Comparative study of methods to measure the density of Cementitious powders

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Abstract

The accurate measurement of the density of hydraulic cement has an essential role in the determination of concrete mixture proportions. As more supplementary cementitious materials (SCM), such as fly ash, and slag, or cement replacements materials such as limestone and calcium carbonate are used in blended cements, knowledge of the density of each powder or of the blended cement would allow a more accurate calculation of the proportions of a concrete mixture by volume instead of by mass. The current ASTM standard for measuring cement density is the "Test Method for Density of Hydraulic Cements" (ASTM C188-14), which utilizes a liquid displacement method to measure the volume of the cement.

This paper will examine advantageous modifications of the current ASTM test, by alcohol substitutions for kerosene. In addition, a gas (helium) pycnometry method is evaluated as a possible alternative to the current standard. The described techniques will be compared to determine the most precise and reproducible method for measuring the density of hydraulic cements and other powders.

Keywords: density, hydraulic cement, volume, pycnometer, liquid displacement

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Introduction

Concrete is sold by volume, but typically, the proportioning is done by mass. However, when using binary and ternary binder mixtures, there are advantages to proportioning by volume. To accomplish this, the density of the component powders should be known and accurately measured because the density of the powders used could vary as low as 2200 kg/m^3 [1] for fly ash to 3150 kg/m^3 for portland cement. One composition of cement is to blend supplementary cementitious materials (SCMs) or other materials that serve as fillers with portland cement. The current interground limestone cements (ASTM C150) [2] and blended cements (ASTM C595) [3] have a density lower than conventional portland cement (a mixture of ground cement clinker and gypsum). The commonly assumed density of 3150 kg/m^3 (3.15 g/cm^3) for portland cement [4] will not be correct when limestone is interground into the cement. Neither ASTM C150 nor ASTM C595 requires the density to be reported or measured. As a result, the lower density of the fly ash [5] will result in a higher volume of fly ash replacing the cement powder when replacement is performed on a mass basis, possibly affecting the water demand compared to conventional concrete [1].

Therefore, accurate density measurements for the cement and other cementitious materials are essential to properly design concrete mixtures by volume. In an effort to meet industry needs, research was conducted to improve the feasibility and efficiency of current standard hydraulic cement density measurements, and their applicability to density measurement of SCMs.

Currently, ASTM C188 [6] is the standard test method to measure the density of hydraulic cement. The current testing standard is time consuming and requires disposal of the chemicals. A specially designed flask, called a Le Chatelier flask, and 300 mL/test (or 900 mL for a 3 replica) of kerosene are required to perform the standard test, producing. Specific directions for handling and disposing of the equipment and materials consume time and resources. Kerosene requires extra precaution when handling, as its vapors are not pleasant and toxic, requiring the use of a fume hood. Currently, when measuring density of cement powders, scientists often substitute kerosene with isopropyl alcohol (IPA) or another readily available organic chemical. It should be noted that this practice would be acceptable by ASTM C188 if it is first verified that " a single operator can obtain results within ± 30 kg/m^3 of the results obtained using the flask method" [6]. The verification is time consuming as a cement needs to be measured with Kerosene and then again with the new liquid. The standard does not state how often this verification needs to be conducted. NIST started using gas pycnometry, instead, to quantify hydraulic cement density since 2012 as it is faster, generates no chemical to dispose. Gas-comparison pycnometry has been approved since 1967 as the standard testing method following ASTM C604, "True Specific Gravity of Refractory Materials" [7]. Helium pycnometers may be a practical replacement or alternative to the current standard method, once their consistency for measuring the density of cementitious materials is proficiently shown.

In this paper, the standard volumetric displacement procedure will be tested using IPA, kerosene, and ethyl alcohol (ethanol). Data from a helium pycnometer will be obtained as an alternative measurement technology. Some discussion on the advantages and disadvantages of each method will be presented. Each suggested method will be tested on a variety of hydraulic cements and limestone powders. Ultimately, changes in the standard are suggested.

Test Methods Used

Volume Displacement or Standard C188

The principle of the ASTM C188 test method [6] is based on the measurement of the displaced volume of a liquid by the addition of a powder specimen. The density can thus be calculated using the mass of powder. A special flask, the Le Chatelier flask (Figure 1 [8]), is used to facilitate the volume measurement. Between 60 mL and 80 mL, or the equivalent of about 64 g (for cement), of the powder material is used. Kerosene or naptha are specified in the standard, although other liquids could be considered if it is demonstrated that the difference in measurements between the standard and the proposed modification is less than 30 kg/m^3. The temperature shall be also monitored, as it should remain within 0.2 °C throughout the test.

To maintain stable conditions, the procedure recommends placing the flask in a constant-temperature water bath to avoid fluctuations greater than 0.2 °C between the initial and final measurements. The key measurement is reading the meniscus of the liquid at two different levels to determine the volume of liquid displacement. These readings are a source of operator error due to parallax error if the meniscus is not read correctly.

Figure 1: *Le Chatelier flask [8]*

When calculating density, equation [1] is used:

$$D = \frac{M}{V} \qquad \textbf{[1]}$$

where D is the density (in kg/m^3), M is the mass of the cement sample (kg), and V is the liquid (i.e., kerosene) displaced volume after adding the material (m^3). The precision statement in ASTM C188 states that for a single operator, the standard deviation is 12 kg/m^3, thus "two measurements by the same operator should not differ by more than 30 kg/m^3"[6].

Sources of error

Upon examination of the standard procedure, the sources of error involved in executing the experiment become evident. Temperature changes of the kerosene can cause volume changes of the fluid. The volumetric coefficient of expansion [9] for kerosene is 9.9 x 10^{-4} °C^{-1}. The temperature control of the fluid in the flask is essential to reduce error. Human error must also be considered in this experiment. Although the meniscus should always be read at eye level and at the center of the meniscus, there could be an error of parallax in the readings.

Another source of error is the amount of material that is introduced into the flask. The mass of powder is typically measured into a container and then transferred carefully into the Le Chatelier

flask. If some material is lost in the transfer, then the mass used in the calculation (equation 1) is incorrect. In the present study, the flask was weighed before and after the addition of the tested specimen on a balance with a readability of 0.01 g to reduce this source of error. The standard C188 states that the mass needs to be measured to the nearest 0.05 g. For this study, this difference between these two mass values was used as the mass of the introduced specimen.

Accuracy of the volume of the flask could be another source of error. To verify the magnitude of this error, several flasks purchased at different times (within years) were filled with distilled water. The same method ASTM C188 procedure was used but in the place of powder, water was used. Then, the same density equation [1] was applied to calculate the density of the water. Four Le Chatelier flasks were tested at a temperature of 20.5 ◦C ± 0.1 °C. The overall average density calculated is 996 kg/m^3 ± 4 kg/m^3. That is certainly below the standard deviation for the test (12 kg/m^3) by a factor of 3. The reported density of the water at that temperature is 998 kg/m^3 [10]. Thus, the error in comparison to the known density of the water is 2 kg/m^3. This process demonstrates that the variability in the actual volume among different flasks should have a negligible contribution to the overall uncertainity.

Pycnometer

The procedure to measure density by means of a helium pycnometer is based on a gas displacement method to accurately measure the volume of specimen (Figure 2a [11]). The process consists of obtaining the specimen mass using a balance reading to the nearest 0.0001 g. An empty metal cylindrical container with its two corresponding pieces, a cap and metal insert (Figure 2b), are cleaned using a clean brush and weighed. A specimen containing about 5 g of material is next placed and compacted into the small insert. The outside of the insert is cleaned once more, and placed inside the cylindrical container. The cap is set in place and the whole vessel is weighed again and enclosed inside the helium pycnometer. The measurement system is typically controlled by a computer. The device then performs the density test by filling the cavity with helium gas to measure the volume of the material. The density is calculated from the volume measured and the material mass.

A ***B***

Figure 2*: Helium Pycnometer*[1]*. A) the overall view; B) accessories: cap, metal container and calibration ball*

[1] Certain commercial products are identified in this paper to specify the materials used and procedures employed. In no case does such identification imply endorsement or recommendation by the National Institute of Standards and Technology, nor does it indicate that the products are necessarily the best available for the purpose.

Sources of error

While the method reduces the potential for test-related bias due to a reduced possibility for human error, experimental errors may still occur. The calculations are done from the volume of helium, volume of the container, and the specimen mass entered in the computer by the operator. If the mass is incorrect due to dust particles, if the material is left exposed for too long (absorption of water for instance), or if the volume of the container is incorrect due to improper cleaning, errors in the measurements will result. Thus, it is important to ensure that all the weighed material is actually inside the container and that no extra particles are incorporated. For instance, in the container used in this study, a small hole was present on the side near the top of the container; thus when the material is not kept below that hole, there is a possibility that some of the tested specimen can be carried out with the gas used, thus reducing the actual specimen mass used in the measurement. The change in specimen mass will affect the results of the test, as the amount of powder in the container might not be known.

A metal sphere (that has a diameter similar to the diameter of the container) of known volume is used to verify the accuracy of or calibrate the pycnometer. Care should be taken that all the parts, e.g., container and sphere, are very clean and do not have particles such as dust on the surface and also that no dents or scratches are introduced on their surface. Serious damage to the sphere would require the replacement of a calibrated sphere. Any measurements for the sphere volume not within 0.01 cm^3 of the certified amount will signify that the machine requires recalibration.

Specimen moisture content could also affect the measurement or its duration, as the first step consists in creating a vacuum. A sufficient vacuum pressure could be difficult to obtain if the specimen contains too much moisture.

Materials

Alcohols for the Volume Displacements

To investigate the applicability of ASTM C188 to the usage of different liquids, three different organic liquids were employed: 1) kerosene as recommended by the standard; 2) IPA; and 3) 190 proof and 200 proof ethanol. The selection of IPA was motivated by its ability to act as a carrier liquid to measure the particle size distribution (PSD) of cement by laser diffraction [12], a method to assess the fineness of the material. Ethanol is commonly found in laboratories and both 190 proof and 200 proof were used for testing. However, it was discovered that the difference in powder densities measured between the two ethanols was less than 5 kg/m^3. Thus, to simplify the experimental plan, the remainder of the study was limited to the usage of 200 proof ethanol.

One means of selecting the liquid medium is to examine the hazards found in the corresponding Safety Data Sheets (SDS). Table 1 displays the hazard levels of the three liquids.

Table 1: *Hazards from SDS of the liquids considered. NFPA is National Fire Protection Association*

Hazard	Kerosene	IPA	Ethanol
Eyes	Mild	Moderate	High
Dermal	Moderate	Mild	Mild
Inhalation	High	Mild	Mild
Ingestion	High	Moderate	Moderate
Environment	High	Mild	Mild
NFPA Health	2	2	2
NFPA Fire	2	3	3
NFPA Reactivity	0	2	0

A careful review of the SDS reveals more hazard specifics actions. The primary kerosene health hazards came from inhalation, skin and eye contact. For ethanol, the main hazard is eye irritation. In general, the use of personal protective equipment (PPE) such as gloves and safety glasses, helps prevent skin and eye contact. To protect the operator from inhalation hazards, the experiment should be conducted in a fume hood. However, not all laboratories are equipped with this device and it is expensive to install.

Powders tested

A variety of materials used in concrete production were tested: 1) two limestones (designated as MA2-182 and MA3-112) from different lots but from the same origin; 2) five cements of different origin. The two limestones were micro-limestone flour; their surface area by BET[2] nitrogen adsorption method and the particle size distribution (PSD) by laser diffraction are shown, respectively, in Table 2 and Figure 3. This information is provided as supplemental information on the materials used.

The five cements included two ASTM Type I cements from the Cement and Concrete Reference Laboratory (CCRL), designated CCRL 115 [13,14] and CCRL 192 [15]. The other cements included Standard Reference Material (SRM) 114q, a white ASTM C150 Type I cement (designation MA3-94C), and an ASTM C150 Type III portland cement (MA3-94A). Their BET surface areas are shown in Table 2 and their PSDs in Figure 3.

The materials used in this study were obtained in sufficient quantities and blended using a 3-D mixer[3,1] to ensure homogeneity. The SRM 114q is delivered in small packages of 5 g each and a sufficient number of packages were opened and blended together before performing the density measurements.

[2] BET stands for Brunauer, Emmett and Teller

[3] A Turbula was used for this mixing

Table 2: *BET surface area and the one standard deviation of the materials used.*

Material	Limestone MA2-182	Limestone MA3-112	CCRL 115	CCRL 192	SRM 114q	White Type I cement MA3-94C	Type III cement MA3-94A
BET [m^2/g]	1.05 ± 0.02	1.04 ± 0.01	0.60 ± 0.03	1.14 ± 0.03	1.32 ± 0.03	1.16 ± 0.03	1.68 ± 0.03

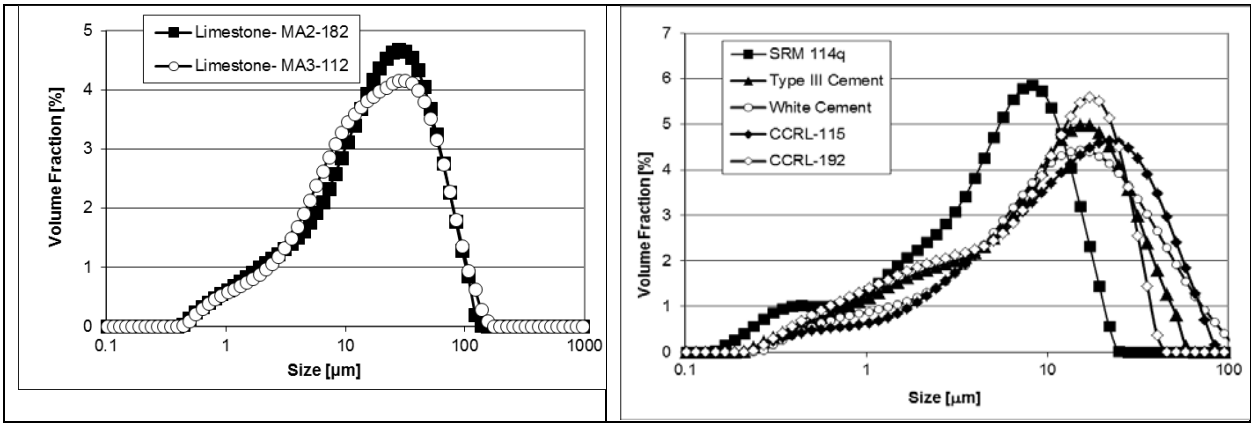

Figure 3: *Particle size distributions of the powders*

Results

Each powder was tested according to ASTM C188 standard test method using three trials of each testing media. A preliminary set of experiments were conducted to determine the standard deviation for the pycnometer which was found to be ± 1.7 kg/m^3. This was also corroborated by a number of tests performed at NIST for a wider variety of materials. Because, the uncertainty is far below the required 12 kg/m^3, for the rest of this study only one specimen will be measured using the pycnometer while three replicas will be performed according to ASTM C188 using the various media selected. The results are tabulated in Table 3.

The standard test method states that the standard deviation in one lab should be below 12 kg/m^3. From Table 3 and Table 4, that about half of the data meet this criterion. The standard deviations that are too high (above 12 kg/m^3) are shown in the table in bold. These high standard deviations are found as often with Kerosene and IPA (3 times each), while ethanol results in five high uncertainty measurements. IPA provides the most consistent results as the standard deviation varies only from 1 kg/m^3 to 25 kg/m^3, while both kerosene and ethanol have a maximum as large as 49 kg/m^3 and 45 kg/m^3, respectively.

The ASTM C188 also stated that the temperature should be controlled and should not vary more than 0.2 °C between the initial measurement and the final readings (Table 4).

Table 3: *Average density value and the standard deviation for each material by method for three separate measurements. The standard deviation that are larger than the precision statement in ASTM C188 are in bold.*

	Average Density Value [kg/m^3]			
	Le Chatelier Method ASTM C188			Pycnometer Method (1 σ = ± 1.7 kg/m^3)
	Kerosene	IPA	Ethanol 200 proof	
Limestone MA2-182	2735 ± **49**	2752 ± **25**	2747 ± **30**	2753
Limestone MA3-112	2741 ± 8	2740 ± 10	2739 ± 8	2772
Cement CCRL 115	3138 ± **19**	3150 ± **17**	3135 ± **17**	3197
Cement CCRL 192	3124 ± 8	3140 ± **24**	3120 ± **23**	3178
Cement SRM 114q	3145 ± 11	3156 ± 1	3120 ± 9	3162
White Cement MA3-94C	3107 ± **19**	3122 ± 10	3087 ± **45**	3136
Type III Cement MA3-94A	3035 ± 5	3026 ± 8	3022 ± **27**	3066

Table 4. *Uncertainty in the measurements both for temperature and density*

		Kerosene	**IPA**	**Ethanol 200**
Temperature change during the experiment (°C)		0.2	0.1	0.2
Range of density standard deviation (kg/m^3)	Min	5	1	8
	Max	49	25	45

Discussion

Comparison of the two methods: Le Chatelier and pycnometer

The ASTM C188 (section 3.3) standard test method states that other methods could be used if it is demonstrated that the results do not differ by more than 30 kg/m^3 from those using kerosene. Table 5 shows the calculated differences for the two alcohols and the helium pycnometer. As compared with the results obtained with kerosene, the densities measured by the pycnometer are on average higher by 34 kg/m^3, and in some cases the difference exceeds the ASTM C188 requirement. This could be explained by the fact that the helium gas penetrates in small pores within the material compared to the liquids selected, allowing the pycnometer test to assess porosity within the powder particles that might be impenetrable to the liquids using the ASTM

C188 test method. Therefore, the liquid methods are unable to detect a portion of this intra-particle porosity affecting the density calculation. As stated in ASTM D3766 [16], this is the difference between the skeletal density (Helium pycnometer) and the bulk density (ASTM C188). It may be debatable as to which density would be best suited for mixture proportioning for concrete. During concrete mixing, it is relevant to know if water penetrates the cement and other powders. If it does penetrate, then the density measured by the pycnometer might be the most relevant. On the other hand if water does not penetrate into the cement particles during mixing as with helium during the pycnometer testing, then the density should be better measured by ASTM C188 as it provides a more relevant value.

Additionally, the pycnometer method exhibits a much lower standard deviation, just under 2 kg/m^3. This is lower than nearly all measurements by the ASTM C188 method by over 80 %. The increased precision of the pycnometer test can be attributed to the more exact mass specifications that originate during the experiment, as the mass is measured using a high-precision balance (readability 0.0001 g instead of 0.01 g for ASTM C188). Gas-comparison pycnometer data also has the benefit of reduced opportunity for operator error.

Table 5. *Difference in density values between various methods and kerosene data (kg/m^3). Bold values exceed the C188 specification limit of 30 kg/m^3.*

	IPA	**Ethanol 200**	**Pycnometer**
Limestone MA2-182	17	13	18
Limestone MA3-112	-1	-2	30
Cement CCRL 115	13	-2	**59**
Cement CCRL 195	16	-4	**54**
Cement SRM 114q	10	-26	17
White Cement MA3-94C	15	-20	29
Type III Cement MA3-94A	-9	-13	**31**
Average Difference	**9**	**-8**	**34**

Improvement in the methodology for the Le Chatelier flask

While performing the tests, some deviations from the description in the standard method were considered to improve the test. The first one was to use funnels to introduce the powder into the Le Chatelier flask. Two funnels were used, one to introduce the fluid (wet funnel) and one to introduce the powder (dry funnel). The wet funnel delivered the fluid to the bottom of the flask avoiding wetting the flask neck. The dry funnel directed the powders into the central region of the flask to prevent material from sticking to the sides. Even when funnels are utilized, traces of materials from the specimen and fluid may stick along the sides of the flask, which misleadingly increases the mass without displacing the fluid. Even when rolled or given time for the kerosene to descend, not all of the excess specimen remaining on the neck of flask can be removed.

The experiment was also adjusted to better control the amount of cement being used. Between 60 mL and 80 mL of powder material was placed into a beaker as specified in the standard procedure. The mass of the flask with liquid up to the 1st graduation was measured. Then after adding the specimen to ensure that the level of the liquid will reach the upper graduations, the flask mass was measured again. This allows for greater accuracy of the mass measurements and

develops means for data verification. Some residual powder may remain within the beaker at the end of the test. Furthermore, some materials such as limestone have a natural lower density. Less powder is required for the volume to reach appropriate levels on the flask neck. Therefore, inserting 64 g of material, as used for cement, for every trial would overflow the flask. These procedural amendments were crucial in making the experiment suitable for a wider variety of powders.

Influence of liquid in the Le Chatelier flask

Comparing results of the different organic liquids within the standard procedure also supports several suppositions. When comparing the temperature changes in each experiment, the IPA averaged the least change in temperature from the time the alcohol was introduced into the flask until the final mass was recorded (Table 4) or just 0.1 °C without the aid of a water bath. Kerosene and ethanol both displayed a temperature change during the test of 0.2 °C, still within the limit of the standard test.

An increased difficulty was also observed in utilizing the kerosene with cement powders, as it generally increases the time spent on each test compared to the alcohols. Clumping of the hydraulic cements increased with the use of kerosene. The longer time period required to complete the steps allowed for a greater interval in which the liquid could either heat or cool.

In a majority of cases, the IPA offered a lower standard deviation when compared to the kerosene and ethanol tests. Kerosene exhibited the widest range of standard deviations (44 kg/m^3) (Table 3 and Table 4) or from 5 kg/m^3 to 49 kg/m^3. Ethanol exhibited a higher range in standard deviation of 37 kg/m^3 (8 kg/m^3 to 45 kg/m^3). While IPA has the lowest range of standard deviation (1 kg/m^3 to 25 kg/m^3). The IPA yielded more reproducible results overall. It could also be observed that both IPA and ethanol had about half the single operator uncertainty as specified in ASTM C188.

With respect to health and safety in handling the liquids, it should be noted that kerosene emitted pronounced fumes. While all the tests were performed under a working fume hood, it was still common for the kerosene odor to permeate the laboratory. The persistent kerosene fumes could be a discomfort for people conducting this testing, or for shared laboratory environments.

It was also found that kerosene is increasingly problematic to remove from the flask after performing the ASTM C188 testing. The shape of the flask is conducive for experimentation; however it is not favorable to a thorough cleaning, leaving stains and clumps of material. When checking the flask error by conducting density testing of distilled water with no powder, the dirty flasks received a higher percentage of uncertainty (0.5 % for three replicates) than was identified for the cleaner flasks (0.1 % for three replicates). Cleaning the flask after density testing using IPA or ethanol is considerably easier than when testing with kerosene. As several measurements need to be done for each material to be tested, harder or lengthy cleaning will add to the duration of the overall test.

The wetting properties of IPA and ethanol results in free flow as they have a 0° angle of contact with glass [17]. Less time will be expended waiting for the alcohol to collect at the bottom of the flask. Meanwhile, kerosene has a 26° angle of contact with glass [17], which creates greater beading that does not easily flow down to the meniscus.

However, the powders often cloud the liquid within the flask to the point of that it is difficult to discern meniscus when using IPA or ethanol. An advantage to the utilization of kerosene exists in the time needed for the powder to settle. The unique interactions of the kerosene and powder leave the liquid transparent towards the neck and allow for the meniscus to be read clearly.

A review of data obtained during this study suggests IPA as an ideal medium. The liquid generates the most precise data, as the range of standard deviation calculated for IPA is only 24, while kerosene and ethanol have 44 and 37, respectively.

Conclusions

Based on all observations and data measurements obtained in this study, it can be concluded that gas-comparison pycnometer testing is the most precise and most convenient test method for hydraulic cement density. Helium pycnometer measurements revealed the smallest standard deviation, the easiest cleaning procedure, and the least amount of material waste (no liquid to dispose safely). Nevertheless, the issue that measured densities are higher by 1 % on average when compared to the kerosene value must be considered. This value is also larger than allowed in ASTM C188 (section 5.3), thus rendering the authorization to use void. On the other hand, IPA and ethanol in this study differ by only 0.3 % from the kerosene. This is due to the methodology that allows the helium to penetrate deeply within the porosity of the powder, while the liquids (kerosene or alcohols) do not penetrate as significantly.

The unresolved issue is which density is best to use when calculating the volumetric proportions of a concrete mixture for cement replacement by SCMs. If it is assumed that the water will not penetrate the cementitious materials in the same way as a helium atom does, then either a systematic correction factor should be applied or the ASTM C188 method is more appropriate even if more tedious to perform.

When considering only the alcohol substitutions for the kerosene, IPA would be the best methodology for use in the current standard procedure. IPA results in the lowest standard deviation, smallest average percent error, and minimal temperature sensitivity. In addition, IPA has greater market accessibility and cleaning feasibility. Some improvements in the test were also identified such as using funnels for material introduction and weighing the flask before and after introduction of the powder.

An inter-laboratory study is recommended to corroborate these findings and discussion with the ASTM subcommittee could yield changes in the C188 standard [6], and either the creation of another standard using the pycnometer method or the adoption of ASTM C604 (for refractory materials) for hydraulic cements [7] as well.

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