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UNCERTAINTY OF MEASUREMENT OF BULK AND SHAKEN DENSITY OF PULGRAN AND SALMAG

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ABSTRACT

The article presents results of research on the bulk and shaken density of two commercial fertilizers: Pulgran urea and universal nitrogen fertilizer Salmag. A statistical analysis of the obtained results of average density at the assumed level of significance proved a significant difference between shaken and bulk density investigated with the use of Engelsmann apparatus and shaken density tested with a laboratory shaker for both investigated fertilizers. The obtained test results and uncertainty of the measured values served for calculation of uncertainty of the standard complex bulk and shaken density determined in case of many uncertainties. Further, an analysis of the impact of error sources on the value of complex uncertainty was conducted. The final results of the measurement were presented according to the convention of the Central Office of Measures.

Introduction

One of the basic principles of precise agriculture is adjustment of the amount and quality of applied fertilizers depending on the soil richness and nutritive needs of cultivated plants (Søgaard and Kierkegaard, 1994; Bovolenta and Pezzi, 1996; Paré et al., 2009; Antille et al., 2013). Popular centrifugal distributors equipped with satellite control systems may ensure required distribution of fertilizers on the field surface if its technological properties preconditioned with physical features are known (Allaire and Parent, 2004; Miserque and Pirard, 2004; Miserque, 2005 ; Paré et al., 2010; Biskupski et al., 2012; Maldaner et al., 2016). Based on the literature data (Schenkel and Miserque, 2005; Scharf, 2009; Przystupa, 2013; Virk et al., 2013) one may conclude that density is one of the most important properties of fertilizers which affect the work of centrifugal distributors. It depends on the fertilizer type, particles size, porosity, and size distribution. Knowledge of the bulk and shaken density of fertilizers is also indispensable when determining the stability of aggregates that consist of the drive unit and suspended distributor. Decisions taken based on unreliable measurements may generate threats for the natural environment (pollution or degradation) and operators of fertilization aggregates and economic losses (Maldaner et al., 2016).

The objective of the paper was to calculate standard uncertainty of the complex bulk and shaken density of Pulgran urea and universal nitrogen fertilizer Salmag based on the test results and uncertainty of the measured sizes. Engelsmann apparatus and adjusted laboratory shaker for determination of the grain composition of loose materials were used.

Materials and methods

Two commercial fertilizers: Pulgran urea produced by Grupa Azoty, Zakłady Azotowe Puławy S.A, and a mixture of nitrogen with the calcium carbonate and magnesium carbonate named Salmag produced by Grupa Azoty, Zakłady Azotowe Kędzierzyn S.A. were the object of research.

A measurement cylinder of Engelsmann apparatus was used for measurement of the bulk density, for which samples of fertilizers collected and prepared according to the standard PN-EN 1236:1999 Fertilizers - Determination of bulk density (loose) and PN-EN 1237:2000 Fertilizers – Determination of bulk density (shaken) were poured through a funnel to the moment a cone was formed over it. A distance between a lower edge of the funnel outlet and the upper edge of the measurement cylinder was 0.05 m. Excess of fertilizer which was over the edge of Engelsmann cylinder was stroked off with a smooth blade. Engelsmann apparatus was used for measurement of shaken density (quivering motion amplitude 0.004 m, frequency 5 Hz, tapping time 4 minutes). Moreover, an adjusted laboratory shaker MULTI-SERW-Morek type LPzE-2e designed for determination of the grain composition of loose materials was used for measurement of shaken density (amplitude of quivering 0.004 m, frequency 30 Hz, shaking time 1 minute). In the place of the set of sieves, a roller made of foamed polystyrene with hollow opening for the measurement cylinder from Engelsmann apparatus was placed. A cover, bottom and straining belts were used for connection of a roller with a shaker system. Bulk density γ_n of fertilizer was calculated with the expression (1):

$$\gamma_n = \frac{m_1 - m_0}{V} \tag{1}$$

where:

m_1 – mass of the measurement cylinder with a sample of fertilizer, kg
m_0 – mass of the measurement cylinder, kg
V – volume of the measurement cylinder, m^3.

Bulk density γ_{us} (with the use of Engelsman apparatus) and shaken density γ_{uw} (with the use of a laboratory shaker) was calculated with the expression (2):

$$\gamma_{us}\,(\gamma_{uw}) = \frac{m_1 - m_0}{V - V_0} \tag{2}$$

Where V_0 stands for a part of the measurement cylinder capacity which is not taken by fertilizer after shaking.

Tests of each type of density were conducted on fifteen samples that were collected in the number of five from three 50-kilo bags of fertilizers that come from the same production batch. A laboratory scales PA4102CM by Ohaus was used for measurement of the mass of the measurement cylinder and the cylinder filled with fertilizer.

Standard complex uncertainty (De, 1989; Lira, 2002; Dorozhovets and Warsza, 2007; Coleman and Steele, 2009; Sousa e Silva et al., 2013) of bulk density $u(\gamma_n)$ of the investigated mineral fertilizers was calculated from the formula (3), and the standard complex uncertainty of shaken density $u(\gamma_{us})$ and $u(\gamma_{uw})$ with the formula (4).

$$u(\gamma_n) = \sqrt{\left(\sigma_{\overline{\gamma_n}}\right)^2 + \left[\frac{\partial \gamma_n}{\partial m_1} u_B(m_1)\right]^2 + \left[\frac{\partial \gamma_n}{\partial m_0} u_B(m_0)\right]^2 + \left[\frac{\partial \gamma_n}{\partial \mathrm{V}} u_B(V)\right]^2} \qquad (3)$$

$$u(\gamma_u) = \sqrt{\left(\sigma_{\overline{\gamma_u}}\right)^2 + \left[\frac{\partial \gamma_u}{\partial m_1} u_B(m_1)\right]^2 + \left[\frac{\partial \gamma_u}{\partial m_0} u_B(m_0)\right]^2 + \left[\frac{\partial \gamma_u}{\partial \mathrm{V}} u_B(V)\right]^2 + \left[\frac{\partial \gamma_u}{\partial \mathrm{V}_0} u_B(V_0)\right]^2} \qquad (4)$$

where:

$\sigma_{\overline{\gamma_n}}$ – standard deviation of average density of γ_n fertilizer calculated with A type method, kg·m^{-3}

$\sigma_{\overline{\gamma_u}}$ – standard deviation of average density of γ_{us} (γ_{uw}) fertilizer calculated with A type method, kg·m^{-3}

$u_B(m_1)$ – standard uncertainty type B of the measurement of the measurement cylinder mass with a sample of fertilizer, kg

$u_B(m_0)$ – standard uncertainty type B of the measurement of the measurement cylinder mass, kg

$u_B(V)$ – standard uncertainty type B of the measurement of the measurement cylinder capacity, m^3

$u_B(V_0)$ – standard uncertainty type B of the measurement of the capacity of the part of the measurement cylinder not taken by fertilizer after shaking, m^3.

Formulas (3) and (4) are based on the uncertainty propagation law (Taylor and Kuyatt, 1994; Taylor, 1997; Arendarski, 2013). This law determines a relation of input and output uncertainties. According to the assumed methodology of research, the input values are values measured: (m_1, m_0, V_0) and values that are in the model: $V, \sigma_{\overline{\gamma_n}}, \sigma_{\overline{\gamma_u}}$ and standard uncertainties resulting from uncertainties of the applied measurement devices and readout of capacities of a part of the measurement cylinder not taken by fertilizer after shaking $[(u_B(m_1), u_B(m_0), u_B(V), u_B(V_0)]$. First three of the mentioned uncertainties were determined based on information on the possible scope of variability of the measured values which were obtained from specification of the applied measurement devices developed by producers. While, uncertainty $u_B(V_0)$ was assumed as a border error of the measurement of the cylinder free space capacity not taken by fertilizer after shaking at the level of the half of the smallest scale.

To check significant differences, the obtained average results of measurement of bulk and tapped density were subjected to statistical analysis. At the assumed level of significance which is α=0.05, the analysis of variance equality with the use of Fisher test and then analysis of equality of average values of the density measurements with t-Student test was performed.

Results and Discussion

Table 1-6 presents budgets of uncertainties of Pulgran and Salmag density measurements. A manner of record of uncertainty of the density measurement was assumed based on the recommendations of the Central Office of Measurements (JCGM, 2008).

Table 1.
Budget of uncertainty of measurement of bulk density of (γ_n) Salmag with the final record of the result

Size	Estimation	Standard uncertainty	Coefficient of the impact	Element of the complex uncertainty
m_1	0.67505 kg	$5.77 \cdot 10^{-6}$ kg	2000 m^{-3}	$1.155 \cdot 10^{-2}$ $kg \cdot m^{-3}$
m_0	0.17138 kg	$5.77 \cdot 10^{-6}$ kg	-2000 m^{-3}	$-1.155 \cdot 10^{-2}$ $kg \cdot m^{-3}$
V	0.0005 m^3	$1.155 \cdot 10^{-6}$ m^3	-2014680 $kg \cdot m^{-6}$	-2.33 $kg \cdot m^{-3}$
$\sigma_{\overline{\gamma_n}}$	8.37 $kg \cdot m^{-3}$	2.16 $kg \cdot m^3$	–	2.16 $kg \cdot m^{-3}$
γ_n	1007.34 $kg \cdot m^{-3}$	–	–	3.18 $kg \cdot m^{-3}$

U(γ_n) = 2u(γ_n) = (1007.34 ± 6.36) $kg \cdot m^{-3}$

Table 2.
Budget of uncertainty of measurement of bulk density of (γ_{us}) Salmag with the final record of the result

Size	Estimation	Standard uncertainty	Coefficient of the impact	Element of complex uncertainty
m_1	0.67505kg	$5.77 \cdot 10^{-6}$ kg	2042.19 m^{-3}	$1.18 \cdot 10^{-2}$ $kg \cdot m^{-3}$
m_0	0.17138 kg	$5.77 \cdot 10^{-6}$ kg	-2042.19 m^{-3}	$-1.18 \cdot 10^{-2}$ $kg \cdot m^{-3}$
V	0.0005 m^3	$1.155 \cdot 10^{-6}$ m^3	-2100579.34 $kg \cdot m^{-6}$	-2.43 $kg \cdot m^{-3}$
V_0	$1.033 \cdot 10^{-5}$ m^3	$1.44 \cdot 10^{-6}$ m^3	2100579.34 $kg \cdot m^{-6}$	3.03 $kg \cdot m^{-3}$
$\sigma_{\overline{\gamma_{us}}}$	8.90 $kg \cdot m^{-3}$	2.3 $kg \cdot m^3$	–	2.30 $kg \cdot m^3$
γ_{us}	1028.59 $kg \cdot m^{-3}$	–	–	4.51 $kg \cdot m^3$

U(γ_{us}) = 2u(γ_{us}) = (1028.59 ± 9.02) $kg \cdot m^{-3}$

Table 3.
Budget of uncertainty of measurement of bulk density of (γ_{uw}) Salmag with the final record of the result

Size	Estimation	Standard uncertainty	Coefficient of the impact	Element of complex uncertainty
m_1	0.67505 kg	$5.77 \cdot 10^{-6}$ kg	2215.66 m^{-3}	$1.28 \cdot 10^{-2}$ $kg \cdot m^{-3}$
m_0	0.17138 kg	$5.77 \cdot 10^{-6}$ kg	-2215.66 m^{-3}	$1.28 \cdot 10^{-2}$ $kg \cdot m^{-3}$
V	0.0005 m^3	$1.155 \cdot 10^{-6}$ m^3	-2472621.72 $kg \cdot m^{-6}$	-2.86 $kg \cdot m^{-3}$
V_0	$4.87 \cdot 10^{-5}$ m^3	$1.44 \cdot 10^{-6}$ m^3	2472621.72 $kg \cdot m^{-6}$	3.56 $kg \cdot m^{-6}$
$\sigma_{\overline{\gamma_{uw}}}$	13.51 $kg \cdot m^{-3}$	3.51 $kg \cdot m^3$	–	3.51 $kg \cdot m^3$
γ_{uw}	1116.11 $kg \cdot m^{-3}$	–	–	5.76 $kg \cdot m^3$

γ_n= (1116.11 ± 11.52) $kg \cdot m^{-3}$

Table 4.
Budget of uncertainty of measurement of bulk density of (γ_n) Pulgran with the final record of the result

Size	Estimation	Standard uncertainty	Coefficient of the impact	Element of complex uncertainty
m_1	0.53081 kg	$5.77 \cdot 10^{-6}$ kg	2000 m^{-3}	$1.155 \cdot 10^{-2}$ $kg \cdot m^{-3}$
m_0	0.17138 kg	$5.77 \cdot 10^{-6}$ kg	-2000 m^{-3}	$-1.15 \cdot 10^{-2}$ $kg \cdot m^{-3}$
V	0.0005 m^3	$1.155 \cdot 10^{-6}$ m^3	-1437720 $kg \cdot m^{-6}$	-1.661 $kg \cdot m^{-3}$
$\sigma_{\overline{\gamma_n}}$	4.592 $kg \cdot m^{-3}$	1.186 $kg \cdot m^3$	–	1.186 $kg \cdot m^{-3}$
γ_n	718.87 $kg \cdot m^{-3}$	–	–	2.04 $kg \cdot m^{-3}$

U(γ_n) = 2u(γ_n) = (718.87 ± 4. 08) $kg \cdot m^{-3}$

Table 5.
Budget of uncertainty of measurement of shaken density of (γ_{us}) Pulgran with the final record of the result

Size	Estimation	Standard uncertainty	Coefficient of the impact	Element of complex uncertainty
m_1	0.53081 kg	$5.77 \cdot 10^{-6}$ kg	2053.39 m^{-3}	$1.184 \cdot 10^{-2}$ $kg \cdot m^{-3}$
m_0	0.17138 kg	$5.77 \cdot 10^{-6}$ kg	-2053.39 m^{-3}	$1.184 \cdot 10^{-2}$ $kg \cdot m^{-3}$
V	0.0005 m^3	$1.155 \cdot 10^{-6}$ m^3	-1515501.6 $kg \cdot m^{-6}$	-1.750 $kg \cdot m^{-3}$
V_0	$1.3 \cdot 10^{-5}$ m^3	$1.44 \cdot 10^{-6}$ m^3	1515501.6 $kg \cdot m^{-6}$	2.182 $kg \cdot m^{-3}$
$\sigma_{\overline{\gamma_{us}}}$	4.796 $kg \cdot m^{-3}$	1.238 $kg \cdot m^3$	–	1.238 $kg \cdot m^3$
γ_{us}	738.06 $kg \cdot m^{-3}$	–	–	3.06 $kg \cdot m^3$

U(γ_{us}) = 2u(γ_{us}) = (738.06 ± 6.12) $kg \cdot m^{-3}$

Table 6.
Budget of uncertainty of measurement of bulk density of (γ_{uw})Pulgran with the final record of the result

Size	Estimation	Standard uncertainty	Coefficient of impact	Element of complex uncertainty
m_1	0.53081 kg	$5.77 \cdot 10^{-6}$ kg	2267.57 m^{-3}	$1.308 \cdot 10^{-2}$ $kg \cdot m^{-3}$
m_0	0.17138 kg	$5.77 \cdot 10^{-6}$ kg	-2267.57 m^{-3}	$1.308 \cdot 10^{-2}$ $kg \cdot m^{-3}$
V	0.0005 m^3	$1.155 \cdot 10^{-6}$ m^3	-1848150 $kg \cdot m^{-6}$	-2.135 $kg \cdot m^{-3}$
V_0	$5.9 \cdot 10^{-5}$ m^3	$1.44 \cdot 10^{-6}$ m^3	1848150 $kg \cdot m^{-6}$	2.661 $kg \cdot m^{-6}$
$\sigma_{\overline{\gamma_{uw}}}$	3.109 $kg \cdot m^{-3}$	0.803 $kg \cdot m^3$	–	0.803 $kg \cdot m^3$
γ_{uw}	815.05 $kg \cdot m^{-3}$	–	–	3.51 $kg \cdot m^3$

γ_n= (815.05 ± 7.02) $kg \cdot m^{-3}$

The statistical analysis of equality of variances obtained with a series of Fisher test did not give basis to reject the hypothesis on the equality of variance. It enabled, in a further part of the statistical analysis, to use t-Student test for assessment of differences between average results of density measurements. This analysis proved statistically significant differences between the average values of measurements of bulk density and shaken density with the use of Engelsmann apparatus as well as shaken density with the use of a laboratory shaker.

Bulk density of new generation urea in the form of white semi-sphere tablets is 718.86 $kg \cdot m^{-3}$. This value constitutes 71.36% of Salmag density which is 1007.34 $kg \cdot m^{-3}$. Shaken density (γ_{us}) measured in Engelsmann apparatus is slightly higher when compared to bulk density. A ratio γ_{us} to γ_n for Pulgran is 1.03, and for Salmag 1.02. According to the studies by Przywara (Przywara, 2012) bulk density of Salmag was 1029 $kg \cdot m^{-3}$ and the shaken one 1062 $kg \cdot m^{-3}$. These differences are considerable and may result from diversification of production batches.

Shaken density γ_{uw} measured with a laboratory shaker LPzE-2e is higher than bulk density by 10% for Salmag and by 13% for Pulgran. This results from restrictive shaking of a fertilizer which considerably reduced the capacity taken by it in the measurement cylinder. Statistical analysis also proved statistically significant differences between average values of the measurement of shaken density γ_{uw}, and shaken density γ_{us} for both investigated fertilizers. For Salmag, the shaken density γ_{uw} is higher for the shaken density γ_{us} by 9%, and for Pulgran by 10%.

From the analysis of the impact of sources of errors on the value of complex uncertainty one may conclude that participation of the first and second element (uncertainty from the measurement of the cylinder mass with a fertilizer and a mass of the cylinder) is scarcely low. Ratio of the highest value of this element of the complex uncertainty ($1.308 \cdot 10^{-2}$ $kg \cdot m^{-3}$) and the lowest value of another element (0.803 $kg \cdot m^{-3}$) does not exceed 0.0163 (tab. 6). The lowest value of this ratio refers to the results of calculations placed in table 3. The ratio of the first element of the complex uncertainty and an element with the highest value does not exceed 0.0036. The highest participation in the complex standard uncertainty of the measurement of density of tested fertilizers is in case of the element $u_B(V_0)$. It is particularly visible on the example of the uncertainty of measurement of shaken density of γ_{uw} Pulgran. The ratio of this component and standard complex uncertainty of density γ_{uw} is almost 0.76.

The analysis of relative extended uncertainties show that they are within 0.57% to 1.03%. The lowest values refer to the uncertainty of measurement of bulk density of both investigated mineral fertilizers (0.57% for Salmag and 0.63% for Pulgran). It is also worth adding that the average relative uncertainty of measurements of Pulgran density is ca. 88% of the average relative extended uncertainty of measurements of Salmag density. It results mainly from a higher degree of uniformity of Pulgran and Salmag grain distribution (UI = 71.09 for Pulgran and UI = 62.52 for Salmag).

Summary

Based on the research and analyses one may make the following conclusions:

1. Bulk density of Pulgran (718.87 kg·m^{-3}) constitutes ca. 71% of the shaken density of Salmag (1007.33 kg·m^{-3}).
2. The statistical analysis at the assumed level of significance of α=0.05 proved statistically significant difference between average results of bulk, shaken density measured in Engelsmann apparatus and shaken density measured with the use of a laboratory shaker.
3. Shaken density of these fertilizers measured with Engelsmann apparatus is slightly higher than bulk density (on average by 2.5%).
4. Restrictive shaking of fertilizers with the use of a laboratory fertilizers with the use of a laboratory shaker influenced considerable increase of bulk shaken density (by 10% for Salmag and by 13% for Pulgran).
5. Shaken density γ_{uw}, reaches higher values from shaken density γ_{us} for Salmag by 9%, and for Pulgran by 10%.
6. Participation of uncertainty from the measurement of the cylinder mass along with fertilizer and the mass of the cylinder itself on the value of complex uncertainty is scarcely low.
7. The highest participation in the standard complex uncertainty of the measurement of density of investigated fertilizers has standard uncertainty of the measurement of capacity of a part of the measurement cylinder not taken by fertilizers after shaking.

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NIEPEWNOŚĆ POMIARU GĘSTOŚCI NASYPOWEJ I UTRZĘSIONEJ PULGRANU I SALMAGU

Streszczenie. W artykule zaprezentowano wyniki badań gęstości nasypowej i utrzęsionej dwóch komercyjnych nawozów: mocznika Pulgranu i uniwersalnego nawozu azotowego Salmagu. Przeprowadzona analiza statystyczna otrzymanych wyników średnich gęstości na założonym poziomie istotności, wykazała istotne różnicę między gęstością utrzęsioną oraz nasypową badaną przy zastosowaniu aparatu Engelsmana, jak również gęstością utrzęsioną badaną z wykorzystaniem wytrząsarki laboratoryjnej dla obu badanych nawozów. Uzyskane wyniki badań oraz niepewności mierzonych wielkości posłużyły do obliczenia niepewność standardowej złożonej gęstości nasypowej i utrzęsionej wyznaczanej w przypadku występowania wielu niepewności. W dalszej części pracy przeprowadzono analizę wpływu źródeł błędów na wartość niepewności złożonej. Końcowe wyniki pomiaru przedstawiono według konwencji Głównego Urzędu Miar.

Słowa kluczowe: niepewność standardowa złożona, gęstość nasypowa, gęstość utrzęsiona