2015 INSENSITIVE MUNITIONS AND ENERGETIC MATERIALS TECHNOLOGY SYMPOSIUM
May 2015

Brookfield Viscosity Test Development for IMX Formulations (Abstract #17284)

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INTRODUCTION

IMX melt pour explosive formulations were designed to be reduced vulnerability general purpose explosives for use in both artillery and bomb fill programs. During the early stages of the formulation development phase, the primary focus was to provide a low cost formulation with performance equal to or better than TNT with improved IM characteristics. The viscosity of these types of formulations is normally tested using the Efflux method and this measurement is used to determine the loading characteristics of the explosives. However, this method was developed for TNT and TNT based formulations and may not correlate to changes in viscosity and flow with regards to IMX melt pour formulations. As a result, alternate methods of measuring viscosity must be evaluated. One method of interest utilizes a Brookfield viscometer which measures viscosity through the use of rotational resistance in the material.

BACKGROUND

The Brookfield viscometer utilizes the well-known theory of rotational viscometry, also known as drag-flow viscometry, to measure the viscosity of a sample. The theory is centered on the idea that the resistance of an object in a fluid is a function of the viscosity of that fluid. The Brookfield viscometer measures the torque of a spindle immersed in a material spinning at a constant speed. Brookfield viscometers have been used successfully in multiple industries to provide accurate and repeatable measurements of various sample types and viscosity. As with any test method, the data will contain random variation. Part of this variation is due to individual differences and part will be due to variability in the measurement equipment and process. One method to determine the variability in a measurement system is a gage repeatability and reproducibility (R&R) study. Gage R&R helps determine the magnitude of the variation in a measurement system as well as the sources of the variation. Through the measurement of multiple samples at multiple sites, statistical analyses of the Brookfield viscosity measurement can be performed.

MELT POUR VISCOSITY MEASUREMENTS

The question is often asked, “Why should I make a viscosity measurement?” One reason for measuring viscosity is in the area of quality control where raw materials must be consistent from batch to batch. For this purpose viscosity is an indirect measure of product consistency and quality. Another reason is that a direct assessment of processability can be obtained. It is with both these goals that viscosity measurements of melt pour formulations can be useful.

The Brookfield Viscometer

Brookfield laboratory viscometers are available in three basic types: dial-reading (analog), digital, and programmable. These three types differ only in the manner in which the viscosity reading is displayed. The Brookfield viscometer is manufactured by Brookfield Engineering and measures viscosity through the use of various spindles immersed in the sample. It measures the torque required to rotate the spindle in the fluid. By using a multiple speed transmission and a variety of spindles a variety of viscosity ranges can be measured. For a given viscosity, the viscous drag, or resistance to flow, is proportional to the spindle’s size and shape. The drag will increase as the spindle size increases or the speed increases. It follows that for a given spindle geometry and speed, an increase in viscosity will be indicated by an increase in deflection of the spring. Figure 1 contains a schematic view of the various components of a basic Brookfield viscometer.

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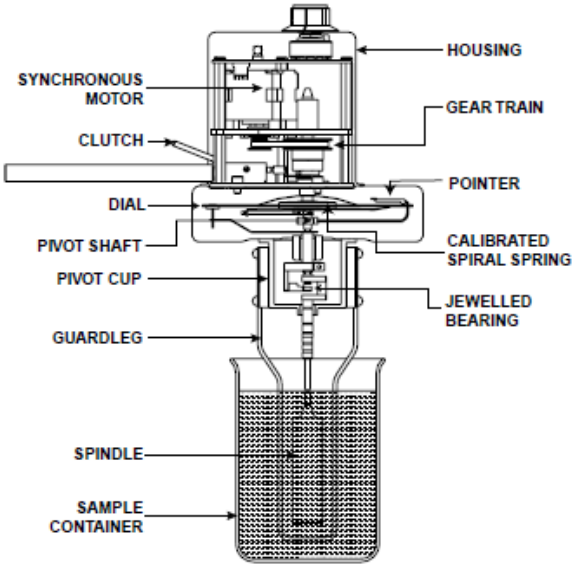

Figure 1: Schematic Drawing of a Basic Brookfield Viscometer

Brookfield Viscometer Setup

The Brookfield viscosity test involves measuring the viscosity of the melt pour sample over a set duration to determine the viscosity of the sample. A Brookfield model RV DV-II+ Pro Viscometer in conjunction with a Brookfield model TC-550SD circulation bath measures the sample at an elevated temperature such that the melt pour material is molten during measurement. Figure 2 contains a photo of the viscometer unit and TC-550SD bath.

Figure 2: RV DV-II+ Pro Viscometer and TC-550SD Circulating Bath

RHEOCALC Data Collection Software

In normal operation of the Brookfield unit, the supplied RHEOCALC Software can be used to collect real time data. The RHEOCALC software collects both sample temperature and sample

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viscosity data in whatever desired time increments. Figure 3 is a screen capture of a typical RHEOCALC data collection.

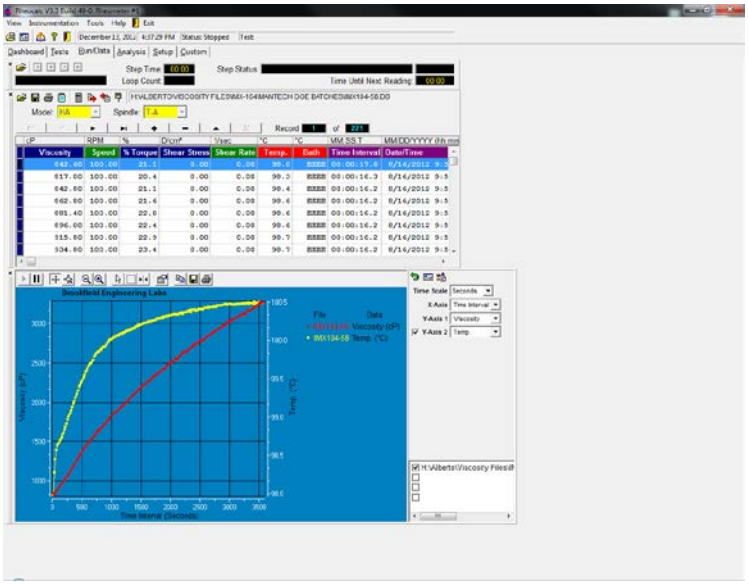

Figure 3: RHEOCALC Data Collection Screen

Measuring Brookfield Viscosity of Melt Pour Formulations

A standard method for operation and data collection was provided by BAE Systems Holston Army Ammunition Plant, HSAAP, to each site in an effort to minimize the variations in data collection from site to site. However, the method provided was only to be used as a guide as some parameters could still vary from site.

Measuring Melt Pour Samples

Using a 1000 milliliter or 600 milliliter glass beaker, approximately one pound of material is placed into a circulating oil bath set at a temperature above the melting point of the material. Additionally, the temperature of the sample is monitored to ensure that the sample temperature is within the range for study. (97°C to 101°C) For this study a target value of 113°C was used for the temperature bath as this consistently resulted in sample temperatures in the desired range for the study. The sample is allowed to melt over a period of time until the entire sample is molten and contains no solid pieces. The sample is mixed thoroughly with a plastic spatula to ensure homogeneity of the sample. The T-B spindle was selected for use in measuring the viscosity of IMX melt pour formulations in this study. The spindle is placed such that the bottom of the T-Bar spindle is just above the bottom of the beaker. Additionally, the temperature probe is placed in such a way that the probe is just submerged below the surface of the molten material to monitor the temperature nearest the surface and avoid crusting of the sample during the analysis. Upon obtaining a molten sample, the RHEOCALC software is programed such that the speed is set at 100 RPMs and data is collected every second over a fifteen minute time frame. During the initial measurements observed by all of the participating sites, it was noted that the viscosity stabilized after approximately fifteen seconds and held steady for approximately thirty seconds prior to beginning to experience settling. As a result, it was agreed upon by the team members that the reported viscosity would be the average value of the recorded viscosities from fifteen seconds to forty five seconds during the initial minute of measurement.

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Viscosity Results

Six melt pour samples were provided to the three sites for evaluation. Each site was to analyze each sample in duplicate and report the data as a single viscosity value for the six samples. Table 1 contains the resulting viscosity values for all three sites.

Table 1: IMX Melt Pour Viscosity Values – All Sites

Site	Site #1 BAE, HSAAP		Site #2		Site #3	
Sample	Trial 1	Trial 2	Trial 1	Trial 2	Trial 1	Trial 2
1	394.9	411.7	434.6	404.7	409.1	Not Tested
2	532.4	535.4	589.4	561.8	502.0	Not Tested
3	535.2	528.6	547.1	603.5	527.2	Not Tested
4	620.1	622.9	586.1	555.1	461.2	Not Tested
5	642.5	657.3	686.3	650.8	407.6	Not Tested
6	377.8	371.1	357.2	354.6	433.7	Not Tested

The resulting viscosities vary between the sites; however some similar values are obtained between the three sites.

STATISTICAL ANALYSIS OF VISCOSITY DATA

As seen in Table 1 the viscosity data obtained across the three sites did show some variability. In particular one site, Site #3, appeared to have values significantly lower than those of the other sites. In an effort to quickly determine if these differences are real a boxplot was created to illustrate the differences in the group of data. Figure 4 contains the box plot for the three sites data.

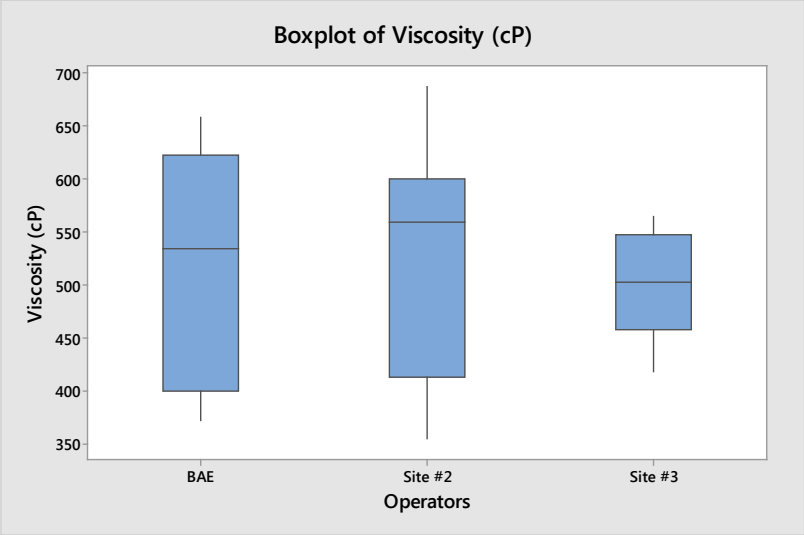

Figure 4: Boxplot of IMX Melt Pour Viscosity Data for All Sites

In Figure 4 one can quickly see the difference in the data distribution for all sites. The overall distribution of data for Site #3 is significantly lower in value than the values obtain by the other sites. In order to observe where the difference in the data may have originated, a plot of the

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viscosity values obtained for the individual samples must be compared. This comparison can be found in Figure 5.

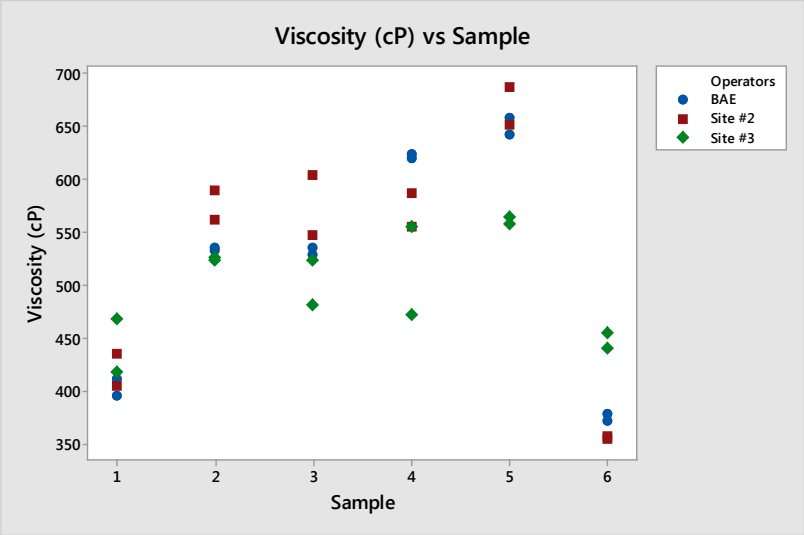

Figure 5: Viscosity by IMX Melt Pour Sample (All Sites)

From the graph you can see the difference in the measured viscosity for samples 4, 5, and 6 which contributes to the lower overall distribution of the data collected by Site #3. Further examination of the data also indicates a difference in the temperature of the samples during the data collection. Table 2 contains the average temperature during of the sample during the data collection.

Table 2: IMX Melt Pour Viscosity Values – All Sites

Site	Site #1, BAE	Site #2	Site #3
Average Temperature (°C)	100.6	98.8	106.4

The sample temperature of the IMX samples was to be between 97°C to 101°C during the viscosity measurement in order to minimize the potential for settling. In Table 2 the average value for the sample measurements made at Site #3 exceeded, 106°C. This variation in sample temperature helps explain the lower values obtained during the study. As a result of the difference in the viscosity values collected by Site #3, as well as the potential for product settling, it was determined that the gage R&R study would only be performed on the remaining two sites.

Gage R&R Study on Brookfield Measurement System

A gage R&R is a two-way analysis of variance (ANOVA) used to estimate the repeatability and reproducibility of intra-laboratory and inter-laboratory studies on test data. For the purposes of this study, the repeatability can be defined as the within laboratory precision, measured through the assessment of the duplicate measurements at each site. The reproducibility can be defined as the between laboratory precision, measured through the assessment of multiple measurements of the same set of samples at different sites. Both of these measures are then associated with random error in the measurement system. A good measurement system will have low variation from repeatability and reproducibility relative to the total variation. Traditionally, the gage R&R will report the variance as the percentage contribution (%R&R) to

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the overall variance. A poor measurement system will have %R&R greater than or equal to 9% of the total variance, an adequate measurement system will have %R&R less than 9%, while a good measurement system will have %R&R values less than 1% of the overall variation in the system. The resulting gage R&R analysis was done on six parts (IMX Melt Pour samples), two replicates (two measurements of each sample), and two operators (Site #1, BAE and Site #2) to determine the variation in the measurement system (Brookfield Viscometer).

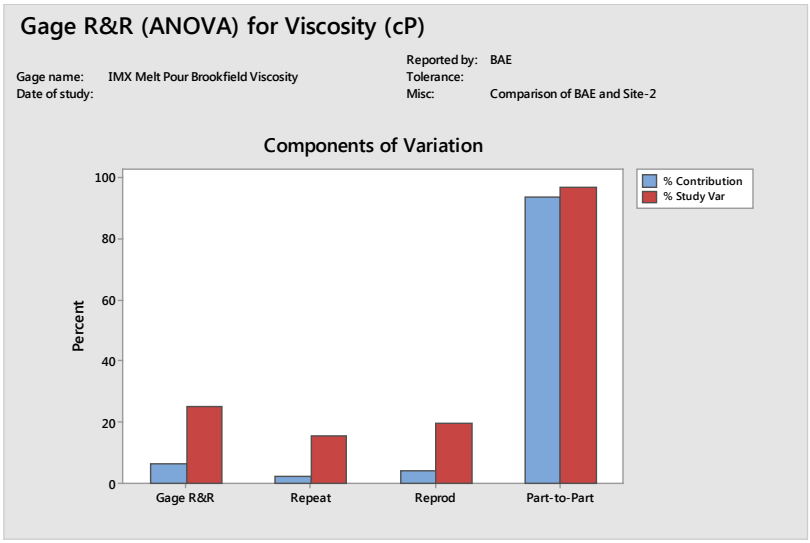

Figure 6: Gage R&R – Components of Variation Graph

The component of variation graph, Figure 6, uses a bar chart to graphically show the contribution from repeatability and reproducibility in relation to the part-to-part variation. One can quickly see that the part-to-part contribution is much higher than that of R&R and indicates that the measurement system has little variation in the inter-laboratory (repeatability) and intra-laboratory (reproducibility) measurements. This can be further evaluated in the measurement by part graph in Figure 7.

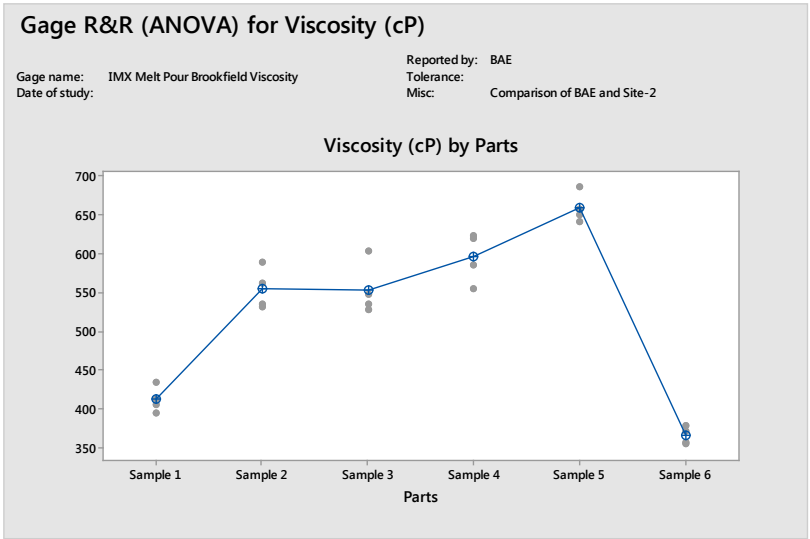

Figure 7: Gage R&R – Viscosity (cP) by Parts

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The measurement by part graph summarizes the repeatability of the measurements. A tight grouping of values for each part indicates that the repeatability for the individual parts is fairly high and contributes only slightly to the variation in the measurement system. Additionally it shows that the variations in the measurements are mainly occurring due to variations in the parts themselves. The remaining two graphs look at the interaction between operators, or in this case the different sites. Figure 8 is the measurement by operator graph, while Figure 9 is the parts*operator interaction graph.

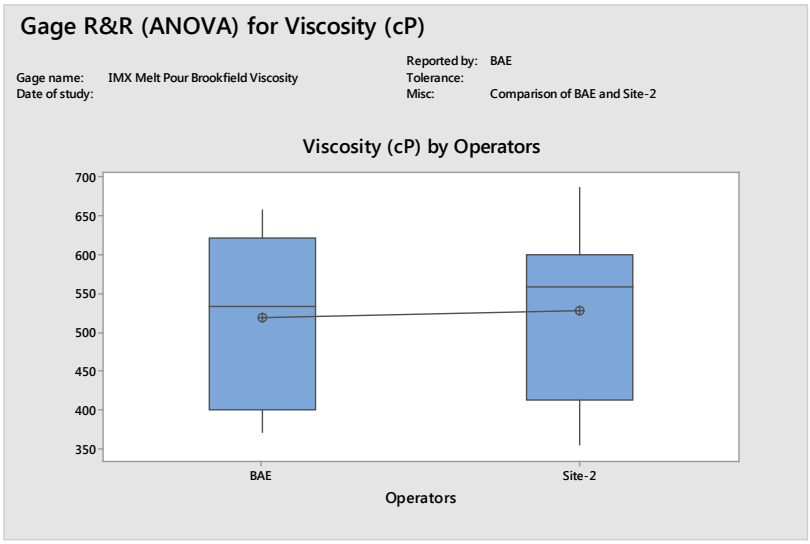

Figure 8: Gage R&R – Viscosity (cP) by Operators

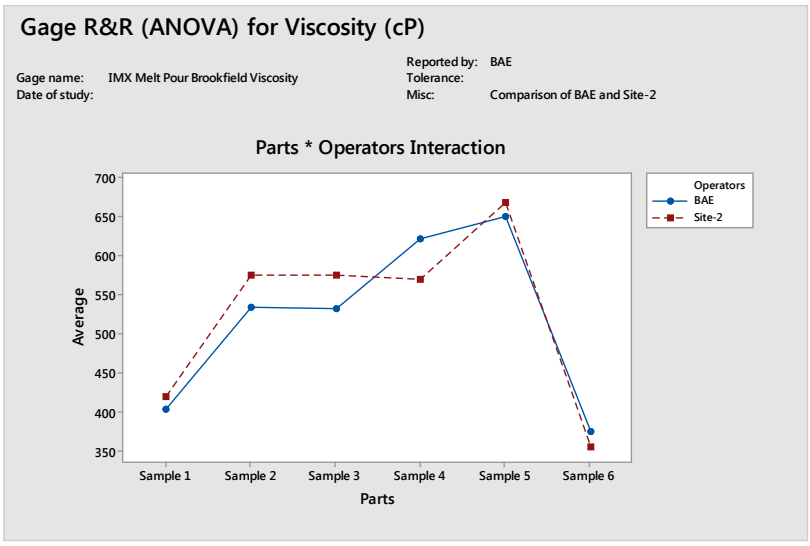

Figure 9: Gage R&R – Parts*Operators Interaction

Figure 8 displays the slight variations in the viscosity measurement that occur between the two sites. However this variation is quite low and can be further evaluated in Figure 9. The parts*operators graph shows that the main source of variation in the operators is due to part-to-

part variations just as it was indicated in the viscosity by parts graph. The results in these two graphs together further strengthen the conclusion that the largest contribution to the variation in the measurement system appears to be coming from the variations in the part and not from the repeatability or the operator making the measurement. However, the full ANOVA analysis must be evaluated to determine if the measurement system is adequate. Table 3 contains the full gage R&R and ANOVA analysis output from Minitab.

Table 3: Gage R&R – ANOVA Analysis Minitab Output

Gage R&R Study - ANOVA Method

Gage R&R for Viscosity (cP)
Gage name: IMX Melt Pour Brookfield Viscosity
Date of study:
Reported by: BAE
Tolerance:
Misc: Comparison of Site #1, BAE and Site #2

Two-Way ANOVA Table With Interaction

Source	DF	SS	MS	F	P
Parts	5	252735	50546.9	37.4703	0.001
Operators	1	428	427.6	0.3170	0.598
Parts * Operators	5	6745	1349.0	4.2204	0.019
Repeatability	12	3836	319.6		
Total	23	263743			

Alpha to remove interaction term = 0.25

Gage R&R

Source	VarComp	%Contribution (of VarComp)
Total Gage R&R	834.3	6.35
Repeatability	319.6	2.43
Reproducibility	514.7	3.92
Operators	0.0	0.00
Operators*Parts	514.7	3.92
Part-To-Part	12299.5	93.65
Total Variation	13133.8	100.00

Source	StdDev (SD)	Study Var (5.15 * SD)	%Study Var (%SV)
Total Gage R&R	28.884	148.755	25.20
Repeatability	17.878	92.074	15.60
Reproducibility	22.686	116.835	19.80
Operators	0.000	0.000	0.00
Operators*Parts	22.686	116.835	19.80
Part-To-Part	110.903	571.151	96.77
Total Variation	114.603	590.204	100.00

Number of Distinct Categories = 5

In the two-way ANOVA in Table 3 the P-values for parts, operators and parts*operators are listed. The high P-value (0.598) for operators indicates that the contribution from operators is in fact low and most likely is not a significant source of variation in the measurement, and further strengthens the conclusions determined in the graphical analysis. On the other hand, the low P-value (0.001) for parts confirms that the part-to-part variation is a significant contributor to the variation in the measurement system. Analysis of the gage R&R values allows for determining if the measurement system is adequate for use in measuring the viscosity of melt pour formulations. Looking at the percent contribution values it can be seen that the total gage R&R makes up only 6.35% while the part-to-part contributes 93.65%. Further analysis shows that total gage R&R value (6.35%) can be separated in individual values of 2.43% for repeatability and 3.92% for reproducibility. A value of less than 9% as an indicator of an adequate measurement system. The value obtained for total gage R&R (6.35%) is well below 9% and

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leads to a conclusion that the measurement system can be considered adequate. Also located in Table 3 are the standard deviations obtained for both total gage R&R and part-to-part. These values can be used in the calculation of the signal to noise ratio for the measurement system. From Table 3 the standard deviation for total gage R&R is 28.8 while the part-to-part standard deviation is 110.9, which results in a signal to noise ratio of 3.85 for the measurement system. A value of greater than 2.2 for signal to noise is considered good and the value obtained in the gage R&R study (3.85) indicates that the ability of the measurement system to measure the samples can be considered adequate. The signal to noise value (3.85) combined with the total gage R&R contribution (6.35%) indicates that overall the measurement system in this study is an adequate measurement system for the purposes of measuring the viscosity of melt pour formulations.

CONCLUSION

Traditionally the efflux viscosity method has been used to determine the flow properties of melt pour materials. However this method was developed initially for TNT and TNT based formulations. As melt pour formulations have become the current IM fill for multiple weapons systems, it has been observed that the efflux measurement may not correlate in the same manner as traditional legacy materials when it comes to processability and loading. An alternate method using a Brookfield viscometer, involves measuring the viscosity of a molten sample using rotational viscometry as an indication of the materials overall viscosity. A series of six melt pour samples were prepared and tested independently by three sites. Each site was provided a standard method for evaluating the viscosity of the samples in an effort to minimize variables that may influence the viscosity measurement. The data was initially analyzed and it was observed that one site's data was statistically different the other sites. Upon closer examination this difference was most likely due to temperature differences during the viscosity measurement. Due to a lack of replicate values and the higher temperatures during the measurement the decision was made to exclude this site from further evaluation at this time. A gage R&R study was performed on the available data from the remaining two sites. The gage R&R is a two-way ANOVA analysis which is used to determine the ability of a measurement system to measure the samples in question and breaks down the variability in the system into the three core components of variability. From this study a value of 6.35% was obtained for the total gage R&R contribution which is below the 9% value needed to establish the measurement system as adequate. However the study did lack the third site's data and future work can be done to include this data. Overall, the use of the Brookfield viscometer shows promise as the gage R&R study indicates. Improvements can be made with regards to minimizing sample temperature variations and data collection. Over time, experience with the Brookfield viscometer will resolve some of these issues, thereby improving the Brookfield viscosity method.

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