

Statistical Evaluation of Burning Rate Data of Composite Propellants Obtained from Acoustic Emission Technique

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ABSTRACT

The acoustic emission technique has been considered to be one of the most reliable and robust methods for the measurement of the steady burning rate of composite propellants. In this work, attempts were made to quantify the measurement variability of the burning rate of composite solid propellants by acoustic emission method using statistical tools. A total of 1100 individual measurements were subjected to statistical treatment. The combination of confidence interval and repeatability limit delineated the extent of natural dispersion in the burning rate measurement data. The very high coefficient of variation values for the propellant compositions, having a burning rate of more than 25 mm s⁻¹ raised concerns about the suitability of the acoustic emission method for high burning rate compositions. The Reliability interval approach was employed to determine the statistically significant sample size for different composite propellants having a burning rate range of 5–31 mm s⁻¹. The entire set of data was screened for identification of outlying observation using the Dixon Q test, and the extent of contamination was quantified. Moreover, the application of statistical techniques could have far-reaching implications for quality control perspectives of burning rate measurement by acoustic emission and could be implemented as reference tolerance limits and preventive measures for ensuring the good health of the instrument as well as propellant processing.

Keywords: Acoustic emission burning rate; Confidence interval; Repeatability limit; Coefficient of variation; Outliers

1. INTRODUCTION

The burning rate of composite solid propellant is a convoluted function of several physical, mechanical, and chemical factors, such as temperature, pressure, heat and mass flux, chemical reactions in the gas, interface, and condensed phases, etc.¹⁻³. It is one of the most important measured quality characteristics due to its ability to change the ballistics of the rocket motor. Therefore, the measurement of the burning rate more economically and efficiently way is a task of paramount importance for every propellant processing facility. Since to date no theoretical model is capable of predicting burn rate with accuracies within 1%, therefore burn rate can only be measured by experimentally⁴. Over the years, the strand burner methods have emerged as a standard technique for steady burning rate measurement. However, ensuring the precision and accuracy was considered to be the main challenge of solid propellant burning rate measurement^{5,6}. Jordan⁷ pointed out that the effect of the burning rate variability on the flight performance of the rocket motor was five times higher as compared to other parameters. This higher variability results in increases the on-target delivery cost. Liu⁸ demonstrated the uncertainties in the pressure exponent (n) value due to the variability in the burning rate measurement data. Furthermore, Kubota⁹, *et al.* and Hoque¹⁰, *et al.* observed a direct correlation between

friction sensitivity and burning rate of non-aluminised and aluminised composite propellants respectively.

A considerable amount of work has been carried out to develop non-intrusive methods that do not perturb the combustion process, using advanced technics such as ultrasonic waves, X-ray radiation, microwave, videography, plasma capacitance, fuse wire, acoustic emission etc.¹¹⁻¹⁴. An initial study by Caveny¹⁵, *et al.* reported 94% accuracy in the burn rate measurement by acoustic emission technique for the single base, double base, triple base, and HMX composite propellants at high pressure. Koury¹⁶ & Christensen¹⁷ established a statistically significant correlation between the solid strand burning rates (7 – 9 mm s⁻¹) measured by acoustic emission and full-scale motor, and proposed use of solid strand burning rate technique in lieu of ballistic motors which would incur considerable savings in expenditure and time. In addition, Rampichini¹⁸, *et al.* showed that the accuracy of acoustic emission was higher (0.7%) as compared to videography (4%) and fuse wire (4.4%) technique. The other methods like ultrasonic, X-ray, and microwave require specialised instrumentations, and NATO Standardisation Agreement 4674¹⁴ recommends exclusion of the X-ray and microwave measurement techniques as a routine ballistics characterisation tools. Therefore, acoustic emission is the most used, reliable, and fast technique for solid strand burning rate measurement, and it had been widely implemented as an essential quality control tool in propellant processing facilities across the globe.

Despite the considerable use of acoustic emission burning rate data, little attention has been paid to develop an integrated idea about the variability and limitations associated with the measurement methodology. No comprehensive trend analysis and precision statistics are reported in the literature covering a wide burning rate range and diverse propellant compositions.

Considering these factors, the goal of this study is to analyse and quantify the measurement variability associated with the acoustic emission strand burning rate technique. The data set covered 1100 burn rate data from diverse propellant compositions with a burning rate range of 5–31 mm s⁻¹.

The approach broadly involves quantification of repeatability statistics by determination of confidence interval (CI) and coefficient of variation (CV) of burning rate data of different formulations. Reliability interval (*L*) was used to determine the appropriate sample size to estimate the sample statistics with a specified precision and accuracy. Furthermore, detection statistics have been employed for ‘Outlier’ which is an assignable cause of variability in the experimental data.

2. METHOD

2.1 Burning Rate Measurement of Composite Propellants by Acoustic Emission

The burning rate of the composite propellant was determined in a modified Crawford’s bomb⁵ using acoustic emission technique. The schematic representation of the assembly is shown in Fig. 1. Solid propellant strands were milled from propellant cartons. The dimensions of each strand were 6 mm × 6 mm × 130 mm. A nichrome igniter wire was threaded through the one end of the propellant strand keeping approximately 3 mm of propellant above the igniter wire, and this prepared sample was ignited while immersed in water inside the closed bomb. The water acted as a natural inhibitor to prevent side burning, and medium to carry acoustic signals generated by propellant burning. The bomb was pressurised with nitrogen gas as per the required burning conditions, and a firing pulse was given. These acoustic signals were captured by a sensor mounted externally on the combustion bomb. The output electronic signal was recorded and displayed in voltage vs. time domain by a data acquisition system. By monitoring the time required for the flame to consume the known length of propellant at a preset pressure, the burning rate of the propellant was determined. To obtain a reliable, precise, and unbiased result, repetitive burning rate measurements were carried out from a single propellant batch, and the average

burning rate value and the standard deviation were reported as quality statistics.

Of particular note, the acoustic emission method has a fundamental limitation of measuring a lower value of the burning rate as compared to the main motor. The reason for this aberration is thought to be the higher heat loss to the surrounding water medium for the usage of a thin strand of propellant during testing. Higher loss of heat produces a cooler combustion zone at the propellant burning surface which in turn lowers the burning rate^{6,19-20}. The acoustic emission method is also inefficient to identify combustion instabilities due to heterogeneity, small defects, and porosity. Besides, the applicability of acoustic emission methodology for higher burn rate formulations (more than 20 mm s⁻¹) has also not been well delineated in the literature.

2.2 Data Collection

The measured acoustic emission strand burning rate data were acquired from the in-house quality control data bank, which consisted of a large quantity of coherence burning rate data, accomplished from the last 10 years of disciplined processing of composite solid propellant. The burning rate data of composite propellants were classified according to their differences in formulations, described in Table 1. The majority of the burning rate measurement data were of HTPB/AP/Al-based composite propellants which were broadly grouped in two subcategories; P01: non-catalysed compositions and P02: catalysed by transition metal oxides. Furthermore, we studied the burning rate of active binder/AP/Al/Nitramine based composite propellant. The reported burning rate data ranges from about 5–31 mm s⁻¹.

The propellant formulations: P01 and P02 were further augmented in five derivative compositions based on the coarse to fine ammonium perchlorate (AP) ratio and the amount of catalyst loading. This expanded set of propellant compositions increased the resolution of the study.

Table 1. Summary of burning rate data of composite propellant from acoustic emission measurements

Categories	Description	Burning rate (mm s ⁻¹)	No. of data points
P01	HTPB/Al/AP (Non-catalysed)	5–7	500
P02	HTPB/Al/AP (Catalysed)	14–31	528
P03	Active Binder/AP/Al/Nitramine	11–13	65

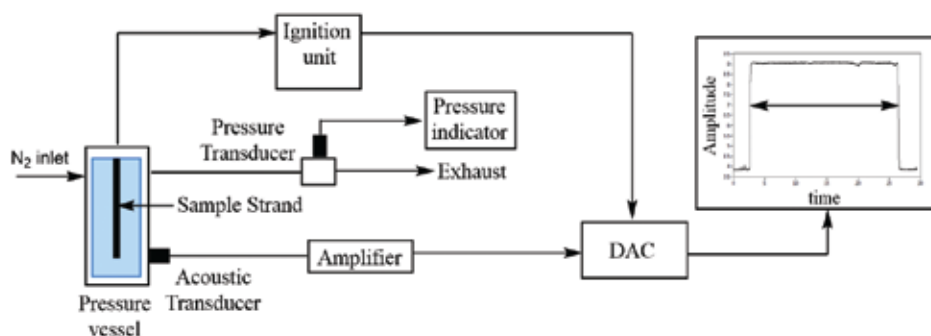


Figure 1. Burning rate measurement using acoustical emission technique.

2.3 Statistical Methods

2.3.1 Statistics of Variability

The confidence interval (CI), repeatability limit (R), and coefficient of variation (CV) were evaluated as variability statistics²¹. For a sample size <30, the equation for confidence interval on the mean (\bar{X}) with sample standard deviation (s) and sample size (n) is as follows:

$$\bar{X} - \frac{t_{\alpha/2, n-1} \times s}{\sqrt{n}} \leq \mu \leq \bar{X} + \frac{t_{\alpha/2, n-1} \times s}{\sqrt{n}} \quad (1)$$

where $\frac{t_{\alpha/2, n-1} \times s}{\sqrt{n}}$ is the $100 \times \frac{\alpha}{2}$ percentage point in t distribution with $n - 1$ degrees of freedom.

The test conditions corroborated with the repeatability criteria, the burning rate measurements for individual samples were carried out with the same standard method on identical propellant compositions in the same laboratory using the same instruments, although, there were minor deviations pertaining to the operator and the time of testing which can be considered negligible.

The fundamental precision statistics for repeatability limit is repeatability standard deviation, and it was obtained by combining the standard deviation values of each set of measurements. Repeatability standard deviation is calculated using the following equation²¹:

$$\text{Repeatability Standard Deviation, } S_r = \sqrt{\frac{(S_1^2 + S_2^2 + S_3^2 + \dots + S_n^2)}{n}} \quad (2)$$

where S_1, S_2, \dots, S_n are standard deviations of measured values of respective batches of identical propellant.

Using the repeatability standard deviation (S_r), the repeatability limits at 95% of probability, was calculated according to the following equation²¹:

$$\text{Repeatability Limit, } R = 2.8 \times S_r \quad (3)$$

Considering the wide burning rate range of 5-31 mm s⁻¹ and different magnitudes of variation, the coefficient of variation (CV) was used for a meaningful comparative study. The coefficient of variation (CV) provided a relative measure of variability and weighted the standard deviation relative to the mean.

2.3.2 Determination of Replication Number

The desired level of measurement precision must be prescribed for the important quality characteristic such as the burning rate of solid rocket propellant. Statistically, it is expressed in terms of margin of error (E), which is defined as the maximum acceptable difference between the true mean (μ) and the sample mean (\bar{X}). This maximum allowable error can also be described as a percentage to the historic mean or true mean (μ) of the population which is known as reliability interval (L)^{22, 23}.

For t-distribution, the margin of error is expressed as:

$$\text{Margin of Error, } E = \text{abs}(\bar{X} - \mu) = \frac{t_{\alpha/2, n-1} \times s_p}{\sqrt{n}} \quad (4)$$

s_p is the pooled estimator of population standard deviation (σ).

The value of margin of error (E) was used to calculate the reliability interval (L) as per the following equation:

$$\text{Reliability Interval, } L = \frac{100 \times E}{Z} \quad (5)$$

where Z is the historical mean of the population.

The value of reliability interval (L) and, its improvement with the increase in repetitive measurements decide the number of replicates. It can be interpreted as if there is a great improvement in reliability interval (L) by carrying out replicates then only the analyst should perform it otherwise repetitive measurements should be restrained to avoid unnecessary time and expenditure.

2.3.3 Detection of Outliers: Assignable Cause of Variability

In view of the problem definition i.e. variability in burning rate measurement, the number of replicates was always less than 10, and Dixon²⁴ criteria for single outlier was the most suitable detection technique as per ASTM-E178. The equation of the criteria is as follows:

$$Q_{cal} = \frac{|X_{suspect} - X_{closest}|}{X_{max} - X_{min}} \quad (6)$$

where $X_{suspect}$ is the suspected data point and $X_{closest}$ is the most nearby data point of the suspected one. The denominator represents the range of the experimental data.

The critical values calculated: Q_{cal} in each of the cases were equated to the standardised statistical criterion: Q_o described by ASTM-E178. If $Q_{cal} > Q_o$ for a certain significance level, it is an indication that the doubtful data is an outlier²⁴⁻²⁶. All burning rate measurement data were scanned for outliers using Minitab18 statistical software²⁷.

3. RESULTS AND DISCUSSIONS

3.1 Analysis of Variability

The primary quantification of variability associated with the acoustic emission burning rate measurement technique was done by calculating the confidence interval ($\alpha=0.05$) for all the burning rate data as summarised in Table 2. The determination of confidence interval (CI) avoided the situation of reporting a single value and provided an interval around the average burning rate value, in which the value of true burning rate can be found with a certain confidence level, usually

Table 2. Summary of confidence interval and repeatability limit for composite propellant burning rate from the acoustic emission method

Categories	Burning rate (mm s ⁻¹)	Average confidence interval ($\alpha=0.05$)	Repeatability limit ($\alpha=0.05$)
P01	5–6	± 0.05	0.13
	6–7	± 0.06	0.15
P02	14–16	± 0.44	1.08
	17–20	± 0.6	1.06
	25–31	± 1.42	4.01
P03	11–13	± 0.2	0.6

95%, and the length of the interval conveyed the precision of the estimation. Furthermore, the precision statement for the acoustic emission method was extended by reporting the repeatability limit corresponding to each range of measured burning rate as illustrated in Table 2. The repeatability limit values represented the extreme allowed difference between two replicates of a strand burning rate sample measured using the acoustic emission method with a probability of 95%.

The burn rate for the first composition of P01 was measured under 4.9 MPa at 24 °C, and the burn rate for the rest of the propellant compositions was reported at, 6.9 MPa at 24 °C. The average burning rate of non-catalysed propellant composition: P01 exhibited a narrow average confidence interval of ± 0.05 and ± 0.06 , and the corresponding repeatability limits were 0.13 and 0.15 respectively. It was found that for catalysed compositions: P02, the length of the average confidence interval broadened up to ± 1.42 for the burning rate range of 25–31 mm s⁻¹, and the associated repeatability limit was 4, which is significantly higher as compared to that of others. Active binder/AP/Al/Nitramine based composite propellant: P03 displayed a narrow confidence interval, and the repeatability limit was determined as 0.6.

The scope of confidence interval and repeatability limit is generally limited to the one-dimensional variability of the method, and they are not statistically appropriate for comparison of variability associated with the different magnitude of measurement scale. For instance, the confidence interval and repeatability limit of the burning rate of range 5–6 mm s⁻¹ should not be directly compared with that of a high burning rate of 25–31 mm s⁻¹. In this context, the coefficient of variation (CV) was used to carry out a statistically eloquent comparative study of variability associated with the measurement of solid strand burning rate by acoustic emission method. Figure 2 outlined the two-dimensional tightness of the burning rate data across all measurement scale in the form of the coefficient of variation vs. burning rate plot.

On comparison of the coefficient of variation, it was found that the non-catalysed propellant composition: P01 showed the most precise burning rate result, and the determined average coefficient of variation was 1% for the data sets comprising 500 data points. For the P02: catalysed propellant composition, the acoustic emission method generated the coefficient variation of average 2% for the burning rate range of 14–20 mm s⁻¹. For active binder/AP/Al/Nitramine based P03 compositions the average coefficient of variation was also around 2%. Moreover, the coefficient of variation was mostly less than 5% for the propellant compositions having a burning rate range of 5–20 mm s⁻¹, these values indicated the reliability of the acoustic emission solid strand burning rate technique for the propellant compositions with burning rate range of 5–20 mm s⁻¹.

However, the variability drastically increased for the high burning rate compositions of 25–31 mm s⁻¹. The average coefficient of variation was found to be more than 5% which is statistically undesirable, and it raised a question about the applicability of acoustic emission

technique for transition metal oxide catalysed HTPB/AP/Al compositions with burning rate beyond 25 mm s⁻¹. Liu⁸ also reported a similar trend of the variability in the Crawford bomb fuse-wire burning rate data for high burning rate compositions of 25–35 mm s⁻¹.

3.2 Calculation of Sample Size

Herein, measurement of reliability interval (L) has been used to estimate the number of replicates to be included in a random sample of composite propellant to determine the burning rate with a prescribed precision using acoustic emission technique. This approach made use of the historical average burning rate (Z) and pooled standard deviation (s_p) for identical propellant formulations for calculation of reliability interval as described in equation 5. First, the reliability interval of the burning rate of different propellant compositions was determined at different replicate numbers (Fig. 3). Following the standard procedure, the percentage difference in the reliability interval ($L_{n-1} - L_n$) was measured with each repetitive measurement. The results are summarised in Table 3. It has

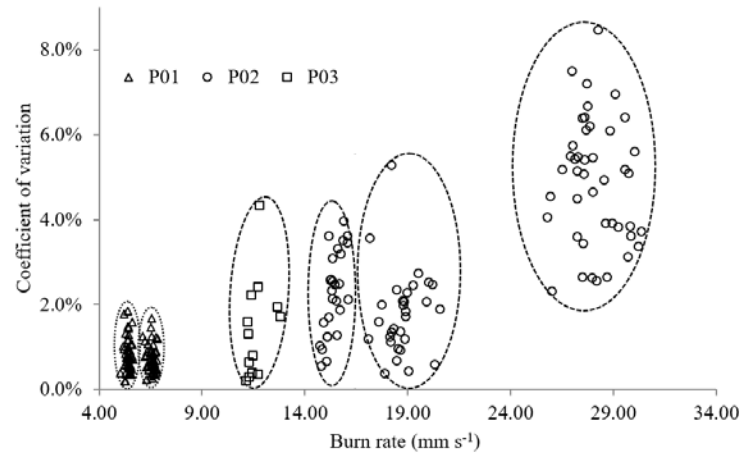


Figure 2. Plot of the coefficient of variation against the burning rate of different propellant compositions ranging 5–31 mm s⁻¹.

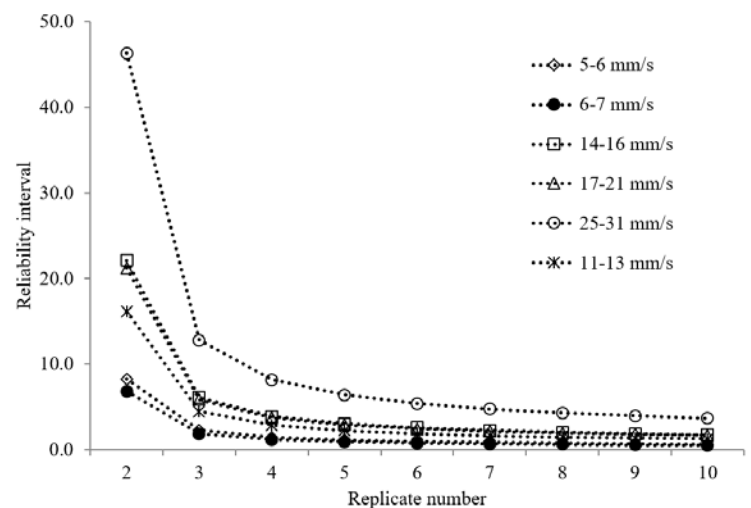


Figure 3. Plot of reliability interval against replicate numbers for the burning rate of different propellant compositions ranging 5–31 mm s⁻¹.

Table 3. Values of reliability interval (RI) and percentage difference with an increase in replicate numbers of burning rate samples for all the propellant compositions

Categories	P01				P02				P03			
	5–6		6–7		14–16		17–20		25–31		11–13	
Burning rate (mm s ⁻¹)	L	% Diff	L	% Diff	L	% Diff	L	% Diff	L	% Diff	L	% Diff
2	8.3		6.9		22.1		21.2		46.3		16.2	
3	2.3	6.0	1.9	5.0	6.1	16.0	5.9	15.4	12.8	33.5	4.5	11.7
4	1.5	0.8	1.2	0.7	3.9	2.2	3.8	2.1	8.2	4.6	2.9	1.6
5	1.1	0.3	0.9	0.3	3.1	0.9	2.9	0.8	6.4	1.8	2.2	0.6
6	1.0	0.2	0.8	0.1	2.6	0.5	2.5	0.5	5.4	1.0	1.9	0.3
7	0.9	0.1	0.7	0.1	2.3	0.3	2.2	0.3	4.8	0.6	1.7	0.2
8	0.8	0.1	0.6	0.1	2.1	0.2	2.0	0.2	4.3	0.5	1.5	0.2
9	0.7	0.1	0.6	0.1	1.9	0.2	1.8	0.2	4.0	0.3	1.4	0.1
10	0.7	0.0	0.5	0.0	1.8	0.1	1.7	0.1	3.7	0.3	1.3	0.1

been observed that after a certain number of repetitive measurements, there was no significant improvement in reliability interval.

The reliability interval data suggested that for P01: non-catalysed propellant formulations, the reliability interval was substantially improved by carrying out the third repetitive measurement i.e. 6% and 5% respectively, and no substantial improvement was achieved by performing more than three replicates. Hence, three replicates would be sufficient for P01 compositions to determine the burning rate with considerable accuracy.

The comprehensive results of Table 3 advocated carrying out four replicate measurements for the first two propellant compositions of P02 (catalysed by burning rate modifier) and active binder/AP/Al/Nitramine based P03 formulation to obtain a strand burning rate with an acceptable level of accuracy. However, it was found that the acoustic emission technique of burning rate measurement would require at least seven repetitive measurements for the propellant composition having a burning rate range of 25–31 mm s⁻¹ to produce a more explicit result. The percentage difference rendered improvement around 2% and 1% with carrying out fourth and fifth repetitive measurements respectively. The choice of seven replicates was attributed to the value of reliability interval which should be less than 5% as better statistical practice.

3.3 Identification of ‘Outliers’ in the Experimental Data

The origin of observed measurement variability in the burning rate data was further investigated. The common assignable cause of measurement variability is one-time systematic errors in the experimental procedure of burning rate determination, and it may lead to aberrant observations; in statistics, they are known as ‘Outliers’. These anomalous data points result in contamination in the accuracy and precision statistics of burning rate measurement.

The statistical criterion of the Dixon Q test was applied to the full set of data to identify the outliers, and the observations are summarised in Table 4.

Table 4. Summary of identification of ‘Outliers’ in the composite propellant burning rate data from the acoustic emission method

Categories	Burning Rate (mm s ⁻¹)	No. of batches	No. of Batches with ‘Outliers’	Probability (%)
P01	5–7	104	8	7.6
P02	14–31	100	7	7
P03	11–13	13	1	7.6

Despite huge variability corresponding to high burning rate compositions: 25–31 mm s⁻¹, moderate contamination in the full set of burning rate data was found. Thus, the apparently large coefficient of variation (CV) observed in the experimental data of high burning rate composition was not due to the presence of outlying observation, rather it might be an extreme manifestation of the random variability inherent to that particular propellant composition.

In addition, the probability of the appearance of outlying observation for a single batch was around 7% for all the propellant compositions. Table 5 depicts the extent of contamination in the average burning rate and standard deviation due to the presence of outlying observations in the sample.

There was no significant contamination in the average burning rate for all the compositions, but the precision of the data set drastically increased when the outlying observations were removed. For instance, when the ‘Outlier’ was omitted,

Table 5. The extent of contamination in average burning rate and standard deviation due to the presence of ‘Outliers’ in the composite propellant burning rate data

Categories	Burning rate (mm s ⁻¹)	Contamination due to ‘Outliers’	
		Average burning rate (mm s ⁻¹)	Standard deviation (mm s ⁻¹)
P01	5–7	0.01 to 0.04	0.02 to 0.07
P02	14–31	0.11 to 0.63	0.17 to 1.00
P03	11–13	0.13	0.24

the maximum change in average burning rate for P02 composition was 0.63 mm s^{-1} , but the improvement in standard deviation was as high as 1 mm s^{-1} . Hence, from a quality control perspective, it is imperative to identify the outlying observations in the experimental data to rectify, adjust, and improve the measurement procedure.

4. CONCLUSION

In summary, the variability in 1100 repeated measured acoustic emission solid strand burning rate data was assessed and quantified using confidence interval (CI) and repeatability limit (R). The use of the coefficient of variation (CV) indicated that the acoustic emission method might not be the appropriate methodology for measuring the burning rate of more than 25 mm s^{-1} for HTPB based composite propellants, catalysed by transition metal oxides. Furthermore, statistically reasonable sample numbers were obtained for steady burning rate measurement of composite propellants having different ranges of burning rates.

Additionally, the whole data set was investigated for the identification of 'Outliers' and moderate contamination was noted. However, it has been envisioned that the 'Outlier' detection statistics should be implemented as a regular quality control activity to prevent unintended adulteration in the burning rate measurement data.

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