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ON SAMPLING ACCURACY OF GRAINED MATERIAL MEASUREMENTS

Summary. It is a basic purpose of the paper to present a probabilistic description of grain material sampling as a process inherent in identification and control process of many industrial processes. The so-called sampling error of a grain composition of a material has been presented in the paper. The mass fractions of different types of grains in a sample are random variables. The formulas of distribution functions of the variables have been determined on the basis of random schemes. The variances and variation coefficients of selected grain fraction content in a sample have been used as criteria of sampling error. Some experimental and simulation results are presented.

1. INTRODUCTION

Testing granular materials, including mineral deposits such as coal and metal ores, is a very specific field of knowledge due to a variety of physical and chemical properties of particular grains making up a parent population. The physical properties, such as the diameter and density of a single grain, metal content in ore grains, ash content, sulphur content or humidity in given coal grains, in view of the observations of the whole mass of a material, should be regarded as random variables.

With regard to grain materials characteristic useful for systems of automation of particular processes applied in industry the following characteristics are basic characteristics of material utilised by process engineers and automation engineers:

- a characteristic of the grain composition presenting portions of grains belonging to different size fractions, where grain diameters d are contained within certain disjoint intervals: $d_i \in (d_{i,min}, d_{i,max})$, $i=1,2,\ldots,l$;
- a densimetric characteristic presenting portions of grains belonging to different fractions where densities of grains δ are contained within certain disjoint intervals: $\delta_j \in (\delta_{j,min}, \delta_{j,max})$, $j=1,2,\ldots,r$.

- characteristic of components content such as e.g. metal content in total mass of ore material, content of useless components such as ash and sulphur in coal, content of clean coal substance and other.

Nowadays available modern technical equipment makes it possible to designate the examined characteristics of granular materials, to test materials susceptibility to flotation reagents and magnetic field impact etc. Moreover, such equipment is furnished with devices for digital processing of measurement results and their graphic representation and print. A common feature of modern engineering equipment is a small mass of the material tested in a unitary measurement. The estimation of the unknown material characteristics that comprise the parent population of grains, on the basis of the measurement of a small sample, may entail an error, the extent of which shall be discussed in the paper.

2. THEORETICAL ASPECTS OF SAMPLING ACCURACY OF GRAIN MATERIALS

2.1. Sampling errors of a single fraction

Sample tests on heterogeneous granular materials involve selecting certain elements out of the population consisting of various types of grains. In the theoretical analysis included in this research work it has been assumed that the size of the population subjected to sampling far exceeds the number of sampled elements. Furthermore, it has been assumed that the granular material is well mixed, so that the values of probability p_{ij} of the occurrence of a grain with properties $(d_i\ \delta_j)$ are the same in different parts of the granular material, irrespective of the samples taken.

In view of the above assumptions, the selection may be regarded as an independent lot. The number of grains in the selected fraction is a random variable $X=k$, where $k= 0, 1, 2, ...n$ and n denotes the sample size. Thus, the frequency rate of a certain type of grain $Y=k/n$, as well as mass fraction $\gamma=ak/n$ (where a represents the coefficient depends on mean values of diameter and density of grain in a sample) are random variables [2].
Each of the random variables that determine the number of grains in the selected fraction in the sample of a constant number of grains n, is subject to binomial distribution:

$$P(X=k)=\binom{n}{k}p^k(1-p)^{n-k} \qquad (1)$$

where p is a probability of the occurrence of a defined type of grain in a parent population.
Additionally, if a sample size n and a are assumed as constant the probability distribution of a portion of chosen grains is equal to:

$$P\left(\gamma=a\frac{k}{n}\right)=P\left(Y=\frac{k}{n}\right)=P(X=k) \qquad (2)$$

The expected values and variances are:

$$E(X)=np, \quad D^2(X)=np(1-p) \tag{3}$$

$$E(Y)=p, \quad D^2(Y)=p(1-p)/n \tag{4}$$

$$E(\gamma)=ap, \qquad D^2(\gamma)=\frac{a^2p(1-p)}{n} \tag{5}$$

The coefficient of variation, expressed as the quotient of standard variation and mean value, is the same for all variables (i.e. for random variable X, Y and γ)

$$\varepsilon(X)=\varepsilon(Y)=\varepsilon(\gamma)=\sqrt{\frac{1-p}{np}} \tag{6}$$

and depends on the sample size n and the probability p of the occurrence a single chosen grain in a parent population

2. 2. Sampling errors while estimating the granular characteristics or densimetric characteristics

In the material subjected to sampling the number of grains in particular grain size grades constitutes a multidimensional random variable $(X_1, X_2, ..., X_J)$ that may take any combination of values:

$$(X_1=n_1, X_2=n_2, ..., X_J=n_J) \tag{7}$$

where $n_1+n_2+...+n_J=n$. The probability distribution of multidimensional random variable (7), with the reservation of the selection being independent, is determined by means of the following polynomial distribution:

$$P(X_1=n_1, X_2=n_2, ..., X_J=n_J)=\frac{n!\,p_1^{n_1}p_2^{n_2}\dots p_J^{n_J}}{n_1!\,n_2!\dots n_J!} \tag{8}$$

Numbers $p_1, p_2, ... p_J$ are the respective boundary probabilities of selecting a single grain with given diameter (or density) out of the granular material subjected to sampling.

The frequency rates of grains belonging to the established size grades in the tested sample containing n-elements also constitute multidimensional random variable $(Y_1, Y_2, ..., Y_J)$, with following values:

$$(Y_1=\frac{n_1}{n}, Y_2=\frac{n_2}{n}, ..., Y_J=\frac{n_J}{n}) \tag{9}$$

subjected to the polynomial distribution, expressed by (8) when $n=const$.

The variance-covariance matrix of multidimensional random variable (7), representing the numbers of grains in all the size grades of the sample consisting of n-elements, has the following form:

$$\mathbf{D}^2_{\mathbf{x}} = n\,\Phi, \tag{10}$$

where Φ is the following matrix of dimensions JxJ:

$$\begin{bmatrix} p_1(1-p_1) & -p_1p_2 \cdots & -p_1p_j \cdots & -p_1p_J \\ -p_2p_1 & p_2(1-p_2)\cdots & -p_2p_j\cdots & -p_2p_J \\ \cdots & \cdots & \cdots & \cdots \\ -p_jp_1 & -p_jp_2 \cdots & p_j(1-p_j)\cdots & -p_jp_J \\ \cdots & \cdots & \cdots & \cdots \\ -p_Jp_1 & -p_Jp_2 \cdots & -p_Jp_j \cdots & p_J(1-p_J) \end{bmatrix} = \Phi \tag{11}$$

It shows that numbers of grains in different fractions in the same sample are correlated random variables.

Conversely, the variance-covariance matrix of multidimensional random variable (9), representing the frequency rate of grains belonging to particular grades in the sample consisting of n-elements is:

$$\mathbf{D}^2_{\mathbf{Y}} = \Phi / n \tag{12}$$

Vector e of the estimation error of granular material characteristics $(p_1, p_2, \ldots, p_J)$ by sample characteristics (9), equal to the difference between the two vectors, is a multidimensional random variable. To measure the accuracy of the approximation a square of the random error vector norm may be applied:

$$\mathbf{e}^{\mathrm{T}}\mathbf{e} = \left(p_1 - \frac{n_1}{n}\right)^2 + \left(p_2 - \frac{n_2}{n}\right)^2 + \ldots + \left(p_J - \frac{n_J}{n}\right)^2 \tag{13}$$

or the expected value of this random vector:

$$E\{e^Te\} = \sum_r \left[\left(p_1 - \frac{n_1}{n}\right)^2 + \left(p_2 - \frac{n_2}{n}\right)^2 + \ldots + \left(p_J - \frac{n_J}{n}\right)^2\right] \frac{n!\,p_1^{n_1} p_2^{n_2} \ldots p_J^{n_J}}{n_1!\,n_2! \ldots n_J!} \tag{14}$$

where the summation pertains to all the combinations of numbers $n_1, n_2, \ldots n_J$ at constant size of sample n. In equation (14) the components of deviation vector e, in reference to the assumed characteristics, constitute simultaneous events, occurring with the probability determined by polynomial distribution (8).

3. EXEMPLARY RESULTS OF MEASUREMENTS AND SIMULATION TESTS

To examine the nature and range of changes in the granulometric characteristics of samples, multiple measurements of fine-granular materials such as magnesia and quartz were taken [2]. The measurements were made by means of diffraction laser analyser. The content

(percentage) of 31 grain size grades in the range of 0.9 to 200μm was measured in each sample. On the grounds of the measurement series of the characteristics in the samples, mean values, variances and variability coefficients (relative error, %) of the content of particular grain grades were calculated. The results for 50 quartz samples are presented in Table 1. The mean percentages of particular grades are the estimators of the granular characteristics of a given material. The spread of the results for particular variables is different, with the relative error reaching even 90 %.
Figures 1 – 3 present the bar charts of the percentage of certain grain grades, where horizontal axis (p) is the percentage of the selected grade, whereas vertical axis (n) is the number of the occurrence of given value *p* in 50 measurements. The nature of the empirical distributions for particular grain grades is diversified. The distributions are not symmetrical, but often irregular.

The measurement results clearly confirm a random nature of sampling tests and diverse range of their variability.

The subject of the simulation tests was the shape of polynominal distribution (8) and the estimation error of granular characteristics for given values of the material characteristics (9) and different values of *n*, determining the number of grains in a sample.
The estimation errors of given material characteristics for the initial range of changes in the number of grains in samples, calculated on the grounds of expression (14), are presented in Fig. 4,5. The calculations were made for the samples with the number of grains from 1 to 450. The conclusion is that the estimation error of the unknown material characteristics by sample characteristics decreases in inverse proportion to the sample size *n* (similarly, as in the case of a single fraction – expressions (4) and (5)), but increases together with increasing of the number of the designated fractions.

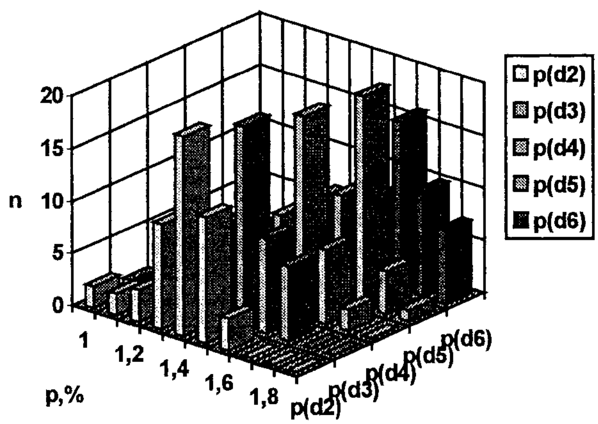

Fig.1. Empirical distributions (for 50 measurements) of grain size fractions i=2,3,4,5,6.

Tab.1. The grain size sample characteristics as the result for 50 quartz samples measurements

Grain size fraction number	*Grain diameter*	*Mean value of fraction contents*	*Variance*	*Relative error*
i	**d_i**, μm	**$E(p_i)$**, %	**$D^2(p_i)$**, [%]^2	$\varepsilon(p_i)$, %
1	0.9 - 1.2	12.712	2.81	13.2
2	1.2 - 1.3	1.376	0.0626	18.2
3	1.3 - 1.5	1.448	0.0213	10.1
4	1.5 - 1.7	1.528	0.0257	10.5
5	1.7 - 2.0	1.604	0.0257	9.99
6	2.0 - 2.3	1.678	0.0275	9.87
7	2.3 - 2.6	1.754	0.0319	10.2
8	2.6 - 3.0	1.960	0.0547	11.9
9	3.0 - 3.4	2.176	0.0557	10.8
10	3.4 - 3.9	2.478	0.0679	10.5
11	3.9 - 4.5	2.702	0.0843	10.75
12	4.5 - 5.2	3.114	0.0685	8.44
13	5.2 - 5.9	3.334	0.3566	17.91
14	5.9 - 6.8	3.636	0.0852	8.03
15	6.8 - 7.8	3.911	0.1219	8.92
16	7.8 - 8.9	4.05	0.0995	7.79
17	8.9 - 10.2	4.136	0.0751	6.625
18	10.2 - 11.7	4.188	0.0692	6.283
19	11.7 - 13.4	4.468	0.0879	6.636
20	13.4 - 15.4	4.402	0.0997	7.174
21	15.4 - 17.6	4.330	0.2046	10.45
22	17.6 - 20.2	4.269	0.2232	11.07
23	20.2 - 23.2	4.214	0.2596	12.09
24	23.2 - 26.8	3.792	1.1881	28.7
25	26.8 - 31.3	3.232	0.5753	23.47
26	31.3 - 36.7	3.484	1.2812	32.49
27	36.7 - 43.8	3.634	0.5992	20.76
28	43.8 - 53.1	3.220	0.8212	28.14
29	53.1 - 66.6	1.626	0.7776	54.23
30	66.6 - 89.1	0.804	0.5265	90.2
31	89.1 - 131.9	0.600	0.2711	86.8

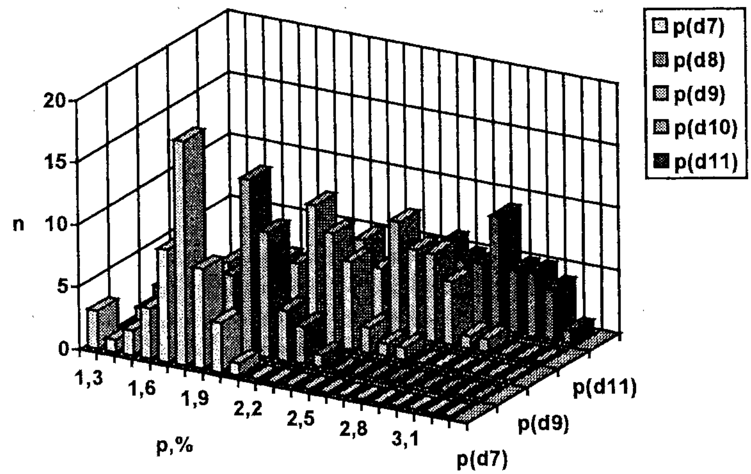

Fig.2. Empirical distributions (for 50 measurements) of grain size fractions i=7-11.

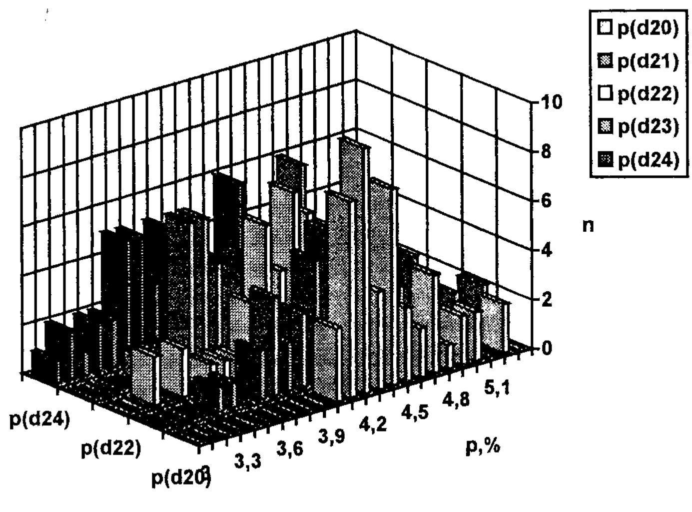

Fig.3. Empirical distributions (for 50 measurements) of grain size fractions i=20-24.

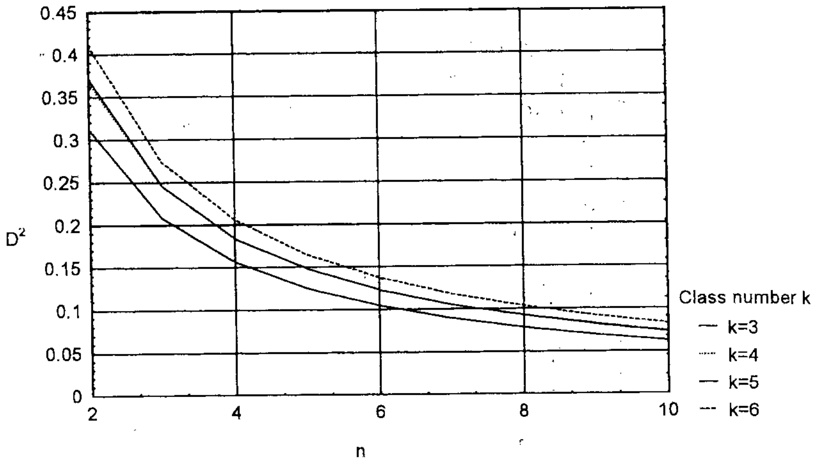

Fig.4. Error of estimation of grain size characteristics; simulation tests, n=10.

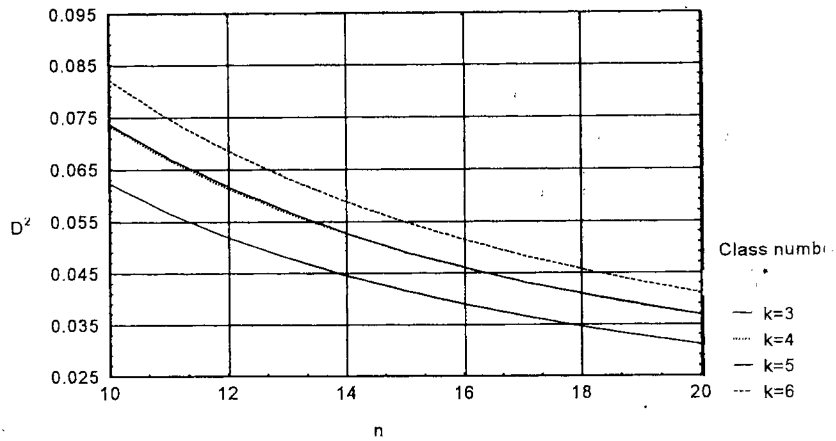

Fig.5. Error of estimation of grain size characteristics; simulation tests, n=20.

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