Estimating the Heating Values of Napier Grass (*Pennisetum purpureum* Schumach) and Wild Sugarcane (*Sacharrum spontaneum* L.) Biomass using Proximate Composition

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**ABSTRACT**

The study aimed to determine the heating values (HV) and proximate composition of the four varieties of Napier Grass (*Pennisetum purpureum* Schumach) and native wild sugarcane (*Sacharrum spontaneum* L.) to generate a mathematical model for predicting HV of Napier Grass varieties and wild sugarcane biomass. The four varieties of Napier Grass namely King, Florida, Dwarf, and Princess Caroline as well as the wild sugarcane were used in this study. The proximate composition such as moisture, ash, organic matter (OM), carbon (C), nitrogen (N), C/N ratio, and heating value (HV) were determined to generate mathematical models. Only ash, nitrogen, organic matter (OM) and carbon (C) have statistically significant r values toward the heating value. Positive correlation was obtained in OM and C whereas negative correlations were obtained in ash and N. Linear regression using Waikato Environment for Knowledge Analysis (WEKA) machine learning generated mathematical models for group variables for predicting the heating value (HV). The organic matter (OM) content showed the most accurate model among the group variables used with adjusted R² value of 0.900, Pearson’s r value of 0.915 and root mean square error (RMSE) value of 481.64 MJ/kg. Among the proximate composition parameters tested, OM is the most accurate predictor of the HV of the Napier Grass varieties and wild sugarcane biomass.

**Keywords:** Heating Value; biomass; machine learning

**INTRODUCTION**

Biomass is the collective term used to describe plant matter and derivatives such as residues from forests and crops, animal wastes, and the organic contents of municipal and domestic wastes. It
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Napier grass (Pennisetum purpureum Schumach) and wild sugarcane (Saccharum spontaneum L.) are grown to be used as feedstock to ruminants. At young stage (45 days) they are palatable and nutritious for ruminants. However, at the mature stage, these are unpalatable and the feed value of these grasses is obviously underappreciated (Feedipedia, 2016). At this stage, they have high lignin, cellulose, and hemicellulose content, which is the main combustible component of the grass that makes them a suitable material for energy production (Flores et al, 2012). Specifically, the mature cuts can be used as feedstock for energy (Mendoza et al., 2002). Brioness (2011) reported that Napier grass can produce 18.4 M J/kg of energy, which is comparable to a low-grade coal. Thus, using the mature parts of these grasses as feedstock for energy may open doors for its potential use for industries and households.

The high heating value (HHV), also known as the gross heating value (GHV), refers to the heat released by the complete combustion of a unit volume of fuel leading to the production of water vapor and its eventual condensation. The total energy released is measured at this point (Friedl et al., 2005). The energy derived from fossil fuels is more expensive (Chum and Overend, 2001; and Demirbas, 2001). On the other hand, the production of biomass fuel from wastes at specialized sites is cheap, which can be profitable to farmers (Bain et al., 1998). However, the major disadvantage of biomass as a fuel is its high moisture content, which is inversely correlated with its heating value (HV) (Vassilev et al., 2010). The determination of the HV of the biomass for grading and for gross energy estimation is complex and can be determined only by accredited laboratories. While most estimation of HV is based on ultimate analysis, which is complicated and difficult to obtain considering the limited laboratories that conduct this analysis, the use of the proximate composition of the grasses for HHV estimation have been demonstrated. Proximate compositions such as volatile matter (VM), fixed carbon (FC), and ash were developed by the American Standards Testing and Materials (ASTM) to estimate HHV values. However, the VM determination requires nitrogen gas for oxygen-free analysis condition, which adds cost to the analysis. On the other hand, the protocol developed by the Association of Official Analytical Chemist (AOAC) for ash and moisture determination, does not use nitrogen gas making it simpler and readily applicable in most laboratories. Since carbon (C) is a widely used estimator of HV, this study explores the principle of loss of ignition (hundred percent less ash as dry weight) and assumed it to be the organic matter (OM), which is mostly carbon, of the biomass. The organic matter content was then used to generate mathematical models for HV prediction. Validation techniques were used to determine the accuracy and repeatability of the mathematical models generated.

The objective of the study is to determine the heating value (calorific value) of the different varieties of Napier grass and wild sugarcane and determine their relationship with its proximate composition by Pearson correlation. Moreover, mathematical models using different algorithms in Waikato Environment for Knowledge Analysis (WEKA) machine learning were generated to determine the variable set for HV prediction.
METHODS

**Materials and Equipment.** Mature Napier grass varieties and native variety of wild sugarcane (approximately 3 months of age) samples were sourced out at the Philippine Carabao Center (PCC) of the University of Southern Mindanao, Kabacan, North Cotabato. Four varieties of Napier grass were used in the study namely; King, Florida, Dwarf, and Princess Caroline. Sampling and analyses were replicated three times and sampling was done in every 45 days. Above ground biomass was cut 3 cm from the ground. Fresh cuttings were washed and air-dried before shredding. The shredded biomass was placed inside a paper bag and dried in the oven at 60 °C. The dried biomass was milled using a laboratory mill and the obtained powder samples were stored in zip lock bags.

Proximate analysis such as ash and nitrogen content using Kjeldahl method were determined using the methods of the Association of Official Analytical Chemists Official Methods of Analysis (1975). Moisture content was determined using the procedure set by Mettler-Toledo Moisture Analyzer. For heating value determination, ASTM Method 3286-96 Isoperibol Bomb Calorimetry (ASTM International, 1996) was used.

**Data Analysis.** Organic Matter and Carbon Content. Organic matter (OM) was determined by using Eq. 1 (Machado et al., 2018) with slight modification and carbon content (C) was determined by dividing OM by a factor which is 1.72 Eq. 2.

\[
OM(\%) = 100 - ash(\%) - moisture(\%)
\]

\[
C(\%) = \frac{OM(\%)}{1.72}
\]

Statistical Treatment. Data were analyzed using analysis of variance and dependent variables with significant differences were further analyzed using Tukey’s test and the test of homoscedasticity (homogeneity of variance) using Levene Test was conducted. Pearson correlation was run to determine the relationship of the proximate composition to the heating value of the biomass. Linear regression algorithm was run using the free machine learning platform WEKA (Waikato Environment for Knowledge Analysis) version 3.8.4 of the University of Waikato, NZ (https://www.cs.waikato.ac.nz/ml/weka/) and generated mathematical model was used to determine the predicted HV values. Different variables were run separately whether it was a cluster of variables or individual variables to generate mathematical models. Meanwhile, mean absolute error (MAE) and mean bias error (MBE) were calculated in Microsoft Excel 2010 software using the equations 3 and 4

\[
MAE = \frac{1}{n} \sum_{i=1}^{n} \left| \frac{HV_{predicted} - HV_{experimental}}{HV_{experimental}} \right| \times 100
\]

\[
MBE = \frac{1}{n} \sum_{i=1}^{n} \left( \frac{HV_{predicted} - HV_{experimental}}{HV_{experimental}} \right) \times 100
\]

where \(n\) is the number of observations, \(HV_{predicted}\) is the predicted HV and \(HV_{experimental}\) is the experimental HV. These statistics are used to determine the accuracy and reliability of the predicted HV.

Paired t-test at 95% level of confidence was run to determine if there was significant difference between the experimental and predicted HVs. All statistical analyses – except the linear regression – were run using the free trial version of IBM SPSS Statistics 20 (https://www.ibm.com/account/reg/ph-en/signup?formid=urx-19774).
RESULTS AND DISCUSSION

Proximate composition of Napier and Wild Sugarcane Grass. The proximate composition (moisture, ash, N, OM, and C), C/N, and HV of the different varieties of Napier grasses and wild sugarcane are presented in Table 1. Figure 1 shows the proximate (organic, inorganic, and moisture) composition of the lignocellulosic biomass. The combined biomass is composed of 78% OM, 15% ash, and 7% moisture. Most of the OM is composed of cellulose, hemicellulose, and lignin, which provide the biomass a high amount of latent energy that can be exploited for energy generation (Kim et al., 2016; Corbo et al., 2011).

![Composition of the Biomass (percent by mass)](image)

Figure 1. Proximate composition of the lignocellulosic biomass used in the study. Organic matter represents most part of the proximate composition; while the inorganic matter (ash) comes next with high margin from the organic matter.

One assumption of running parametric statistics is the homogeneity of the variances and the normal distribution of the data. Once violated, the data must be transformed to meet the criterion. Using Levene Test, one can determine what appropriate statistical test will be used. When the p-value of the test is greater than 0.05, the data is normally distributed, thus its variance; otherwise, data transformation should be applied. All the data except for C/N ratio had normal distribution and homogeneous variances (Table 1). The C/N ratio data was log transformed so that the assumption of equal variance and normal distribution will be met.

Mean moisture contents of the dried biomass ranged between 6.60% and 7.78% and they were comparable at p>0.05. The King variety had the numerically lowest moisture content of 6.60±0.43% whereas the wild sugarcane variety had the numerically highest moisture content of 7.78±1.32%.

Wild sugarcane’s ash content (7.87±0.40%) had the lowest ash content significant at p<0.01. The ash contents of Napier grass varieties are not significantly different indicative of similar ash contents regardless of the variety. Results showed that the Napier grass contains higher amounts of mineral contents as compared to the wild sugarcane suggesting that the latter is a better biomass for energy consumption. In terms of percentage organic matter (OM) and percentage carbon (C), the Napier King, Napier Florida, and wild sugarcane have comparable OM and C contents at p>0.05. However, wild sugarcane has significantly higher OM and C contents at p<0.01 compared to the Napier Dwarf and Napier Princess Caroline varieties (OM: 84.36±0.92%, 75.80±2.06%, and 75.27±0.35% respectively; C: 49.04±0.54%, 44.07±1.20%, and 43.76±0.20% respectively).
Table 1. Proximate Composition and Calorific Value Contents for the Napier varieties and wild sugarcane (Mean±S.D.)

| Variety          | %Moist | %Ash* | %OM* | %C* | %N* | C/N² | HV (MJ/kg)*1 |
|------------------|--------|-------|------|-----|-----|------|--------------|
| Napier King      | 6.60±0.43* | 16.26±0.86* | 77.14±0.43* | 44.85±0.25* | 1.12±0.036* | 40.07±1.09* | 15.21±0.412* |
| Napier Florida   | 7.39±1.73* | 15.37±0.02a | 77.24±1.71b | 44.91±0.99b | 1.03±0.21a | 46.00±10.35a | 15.60±0.602b |
| Napier Dwarf     | 7.13±1.28b | 17.08±0.78a | 75.80±2.06a | 44.07±1.20b | 2.00±0.02b | 22.03±0.38b | 14.83±0.401b |
| Napier P. Caroline | 6.67±0.13a | 18.07±0.22a | 75.27±0.35a | 43.76±0.20a | 1.99±0.026b | 21.99±0.39b | 14.98±0.046b |
| Wild sugarcane   | 7.78±1.32a | 7.87±0.40b | 84.36±0.92b | 49.04±0.54b | 0.84±0.24b | 61.98±19.12b | 17.90±0.477b,c |

* Denotes p<0.01
Values with the same letters have no significant difference.
¹Organic matter
²Carbon-nitrogen ratio
³Heating value
†Test for variance homogeneity. If p>0.05, the data has equal variance.
‡The data was Log transformed to meet the criteria for conducting one-way ANOVA.

Comparable %N values were observed in Napier King, Napier Florida, and wild sugarcane and the same went to Napier Dwarf and Napier Princess Caroline. Significant differences at p<0.01 were observed between the two species of grasses with Napier Dwarf the highest (2.00±0.020%) and wild sugarcane had the significantly lowest value (0.84±0.24%).

The gross heating value refers to the heat released by the complete combustion of a unit volume of fuel leading to the production of water vapor and its eventual condensation. Lignite coal which is mainly used for electricity generation has a heating value of 16.08 MJ/kg (Engineering Toolbox, 2010). The HV of wild sugarcane is significantly higher than the rest of the Napier varieties at p<0.01 (17.90±0.477 MJ/kg), which is also higher than the lignite coal. The observed high HV of wild sugarcane is associated with its high % of OM and carbon content (%C), which are related to the inherent energy of carbon-based materials that are released upon combustion process most notably the lignin which is highly correlated to HV (Demirbas, 2002). The bond enthalpies of the functional groups present in the structural carbohydrate contain stored energies (C-H, 413 kJ; C-C 348, kJ; C-O, 358kJ; O-H, 463, kJ), which is higher in functional groups with multiple bonds (C=C, 839 kJ and C=O, 799 kJ) (Whitter et al., 2005). The combustion of lignocellulose produces high energy release which can be exploited for energy generation.

Carbon to nitrogen ratio (C/N). The choice of the specific process is affected by evaluating the characteristics of the initial biomass, in particular, its carbon and nitrogen content (C/N ratio) and humidity. High nitrogen content (low C/N ratio) implies green vegetable material, more suitable for biochemical reactions, and suitable for soil conditioning; whereas high carbon content (high C/N ratio) indicates that the biomass can usefully be exploited by combustion (Corbo et al., 2011). According to Dioha et al., (2013), C/N ratio ranging between 20/1 and 30/1 are the best. Native Napier variety in mature stage has a C/N ratio of 37.3/1 with a heating value of 17.15 MJ/kg (Flores et al., 2012). As shown in Table 1, the wild-sugarcane had the significantly highest C/N ratio of 61.98±19.12 (p<0.01), which is comparable to Napier King and Napier Florida varieties; the Napier Princess Caroline variety had the significantly lowest C/N ratio of 21.99±0.39, which is comparable to the Napier Dwarf variety. In the mature stage, grasses contain more structural carbohydrates than in vegetative stage (Brink and Fairbrother, 1994; Albrecht et al., 1987;
Cherney et al., 1993) and the N levels decrease as a result of the decrease of the rate of protein synthesis (Baranga, 1983; Walkley, 1940). This implies that the grass varieties/species are suitable for energy production.

**Relationship of the proximate composition to calorific value.** Table 2 shows the Pearson’s r of the respective proximate compositions to the calorific value (HV). Significant relationships were obtained on nitrogen (N) (p<0.01) as well as on ash, organic matter, and carbon (p<0.01). Significant but negative correlations were obtained from ash and N. In contrast, statistically significant and positive correlation was obtained from OM and C.

**Table 2. Pearson’s r values for the proximate composition correlated with the calorific value**

| Parameter (MJ/kg) | Statistics | Moisture | Ash | OM | C | N |
|------------------|------------|----------|-----|----|---|---|
| HV               | r          | 0.382    | -0.939* | 0.952* | 0.952* | -0.661* |
| Sig. (2-tailed)  | r          | 0.160    | 0.000 | 0.000 | 0.000 | 0.007 |
| n                |            | 15       | 15   | 15  | 15  | 15  |

*Denotes significant at p<0.01

Organic matter (OM) and thus, the carbon content, have higher levels corresponding to higher heating value (r=0.952; p<0.001). This can be supported by the fact that the chief combustible material in plant materials is the fiber content, which is mostly composed of carbon-containing compounds. The fiber content increases as the plant matures, thus its carbon content and the heating value also increases. Plant fibers consist of lignin, cellulose, and hemicellulose, which contain high HV (lignin: 20.4 MJ/kg; cellulose: 16.5 MJ/kg; and xylan (hemicellulose): 13.9 MJ/kg) (Kim et al., 2016). Combination of these structural carbohydrates (in proportion) makes up the gross heating value of the biomass.

After combustion of plant materials, a silica-rich residue is left in the form of the ash. It contains no calorific value thus it does not contribute to the total energy content of the plant material. Moreover, “high ash content has been related to certain ignition and combustion problems, while a low dissolved ash fusion temperature can lead to furnace dirtying and the formation of slag” (Saidur et al., 2011). Low ash content means high calorific value confirming the significant (p<0.001) Pearson’s r negative correlation (-0.939) of ash to the calorific value.

High nitrogen is suitable for feedstock to ruminants whereas low nitrogen but high carbon content is more suitable as biomass for energy production. Nitrogen has r value of -0.661, which is significant at p<0.01. This means that as the nitrogen content decreases, the calorific value increases, which proves the ideal C/N ratio (higher carbon content to nitrogen). It should be noted that the heat of combustion of N is 33.10 kJ mol\(^{-1}\) to produce NO\(_2\), which is endothermic or it requires energy to proceed the reaction rather than producing heat (energy) (Huang and Lo, 2020). Thus, the correlation is negative.

**Theoretical Heating Values.** The theoretical heating value is the predicted values obtained from the mathematical model. Using WEKA Machine Learning version 3.8.4 (developed by the University of Waikato, NZ), a linear regression analysis was run to determine the underlying mathematical model from the given parameters to predict the heating values. There were four mathematical models generated from mining the data. Among the four equations, the equations below were the most accurate in HV prediction:
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\[
HV \left(\frac{MJ}{kg}\right) = 0.325(OM) - 9.647 \\
HV \left(\frac{MJ}{kg}\right) = 23.105 - 0.327(Ash) - 0.353(Moisture)
\]

where HV is the heating value of the biomass in MJ/kg and OM as the percentage organic matter of the biomass. The scatter plot for Eq. 5 and Eq. 6 are shown in Fig. 2 and Fig. 3, respectively.

Figure 2. Scatter plot of organic matter (OM) vs. heating value (MJ/kg).

Figure 3. Scatter plot of ash (%), moisture (%), and HV (MJ/kg).

Adding a variable to improve the R\(^2\) value can be misleading because it may cause over-fitting of the model. To compensate for this, adjusted R\(^2\) is calculated to determine if the added variable has significance to the model. Adding useless variables always increase R\(^2\) and adjusted R\(^2\) may also increase but most of the time it decreases. Moreover, it explains the variance made by the independent variable to the dependent variable. The R\(^2\) of the model is always higher than the adjusted R\(^2\). Adjusted R\(^2\) can be negative when R\(^2\) is almost zero which implies poor variables relationship (Glen, 2013).

The adjusted R\(^2\) of the two equations are 0.900 (p<0.01: df=13) and 0.891 (p<0.01: df=12) respectively (Table 3). In terms of root mean square error (RMSE), Eq. 5 had lower RMSE of 481.64 MJ/kg with Pearson’s r of 0.915 than Eq. 6 with RMSE of 519.86 MJ/kg with Pearson’s r of 0.907.
Table 3. Accuracies of the generated mathematical models for HV prediction using WEKA 3.8.4.

| Equation | Independent variables | Adjusted R² | df | Significance | RMSE² (MJ/kg) | Pearson’s r | MBE (%) | MAE (%) |
|----------|-----------------------|-------------|----|--------------|---------------|-------------|----------|---------|
| 5        | Organic matter (OM)   | 0.900       | 13 | p<0.001      | 481.64        | 0.915       | 0.047    | 1.65    |
| 6        | Ash and moisture      | 0.891       | 12 | p<0.001      | 519.86        | 0.907       | 0.046    | 1.67    |

1 Degrees of freedom
2 Root square mean error
3 Mean bias error
4 Mean absolute error

The mean bias error (MBE) and mean absolute error (MAE) are statistical measures used to determine the accuracy of the predicted heating values from the measured heating value (experimental). A MAE value close to zero means that the predicted value is close to the experimental value, affording high accuracy. Meanwhile, an MBE with positive value underestimates the predicted value and a negative MBE otherwise. The MAE of Eq. 5 is 1.65%, which means that the predicted value is 1.65% higher than the experimental value; the MAE of Eq. 6 is 1.67%, which means that using Eq. 6, the predicted value is 1.67% higher than the experimental value. On the other hand, the MBE of Eq. 5 (0.047%) is slightly higher than Eq. 6 (0.046%). Both equations underestimate the predicted HV only by 0.046 to 0.047%.

Eq. 5 is better in predicting HV compared to Eq. 6 because it has the lowest RMSE and MAE and highest adjusted R² and Pearson’s r. In terms of MBE, Eq. 6 is lower than Eq. 5 but only in a very small margin.

Using paired t-test at 95% level of confidence, Eq. 5 and Eq. 6 were compared. There is no significant difference between the measured and predicted heating values using the mathematical models (Eq. 5 and Eq. 6) (Table 4). This implies that the generated mathematical models can be used to predict the heating values of the Napier grass and wild sugarcane biomass at mature stage (3 months and so) although Eq. 5 gives a better fit than Eq. 6.

Table 4. Paired t-test for measured (experimental) and theoretical (predicted) HV.

| Heating Value | Mean (MJ/kg) | SD (MJ/kg) | SEM (MJ/kg) | df | Significant? |
|---------------|--------------|------------|-------------|----|--------------|
| Experimental  | 15704.45     | 1220.77    | 315.20      | 14 | No (p>0.05)  |
| Equation 5    | 15704.57     | 1162.30    | 300.10      |    |              |
| Equation 6    | 15704.54     | 1162.32    | 300.11      | 14 | No (p>0.05)  |
| Equation 5    | 15704.57     | 1162.30    | 300.10      |    |              |
| Equation 6    | 15704.54     | 1162.32    | 300.11      | 14 | No (p>0.05)  |

1 Standard deviation
2 Standard error mean
3 Degrees of freedom

The obtained mathematical models were compared to mathematical models obtained by Sahito et al., (2013). Instead of AOAC proximate composition parameters, they used ASTM protocol. The parameters were fixed solids (FS), volatile matter (VM), and ash. Table 5 shows the summary of the equations with their respective R², mean bias error (MBE), and mean absolute error (MAE). Unlike this current study, which used Napier and wild sugarcane biomass to estimate heating values by generating mathematical models (with a total number of 15 samples), Sahito et al., (2013) used a total of 22 biomass from different sources to determine FS, VS, ash, and heating value (MJ/kg). Such biomass used were corn cob, rice straw, coconut coir, saw dust to name a few.
Table 5. Adjusted $R^2$, MBE, and MAE of the equations

| Equation                                      | Independent variables          | Unadjusted $R^2$ | MBE(%)$^1$ | MAE(%)$^2$ | Reference                  |
|----------------------------------------------|--------------------------------|-----------------|------------|------------|----------------------------|
| $HV = 0.325(OM) - 9.647$                     | Organic matter (OM)           | 0.907           | 0.047      | 1.65       | This study                 |
| $HV = 23.104 - 0.327(Ash) - 0.353(Moisture)$ | Ash and moisture               | 0.907           | 0.046      | 1.67       | This study                 |
| $HV = 0.22551(VS) + 0.02505(FS)$            | Volatile solids & fixed solids| 0.783           | 0.17       | 3.36       | Sahito, et al (2013)       |
| $HV = 0.21575(VS) + 0.07492(FS) - 0.08426(ash)$ | Volatile solids, fixed solids, & ash | 0.822           | 0.13       | 2.71       | Sahito, et al (2013)       |

$^1$Mean bias error

$^2$Mean absolute error

Many literatures cited that the carbon (C), nitrogen (N), sulphur (S), hydrogen (H), oxygen (O) or collectively known as CHNS/O as the most reliable predictors of heating value. Moreover, fixed carbon and volatile matter are also reliable information in predicting the HV of the lignocellulosic biomass (Cordero et al., 2001; Machado et al., 2018; Sahito et al., 2013; Friedl et al., 2005). Elemental analysis requires sophisticated equipment and high cost input to be used for predicting HV. By determining the proximate composition of the biomass (such as ash, moisture, and OM) can be enough to obtain a reliable HV, which is true by using the equation generated in this study (Eq. 5 and Eq. 6). Moreover, to highlight this claim, the obtained mathematical models in this study had higher accuracy compared to the mathematical models obtained by Sahito et al., (2013) using the VM, FS, ash content of the biomass.

In this study, N does not have significant bearing on the HV of the biomass. The N level of biomass is more significant for forage and biochemical reactions than in energy generation for combustion (Corbo et al., 2011; Feedipedia, 2016). The result is consistent with the observed nonsuitability of mature grass for ruminant consumption because of its low nutritional level and indigestibility. The results in this study showed that mature grasses can instead be utilized for bioenergy production.

CONCLUSION

The study aimed to determine the mathematical model that best fits the HV prediction using proximate composition. Using cross-validation and accuracy determination, the mathematical model with the lowest root mean square error (RMSE), highest Pearson’s r, highest adjusted $R^2$, lowest mean absolute error (MAE), and mean bias error (MBE) was chosen. The variable organic matter (OM), which can be determined by loss of weight, was the most reliable proximate component that gave the most accurate HV prediction. By determining the moisture and ash contents of the Napier and wild sugarcane biomass, one can estimate the HV of biomass by determining the OM (by subtracting ash and moisture percentages from 100). The validated mathematical model can be used to estimate heating values of biomass for Napier Grass varieties and wild sugarcane without using the conventional and expensive analysis using bomb calorimeters.

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