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Independent Expert Report

This is a report on my review of on an internal paper of Ultimo Measurement LLC (Ultimo) titled "On the Precision of Gamma-Ray and MPD ("Percussion") Densitometers for Loose Solid Materials".

In the above paper two non-invasive methods for density measurements of non-gaseous free flowing materials are described and analyzed: a gamma-ray absorption method and the Percussion method developed by the Ultimo Measurement LLC.

Gamma Ray Absorption Method

Extensive literature analysis on the precision of gamma absorption method is presented in the paper. It was described that the accuracy of measurements can be increased by accumulating the number of counts and gamma method accuracy losses depend on variations of absorption of path length and how material range density is defined. It was concluded that practical standard deviation of gamma absorption method is in the range between 0.13 and 0.4%. As it was correctly mentioned in the paper the further increase in the path length will substantially reduce the gamma ray counts rate and the precision of measurements.

Using modified formula 1.1 presented in the paper and Mathcad 2000 software the precision of measurements F(n,l,N) by the gamma absorption method can be determined by formula

$$F(n,l,N) = \frac{100}{\sqrt{N \cdot \exp(-l \cdot n)}} \qquad (1)$$

In this formula N is the number of counts of a detector; n - density of sample; l – coefficient characterizing absorption properties of a sample and the path length; $l = \frac{\sigma \cdot S}{p \cdot 1000}$ where σ is absorption cross section of gamma rays by one atom; S - absorption path length; p – weight of one atom in the sample. It can be seen from the Fig. 1 (two upper red and blue dot lines) when l is increased from 1 to 1.1 the error for high density samples (for example 6g/L) is increased from approximately 15.3 to 18.2 %. At the same time for 2 g/L sample the error is changed from 8.5 to only 9.2 %. As we can see in all cases when density of sample is increased the precision of measurements can dramatically be deteriorated. For example, in the case when million counts are detected (bottom blue line in Fig.1) and when we have the sample with the density 2mg/L the error is in rather acceptable region 0.2 - 0.3 %. If it is 8 g/L the error become 5 - 6 % what is in many cases unacceptable. Based on these data we can say that the gamma ray absorption methodology is applicable mainly in cases when there are no substantial variations in the sample absorptivity, for example, when measuring the thickness of a metal strip during rolling. In the case when there is a substantial sample density difference (for example from 20 to 150 g/L described in the paper) the problem of precision deterioration for high density samples can occur.

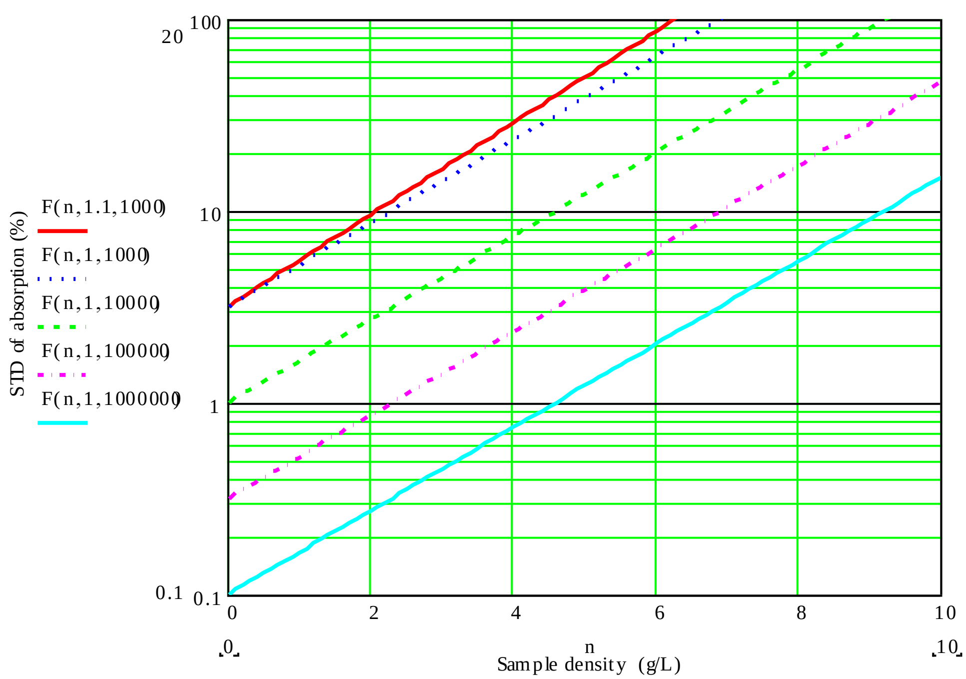

Fig1. Precision of absorption measurements.

It needs to be emphasized that a substantial absorption of gamma rays can also occur in the walls of a pipe with the sample. Despite of the fact that the pipe wall has much less thickness (6 -10 mm) than its diameter (300 -500 mm) the background absorption in the walls can be even more than in the sample. It is not difficult to evaluate the absorptivity of gamma rays in this case by taking into the account that the density of the wall might be 2000 -8000 g/L but the sample might have density only 20 g/L. If the diameter of a pipe is 35 cm and the two walls have 1.5 cm it is understandable that 8000 times 1.5 is 1.7 times more than 20 times 35 ($\frac{8000 \cdot 1.5}{20 \cdot 35} = 1.7$). In this case two additional factors can increase the errors of measurements.

One of them is due to fact that the walls can transfer the range of measurements in to nonlinear area and another one is due to possible variations in the wall thickness. Due to these nonlinearities, one percent variations in the wall thickness can produce much more than 1% systematic error, which can only be partially eliminated when the absorption signal is calibrated to an empty pipe.

There are also many practical problems while working with radioactive substances used in gamma ray detectors. The better precision of measurements necessitates the higher intensity of gamma ray source. The higher is the source intensity the more expensive it is and much more maintenance problem can occur. Only specially trained and certified personnel can work with the sources, they have to be kept in places with high level of security. The personnel have to carry thin film badges-detectors which periodically should be checked to evaluate to what dosage of harmful radiation people were exposed. High intensity sources with the system of protection against radiation are very heavy and require special mechanics for their movement, assembling and installation. Very often lead is used to protect personnel from gamma radiation, but this metal is also considered toxic for human and require some special handling to avoid exposure to it.

Percussion Method

In the paper an attempt is undertaken to define the precision (repeatability) of the PERCUSSION method based on the assumption that it will depend on the significant figures values. “Common convention in science and engineering is to express accuracy and/or precision implicitly by means of significant figures. Here, when not explicitly stated, the margin of error is understood to be one-half the value of the last significant place.” Based on the concept of significant figures the repeatability of the PERCUSSION method was evaluated for the case when electronic hardware is capable to register up to 1 and 0.1 % of the signal. For the first case the repeatability 0.1% and for the second one 0.01% was obtained. From these data the conclusion was drawn that repeatability of the PERCUSSION method is expected to be 0.06 %.

Ideally, in order to determine the accuracy and precision (reproducibility) of the PERCUSSION method one would need to have several reference pipes with certified sample density in the range 20 – 150 g/L (it might be 5 -15 pipes). After that for every pipe 15 - 20 measurements should be done, STD calculated and a calibration curve should be drawn. The STD of measurements for every density can be expressed in g/L values. For example, in the case of linear calibration curve which begin from 0 if the STD of measurements for the 50 g/L sample is 1% the precision of measurements is $50 \pm 0.5 g/L$. In the general case when calibration curve is nonlinear for different concentrations the STD might be different. It needs to be mentioned that for the MDM method it also could be nonlinear. In the regions of considerable nonlinearities it might be necessary to create, design and to attest some additional reference pipes. In the case if the reference pipes are reasonably identical to the pipes which should be measured there is no need to worry about the accuracy of the procedure of measurement. In the theory of nondestructive testing and analytical chemistry the case described above is considered to satisfy the criteria of accurate measurements. If, for example, it is very difficult or impossible to design and create identical reference sample, a more accurate test method should be found to get accurate results. For example, to check the accuracy of PERCUSSION method the gamma ray absorption method can be used if previously it was certified. Sometimes creation of the reference samples (pipes in our case) can be more expensive and time consuming than the development and creation of a device and a methodology of measurements with this device.

In general case, all errors leading to deterioration of the precision of the PERCUSSION method are considered systematic and random. Analyzing data presented to me I could not find the physical process which can lead to systematic error for the PERCUSSION measurements. Typically, a careful design of the experiment can and should eliminate such sort of errors. Assuming the processes leading to the deterioration of the precision of the PERCUSSION method as random and Gaussian, the expression for the error can be as follows

$$E_{Tot}^2 = E_W^2 + E_p^2 + E_M^2 + E_E^2 + E_D^2 \qquad (2)$$

Where E_W - error stipulated by the inaccuracy of PERCUSSION device installation on the wall of a pipe;

E_p - error due a plunger impulse energy variations; E_M - error of the vibrations detector; E_E - noises of the detector electronics; E_D - error of a signal reading device.

In the paper the conclusion about repeatability of PERCUSSION method was drawn taking in to account only last E_D error. This approach can be considered correct only if all other errors are negligible in comparison with this one.

Another piece of tabulated information about standard deviation of the Percussion signal measurements was presented to me. The signal was recorded on the pipe filled with various sample powders with densities 18 -25 g/L.

Test ID	# Measurements	Mean Read Value	Read Value STD	Variation Coefficient, %
1	8	329,5625	4,988	1,513521714
2	8	326,5	5,99	1,834609495
3	6	323,92	3,125	0,964744381
4	8	326,469	3,312	1,014491422
5	5	211,7	1,745	0,824279641
6	8	212,781	1,333	0,626465709
7	8	210,9	0,906	0,429587482
8	8	212,125	2,13	1,004124926

As can be seen the best variation coefficient value is 0.429%, worst - 1.8%. The most probable reason for such a large volatility in data might be the fact that the process of measurement has some influence on the density of the sample. The observations performed with an empty pipe showed that volatility of readings might be 3 -5 times less and correspondingly the precision of the method actually is 3 -5 times better. Based on these data it can be concluded that repeatability 0.06% shown in the paper should be considered potentially achievable if all other errors (the most contributing to the total error might be $E_p ; E_M$ and density variations due to the process of measurement itself) are diminished at least to the level of E_D error. Considerable variations in the errors values (from 1.8 to 0.43) shown in the table is clear indication that there are significant reserves in the PERCUSSION method precision improvement.

Conclusions.

1. Theoretically gamma ray absorption method can provide 0.3 – 0.5 % precision of measurements. However, rather high intensity gamma source has to be used to reach these figures in practice when measuring loose solid materials density in metallic pipes. The precision of measurements can suffer due to the pipe wall thickness variations, due to nonlinearities and because of rather large range of densities (20 -150 g/L) to be measured as well as strong gamma ray absorption by the walls.
2. The Percussion method potentially can provide a 0.1 – 0.01% precision of sample density measurements if, as a result of further development, all other errors are eliminated or become insignificant. The data on the STD measurements show that there are substantial reserves to further improvement of the precision of the Percussion method.

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