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Integrated Data Collection Analysis (IDCA) Program-Sandpaper Issues in Drop Hammer Testing

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Integrated Data Collection Analysis (IDCA) Program —Sandpaper Issues in Drop Hammer Testing

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ABSTRACT

This report summarizes the IDCA observations of the effect of sandpaper in the drop hammer experiment on measured sensitivity of energetic materials.

Keywords: Small-scale safety testing, Proficiency Test, round-robin test, safety testing protocols, HME, RDX, potassium chlorate, potassium perchlorate, sugar, dodecane, aluminum, sandpaper in drop hammer

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1.1 Sandpaper Discussion from the $KClO_4$/Al Report (014)

Table 3 shows the results of impact testing of the $KClO_4$/Al mixture as performed by LANL, LLNL, IHD, and AFRL. Differences in the testing procedures are shown in Table 2, and the notable differences are the sandpaper grit size, amount of sample, and the methods for detection of a positive event. All laboratories used 180-grit sandpaper, and LLNL used 120-grit flint paper, in addition, for the impact testing. All participants performed data analysis by modified Bruceton method[13,14] and LANL and AFRL also performed data analysis by the Neyer method[15].

Table 3. Impact testing results for $KClO_4$/Al mixture

Lab[1]	Test Date	T, °C	RH, %[2]	DH_{50}, cm[3]	s, cm[4]	s, log unit[4]
LLNL (120)	5/3/10	23.3	22	> 177	NA[5]	NA[5]
LLNL (120)	5/19/10	22.8	28	> 177	NA[5]	NA[5]
LLNL (120)	5/26/10	23.3	24	> 177	NA[5]	NA[5]
LLNL (180)	10/19/11	23.3	29	17.9	2.9	0.07
LLNL (180)	10/19/11	23.9	30	16.8	2.0	0.04
LLNL (180)	10/20/11	23.3	31	16.1	0.7	0.02
LANL (180)	4/16/10	22.3	24.8	56.7	21.4	0.16
LANL (180)	4/19/10	21.1	26.0	60.0	5.5	0.04
LANL (180)	4/20/10	21.5	24.0	69.7	6.4	0.04
IHD (180)	11/11/10	22	42	43	8.0	0.08
IHD (180)	11/15/10	22	43	39	6.3	0.07
IHD (180)	11/16/10	20	48	42	10.8	0.11
AFRL (180)	9/20/11	23.9	56	42.9	4.9	0.05
AFRL (180)	9/21/11	23.9	54	45.1	8.4	0.08
AFRL (180)	10/19/11	22.8	40	45.4	7.3	0.07
AFRL (180)	10/19/11	23.9	39	36.8	7.7	0.09
AFRL (180)	10/19/11	23.9	38	36.9	2.6	0.03

1. Number in parentheses indicates grit size of sandpaper; 2. Relative humidity; 3. Modified Bruceton method, in cm, load for 50% probability of reaction (DH_{50}); 4. Standard deviation; 5. NA = not applicable.

The test results from the three participating laboratories for impact show a large range for DH_{50} from 16 cm to insensitive. The average values for 180-grit sandpaper are (in cm) LLNL 16.9 ± 0.9; IHD, 41.3 ± 2.1; AFRL 41.4 ± 4.3; LANL, 62.1 ± 6.8. LLNL used two sandpaper sizes, 120-grit and 180-grit. The average values based on grit size are: 120, insensitive (exceeds equipment response): 180, 40.6 ± 15.8 cm (14 determinations). The standard deviation is below the 0.16 log unit range where applicable. The impact of step spacing will be evaluated in detail in a later report.

There are differences in methodologies and equipment configurations among the participating laboratories, so comparison of results for the same test is useful to highlight any differences in SSST testing techniques. For impact testing, when using 180-grit sandpaper, all participants show the $KClO_4$/Al mixture to be relatively insensitive. LLNL found it more sensitive than the LLNL-determined sensitivity of RDX while the other participants found the $KClO_4$/Al mixture value less sensitive than their determined sensitivity of RDX. This is further complicated by the LLNL determined sensitivity of the $KClO_4$/Al mixture using 120-grit sandpaper. LLNL could not measure a positive event in the drop hammer testing range, which has a high limit of > 177 cm.

There are two issues presented by these results: 1) a substantial difference in DH_{50} values when different grit size sandpapers are used, and 2) a difference in DH_{50} values when the same grit size sandpaper is used. Sandpaper grit size has been determined a factor in previous materials in the Proficiency Test. The sensitivity of the RDX standard, as measured by LLNL using 120-grit sandpaper, LANL using 150-grit sandpaper, and IHD and AFRL using 180-grit sandpaper, has been in the range of 15 to 25 cm. Over the course of the Proficiency Test, when all the participants use 180-grit sandpaper, the average DH_{50} value for RDX is 19.8 ± 2.8 cm (19 determinations), or a 14% relative deviation. A much bigger variation was observed in the average DH_{50} value for the $KClO_3$/dodecane mixture[23] when the participants used the different grit size sandpapers shown above—15.9 ± 12.3 cm, or a relative deviation of 77%. Much less variation was observed in the average DH_{50} value for the $KClO_3$/dodecane mixture when only 180-grit sandpaper was used—9.2 ± 1.7 cm, or a relative deviation of 18%.

In this case for the $KClO_4$/Al, use of the standardization of the sandpaper did not bring the DH_{50} values in agreement, with LLNL showing the material more sensitive than the other participants, even when the same adjustment in method worked well for RDX and $KClO_3$/dodecane. This would suggest that a different parameter is affecting the comparison. At this time, the cause is unknown.

The radical difference in sensitivity found using 120-grit sandpaper compared to 180-grit sandpaper can be explained a number of ways. The sandpaper in the impact experiment provides two functions—to hold the sample in place and to provide sites for reactions to occur. Sandpapers are generally made of very hard solid materials to withstand erosion during use. In the drop hammer application, erosion is not the primary affect on structure of the sandpaper, fracturing is. The particles that make up the grit are subjected to impacting shock in the drop hammer as opposed to friction wear by a constant rubbing action in normal use. These particles make the sites for reaction during the drop hammer experiment, and if the materials are reactive, the amount of these sites can determine whether the reaction will be detected during the experiment.

Table 3 shows different sensitivity of the $KClO_4$/Al mixture based on the grit size of the sandpaper. The reason or reasons are not clear at this time. However, there are several potential reasons for differences between 120-grit sandpaper DH_{50} values vs. the 180-grit sandpaper DH_{50} values. These potential sources are being examined with further experimentation.

- Particle size of mixture vs. grit size of sandpaper—the 120-grit and the $KClO_4$/Al mixture are greatly mismatched and the fine powder may fall between the grains of the sandpaper, preventing much contact of the striker. In the 180-grit case, the grit of the sandpaper and the particle size of the $KClO_4$/Al mixture are closer in size allowing for better contact.

  Figure 1 illustrates the size differences. Shown are the Scanning Electron Micrograph (SEM) images of $KClO_3$ (top left), $KClO_4$ (top right), 120-grit sandpaper (bottom left) and 180-grit (bottom right). The $KClO_4$ image shows a very fine material. The Al used in these tests is even finer (image not shown). Mixed together, they make an extremely fine material. The 120-grit sandpaper has very large grain size compared to this mixture. If the $KClO_4$/Al mixture is put on the 120-grit sandpaper, the $KClO_4$/Al mixture could possibly get lost in the grit matrix. Then the striker of the Drop Hammer does not really have much contact with the $KClO_4$/Al mixture because the grit of the 120-grit sandpaper physically prevents much contact and therefore does not provide enough sites for the reactions to start. With the 180-grit sandpaper, however, the grit size is small, the density of grains is about the same as the 120-grit sandpaper[23], and so the striker has better contact and more sites for reactions to start. For $KClO_3$, this mismatch of mixture particle size and sandpaper grit size is not as extreme, so there are more sites for reactions for both sandpapers. As a result, our particular $KClO_3$/fuel mixtures show less sandpaper grit size dependency than the $KClO_4$/Al mixture.

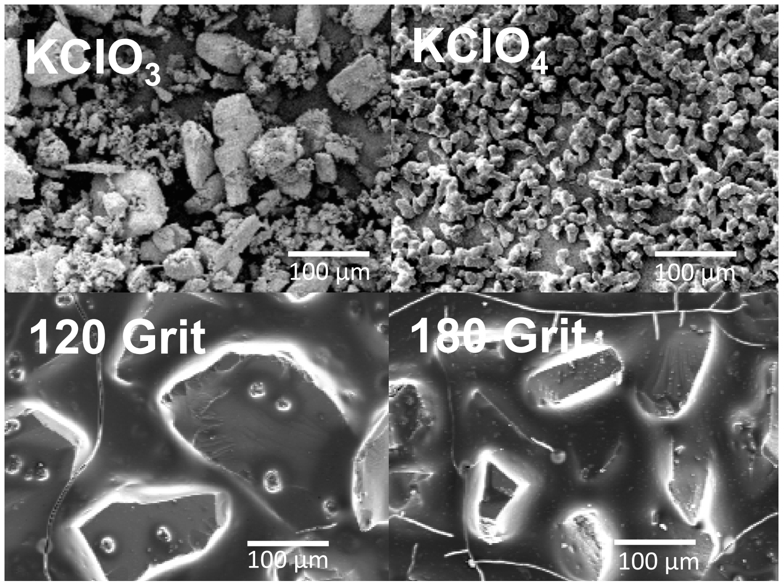


Figure 1. Scanning Electron Micrographs of $KClO_3$, $KClO_4$, 120-grit sandpaper, and 180-grit sandpaper at the same magnification.

The mismatch of the particle size of the $KClO_4$ and the 120-grit Si/C sandpaper is further illustrated in Figure 2 that displays the particle size distribution (by laser light scattering) for both the $KClO_3$ and $KClO_4$ starting materials. The $KClO_4$ distribution is shifted significantly to small size compared to the $KClO_3$ distribution. Also shown are the mean diameters of the grit particles of the 120- and 180-grit sandpapers based on the CAMI specification[24]. For the $KClO_3$, both the 120- and 180-grit average size fall in the size range of the oxidizer. For the $KClO_4$, only the 180-grit average size fall in the particle size range. The 120-grit average size does not overlap at all with the $KClO_4$ particle size range further supporting the argument above. A similar grit size particle size distribution relationship is seen when comparing particle size distributions as measured by Coulter Counter[4,5].

- Grit composition of the sandpaper—the 180-grit sandpaper is garnet while the 120-grit composition is silicon carbide (previously mislabeled as flint). The latter material has different crystal morphology than the garnet paper, and, as well has different hardness and fracture properties than garnet (garnet—6.5 to 7.5, silicon carbide—9 to 10, on Mohs' hardness scale)[25]. This could greatly affect the interaction of the sandpaper with the $KClO_4$/Al, and therefore the number of sites for reaction. The action of the striker on the more friable garnet paper could generate more sites for reaction compared to the Si/C paper.
- Thickness of the sandpaper backing—the 120-grit paper is a "wet" paper indicating that it can be used in wet or dry applications. The backing appears almost like a woven fabric (consistent with wet/dry papers). The 180-grit garnet paper is visibly

much thinner and appears more paper like. Simple measurements of the thickness of the unused intact paper are: 180-grit garnet—0.229 mm (0.009 in); 120-grit Si/C—0.406 mm (0.016 in). Because the drop hammer is a shock experiment, the thicker paper could absorb more of the impact and diffuse the amount of energy transferred from the striker to the $KClO_4$/Al.

- Bonding agent on the sandpaper—the 120-grit paper is a "wet" paper indicating that it can be used in wet or dry applications, while the 180-grit sandpaper is for dry use only. Figure 3 shows photographs of the front and back of both sandpapers. The color and coding show the differences in the two types of paper. The adhesive to keep the grit in place is likely to be different[26]. The standard garnet generally has an adhesive, such as hide glue (animal connective tissue). The wet type sandpaper is likely to have a water insoluble resin. The effect on the DH_{50} is unknown. However, NSWC-IHD has seen effects of adhesive on impact testing of ammonium perchlorate (NH_4ClO_4) mixtures. These effects are large enough that IHD does not use sandpaper when testing mixtures containing NH_4ClO_4.

Experimentation is under way to clarify these issues.

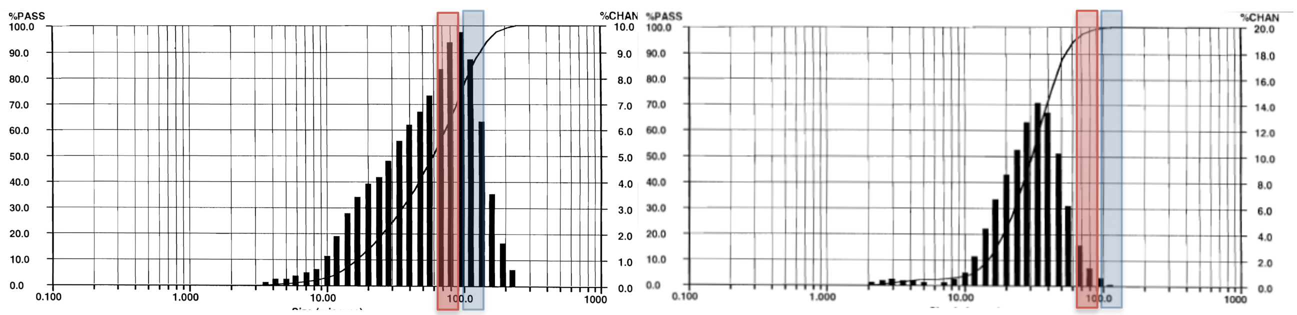


Figure 2. Particle size distribution of $KClO_3$ (left side) and $KClO_4$ (right side) and 180-grit sandpaper (red overlay) and 120-grit sandpaper (blue overlay) from CAMI specifications.

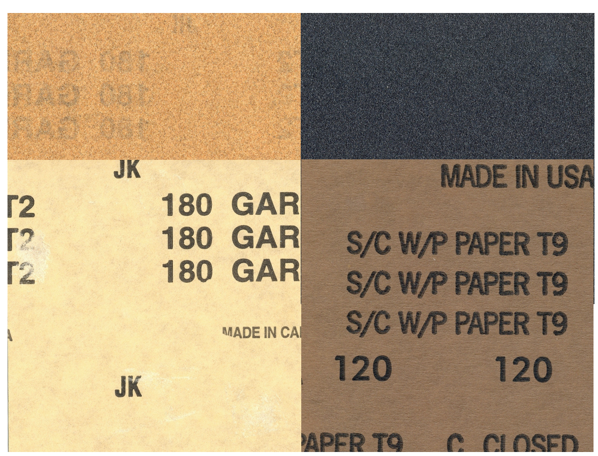


Figure 3. Photographs of the front and back of sandpapers used in drop hammer testing—180-grit garnet paper on the left side, 120-grit Si/C paper on the right side.

1.2 Sandpaper issue from Methods Report (009)

Modification—Sandpaper Type. The first material tested in the Proficiency Test was the RDX standard. The results from testing by LLNL, LANL, IHD and AFRL went as expected, because RDX has been extensively tested over the years. When the solid oxidizer/sugar mixtures were tested, an interesting feature appeared in the impact sensitivity data—the 50% probability of reaction level was sandpaper dependent. Three different sandpapers were being used for the testing, the type depending upon the participant. LLNL and LANL were using 120-grit Si/C and 150-grit garnet sandpapers, respectively. IHD and AFRL were using 180-grit garnet sandpaper. In testing RDX, this made little difference. However, in testing most of the HMEs, the difference among results from the different sandpapers was dramatic.

Table 5 shows the DH_{50} values of several Proficiency Test materials relative to RDX DH_{50} values, in cm, using 120-grit and 180-grit sandpapers. The sandpaper used for the RDX measurements that the mixtures were compared to the same.

Table 5. DH_{50} values[1], in cm, of selected test materials, relative to DH_{50} values, in cm, of RDX[2]

Grit[3]	$KClO_3$/Dodecane	$KClO_4$/Al	$KClO_4$/Dodecane	PETN	Ammonium Nitrate	Ammonium Nitrate/Gunpowder	HMX
120	14.4	152.9	152.9	-13.3	131.9	61.7	21.9
180	-12.5	-4.9	8.7	-13.5	60.2	25.0	16.4

1. DH_{50}, in cm, by modified Bruceton method, height for 50% probability of reaction; 2. All RDX testing was performed using the same sandpaper as used for the compared material, formula, DH_{50} material – DH_{50} RDX; 3. 120 is 120-grit Si/C wet/dry sandpaper, 180 is 180-grit garnet dry sandpaper.

In the table, a 0 value would indicate that the material is sensitive as RDX. A "+ " value indicates the materials are less sensitive than RDX. A "-" value indicates the material in more sensitive than RDX. The table shows obvious discrepancies in evaluating the sensitivity of a material. For example, $KClO_4$/Al is completely non-sensitive relative to RDX when using 120-grit sandpaper, but more sensitive relative to RDX when using the 180-grit sandpaper.

Table 6. DH_{50} values[1], in cm, of selected test materials, relative to DH_{50} values, in cm, of RDX[2]

Grit[3]	$KClO_3$/Sugar (100)	$KClO_3$/Sugar (AR)	$KClO_3$/Dodecane
150	-8.4	-10.4	-14.2
180	-10.2	-10.4	-12.2

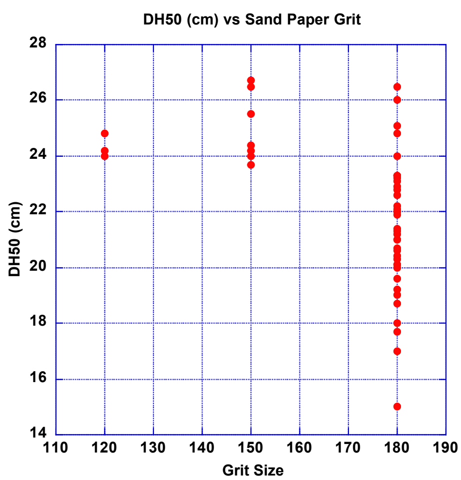
1. DH_{50}, in cm, by modified Bruceton method, height for 50% probability of reaction; 2. All RDX testing was performed using the same sandpaper as the compared material, formula, DH_{50} material – DH_{50} RDX; 3. 120 is 120-grit Si/C wet/dry sandpaper, 180 is 180-grit garnet dry sandpaper.

Table 6 shows the DH_{50} values, in cm, relative to RDX DH_{50} values, in cm, taken under the same testing conditions for several of the Proficiency Test materials using 150-grit and 180-grit sandpapers. The differences in the sensitivity of the materials relative to RDX determined using 180-grit and 150-grit sandpapers are not as dramatic as in Table 5. In addition, in both tables, the differences in the results for military standards among all the sandpapers are very little.

The variability in these results highlighted a real issue in standardization of the test method. As a result, the IDCA decided to use 180-grit garnet sandpaper as the standard testing configuration. LANL purchased a large quantity and distributed that sandpaper to all the participants in the Proficiency Test.

1.3 Sandpaper issue from RDX Statistics Report (027)

None of the variables showed an influence on the DH_{50} with the possible exception of sandpaper type, which has been shown to matter in other studies of other materials.



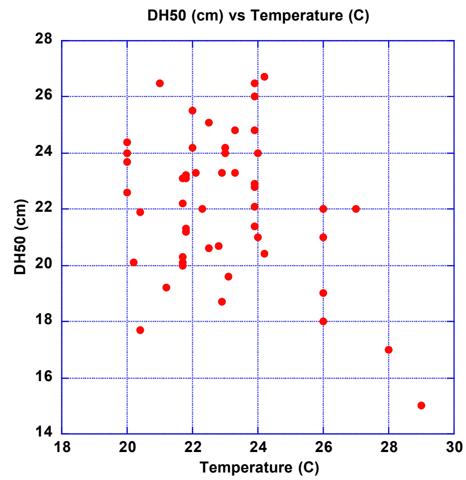


Figure 3. Comparison of DH_{50} with various method and environment variables.

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