁ class weights. Unacceptably large mass losses occurred from spark erosion and as such this process is not recommended for weight marking.

6.6 Future work

The initial mass gains and medium term stabilities (i.e. over an eight month period) of the laser marked OIML shaped kilograms were studied. Longer-term monitoring of the mass values of these kilograms may highlight any differences in their stability

over their full calibration term, which is two years. Also, the research into the other methods of weight marking - die stamping, spark erosion and acid etching - did not encompass the medium-term stability tests for these processes. It would therefore be useful to include medium and longer-term tests in any future work on weight marking.

7. ACKNOWLEDGEMENTS

The authors wishes to thank South Yorkshire Trading Standards Unit for laser marking the kilograms. Also thanks to Stephen Brown for his help and advice with the project.

8. REFERENCES

[1] Organisation International de Métrologie Légale, International Recommendation No 111: 2004, *Weights of classes E_1 , E_2 , F_1 , F_2 , M_1 , M_2 , M_3* .