

NIST Internal Report NIST IR 8470

Test Temperature Range for NIST Certified Charpy Specimens for Testing at "Room Temperature"

Enrico Lucon

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Enrico Lucon Applied Chemicals and Materials Division Material Measurement Laboratory

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Abstract

The NIST Charpy Machine Verification Program is ready to introduce low-energy and high-energy certified specimens that can be tested at room temperature instead of -40 °C, thus accommodating the requests of many customers during the past 35 years. An investigation aimed at providing a practical and technically sound definition of "room temperature" (barycentric value and tolerance) was conducted, by examining previously obtained Charpy energy transition curves for different energy levels, as well as by testing certified low-energy and high-energy specimens at 21 °C (ambient temperature of the NIST Charpy Lab in Boulder, Colorado) and additional temperatures in the range 21 °C \pm 5 °C. Results have shown that for low-energy specimens, even within a relatively small 10 °C range, the influence of test temperature on Charpy absorbed energy can be significant enough to cause "good" machines to fail the requirements of the ASTM E23 standard. Therefore, at this energy level, the allowable test temperature range must be restricted to 21 °C \pm 1 °C. For the other two energy levels (high and super-high), the influence of test temperature is small enough that the allowable temperature range can be expanded to 21 °C \pm 3 °C.

Keywords

Charpy Machine Verification Program; Charpy transition curves; NIST certified Charpy specimens; room temperature.

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1. Introduction

Since the inception of the Charpy Machine Verification Program during the 1960s at the Watertown Arsenal in Massachusetts by the US Army [1], certified reference Charpy specimens of low-energy and high-energy level have always been required to be tested at -40 °C \pm 1 °C (-40 °F \pm 2 °F). It appears that this test temperature was selected because it corresponds to the only point in which the two most widely used temperature scales (Celsius and Fahrenheit) coincide. This would exclude the possibility that a user would misinterpret the units to be used for the test temperature, thus invalidating their results. Moreover, this temperature is low enough for results to be representative of brittle failures, such as the ones that were infamously recorded for the Liberty warships, many of which sank unexpectedly with the loss of many lives in the cold waters of the Atlantic Ocean during World War II, [2]-[4]. In one case (S.S. Schenectady, shown in Figure 1), the ship failed before even leaving the shipyard [2].



Figure 1 - Brittle failure of the S.S. Schenectady Liberty ship, which failed before leaving the shipyard in 1943. Reproduced from [2].

When NIST took over the Charpy Program in 1989 [5], the same test temperature was maintained for low- and high-energy reference specimens. However, when a new energy level (super-high-energy specimens) was introduced in the late 1990s, the test temperature was set at 21 °C \pm 1 °C (70 °F \pm 2 °F).

Over the last 34 years, many Charpy Program customers contacted NIST to advocate for the change of test temperature for low- and high-energy specimens from -40 °C to "room temperature"¹ (to be intended in a typical range of 18 °C – 25 °C), or alternatively for the

¹ The reason "room temperature" is shown here in quotation marks is that one can find many different definitions of "room temperature", see Section 2 of this report. For simplicity and following common custom, the quotation marks will not be used hereinafter.

addition of certified specimens to be tested at room temperature. The prevailing motivation for these requests was that many companies only perform Charpy tests at room temperature.

Recently, we decided to certify most future lots of low- and high-energy Charpy specimens at room temperature instead of -40 °C. The question has therefore arisen of how to precisely define room temperature, and more specifically what tolerance with respect to the central value (taken as 21 °C, or 70 °F – ambient temperature of the NIST Charpy Lab in Boulder, Colorado) to allow for the actual test temperature.

A very narrow tolerance, such as $\pm 1 \,^{\circ}C (\pm 2 \,^{\circ}F)$, might be difficult to achieve for many test laboratories, particularly those that are not equipped with (or cannot afford) sufficiently accurate systems of temperature control. On the other hand, a wide temperature range, such as $\pm 5 \,^{\circ}C (\pm 9 \,^{\circ}F)$, might significantly affect the measured absorbed energy values, particularly for low-energy specimens, that exhibit transitional fracture behavior at room temperature. Conversely, both high-energy and super-high-energy specimens exhibit fully ductile (upper shelf) behavior at room temperature, and therefore even a change in temperature of $\pm 5 \,^{\circ}C$ is not expected to significantly affect test results.

An investigation, primarily focused on low-energy samples, was therefore conducted to assess the consequences of varying the test temperature above and below 21 °C on the energy absorbed by NIST certified reference Charpy specimens. Specifically, the following two test temperature ranges were investigated: 18 °C to 24 °C (narrower temperature range) and 16 °C to 26 °C (wider temperature range). Intervals smaller than 6 °C or larger than 10 °C were considered too restrictive or too lenient, respectively.

2. Definitions of Room Temperature

A Google search for "definition of room temperature" returns numerous, sometimes conflicting results, as listed below.

Wikipedia defines room temperature as "a range of air temperatures that most people prefer for indoor settings", adding that "in certain fields, like science and engineering, (...) room temperature can mean different agreed-upon ranges." [6]

The American Heritage Dictionary of the English Language identifies room temperature as around 20 °C – 22 °C (68 °F – 72 °F) [7], while the Oxford English Dictionary states that it is "conventionally taken as about 20 °C (68 °F)." [8]

For the shipping and storage of pharmaceuticals, the United States Pharmacopeia-National Formulary (USP-NF) defines *controlled room temperature* the range between 20 °C and 25 °C (68 °F and 77 °F), with excursions allowed between 15 °C and 30 °C (59 °F and 86 °F) [9]. In contrast, the European Pharmacopoeia defines it as being simply 15 °C to 25 °C (59 °F to 77 °F) [10]. The same temperature range is indicated by the Merriam-Webster Medical Dictionary as being "*suitable for human occupancy*." [11]

The ISO 1:2022 standard specifies the standard reference temperature for geometrical product specification and verification as 20 °C (68 °F) [12]. Among the reasons for choosing 20 °C was that this is a comfortable and practical workshop temperature, which corresponds to an integer value on both the Celsius and Fahrenheit scales.

Both the International Union of Pure and Applied Chemistry (IUPAC) [13] and the U.S. Environmental Protection Agency (EPA) [14] define room temperature as 25 °C (77 °F).

According to the New World Encyclopedia, for scientific calculations room temperature is taken to be roughly 20 °C to 23.5 °C (68 °F to 74.3 °F), with an average of 21 °C (69.8 °F). However, room temperature is *not* a precisely defined scientific term, as opposed to Standard Temperature and Pressure, which has several, slightly different, definitions [15].

For all future Charpy specimen lots certified at NIST for room temperature testing, the barycentric value of the allowable test temperature range will be 21 °C, which is the air temperature of the NIST Charpy Laboratory in Boulder, Colorado, where certification tests are routinely performed.

3. Examination of Previously Obtained Charpy Energy Transition Curves

During the course of previous investigations [16]-[17], we had obtained absorbed energy (KV) vs. test temperature (T) curves for several certified lots of low-, high-, and super-high-energy levels.

Four low-energy lots from 4340 steel (LL-119, LL-133, LL-140, and LL-141) had been characterized by conducting Charpy tests at temperatures ranging from lower shelf (fully brittle behavior) to upper shelf (fully ductile behavior). The corresponding KV vs. T transition curves are illustrated in Figure 2 in the range -50 °C to 50 °C. Experimental data points (between 10 and 14for each lot) are not shown for the sake of clarity.



Figure 2 - Charpy energy transition curves for 4 low-energy lots in the range -50 °C \div 50 °C. The long dash line indicates 21 °C, the dashed lines 18 °C and 24 °C (narrower range, 21 °C ± 3 °C), and the dotted lines 16 °C and 26 °C (wider range, 21 °C ± 5 °C). The dashed-and-dotted lines corresponds to -40°C (current test temperature for low-energy lots).

All the curves shown in Figure 2 show upper transitional behavior at both -40 °C and 21 °C, with qualitatively similar slopes. Energy values at 21 °C from the transition curves correspond to between 63 % and 71 % of the respective upper shelf energies.

Conversely, based on the Charpy energy transition curves shown in Figure 3, a highenergy lot (HH-143) and a super-high-energy lot (SH-38) exhibit upper shelf conditions between 16 °C and 26 °C (100 % and 98 % of the respective upper shelf energies at 21 °C).



Figure 3 - Charpy energy transition curves for a high-energy lot and a super-high-energy lot in the range -50 $^{\circ}$ C \div 50 $^{\circ}$ C.

Based on the transition curves illustrated in Figure 2 and Figure 3, the absorbed energies values at 21 °C and at the limits of the wider (16 °C and 26 °C) and narrower (18 °C and 24 °C) test temperature ranges are listed in Table 1. The corresponding variations of absorbed energy with respect to the barycentric temperature (21 °C) are shown in Table 2.

Energy	Lot id	Charpy absorbed energy (J) at				
level		16 °C	18 °C	21 °C	24 °C	26 °C
	LL-119	17.90	18.03	18.21	18.40	18.52
Low	LL-133	17.19	17.33	17.54	17.75	17.89
LOW	LL-140	18.90	19.04	19.26	19.47	19.61
	LL-141	22.20	22.39	22.68	22.97	23.15
High	HH-143	111.19	111.22	111.25	111.28	111.30
Super-High	SH-38	173.61	173.83	174.14	174.42	174.59

Table 1 - Absorbed energy values at different test temperatures between 16 °C and 26 °C for various low-, high-, and super-high-energy Charpy lots.

Enorgy		Wider range		Narrower range	
Litergy	Lot id	ΔKV_{26-21}	ΔKV_{21-16}	ΔKV_{24-21}	ΔKV_{21-18}
level		(J)	(J)	(J)	(J)
	LL-119	0.31	0.31	0.18	0.19
T	LL-133	0.35	0.35	0.21	0.21
LOW	LL-140	0.36	0.36	0.21	0.22
	LL-141	0.47	0.48	0.28	0.29
High	HH-143	0.05	0.06	0.03	0.03
Super-High	SH-38	0.45	0.53	0.28	0.31

Table 2 - Absorbed energy, *KV*, differences between extreme (16 °C, 18 °C, 24 °C, 26 °C) and barycentric (21 °C) test temperatures for various low-, high-, and super-high-energy Charpy lots.

For low-energy lots, energy differences vary significantly among the four lots considered, ranging from 0.31 J (LL-119) to 0.48 J (LL-141) for the narrower range, and from 0.18 J (LL-119) to 0.29 J (LL-141) for the narrower range. The largest differences (LL-141) correspond to 34 % and 21 %, respectively, of the maximum allowable difference between NIST certified values, KV_R , and average absorbed energy from 5 indirect verification Charpy tests (1.4 J) according to ASTM E23.

For the high-energy lot, the largest energy differences are 0.06 J and 0.03 J for the wider and narrower energy range respectively, corresponding to 0.06 % and 0.03 % of KV_R for HH-143 (100.4 J). For the super-high-energy lot, the largest differences are 0.53 J (0.30 % of KV_R) and 0.31 J (0.18 % of KV_R) for the wider and the narrower range. Considering that the maximum difference allowed by ASTM E23 at these energy levels is 5 % of KV_R , we can conclude that the influence of test temperature within both considered ranges is negligible for high- and superhigh-energy lots.

4. Charpy Tests on Low- and High-Energy Specimens Within the Test Temperature Ranges Investigated

The analyses conducted on existing transition curves indicated that test temperature tolerance should only be critical for low-energy specimens.

In order to corroborate the conclusions reached after analyzing the transition curves in Figure 2, new tests were conducted on low-energy specimens from two lots (LL-176 and LL-192) and high-energy specimens from one lot (HH-181) at the 5 temperatures listed in Table 1 (16 °C, 18 °C, 21 °C, 24 °C, and 26 °C). Five specimens were tested at each temperature and for each lot, with one exception (4 tests at 21 °C for LL-192).

Results are provided in Table 3, in terms of individual absorbed energies, average values, standard deviations SD, and coefficients of variation CV (ratio between standard deviation and average value). Experimental data points are shown in Figure 4 (low-energy) and Figure 5 (high energy), with linear trendlines. All three lots display a generally increasing trend of absorbed energy with test temperature within the wider range ($16 \degree C \le T \le 26 \degree C$, blue linear trendlines), whereas the trend becomes variable inside the narrower range ($18 \degree C \le T \le 28 \degree C$, orange linear trendlines).

Table 3 – Absorbed energy values obtained from Charpy tests performed between 16 $^\circ$ C and 26 $^\circ$ C on two low-energy lots and one high-energy lot.

T . 4 ° J	Charpy absorbed energy (J) at					
Lot 1a	16 °C	18 °C	21 °C	24 °C	26 °C	
	20.150	19.795	19.180	22.057	20.140	
	17.376	18.494	21.265	19.968	21.880	
LL-176	18.415	19.274	20.656	22.144	21.880	
	18.675	17.716	21.700	20.577	20.488	
	17.722	19.621	20.830	21.882	21.532	
Average	18.468	18.980	20.726	21.326	21.184	
SD	1.075	0.865	0.955	0.990	0.816	
CV	5.8 %	4.6 %	4.6 %	4.6 %	3.9 %	
	18.241	18.667	17.882	18.321	18.839	
	18.501	18.581	18.660	17.370	18.839	
LL-192	17.376	19.448	18.747	18.408	19.706	
	18.588	18.927	19.267	18.927	19.012	
	19.021	18.667		19.795	17.368	
Average	18.345	18.858	18.639	18.564	18.753	
SD	0.610	0.354	0.571	0.888	0.853	
CV	3.3 %	1.9 %	3.1 %	4.8 %	4.5 %	
	97.63	106.99	97.82	101.88	105.10	
	102.83	105.38	108.98	103.49	110.30	
HH-181	100.56	109.64	107.37	110.59	113.62	
	111.06	109.55	102.83	112.29	113.05	
	111.25	104.25	96.12	108.32	115.80	
Average	104.67	107.16	102.62	107.31	111.57	
SD	6.20	2.43	5.67	4.49	4.12	
CV	5.9 %	2.3 %	5.5 %	4.2 %	3.7 %	



Figure 4 – Charpy absorbed energies for LL-176 (left) and LL-192 (right). Blue lines are linear fits of data points inside the wider temperature range, while orange lines are linear fits of data points inside the narrower range.



Figure 5 – Charpy absorbed energies for HH-181. The blue line is a linear fit of the data points inside the wider temperature range, while the orange line is a linear fit of data points inside the narrower range.

The results obtained were statistically analyzed to assess the influence of test temperature within the two investigated ranges, as detailed below.

4.1. Single-factor ANOVA for the Equality of Means

Mean values of absorbed energy obtained at each temperature within the wider range (16 °C, 18 °C, 21 °C, 24 °C, 26 °C) and within the narrower range (18 °C, 21 °C, 24 °C) were statistically compared at a confidence level $\alpha = 0.05$ by conducting a one-factor analysis of variance [18], which is a special case of analysis of variance (ANOVA), and a generalization of the two-sample *t*-test.

In the case of the LL-176 lot, means are statistically different (p < 0.05) within both ranges (16 °C $\leq T \leq$ 26 °C and 18 °C $\leq T \leq$ 24 °C). Conversely, mean absorbed energies for the LL-192 and HH-181 lots were found not to be statistically different (p > 0.05) in both test temperature ranges. The results of the ANOVA analyses are summarized in Table 4.

The different behavior of the two low-energy lots may appear surprising, but despite being manufactured from the same steel and subjected to the same nominal heat treatment, lot-to-lot differences in mechanical behavior are to be expected.

Table 4 - Outcome of the ANOVA analyses on mean energy values for the three specimen lots. F and Fcrit
are the calculated and critical values, respectively, of the statistical parameter determined by the ANOVA
analyses. If $F > F_{crit}$, $p < 0.05$ and the differences between the means are statistically significant.

Lot id	T range	F	F _{crit}	р	Outcome
LL-176	16 °C to 26 °C	9.780293	2.866081	0.000150	Significant
	18 °C to 24 °C	8.439217	8.439217	0.005148	Significant
LL-192	16 °C to 26 °C 18 °C to 24 °C	$0.400648 \\ 0.273502$	2.895107 3.982298	$0.805736 \\ 0.765736$	Not significant Not significant
HH-181	16 °C to 26 °C	2.481720	2.866081	0.076714	Not significant
	18 °C to 24 °C	1.832926	3.885294	0.202006	Not significant

4.2. Analyses of Variance on the Slopes of Linear Regressions

The slope of the linear trendlines shown in Figure 4 and Figure 5 were also statistically assessed by additional analyses of variance (ANOVA) calculations, aimed at establishing if the observed trends of absorbed energy as a function of test temperature are statistically significant at a confidence level $\alpha = 0.05$. In this case, the influence of temperature on Charpy absorbed energy is statistically significant if $p \le 0.05$.

For the LL-176 lot and both temperature ranges, the slopes were found to be statistically different from 0, indicating that test temperature does indeed affect absorbed energy. The opposite outcome (slopes not statistically different from 0) was obtained for the LL-192 lot. The results are presented in Table 5.

Table 5 - Outcome of the ANOVA analyses on the slopes of the linear regressions between temperature and absorbed energy. t is the calculated value of the statistical parameter determined by the analysis.

Lot id	T range	Slope (J/°C)	t	р	Outcome
LL-176	16 °C to 26 °C 18 °C to 24 °C	0.29461 0.37568	5.655456 3.745234	9.32×10 ⁻⁶ 0.00245	Significant Significant
LL-192	16 °C to 26 °C 18 °C to 24 °C	0.01481 -0.04721	0.393856 -0.73202	0.697658 0.478210	Not significant Not significant
HH-181	16 °C to 26 °C 18 °C to 24 °C	$0.55303 \\ 0.17840$	2.090526 0.370033	0.047817 0.717316	Not significant Not significant

The results obtained indicate that two low-energy lots, both acceptable according to NIST specifications, can display quite different test temperature sensitivities. This is also underscored by the differences between mean energy values at 21 °C and at the upper and lower limits of the two investigated ranges. As shown in Table 6, for LL-176 differences are large with respect to the allowed tolerance of the ASTM E23 standard (1.4 J) for both temperature ranges, particularly below 21 °C. Contrarily, differences are much smaller or even negative for lot LL-192.

Table 6 – Absolute differences between mean absorbed energy values between extreme (16 °C, 18 °C, 24 °C, 26 °C) and barycentric (21 °C) test temperatures for two low-energy Charpy lots.

	Wider	range	Narrower range		
Lot id	ΔKV_{26-21}	ΔKV_{21-16}	ΔKV_{24-21}	ΔKV_{21-18}	
	(J)	(J)	(J)	(J)	
LL-176	0.46	2.26	0.60	1.76	
LL-192	0.11	0.29	-0.08	-0.22	

5. Conclusions

A new definition of "room temperature" (barycentric temperature and tolerance) was researched in view of the imminent introduction of low-energy and high-energy Charpy reference specimens tested at room temperature (instead of -40 °C) for the indirect verification of impact machines according to ASTM E23. Assuming that the allowable test temperature range must be centered on 21 °C (ambient temperature of the NIST Charpy Laboratory in Boulder, Colorado, where Charpy lots are certified), the most suitable tolerance range was established on the basis of the expected temperature sensitivity of fracture energies for low- and high-energy specimens.

The following conclusions were obtained.

- For high-energy and super-high energy lots, the influence of test temperature on absorbed energy, KV, can be considered negligible. Both materials (4340 and 9310 steels respectively) are in upper shelf around 21 °C, so that KV variations are much smaller than the E23 acceptance limits (\pm 5 % of the certified energy, K_R).
- On the contrary, low-energy specimen lots (4340 steel with different heat treatment) exhibit ductile-to-brittle transitional behavior at room temperature, and can therefore be much more sensitive to test temperature variations.
- Based on the analysis of previously obtained low-energy transition curves, a relatively narrow range of 21 °C ± 3 °C seems to represent a reasonable choice, considering the relevant ASTM E23 requirement ($K_R \pm 1.4$ J).
- Further analyses conducted on a number of Charpy test results generated in the range 16 °C $\leq T \leq 26$ °C for two recently certified low-energy lots showed, however, a different temperature sensitivity between them:
 - For LL-192, test temperature influence was found to be not statistically significant within the range considered. Mean absorbed energy differences were lower than those calculated from the transition curves examined above. A high-energy lot (HH-181) examined in a similar fashion also showed relative insensitivity to test temperature, particularly between 18 °C and 24 °C.
 - \circ For LL-176, all statistical tests performed indicated a significant influence of test temperature on Charpy energy. Even within the narrower range considered (21 °C ± 3 °C), energy differences were large with respect to 1.4 J.

On account of the investigations performed, the following decisions concerning the test temperature requirements for future "room temperature" low- and high-energy NIST certified lots have been taken:

- Low-energy NIST certified specimens will have to be tested at 21 °C \pm 1 °C (same tolerance as the current -40 °C lots). While such a tight tolerance may be impractical for many labs, it should prevent many Charpy machines to fail the ASTM E23 verification process due to test temperature variations. NIST should however recommend that any customer who does not think they can control the laboratory temperature within the required tolerance, use reference Charpy specimens to be tested at -40 °C \pm 1 °C, so that appropriate specimen cooling equipment can be used. If necessary, obviously, the same equipment could be used to maintain the temperature of the test specimens at 21 °C \pm 1 °C.
- High-energy NIST certified specimens will be tested at 21 °C ± 3 °C. This wider tolerance is justified by the fact that, at this energy level and within this interval, test temperature influence is negligible.

For consistency, the test temperature of super-high-energy NIST certified specimens (currently set at 21 °C \pm 1 °C) should also be changed to 21 °C \pm 3 °C.

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