

# Charpy Key Comparison SIM.M.F-S3

## Instrumented Charpy Tests on Low- and High-Energy Specimens

**Final report** 

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### ABSTRACT

This report describes a CIPM Key Comparison through the SIM/MWG-7 organization, concerning instrumented impact testing of low-energy (20 J) and high-energy (100 J) Charpy V-notch specimens.

In this Comparison, various instrumented and non-instrumented data were obtained from instrumented Charpy tests performed at the pilot (NIST) and participant (INMETRO) laboratories using the same instrumented Charpy striker and acquisition system.

The use of instrumented absorbed energy ( $W_t$ ), rather than absorbed energy yielded by the impact machine encoder or dial gage (KV), guarantees traceability to the International System and offers the promise of reducing bias between NMIs and simplifying comparisons between certification systems.



**Figure 1.** Comparison of representative instrumented curves for low-energy specimens (left) and high-energy specimens (right) from the  $1^{st}$  and  $3^{rd}$  test series.



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#### 1. Introduction

In an effort to advance NIST strategic alliances with the institutions participating in the Inter-American System of Metrology (SIM) and to further strengthen SIM, the Associate Director of Laboratory Programs made funds available to support and to contribute to the NIST/USA and INMETRO/Brazil measurement service programs.

The certified absorbed energies of reference materials (RMs) that are used for the indirect verification of Charpy test machines represent a method-specific value, that is traceable first to the reference machines used in the certification, and then to Charpy impact test standard used (such as ISO 148). The current certification system used by NMIs for Charpy RMs has served, and continues to serve, the Charpy community well, but it is difficult to track and maintain equivalence of the current systems, because the certified values are not traceable to primary SI units and the Charpy test is a destructive test. More direct traceability to primary SI units would simplify comparisons and provide a means to better quantify and reduce bias between certification systems used by the various NMIs.

In this comparison, KV measurements are compared at two energy levels, with NIST as pilot laboratory. The Key Comparison was officially designated SIM.M.F-S3 by CIPM/CCM following the specific Charpy absorbed energy levels:

- Low energy (nominal energy level = 20 J).
- High energy (nominal energy level = 100 J).

#### 2. List of Participants

There were two participating laboratories, including the pilot. See Table 1 for the list of participating laboratories and the schedule of the comparison.

Participant	Code Number	Test Date			
NIST (USA)	01	August 2012			
INMETRO (Brazil)	02	March 2013			
INMETRO (Brazil)	02	April 2013			
NIST (USA)	01	July 2013			

**Table 1.** Participating countries and laboratory code numbers used in the report



#### 3. Purpose of the KC

The purpose of this Key Comparison is to explore a means to reduce the bias between National Metrology Institutes (NMIs) that certify reference materials for Charpy impact testing. Here, the National Institute of Metrology Quality and Technology (INMETRO/Brazil) and the National Institute of Standards and Technology (NIST/USA) performed initial comparisons to evaluate how a change in the traceability of Charpy absorbed energy (KV) measurements might help to reduce bias between NMIs. Currently, KV measurements are traceable to reference machines that measure absorbed energy as the loss in energy from a pendulum that impacts and breaks a notched-bar test specimen. The energy loss is determined using a dial gage or encoder on the machine that



measures the final height of the pendulum following impact to determine the energy absorbed in the impact (Figure 2). These measurements are traceable to the specific pendulum machine used for the measurement. A change in the traceability of the KV measurement to primary SI units is considered in this comparison, which may simplify comparisons. The approach is to strain-gage (instrument) the machine striker and compare the output of this particular striker (and its data acquisition system) at several energy levels on two different reference machines (one at INMETRO, one at NIST). For the instrumented measurements, the output from the striker is plotted as force-deflection and the area under the curve corresponds to the absorbed energy of the test. The combined benefit of having more direct traceability to SI and the use of a standardized instrumented striker design is evaluated as a basis to establish a harmonized international scale to measure the absorbed energy in a Charpy impact test. The execution of the Charpy measurements is in accordance with the ISO 148-1 [1] and ISO 148-2 [2] standards.

#### 4. Limitations of the KC

- a. Each measurement loop is independent of the others.
- b. There is no absolute numerical reference value.
- c. Only relative deviations can be compared.

#### 5. Comparison Protocol

Comparisons of various types, as defined by the International Committee for Weights and Measures CIPM-MRA-D-05 document [3], are selected by a Consultative Committee



(CC) to test the principal techniques and methods in a specific field. The comparisons between National Metrology Institutes (NMIs) have the objective of establishing a degree of equivalence for national measurement standards to ensure world-wide uniformity of measurement, and to provide for the mutual recognition of calibration and measurement certificates issued by the NMIs.

The comparison between NIST and INMETRO was a "Supplementary Comparison of Instrumented Charpy Impact Machines". The request for this comparison was submitted to the Consultative Committee of Mass and Related Quantities (CCM) throughout the SIM-MWG7 (Metrology Working Group No. 7 of Mass and Related Quantities of the Inter-American Metrology System) that is in charge of supporting the CMC of National Metrology Institutes. The CCM has analyzed the request and accepted the comparison inside its Working Group on Force. The BIPM allows this new type of comparison (supplementary) to be run by RMOs (Regional Metrology Organizations) to cover areas or techniques not covered by BIPM Key Comparisons, which comprise the case of Absorbed Energy by Impact Charpy. The supplementary comparison was automatically included in the BIPM framework where the value obtained by the work is to be published at the BIPM KCDB (Key Comparison Data Base).

The comparison was performed in a so-called loop format where the instrumented Charpy striker and its components were sent back to the pilot after the participating laboratory's measurements, completing the measurement cycle (pilot laboratory – participating laboratory – pilot laboratory). The pilot's first measurement is denoted A-measurement and its second, after the participating laboratory, is called the B-measurement. The change at the pilot (B-measurement – A-measurement) is called the drift for that particular loop.

#### 6. Methods of Measurement

For this comparison, two sets of 5 verification specimens, supplied by the pilot laboratory, were tested by the participating institute in each testing round. Each institute executed the Charpy impact tests and provided the values obtained for the required measurements, as reported in the results section. Each participant tested verification specimens at low and high energy levels using the same instrumented striker and data acquisition system. The verification specimens tested had been previously certified by the pilot laboratory, in a round-robin exercise, for maximum force (24,06 kN for the high energy specimens tested and 33,00 kN for the low energy specimens tested) [4].

The instrumented impact tests were conducted according to ISO 148-1 [1], ISO 148-2 [2], and ISO 14556 [5]. Figure 3 gives the dimensions of the standard-size Charpy V-notch verification specimen. Nominal dimensions and tolerances are provided in Table 2. Figure 3 shows a cross section of the 8 mm instrumented striker, while Table 3 provides a list of the components that were received by/shipped to the participants in this supplementary comparison.



Figure 3. Dimensions of the Charpy V-notch verification specimen (millimeters).

**Table 2.** Nominal dimensions and tolerances of the Charpy V-notch verification specimen.

Length (L):	55 <sup>+0,00</sup> <sub>-0,30</sub> mm
Width and thickness (W and B):	10,00 mm ± 0,07 mm
Ligament size (b):	8,000 mm ± 0,025 mm
Angle of V-notch ( $\theta$ ):	45,0° ± 1,0°
Root radius of V-notch (ρ):	0,250 mm ± 0,025 mm



**Figure 4.** The striker cross section provides approximate dimensions for the instrumented striker used in this comparison. The strain gauges are positioned on both sides of the 4 mm-thick beam that supports the striking edge. The center of the gauge is approximately 7 mm behind the leading edge of the striker and 12 mm below the top of the striker. The center of strike is about 10 mm from the top of the striker. The radius of the striking edge is 8 mm, in accordance with ASTM E23-18 [6].

Component	Description
Instrumented striker	Commercially available (MPM Technologies)
Strain amplifier	Commercially available (Micro Measurements)
Data acquisition system	Commercially available (National Instruments)
Cables	
Calibration software	NIST/INMETRO
Data acquisition software	NIST/INMETRO
Data analysis software	NIST/INMETRO
Jig for striker calibration	NIST/INMETRO
Supplementary comparison data sheet.xls	

**Table 3.** List of components used in the supplementary comparison.



#### 7. Assembly of the Instrumented Striker

In order to minimize discrepancies, the following procedure was followed by participating laboratories when mounting the striker on their Charpy impact machines.

- a) Lift and support the pendulum and remove the non-instrumented striker.
- b) Position the instrumented striker on the pendulum and engage the positioning pins.
- c) Torque the bolts to 74,6 N.m, as specified by the manufacturer.
- d) Attach the cable to the pendulum arm and ensure there is no interference in the hammer swing path.
- e) Connect the striker cables to the strain amplifier.
- f) Connect the strain amplifier and the encoder to the data acquisition system.
- g) Conduct several (at least 10) preliminary impact tests with specimens not included in the comparison test matrix, in order to seat the striker.
- h) Check the output signals for these preliminary tests and re-check the torque on the striker bolts.

#### 8. Experimental Conditions

- Test temperature: room temperature, *i.e.*, 22,0 °C ± 2,0 °C.
- Test 10 low-energy (nominal absorbed energy = 20 J) verification specimens.
- Test 10 high-energy (nominal absorbed energy = 100 J) verification specimens.

#### 9. Results

#### 9.1 Overview of Tests Performed and Parameters Calculated/Reported

The test matrix for the instrumented impact tests performed at NIST (lab code: 01) and INMETRO (lab code: 02) for the comparison is provided in Table 4. All tests were performed at room temperature (22 °C  $\pm$  2 °C). The instrumented Charpy striker conformed to the requirements of ISO 148-1 [1] and ASTM E23-18 [6] (radius of the instrumented striker = 8 mm, see also Figure 2).

Laboratory	Energy level	Specimen batch	Number of tests	Test date	
01	Low	LL-103	5	0/2012	
01	High	HH-103	5	0/2012	
02	Low	LL-103	5	2/2012	
	High	HH-103	5	5/2015	
02	Low	LL-103	5	1/2012	
02	High	HH-103	5	4/2013	
01	Low	LL-103	5	7/2012	
	High	HH-103	5	//2013	

For every test performed, the following parameters were measured and reported in accordance with ISO 14566 [5]:



Instrumented force values (F)

 $F_{gy}$  = force at general yield (kN)  $F_m$  = maximum force (kN)

Instrumented deflection values (s)

 $s_{gy}$  = deflection corresponding to force at general yield (mm)

*s<sub>m</sub>* = deflection corresponding to maximum force (mm)

*s*<sub>*t*</sub> = deflection corresponding to test termination (mm)

#### Instrumented absorbed energy values (W)

 $W_{gy}$  = absorbed energy corresponding to force at general yield (J)

 $W_m$  = absorbed energy corresponding to maximum force (J)

 $W_t$  = absorbed energy corresponding to test termination, or total absorbed energy (J)

In addition, the following quantities were measured and reported:

KV = absorbed energy provided by the impact machine encoder (J)

 $C_{el}$  = initial elastic compliance (machine + specimen) (mm/kN)

 $KV/W_t$  = ratio between absorbed energy values measured by the encoder and calculated from the instrumented force/deflection record.

Note that all tests, including those performed at INMETRO, were analyzed by NIST.

## 9.2 1<sup>st</sup> Test Series (NIST, August 2012)

Ten verification specimens, five of low-energy level (batch LL-103) and five of highenergy level (batch HH-103), were tested at NIST in August 2012. The Charpy impact machine used had a capacity (initial potential energy) of 358 J and an impact speed of 5,12 m/s.

Test results are provided in Tables 5 (low energy) and 6 (high energy). Tables include individual test values, average values, standard deviations ( $\sigma$ ), and coefficients of variation<sup>1</sup> (*CV*).

Table 5. Low-energy instrumente	${\sf I}$ impact test results for the 1 $^{ m s}$	<sup>t</sup> series of tests (Lab 01, 8/2012).
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Specimen	Fgy	C <sub>el</sub>	Fm	s <sub>gy</sub>	s <sub>m</sub>	s <sub>t</sub>	v <sub>gy</sub>	V m	V <sub>fin</sub>	W <sub>gy</sub>	Wm	Wt	KV	KV /M
id	(kN)	(mm/kN)	(kN)	(mm)	(mm)	(mm)	(m/s)	(m/s)	(m/s)	(J)	(J)	(L)	(L)	KV/VV <sub>t</sub>
131	24.62	0.02340	32.43	0.54	0.88	0.98	5.0683	4.9985	4.9890	7.19	16.79	18.08	19.02	1.052
365	24.71	0.02334	32.63	0.55	0.88	0.96	5.0677	4.9994	4.9915	7.27	16.66	17.74	18.33	1.033
398	25.14	0.02424	33.12	0.56	0.87	0.95	5.0652	4.9990	4.9913	7.62	16.72	17.77	18.07	1.017
562	24.26	0.02283	32.45	0.53	0.90	1.00	5.0702	4.9924	4.9825	6.93	17.62	18.97	19.63	1.035
817	23.74	0.02169	32.40	0.52	0.91	1.04	5.0728	4.9932	4.9840	6.57	17.50	18.76	19.28	1.028
Average	24.49	0.02310	32.61	0.54	0.89	0.99	5.0688	4.9965	4.9877	7.12	17.06	18.26	18.83	1.028
$\sigma$	0.525	9.37E-04	0.301	0.016	0.016	0.036	0.00284	0.00340	0.00418	0.392	0.463	0.569	0.652	0.013
CV, %	2.14	4.05	0.92	2.93	1.85	3.63	0.06	0.07	0.08	5.51	2.71	3.12	3.46	1.24

<sup>&</sup>lt;sup>1</sup> The coefficient of variation, expressed in percent, is calculated by dividing the standard deviation by the average value.



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Table 6. High-energy instrumented impact test results for the 1<sup>st</sup> series of tests (Lab 01, 8/2012).

Specimen	Fgy	C <sub>el</sub>	Fm	s <sub>gy</sub>	<i>s</i> <sub>m</sub>	s <sub>t</sub>	V gy	V m	V <sub>fin</sub>	W <sub>gy</sub>	Wm	Wt	KV	KV /W
id	(kN)	(mm/kN)	(kN)	(mm)	(mm)	(mm)	(m/s)	(m/s)	(m/s)	(L)	(L)	(L)	(L)	<i>kv</i> / <i>vv</i> <sub>t</sub>
74	20.58	0.02568	24.18	0.48	1.70	14.87	5.0802	4.8741	4.2756	5.54	33.56	108.32	105.00	0.969
346	20.44	0.02532	24.23	0.47	1.65	12.65	5.0814	4.8825	4.3146	5.38	32.44	103.74	107.69	1.038
365	20.44	0.02663	24.06	0.47	1.60	15.03	5.0802	4.8901	4.3354	5.54	31.42	101.28	106.94	1.056
667	20.41	0.02952	24.03	0.49	1.73	16.40	5.0781	4.8699	4.2903	5.83	34.11	106.60	112.62	1.056
729	20.21	0.02873	23.99	0.48	1.67	17.35	5.0805	4.8811	4.3077	5.51	32.62	104.56	110.73	1.059
Average	20.42	0.02718	24.10	0.48	1.67	15.26	5.0801	4.8795	4.3047	5.56	32.83	104.90	108.60	1.052
$\sigma$	0.133	1.86E-03	0.102	0.008	0.049	1.781	0.00121	0.00783	0.02293	0.165	1.044	2.700	3.051	0.038
CV, %	0.65	6.86	0.42	1.75	2.96	11.67	0.02	0.16	0.53	2.96	3.18	2.57	2.81	3.62

The comparison between instrumented force/time curves for specimens 365, 667, and 729 (high energy), shown in Figure 5, demonstrates the homogeneity of the material and the consistency of the test records. The specimens selected exhibit almost identical maximum forces (see Table 6 above) and slightly different values of absorbed energy *KV*. We note that curves are virtually indistinguishable up to maximum force, and deviations (justifying the differences in *KV*) are only visible after  $F_m$ .



**Figure 5.** Comparison between instrumented force/time traces for three high-energy specimens from the 1<sup>st</sup> test series (Lab 01).

After the 1<sup>st</sup> series of tests was completed, the instrumented striker and the instrumentation (strain amplifier, data acquisition box, and cables) were shipped from NIST to INMETRO.

## 9.3 2<sup>nd</sup> Test Series (INMETRO, March 2013)

Ten verification specimens, five of low-energy level (batch LL-103) and five of highenergy level (batch HH-103), were tested at INMETRO in March 2013. The Charpy impact machine used was similar to the NIST machine, and had a capacity (initial potential energy) of 408 J and an impact speed of 5,47 m/s.

With respect to the 1<sup>st</sup> series of tests, the following procedural features were different:



- (a) Sampling rate during acquisition: 100 kHz (was 500 kHz for the 1<sup>st</sup> series of tests performed at NIST).
- (b) No shunt calibration<sup>2</sup> of the instrumented striker was performed prior to testing.
- (c) The strain amplifier was not grounded.

Test results are provided in Tables 7 (low energy) and 8 (high energy).

Specimen	Fgy	C <sub>el</sub>	Fm	s <sub>gy</sub>	s <sub>m</sub>	s <sub>t</sub>	V gy	V m	V <sub>fin</sub>	W <sub>gy</sub>	Wm	Wt	KV	KV /W
id	(kN)	(mm/kN)	(kN)	(mm)	(mm)	(mm)	(m/s)	(m/s)	(m/s)	(J)	(L)	(J)	(J)	KV/VV <sub>t</sub>
241	19.21	0.02235	27.52	0.62	0.98	1.25	5.4373	5.3831	5.3673	4.87	12.87	15.19	19.37	1.275
259	19.69	0.02173	27.19	0.61	0.98	1.25	5.4355	5.3819	5.3667	5.14	13.05	15.28	19.67	1.287
321	19.13	0.02415	26.34	0.55	0.92	1.14	5.4347	5.3802	5.3680	5.25	13.30	15.09	19.97	1.323
572	18.51	0.02114	26.59	0.60	0.98	1.30	5.4450	5.3854	5.3673	3.73	12.53	15.20	19.82	1.304
605	18.75	0.01930	26.55	0.55	0.93	1.19	5.4400	5.3860	5.3655	4.47	12.45	15.45	20.27	1.312
Average	19.06	0.02173	26.84	0.59	0.96	1.23	5.4385	5.3833	5.3670	4.69	12.84	15.24	19.82	1.300
$\sigma$	0.454	1.77E-03	0.495	0.034	0.030	0.062	0.004	0.002	0.001	0.616	0.355	0.134	0.335	0.019
CV, %	2.38	8.13	1.85	5.74	3.17	5.05	0.08	0.04	0.02	13.13	2.77	0.88	1.69	1.48

 Table 7. Low-energy instrumented impact test results for the 2<sup>nd</sup> series of tests (Lab 02, 3/2013).

Table 8. High-energy instrumented i	mpact test results for the 2 <sup>nd</sup>	<sup>i</sup> series of tests (Lab 02, 3/2013).
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Specimen	Fgy	C <sub>el</sub>	Fm	s <sub>gy</sub>	s <sub>m</sub>	s <sub>t</sub>	V gy	V m	V <sub>fin</sub>	W <sub>gy</sub>	W <sub>m</sub>	Wt	KV	KV /W
id	(kN)	(mm/kN)	(kN)	(mm)	(mm)	(mm)	(m/s)	(m/s)	(m/s)	(L)	(J)	(J)	(J)	<i>KV / W</i> <sub>t</sub>
135	17.50	0.02195	19.86	0.48	1.45	15.09	5.0904	4.9538	4.5004	4.13	22.85	81.38	114.07	1.402
274	18.00	0.02122	19.69	0.50	1.51	12.14	5.0926	4.9511	4.5084	3.82	23.23	80.39	112.50	1.399
518	17.84	0.02481	19.75	0.56	1.54	12.35	5.0865	4.9510	4.5283	4.66	23.24	77.95	107.81	1.383
645	17.71	0.02064	19.67	0.62	1.61	13.03	5.0895	4.9549	4.5188	4.25	22.71	79.11	111.58	1.410
870	17.94	0.02271	19.84	0.52	1.39	13.80	5.0873	4.9636	4.5270	4.55	21.53	78.10	110.48	1.415
Average	17.80	0.02227	19.76	0.54	1.50	13.28	5.0893	4.9549	4.5166	4.28	22.71	79.39	111.29	1.402
$\sigma$	0.200	1.62E-03	0.086	0.055	0.084	1.201	0.002	0.005	0.012	0.336	0.701	1.481	2.348	0.012
CV, %	1.12	7.28	0.43	10.35	5.62	9.05	0.05	0.10	0.27	7.85	3.08	1.87	2.11	0.87

## 9.4 3<sup>rd</sup> Test Series (INMETRO, April 2013)

Ten verification specimens, five of low-energy level (batch LL-103) and five of highenergy level (batch HH-103), were tested at INMETRO in April 2013 using the same experimental setup as the 2<sup>nd</sup> series.

Following consultations between NIST and INMETRO, the following modifications to the experimental procedure were decided:

- (a) The sampling rate was set to 500 kHz.
- (b) Before testing, the shunt calibration of the instrumented striker was performed.
- (c) The strain amplifier was grounded.

Test results are shown in Tables 9 (low energy) and 10 (high energy).

<sup>&</sup>lt;sup>2</sup> Shunt calibration is a procedure aimed at adjusting the gain factor of the strain amplifier, using a value specified by the striker manufacturer.



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Table 9. Low-energy instrumented impact test results for the 3<sup>rd</sup> series of tests (Lab 02, 4/2013).

Specimen	Fgy	C <sub>el</sub>	Fm	s <sub>gy</sub>	<i>s</i> <sub>m</sub>	s <sub>t</sub>	V gy	V m	V <sub>fin</sub>	W <sub>gy</sub>	Wm	Wt	KV	KV/W.
id	(kN)	(mm/kN)	(kN)	(mm)	(mm)	(mm)	(m/s)	(m/s)	(m/s)	(L)	(J)	(J)	(J)	,t
25	24.44	0.01857	32.49	0.52	0.90	1.02	5.4288	5.3525	5.3395	6.13	17.36	19.25	19.84	1.031
144	23.30	0.01700	32.96	0.45	0.91	1.04	5.4354	5.3445	5.3346	5.15	18.53	19.96	20.65	1.035
386	23.64	0.01669	33.40	0.47	0.92	1.06	5.4349	5.3435	5.3360	5.22	18.67	19.77	19.67	0.995
521	25.15	0.01838	32.83	0.50	0.90	1.04	5.4274	5.3467	5.3380	6.34	18.20	19.47	20.27	1.041
1011	23.30	0.01776	32.65	0.45	0.84	1.05	5.4356	5.3584	5.3355	5.12	16.49	19.84	20.72	1.044
Average	23.97	0.01768	32.87	0.48	0.89	1.04	5.4324	5.3491	5.3367	5.59	17.85	19.66	20.23	1.029
$\sigma$	0.809	8.26E-04	0.348	0.031	0.031	0.015	0.004	0.006	0.002	0.593	0.915	0.291	0.470	0.020
CV, %	3.38	4.67	1.06	6.52	3.50	1.42	0.07	0.12	0.04	10.60	5.12	1.48	2.32	1.93

**Table 10.** High-energy instrumented impact test results for the  $3^{rd}$  series of tests (Lab 02, 4/2013).

Specimen	Fgy	C <sub>el</sub>	F <sub>m</sub>	s <sub>gy</sub>	<b>s</b> <sub>m</sub>	s <sub>t</sub>	V gy	V m	V <sub>fin</sub>	W <sub>gy</sub>	W <sub>m</sub>	Wt	KV	KV /W
id	(kN)	(mm/kN)	(kN)	(mm)	(mm)	(mm)	(m/s)	(m/s)	(m/s)	(J)	(J)	(J)	(J)	<i>KV / VV</i> t
155	22.50	0.02783	24.53	0.51	1.50	14.91	5.4269	5.2645	4.7175	6.41	30.11	104.64	110.84	1.059
372	22.25	0.02549	24.35	0.50	1.51	13.66	5.4293	5.2667	4.7114	6.06	29.79	105.43	111.76	1.060
377	22.33	0.02190	24.40	0.48	1.44	12.66	5.4313	5.2769	4.7394	5.76	28.33	101.81	108.09	1.062
696	22.41	0.02483	24.27	0.50	1.48	13.34	5.4289	5.2695	4.7326	6.12	29.39	102.69	108.36	1.055
1113	22.20	0.02196	24.49	0.51	1.59	14.11	5.4313	5.2561	4.7099	5.75	31.32	105.61	111.40	1.055
Average	22.34	0.02440	24.41	0.50	1.50	13.74	5.4295	5.2667	4.7222	6.02	29.79	104.04	110.09	1.058
$\sigma$	0.121	2.52E-03	0.105	0.012	0.055	0.842	0.002	0.008	0.013	0.276	1.088	1.699	1.736	0.003
CV, %	0.54	10.31	0.43	2.45	3.66	6.13	0.03	0.14	0.28	4.58	3.65	1.63	1.58	0.29

After the 3<sup>rd</sup> series of tests was completed, the instrumented striker was statically calibrated at INMETRO. During calibration, the striker was accidentally overloaded and consequently damaged. The complete instrumentation was then shipped back to NIST.

#### 9.5 4<sup>th</sup> Test Series (NIST, July 2013)

Ten verification specimens, five of low-energy level (batch LL-103) and five of highenergy level (batch HH-103), were tested at NIST in July 2013. The same experimental setup was used as for the 1<sup>st</sup> series of tests, although the instrumented striker was damaged during calibration at INMETRO (see above).

Test results are provided in Tables 11 (low energy) and 12 (high energy).

Specimen	Fgy	C <sub>el</sub>	Fm	s <sub>gy</sub>	<i>s</i> <sub>m</sub>	s <sub>t</sub>	V gy	V m	V <sub>fin</sub>	W <sub>gy</sub>	Wm	Wt	KV	KV /W
id	(kN)	(mm/kN)	(kN)	(mm)	(mm)	(mm)	(m/s)	(m/s)	(m/s)	(L)	(L)	(L)	(L)	KV/Wt
13	26.72	0.01911	33.85	0.54	0.91	1.05	5.0683	4.9822	4.9703	7.19	19.00	20.62	19.97	0.968
208	27.52	0.02157	33.66	0.57	0.92	1.05	5.0617	4.9811	4.9698	8.10	19.15	20.69	20.24	0.978
465	26.67	0.01913	34.34	0.52	0.91	1.04	5.0689	4.9807	4.9709	7.11	19.21	20.54	19.55	0.952
558	26.49	0.01861	33.77	0.52	0.92	1.05	5.0697	4.9802	4.9691	7.00	19.28	20.79	19.89	0.957
701	26.70	0.01889	33.66	0.52	0.94	1.12	5.0688	4.9740	4.9627	7.13	20.12	21.66	20.59	0.951
Average	26.82	0.01946	33.86	0.53	0.92	1.06	5.0675	4.9796	4.9686	7.31	19.35	20.86	20.05	0.961
$\sigma$	0.402	1.20E-03	0.282	0.022	0.012	0.033	0.003	0.003	0.003	0.449	0.442	0.457	0.390	0.012
CV, %	1.50	6.15	0.83	4.10	1.33	3.08	0.06	0.07	0.07	6.15	2.28	2.19	1.95	1.24

 Table 11. Low-energy instrumented impact test results for the 4<sup>th</sup> series of tests (Lab 01, 7/2013).

**Table 12.** High-energy instrumented impact test results for the 4<sup>th</sup> series of tests (Lab 01, 7/2013).

Specimen	Fgy	C <sub>el</sub>	F <sub>m</sub>	s <sub>gy</sub>	<i>s</i> <sub>m</sub>	s <sub>t</sub>	V gy	V m	V <sub>fin</sub>	W <sub>gy</sub>	W <sub>m</sub>	Wt	KV	KV /W
id	(kN)	(mm/kN)	(kN)	(mm)	(mm)	(mm)	(m/s)	(m/s)	(m/s)	(L)	(J)	(J)	(J)	
150	23.04	0.02353	25.36	0.50	1.52	10.86	5.0769	4.8906	4.2797	6.00	31.35	107.84	112.90	1.047
351	22.91	0.02157	25.29	0.48	1.55	11.01	5.0793	4.8847	4.2688	5.66	32.14	109.11	112.43	1.030
587	22.93	0.02343	25.60	0.50	1.51	12.39	5.0764	4.8931	4.2838	6.07	31.01	107.36	106.11	0.988
704	23.07	0.02102	25.48	0.47	1.46	10.71	5.0800	4.8983	4.2947	5.57	30.33	106.08	111.10	1.047
873	23.08	0.02517	25.62	0.51	1.53	12.36	5.0741	4.8866	4.2723	6.39	31.88	108.70	107.24	0.987
Average	23.01	0.02294	25.47	0.49	1.51	11.47	5.0773	4.8907	4.2799	5.94	31.34	107.82	109.96	1.020
$\sigma$	0.080	1.67E-03	0.145	0.016	0.034	0.837	0.002	0.005	0.010	0.331	0.718	1.191	3.093	0.030
CV, %	0.35	7.27	0.57	3.34	2.22	7.30	0.05	0.11	0.24	5.57	2.29	1.11	2.81	2.98



## 9.6 Comparison of Instrumented Impact Results

The results obtained at NIST (1<sup>st</sup> test series) and INMETRO (3<sup>rd</sup> test series) are compared in Tables 13 and 14 in terms of average values and coefficients of variation, respectively. The other two test series (2<sup>nd</sup> series at INMETRO and 4<sup>th</sup> series at NIST) have been excluded from the following comparative analyses due to, respectively, differences in the test procedure (see §10.3) and striker damage (see §10.4).

Specimen	Testing	Test	No. of	Fgy	C <sub>el</sub>	Fm	s <sub>m</sub>	s <sub>t</sub>	W <sub>gy</sub>	Wm	Wt	KV	KV /W
batch	lab	date	tests	(kN)	(mm/kN)	(kN)	(mm)	(mm)	(J)	(J)	(J)	(J)	<i>kv</i> / <i>v</i> <sub>t</sub>
11 102	01	8/12	5	24.49	0.02310	32.61	0.89	0.99	7.12	17.06	18.26	18.83	1.028
LL-105	02	4/13	5	23.97	0.01768	32.87	0.89	1.04	5.59	17.85	19.66	20.23	1.029
111 102	01	8/12	5	20.42	0.02718	24.10	1.67	15.26	5.56	32.83	104.90	108.60	1.052
HH-103	02	4/13	5	22.34	0.02440	24.41	1.50	13.74	6.02	29.79	104.04	110.09	1.058

**Table 13.** Comparison between average values obtained in the 1<sup>st</sup> and 3<sup>rd</sup> test series.

Table 14. Comparison betw	een coefficients of variation	n obtained in the 1 <sup>st</sup> and	d 3 <sup>rd</sup> test series.
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Specimen	Testing	Test	No. of	F <sub>gy</sub>	C <sub>el</sub>	Fm	s <sub>m</sub>	s <sub>t</sub>	W <sub>gy</sub>	Wm	Wt	KV
batch	lab	date	tests	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
11 102	01	8/12	5	2.14	4.05	0.92	1.85	3.63	5.51	2.71	3.12	3.46
LL-103	02	4/13	5	3.38	4.67	1.06	3.50	1.42	10.60	5.12	1.48	2.32
1111 102	01	8/12	5	0.65	6.86	0.42	2.96	11.67	2.96	3.18	2.57	2.81
пп-103	02	4/13	5	0.54	10.31	0.43	3.66	6.13	4.58	3.65	1.63	1.58

## 9.7 Statistical Analysis of Test Results Differences

In order to assess the statistical significance of the observed differences between mean values of total absorbed energy and maximum force for the two test series, the unpaired *t*-test was employed. This test is used to determine if two sets of data are significantly different from each other, under the assumption of a normal distribution, by testing the null hypothesis that the means of the two populations are equal. The *t*-test was applied with a significance level  $\alpha = 0,05$ . If the calculated two-tailed *P* value is lower than  $\alpha$ , the difference between the means of the two series is statistically significant.

The results of the unpaired t-tests are summarized in Table 15 ( $W_t$ ) and 16 ( $F_m$ ).

Based on the analyses performed, the mean values of  $W_t$  for low-energy specimens and  $F_m$  for high-energy specimens are statistically different, while  $W_t$  for high-energy specimens and  $F_m$  for low-energy specimens are not statistically different.

Charpy lot	Test series	Lab	N	Mean (J)	SD (J)	SEM (J)	t	df	SED (J)	Р	NOTES
LL-103	1st 3rd	01 02	5	18.26 19.66	0.569 0.291	0.255	4.8746	8	0.286	0.0012	Means are statistically different
HH-103	1st 3rd	01 02	5	104.90 104.03	2.700 1.699	1.207 0.760	0.6056	8	1.427	0.5615	Means are not statistically different

**Table 15.** Results of unpaired *t*-tests for *W*<sub>t</sub> results.

<u>LEGEND</u> N = number of tests performed

SD = standard deviation

SEM = standard error of the mean

df = degrees of freedom

SED = standard error of the difference between the means.



#### FORCE SUPPLEMENTARY COMPARISON BETWEEN NIST AND INMETRO SIM.M.F-S3

							•				
Charpy	Test	Lab	N	Mean	SD	SEM	+	dt	SED	D	NOTES
lot	series	Lau	IN	(kN)	(kN)	(kN)	L	u	(kN)	Р	NOTES
11 102	1st	01	5	32.61	0.301	0.135	1 2042	0	0.200	0.2417	Magna are not statistically different
LL-103	3rd	02	5	32.87	0.345	0.156	1.2043	ð	0.206	0.2417	Means are not statistically different
1111 400	1st	01	5	24.10	0.102	0.046	4 7200	0	0.000	0.0045	No
HH-103	3rd	02	5	24.41	0.105	0.047	4.7286	8	0.066	0.0015	Means <u>are</u> statistically different

#### **Table 16.** Results of unpaired *t*-tests for $F_m$ results.

## 9.8 General Observations

Graphical comparisons of  $F_m$ ,  $W_m$ , and  $W_t$  are shown in Figure 6. The distributions of maximum force values are reasonable, and slightly larger for the low energy level. The average differences in the maximum force values measured by the two laboratories are small: 0,26 J or 0,8 % at the low energy level, and 0,31 J or 1,3 % at the high energy level. Compared with the certified maximum force values (24,06 kN and 33,00 kN), laboratory 1 and 2 were 1,2 % and 0,39 % low at the low energy level. At the high energy level, laboratory 1 was 1,8 % high and laboratory 2 was 0,37 % low.

The absorbed energy calculated at maximum force for laboratory 2 at the low energy level is slightly higher than that calculated for laboratory 1, which is consistent with the slightly higher maximum force values for laboratory 2. This is not the case at the high energy level:  $W_m$  for laboratory 2 is lower than  $W_m$  for laboratory 1 ( $F_m$  was higher for laboratory 2). The difference in the total energy measured at the low energy level was 1,4 J. The difference measured in total energy at the high energy level was 0,86 J.



**Figure 6.** Box plots comparing results for laboratories 1 and 2 for  $F_m$ ,  $W_m$ , and  $W_t$  for the low- and high-energy levels.

A comparison of individual, representative force/deflection curves from the two series of tests is provided in Figures 7 (low energy) and 8 (high energy). The test with the KV



value closest to the average *KV* of each individual series was chosen as the most representative for the test series. Test records from laboratory 2 (green curves in Figures 6 and 7) exhibit a significant amount of electrical noise (cause not identified), but the shapes of the curves are similar, with the laboratory 2 curve having higher force values at both low and high energy levels. The curves for laboratory 1 have slightly more deflection at maximum force. It is a bit difficult to see in this example, due to noise, but curves at the high energy levels characteristically differ for one test compared to another. This is because fracture initiates just after the maximum force, which introduces the additional variables of crack extension and specimen-machine interactions.



Figure 7. Comparison of representative curves for low-energy specimens from the  $1^{st}$  and  $3^{rd}$  series.



**Figure 8.** Comparison of representative curves for high-energy specimens from the 1<sup>st</sup> and 3<sup>rd</sup> series.

#### 10. Calculation of Normalized Errors

In interlaboratory comparisons, the normalized error  $(E_n)$  compares measurement results and associated uncertainties from participating laboratories, to determine if they are in agreement. If the absolute value of  $E_n$  is greater than one, the different sets of measurements are not in agreement.

For this Key Comparison, normalized errors were calculated for values of maximum force,  $F_m$ , and instrumented energy,  $W_t$ , from the 1<sup>st</sup> and 3<sup>rd</sup> test series for the LL-103 and HH-103 Charpy lots. The data used for  $E_n$  calculation are summarized in Table 17, while the calculated values of  $E_n$  are provided in Table 18. Note that standard uncertainties, u, in Table 17 were calculated using the NIST Uncertainty Machine webbased software [7], assuming Gaussian distribution of the variables, while expanded uncertainties, U, were obtained by multiplying u by the coverage factor k = 1.96, corresponding to a level of confidence of 95 %.

Specimen	Test	Testing	Veriable	11.4.14	Mean	Standard		
batch	series	lab	variable	Unit	value	deviation	u	U
	1 ct	01	F <sub>m</sub>	kN	32.61	0.30	0.30	0.59
11 102	151	01	W <sub>t</sub>	J	18.26	0.57	0.57	1.12
LL-103	) rd	02	F <sub>m</sub>	kN	32.87	0.35	0.35	0.68
	310	02	W <sub>t</sub>	J	19.66	0.29	0.29	0.57
	1 ct	01	F <sub>m</sub>	kN	24.10	0.10	0.10	0.20
1111 102	151	01	W <sub>t</sub>	J	104.9	2.70	2.70	5.29
пп-103	ard	02	F <sub>m</sub>	kN	24.41	0.11	0.11	0.21
	510	02	W <sub>t</sub>	J	104.0	1.70	1.70	3.33

**Table 17.** Data used for the calculation of normalized errors.



 Table 18. Values of normalized errors calculated for this Key Comparison.

Lot ID	Variable	E <sub>n</sub>
11 102	F <sub>m</sub>	-0.29
LL-103	W <sub>t</sub>	-1.12
1111 100	F <sub>m</sub>	-1.08
пп-103	W <sub>t</sub>	0.14

According to the values presented in Table 18, test results for  $F_m$  (LL-103) and  $W_t$  (HH-103) are in agreement between the participating labs, whereas results for  $W_t$  (LL-103) and  $F_m$  (HH-103) are not in agreement. This outcome is consistent with the results of the *t*-tests described in section 9.7 above.

#### **11.** Summary and Conclusions

Examination of Table 13 and Figure 6 reveals that average force values for the 1<sup>st</sup> and 3<sup>rd</sup> test series are in reasonable agreement, with the exception of  $F_{gy}$  for the high-energy specimens (a difference of about 9 % is observed between the two series).  $F_{gy}$  is dependent on human judgment for its determination, so this value is not considered as useful for machine verification purposes. It was determined that more sophisticated curve smoothing and/or algorithms are needed to identify the maximum force for curves at the high energy level. A significant portion of the differences between  $W_m$  values were due to the procedures used. Improvement of procedures should reduce variation in the deflection values identified at maximum force, and help reducing within-laboratory variation.

Based on the examination of Table 14 and Figure 6, the scatter of test results is comparable across the two series. It is interesting to note that values of instrumented absorbed energies  $W_t$  have similar dispersion to KV values (absorbed energy measured by the encoder of the machine) and indicate similar differences between the results of laboratories. With the differences being similar and the traceability to SI more direct for  $W_t$ , instrumented testing is viewed as providing a more desirable measurement system. However, we would hope the  $W_t$  scale can be improved to reduce the bias between our laboratories, with respect to the KV measurement system. These initial comparisons have identified several areas in which improvements in  $W_t$  measurements can be made. Improvements include better curve fitting and algorithms for identifying the maximum force, and a better/"true" dynamic force calibration of the instrumented striker [8].



#### 11. Nomenclature

Energy absorbed in an impact test performed on a Charpy V-notch specimen
International Bureau of Weights and Measures
Consultative Committee
Consultative Committee for Mass and Related Quantities
International Committee for Weights and Measures
Brazilian National Institute of Metrology, Quality and Technology
International Standards Organization
Key Comparison Data Base
National Institute of Standards and Technology
National Metrology Institute
Reference Material
Regional Metrology Organization
International System
Inter-American System of Metrology
Metrology Working Group No. 7 of Mass and Related Quantities
Calibration and Measurement Capabilities

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