



Article

Estimation of Gas Loss in Methodology for Determining Methane Content of Coal Seams

Nikodem Szlázak, Dariusz Obracaj *  and Marek Korzec * 

Faculty of Mining and Geoengineering, AGH University of Science and Technology, Mickiewicza 30 av., 30-059 Kraków, Poland; szlajak@agh.edu.pl

* Correspondence: obracaj@agh.edu.pl (D.O.); mkorzec@agh.edu.pl (M.K.)

Abstract: The determination of natural gas content in coal seams is essential to safe mining activities and to estimate gas resources. It is used to estimate methane emissions into workings, which is very important when using preventive measures of methane ignition and explosion. Methane content in a coal seam is mostly determined using a direct method. However, a problem with estimation of gas loss during collecting a sample in a mine occurs in each method. That gas loss is estimated using different procedures. In the Polish mining industry, methane content in a coal seam is mostly determined during roadway advancing faces in coal seams. The differences in procedures for determining methane content in coal in various laboratories gave rise to starting research into developing a new method. This article presents results of study of gas loss unification for the determination of methane content in coal seams. The research focused mainly on determining a uniform method for estimating gas loss using the early adsorption diffusion process. The method of coal sample saturation with methane was used to measure gas desorption rate. Saturation pressures of samples were determined for coal seams in the Upper-Silesian Coal Basin. The analysis of desorption results allowed to determine the dependence between methane content in a coal sample and the initial methane content in a coal seam. It was concluded that the desorption rate was highest within two minutes. For the procedure of coal sampling, gas loss was determined at 12% of both desorbed and residual gases. The presented research results gave rise to developing a uniform method for determining gas content used in Polish coal mining. The method was accepted as the national standard. Methodology using a new gas loss estimation procedure allows for achieving relatively fast results of the determination of methane content in coal seams, which is very important, especially in gassy mines.

Keywords: coal; methane content of coal; determination methane content; gas loss



Citation: Szlázak, N.; Obracaj, D.; Korzec, M. Estimation of Gas Loss in Methodology for Determining Methane Content of Coal Seams. *Energies* **2021**, *14*, 982. <https://doi.org/10.3390/en14040982>

Academic Editor: Manoj Khandelwal

Received: 20 January 2021

Accepted: 10 February 2021

Published: 13 February 2021

Publisher's Note: MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.



Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

1. Introduction

Coal, which is a sedimentary rock of plant origin, is a valuable energy resource. It depends on coal rank containing from 60% carbon in lignite to more than 90% in anthracite [1]. A characteristic feature of coal deposits is their variable saturation with gases, in particular with methane. Methane occurs in coal seams primarily by sorption in the pores of the coal matrix or on the surface of cleat faces. Secondly, it can occur in free form filling fissures and fractures.

The knowledge of natural gas content in coal is essential to mining activities, especially in underground mines. It is used to estimate the amount of methane emitted into workings, which is very important as far as prevention against ignition and explosion of methane is concerned. Additionally, methane is an efficient energy source.

Many methods for determining gas content in a coal seam have been developed in the world mining industry. Depending on the way of determining methane content in coal seams, each method can be classified as [1,2]:

- direct method—it involves collecting coal samples in a mine and measuring the amount of gas released,

- indirect method—it relies on correlations, equations, and laboratory isotherms to estimate gas content in coal.

Methane content in a coal seam is mostly determined using a direct method, which is more accurate than an indirect method [1–3].

Some of the most popular methods used worldwide include: Bertard's Method developed in France [4], Smith's and Williams Method [5,6], the USBM Direct Method (Bureau of Mines in the Department of the Interior of the United States, Washington, DC, USA) [7], the Modified USBM Method (MDM—the Modified Direct Method) [8–10], the GRI Method (Gas Research Institute, Chicago, IL, USA) [11–13], the Australian Standard [14,15], and the CSIRO-CET method [16]. Some of these methods are still being developed. Zhang et al. [17] used the neural network model method to predict gas content based on structural complexity. Dong Li et al. [18] have used support vector machines and neural network inversion methods for the 3D volume prediction of gas content in a coal seam.

The procedure of determining gas content in a coal seam according to direct methods can be divided into activities connected with drilling boreholes (utilizing cutting drillings or core), collecting samples, measuring emitted gas, and finally crushing coal samples. During these operations, gas is released from the sample. The total gas contained in coal is the sum of three components [6,19]:

$$Q_{tot} = Q_{loss} + Q_{des} + Q_{res}, \quad (1)$$

where Q_{tot} is total gas content, m^3/t ; Q_{loss} is gas loss (free gas and desorbed gas during a sample collection), m^3/t ; Q_{des} is desorbed gas content, m^3/t ; and Q_{res} is residual gas content, m^3/t .

The direct method of measurement does not allow the determination of the gas loss component. Gas loss is released from coal during drilling and other activities since a coal sample is sealed in an airtight canister once collected. There is no technical possibility to measure this component. It is only possible to measure desorbed Q_{des} and residual gas Q_{res} . In slow desorption methods, desorbed gas and residual gas can be measured separately. The main disadvantage of slow desorption methods is that it takes a long time to obtain measurement results. Therefore, these methods are not widely used in mines. In fast desorption methods, when a coal sample is crushed before measuring desorbed gas, desorbed and residual gases are measured together. However, a problem with measuring gas loss still remains.

The first Polish direct method for determining methane content in coal was developed in the 1950s. That method was based on the Peters and Wernceki's method [20], which was applied in the 30s of 20th century in the German mining industry. The method was modified by scientific institutes and the coal mining industry standardized the methodology of collecting coal samples in mine excavations. However, laboratories and research institutes still estimated gas loss according to their individual procedures. The inspiration for conducting comprehensive tests was the results of comparative research into gas content in coal in various laboratories and institutes carried out by Ryszka and Sporysz [21]. The results showed different results for the same samples of coal. The biggest differences in methodologies for determining methane content in coal were from using various procedures for estimating gas loss. That was the basis for starting research into unifying the procedure of determining methane content in a coal seam.

Many researchers who estimate coal seam gas content for particular coal basin conditions or even for individual mines have been trying to solve the problem of gas loss [1,3,10,22]. Coal is a material with a complicated porous structure with enhanced sorption properties, especially for gases. These properties have a great influence on coal extraction and mineral processing methods. Bertard et al. [2] showed in their work that gas loss could be predicted based on the results of methane desorption from coal. They found that gas loss could be estimated by relating it to the square root of time since the desorption process started. Other scientists further considered the concept for estimating gas losses when sampling coal. McCulloch et al. [13] proposed cumulative desorption referenced to

the square root of time. Ulery and Hyman [8] modified the method of McCulloch et al. by relating the desorption studies to standard conditions of temperature and pressure. In turn, Sagafi et al. [16] proved that the initial desorption of gas from coal is the exponential function in time. Whereas Chen et al. [23] stated that the fit of the gas prediction function with gas desorption curves should be tested not to make significant errors in gas loss estimation. Wang et al. [24] reported that sampling time should be up to 5 min based on coal research from the Lubanshan North Mine (LBS) in Sichuan Province. Liu [25] found that gas loss time was 2 and 5 min and the cumulative gas loss increased with an increase in equilibrium pressure. Xu et al. [26] developed a nonlinear curve-fit for measured gas loss and total gas content by best fitting the experimental data using additional residual gas as part of the total desorbed gas.

As it can be observed on the basis of the studies above, gas losses are related to the initial desorption process. The relation of free gas to the total gas is only 2–3%, therefore, mainly desorbed gas is released from a coal seam during mining activities [27]. It is essential to take into account adsorption and desorption mentioned above while establishing methodology for determining methane content in coal. The knowledge about the adsorption process allows to establish methane content in coal. In turn, knowledge about the desorption process can be useful when establishing the amount of desorbed methane from coal. Chen et al. [23] confirmed that gas content calculation methods based on gas desorption studies usually give reliable results. Zhigen et al. [22] presented the pressure adsorption curve method for predicting the theoretical amount of methane adsorption and the free amount of methane based on the example of coal from one of the mines in the Sunan mining area. There are also known models of predicting sorption isotherms depending on both variable pressure and variable temperature [28,29].

It can be concluded from the review above that the majority of these studies focus on the determination of the fit of the gas desorption curve run function. However, each desorption test for coal samples is time-consuming; therefore, the generalization of the initial desorption test results by determining the relative proportion of gas lost during sampling was advisable for developing a standardized method for determining methane content in a coal seam.

Therefore, to develop further a universal method in Polish mining, we conducted our research to determine the dependence between methane content in a coal sample and initial methane content in a coal seam.

2. Materials and Methods

2.1. Coal Samples

Coal samples for determining gas losses were collected in mine roadways, which have been driven in coal seams. Polish regulations [30] specify that samples for determining methane content of coal should be collected:

- in test boreholes drilled to assess coal seams, every 100 m in a borehole;
- in shafts (blind shafts) and roadway advancing faces while crossing coal seams whose thickness exceeds 0.4 m;
- in front of roadway advancing faces, at the minimum distance of 200 m in the plane of a coal seam.

Additionally, at the minimum distance of 25 m from faults or other geological dislocations, which may result in an increasing in methane content.

The most significant number of determined methane content in coal is performed in Polish coal mines by means of roadway advancing faces. According to the Polish mining law & regulations, methane content in coal has to be determined using the direct method. However, the regulation does not specify the methodology of measurements.

Coal samples for planned measurements were collected in various coal mines in Upper Silesian Coal Basin. Polish coal mines operate primarily in the Upper Silesian Coal Basin (USCB). The USCB is located in southern Poland, and it is the largest coal basin in Poland. USCB is characterized by diversity and various methane content in coal

seam occurrences in different parts of the basin. The distribution of methane content in coal seams was confirmed by Kędzior [31]. There are coal seams without impermeable overburden containing only trace amounts of methane. There are also coal seams with high methane contents underneath the overlying impermeable thick siltstone or claystone layers.

During the initial stage of research, physical parameters of various kinds of coal and their influence on methane content determination were analysed. They came from different coal seams and different mining levels. Relationships between these parameters were conducted for 190 samples. The range of examined parameters in samples is presented in Table 1. The analyses of results showed that coal seams' methane content is not correlated to the ash content, water content, volatile matter, Protodyakonov coefficient of coal strength [32,33].

Table 1. Variation of selected physical parameters for coal samples.

Parameter	Minimum	Maximum	Average	Standard Deviation
Methane content in coal seams, $\text{m}^3/\text{t}^{\text{daf}}$	0.00	14.15	4.08	2.89
Protodyakonov coefficient of coal strength, -	0.28	1.72	0.79	0.33
Volatile matter, %	22.20	44.10	31.92	4.72
Moisture content, %	0.91	12.89	3.74	1.90
Ash content, %	0.43	36.50	7.81	5.53

The test samples for sorption measurements were carried out and collected in various coal mines of the Upper Silesian Coal Basin. They came from different coal seams and different mining levels. Twenty-eight coal samples were selected from 25 coal seams from 9 coal mines. A wide range of methane content characterized coal samples. The results of sorption measurements are presented in the next section of this article.

2.2. The Procedure for Gas Loss Estimation

In this study, gas loss related to coal sampling was determined on the basis of methane sorption and desorption tests of coal samples carried out in laboratory conditions with the use of the gravimetric microbalance IGA-001 (Intelligent Gravimetric Analyser). The coal samples previously saturated with methane were subjected to the desorption process. The coal sorption curves for methane were approximated by Langmuir sorption isotherm equations [34]. All the sorption curves are presented in [32,33].

The same samples of drill cuttings as the ones for the determination of methane content were used to examine sorption isotherms. The samples were dried in 25 °C for 8 h and then sieved below 500 μm . The samples weighing 0.1 g were put into the gravimetric microbalance IGA-001, in order to saturate them with methane at pressure p_{sat} of 0.5 MPa, 1.0 MPa and 1.5 MPa respectively. Eighty-four coal samples were prepared for sorption tests. Saturation pressures of samples were determined for coal seams in USCB. The examinations of methane pressure in the strata showed methane pressure in the range of 1.2–12.3 bar (0.12–1.23 MPa) [35,36]. The upper-pressure range's value also refers to the safety limit against the outburst of gases and rocks in USCB mines.

Figure 1 shows the variability of sample saturation with methane M_{sat} (initial methane content) for each of the pressures p_{sat} . It was assumed that methane saturation in coal corresponds to in-situ $M_{\text{sat}} = M$, i.e., before the moment of taking a coal sample for gas content tests.

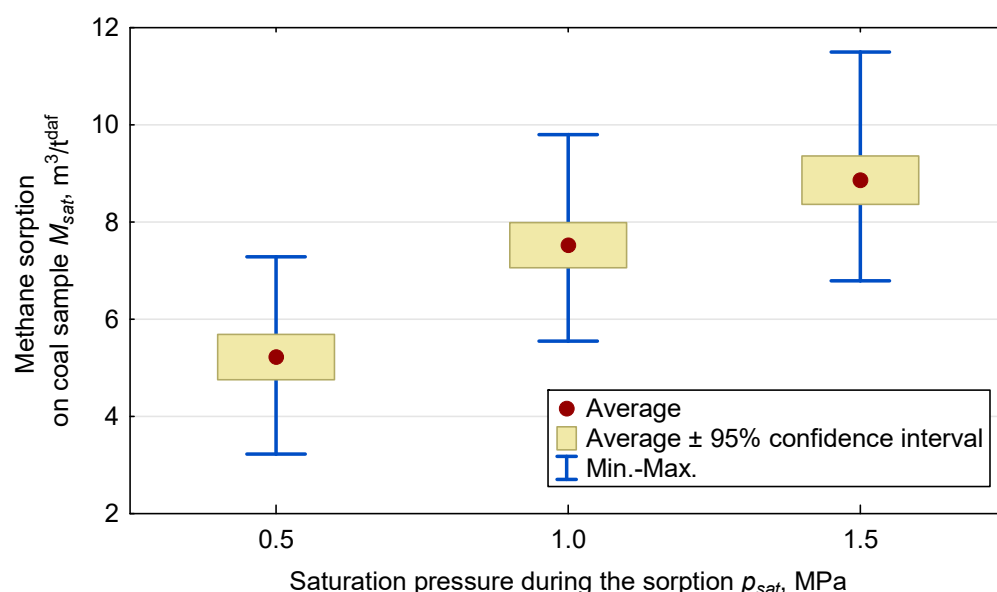


Figure 1. Methane sorption on coal samples M_{sat} for different saturation pressures p_{sat} .

For each of methane saturated coal samples, the pressure in gravimetric microbalance IGA-001 was reduced to 0.1 MPa. Then the process of methane desorption from coal samples was analysed. Particular attention was paid to the first 10 min of the desorption process.

3. Results and Discussion

During the desorption analysis, attention was paid to the initial period when the dynamics of the process was more intensive. Desorption curves for 3 example samples and three analysed values of saturation pressure are presented in Figure 2. The results of all performed examinations for all the tested samples are presented in works [32,33]. The analysis of all the curves and procedures of the collected samples in mines shows that gas loss should be estimated for the first two minutes. Within two minutes, all activities should be finished from the beginning of the sample collection to its closing in an airtight steel canister. The methane content determined in the laboratory is lower than the initial methane content in coal which can be expressed as:

$$M_{lab(2)} = M - M_{loss} \quad (2)$$

where, $M_{lab(2)}$ is methane content in a coal sample determined in the laboratory, m^3/t_{daf} ; M is methane content in a coal seam, m^3/t_{daf} ; and M_{loss} is gas loss during collecting the sample in a mine, m^3/t_{daf} .

The preliminary analysis of gas loss results for all the analysed coal samples shows that gas loss in the second minute changed in a range of 1.79% to 22.99%. The relationship between gas loss in the second minute of desorption $M_{loss\ des}$ and the initial methane content in coal samples M_{sat} is presented in Figure 3.

On the basis of the graph presented, it cannot be concluded explicitly what gas loss in the second minute after beginning methane desorption occurs. There is a much greater variability in losses at lower pressure of saturation. The range of gas loss also results from changeable parameters of coal samples, presented in Table 1. In order to unify research procedures, a decision was taken to analyse the curves of methane desorption from particular coal samples.

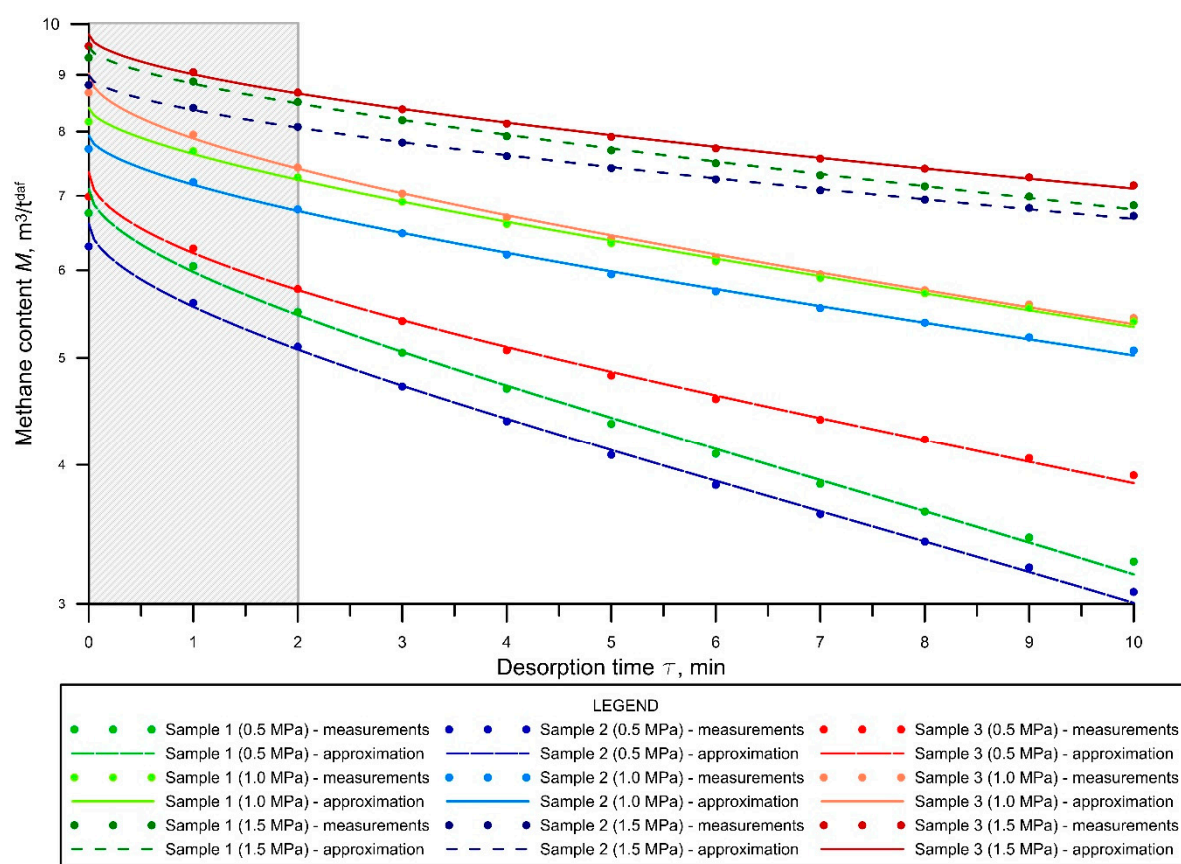


Figure 2. Examples of curves fitted for the desorption results for three samples saturated by methane at pressures 0.5 MPa, 1.0 MPa and 1.5 MPa, respectively.

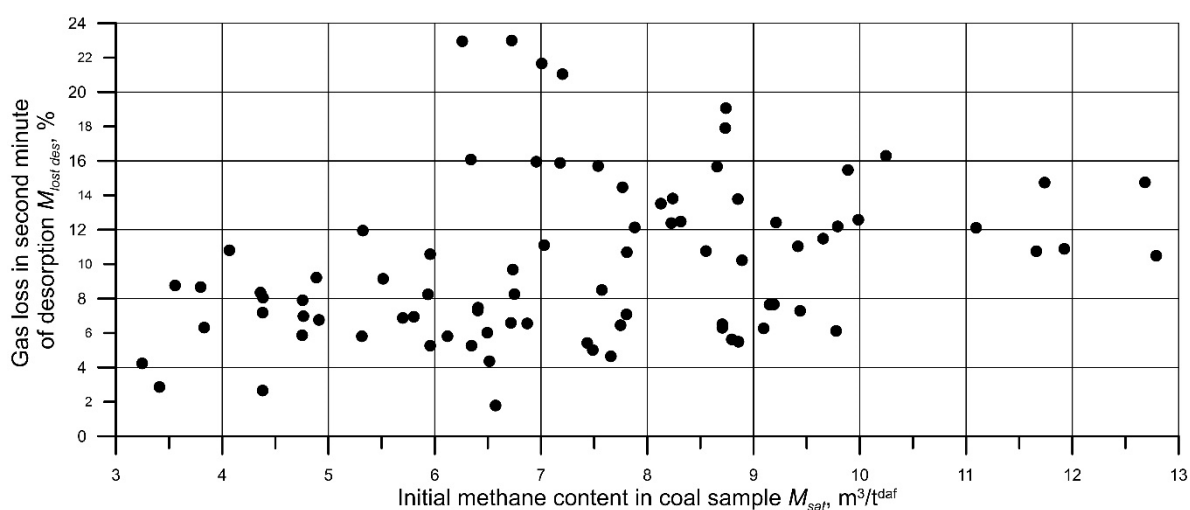


Figure 3. Variation of gas loss $M_{\text{loss des}}$ ranges over initial methane content in tested coal samples M_{sat} .

Methane content in the sample was analysed in the second minute of desorption for all the analysed coal samples to determine gas loss. The approximation of the test results for 3 exemplary desorption runs is shown in Figure 2. The nonlinear least-squares estimation method was applied using the Levenberg-Marquardt algorithm. The conducted

analyses showed that the best fit for the obtained desorption courses was obtained using the following empirical equation:

$$M_{lab(2)} = M - a \cdot (\tau^n) \quad (3)$$

where, a is co-efficient characteristic of coal and reflecting initial desorption rate within $\tau = 2$ min. and exponent n is a coefficient dependent on desorption dynamics.

Co-efficient a and coefficient n may be changeable depending on a kind of coal. Equation (3) was fitted to the obtained results of methane desorption in the second minute.

Table 2 summarizes the parameters for fitting the Equation (3) to the desorption curves for the three exemplary coal samples, which were checked with three saturation pressure values. The values of the lower and upper confidence limits given in Table 2 refer to 95% probability level. The high percentage of the explained variance in the regression model was obtained for each of the desorption series. The results of the analyses for all the coal samples are presented in publications [32,33].

Table 2. Results of fitting Equation (3) to desorption runs for three exemplary coal samples saturated by methane at three different pressures.

Sample Number	Saturation Pressure, MPa	Parameter acc. to Equation (3)	Fitted Value	Standard Error	Lower Confidence Limit	Upper Confidence Limit	Coefficient of Determination
1	0.5	$M=$	7.099	0.021	7.058	7.139	0.9964
		$a=$	1.120	0.019	1.082	1.158	
		$n=$	0.543	0.006	0.532	0.554	
	1.0	$M=$	8.400	0.014	8.372	8.428	0.9969
		$a=$	0.764	0.013	0.738	0.789	
		$n=$	0.604	0.006	0.592	0.615	
	1.5	$M=$	9.534	0.012	9.510	9.558	0.9970
		$a=$	0.698	0.011	0.677	0.720	
		$n=$	0.592	0.005	0.581	0.602	
2	0.5	$M=$	6.606	0.017	6.574	6.639	0.9974
		$a=$	1.045	0.015	1.015	1.075	
		$n=$	0.537	0.005	0.528	0.547	
	1.0	$M=$	7.933	0.012	7.910	7.957	0.9975
		$a=$	0.769	0.011	0.747	0.791	
		$n=$	0.577	0.005	0.568	0.587	
	1.5	$M=$	8.985	0.009	8.967	9.004	0.9976
		$a=$	0.618	0.009	0.601	0.635	
		$n=$	0.573	0.005	0.564	0.582	
3	0.5	$M=$	7.352	0.020	7.312	7.391	0.9968
		$a=$	1.134	0.019	1.097	1.172	
		$n=$	0.489	0.005	0.479	0.500	
	1.0	$M=$	9.024	0.020	8.985	9.063	0.9969
		$a=$	1.136	0.019	1.099	1.173	
		$n=$	0.508	0.005	0.498	0.518	
	1.5	$M=$	9.783	0.012	9.759	9.808	0.9974
		$a=$	0.773	0.011	0.751	0.796	
		$n=$	0.539	0.005	0.530	0.549	

Variation of co-efficient a in function $M_{lab(2)}$ is presented in Figure 4. Co-efficient a was approximated by straight line for all the samples.

The results presented in Figure 4 show that the value of coefficient a depends on methane content in coal samples in the second-minute of desorption time $M_{lab(2)}$. When using linear regression, the slope of linear function equal to 0.085 was determined. Correlation coefficient for estimating that value reaches 0.704. The estimated value of coefficient a may be presented by linear function $a = 0.085 \cdot M_{lab(2)}$.

Figure 5 shows the relationship between coefficient n and methane content. The estimation of coefficient n in equation (Equation (3)) shows that value n is 0.5. The value

of constant n does not depend on methane content in a coal sample in the second minute, which Table 2 and Figure 5 confirm.

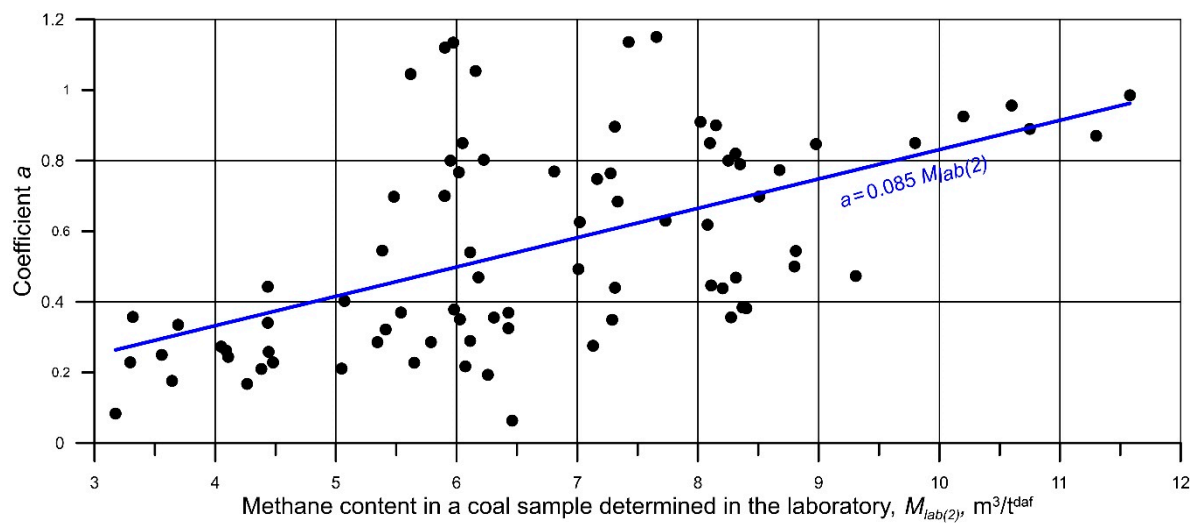


Figure 4. Relationship between coefficient a and methane content in coal samples in second minute of desorption $M_{lab(2)}$.

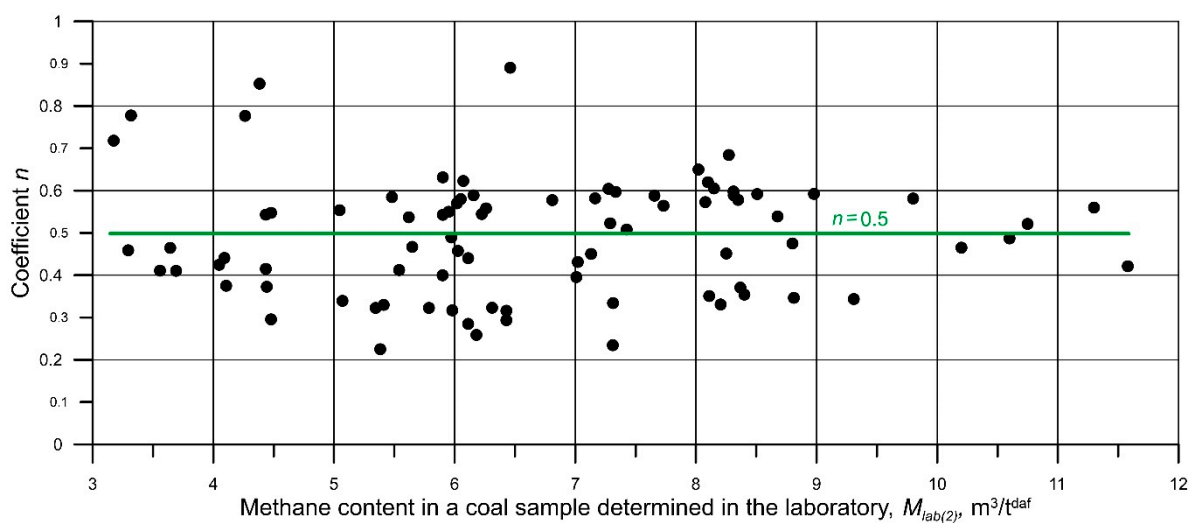


Figure 5. Relationship between coefficient n and methane content in coal samples in second minute of desorption $M_{lab(2)}$.

Taking into account the values of estimated coefficients a , n and equation (Equation (3)), gas loss may be determined. This gas loss according to Equation (2) is the difference between methane content in a coal sample and initial methane content in coal. It can be determined according to:

$$M_{loss} = M - M_{lab(2)} \quad (4)$$

When substituting Equation (3) to Equation (4), the following is obtained:

$$M_{loss} = a \cdot (\tau^n) \quad (5)$$

When assuming obtained coefficients a , and constant n , gas loss is presented as follows:

$$M_{loss} = 0.085 \cdot M_{lab(2)} \cdot \sqrt{\tau} \quad (6)$$

For $\tau = 2$ min, the gas loss amounts to:

$$M_{loss} = 0.12 \cdot M_{lab(2)} \quad (7)$$

The test results showed gas losses amounting to 12% during two minutes of coal sampling in the mine.

4. Application of Developed Procedure for Gas Loss Estimation

4.1. Stages of the Method for Determining Methane Content in Coal

The determined share of gas losses in the initial methane content was adopted to establish a uniform method for determining methane content in coal seams in Polish mines.

The method for gas in coal seam determination was developed based on both mine and laboratory tests. The method defines the procedure for determining methane content in coal seams [32,33]. The procedure is divided into stages, including coal sampling in a mine and determining gas content in a sample under laboratory conditions, respectively. A coal sample is taken in drill cuttings when drilling a borehole in a coal seam. For the developed method, the measurement uncertainty was analysed. Relative standard uncertainty of determining methane content in a coal sample does not exceed 2.5%, while relative maximum uncertainty does not exceed 7.0% [37].

The stages of the method are divided into:

- collection of drill cuttings into airtight steel canisters and measurement of environmental conditions,
- transport of samples to a laboratory,
- determination of desorbable and residual gas components,
- estimation of gas loss.

The method for determining gas content in coal relies in degassing coal samples and measuring the volume of methane separated. For this purpose, drill cuttings are sampled while a test hole is drilled in a coal seam and delivered to the laboratory.

4.2. Collecting Samples in a Mine

To collect samples in a mine, a drilling rig with a minimum 42 mm diameter drilling bit, two airtight steel canisters with steel balls (Figure 6), a stopwatch, a thermometer, a barometer, a methane detector and a calibrated vessel (100 cm³ volume) have to be prepared.



Figure 6. Airtight steel canister with steel balls for collecting coal samples.

Two coal samples are taken to determine methane content in a coal seam. The samples are drill cuttings collected during drilling two horizontal bore holes in the coal seam face—one sample for each borehole. The bore holes should be 1.0 m or more apart from each other in the coal face. Cuttings from each bore hole are sampled from the last 3.5 m of drilling (i.e., between 3.5 and 4.0 m deep). The example of the boreholes layout for sampling coal from the roadway advancing face is shown in Figure 7. Activities from the beginning of collecting drill cuttings from the hole at the depth of 3.5–4.0 m to the closing of the sample in an airtight steel canister should be completed within 120 s.

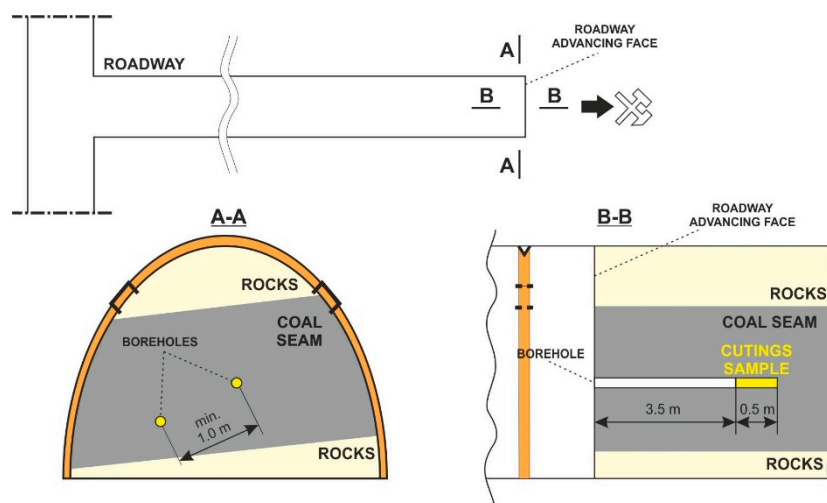


Figure 7. Example of boreholes localization for collecting samples in the roadway advancing face.

A gas sample volume of 100 cm³ should be taken from each of the 2 boreholes. Before a coal sample is closed in an airtight steel canister, it is screened through a sieve with apertures of 1.0 mm. The fraction above 1.0 mm is collected to the canister.

During the collection of the samples on-site, methane concentration in the air, air temperature and atmospheric pressure have to be measured.

Samples are taken to the laboratory in order to carry out further steps and measurements. Two steel canisters with coal samples are transferred to the laboratory.

4.3. Determination of Desorbed and Residual Gases in a Laboratory

One of the goals of the developed method is to obtain the test result relatively quickly. Thus, the method uses the principle of fast desorption, which consists in crushing the sample and determining the desorbed gas and the residual gas as one component. The steel canisters with coal samples are placed in a shaker to grind the coal sample. Steel canisters in which the inspection revealed leakage leading to gas loss should not be used in further tests. Next, the steel canisters should be placed in the shaker and the process should be continued for 2 to 4 h, depending on the coal compactness. Grinding coal should be performed immediately after a steel canister is delivered to the laboratory.

The procedure of determining desorbed and residual gases in the laboratory includes:

- Placing steel canisters with coal samples in a shaker to grind the coal sample for 2 to 4 h, depending on coal compactness;
- Degasifying the samples;
- Pumping gas from a steel canister to a measuring tank;
- Recording pressure in a measuring tank;
- Determining methane concentration in gas mixture;
- Measuring coal mass and determining coal physical parameters.

Degasification of the sample is conducted after its crushing in a steel canister. A schematic diagram of that equipment is presented in Figure 8a. In the first step, a

steel canister with a coal sample is connected to the aperture (Figure 8b). In the next step, a vacuum in the measuring system is created using a vacuum pump (Figure 8c). After this operation, the whole gas from the steel canister is pumped over to the measuring tank (Figure 8d). The volume of the measuring tank is 2250 cm³. Then the pressure in measuring tank should be recorded. The last action is the connection of the system to the gas chromatograph for a chromatography analysis of the gases (Figure 8e). Methane concentration in gas mixture is determined. It is also necessary to read the ambient environmental condition during the activities mentioned above, which is necessary for the next stages of gas content determination.

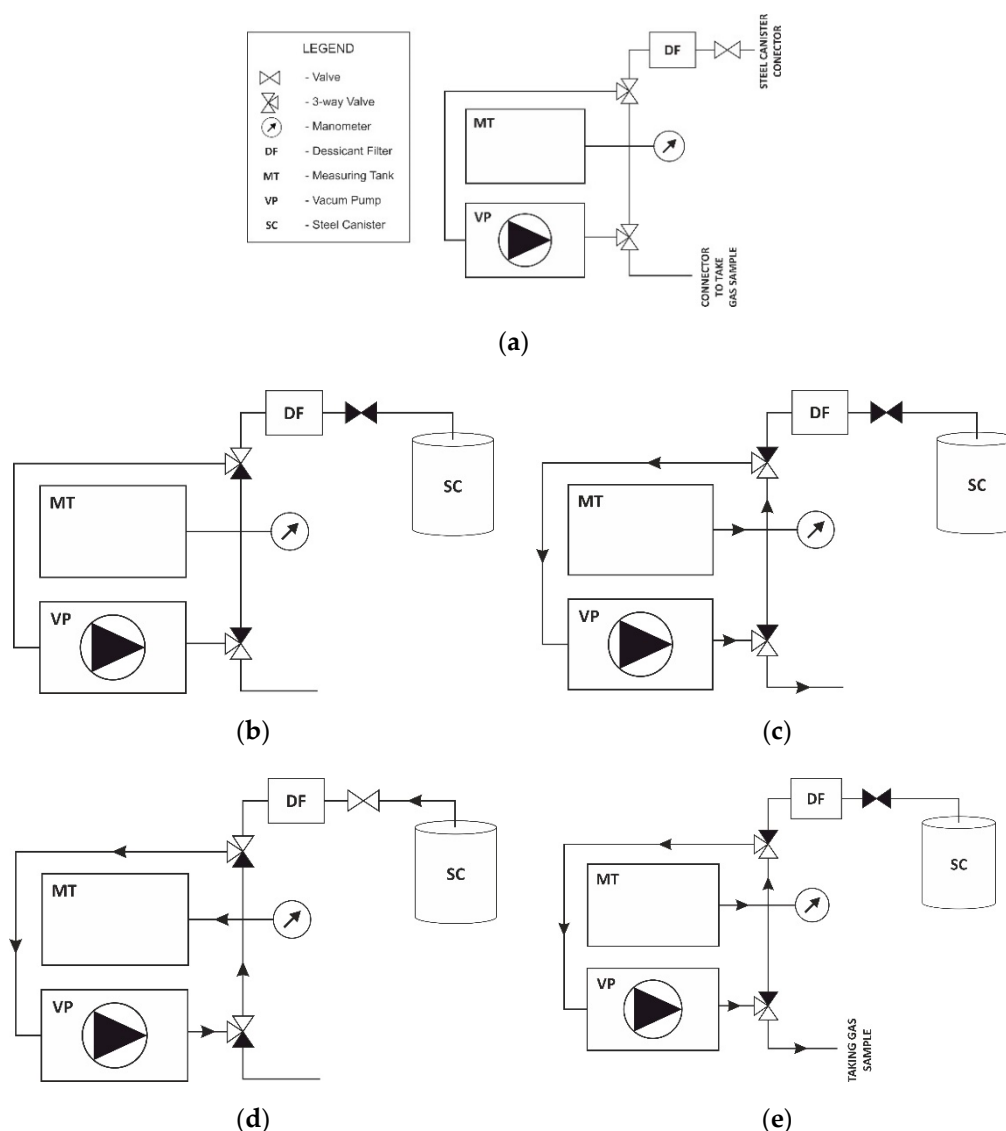


Figure 8. Schematic diagram of equipment used for degassing coal samples: (a) components of degassing apparatus; (b) connecting a steel canister with a coal sample; (c) creating vacuum in the measuring system; (d) degassing a coal sample; (e) taking a gas sample for chromatography analysis.

The last stage of laboratory tests is the measurement of the coal mass and next, the determination of the selected coal physical parameters. After degassing the coal sample and detaching the canister, air should be supplied by opening the clamp on the hose. After opening the canister, the balls are taken out on the tray by a spoon. Next, the canister is cleaned from coal dust by thoroughly brushing the canister, the inner surface of the cover and the steel balls with a brush. Then the weight of coal sample, total moisture

content and ash content are determined, respectively. Coal density is also determined. These parameters are determined according to Polish national standards. Determining the parameters above is necessary because the result of methane content determination is related to dry-ash free coal.

4.4. Determination of Methane Content in a Coal Seam

The procedure of determining methane content of coal, which is related to sampling activities carried out in a mine and laboratory is presented by means of the algorithm shown in Figure 9.

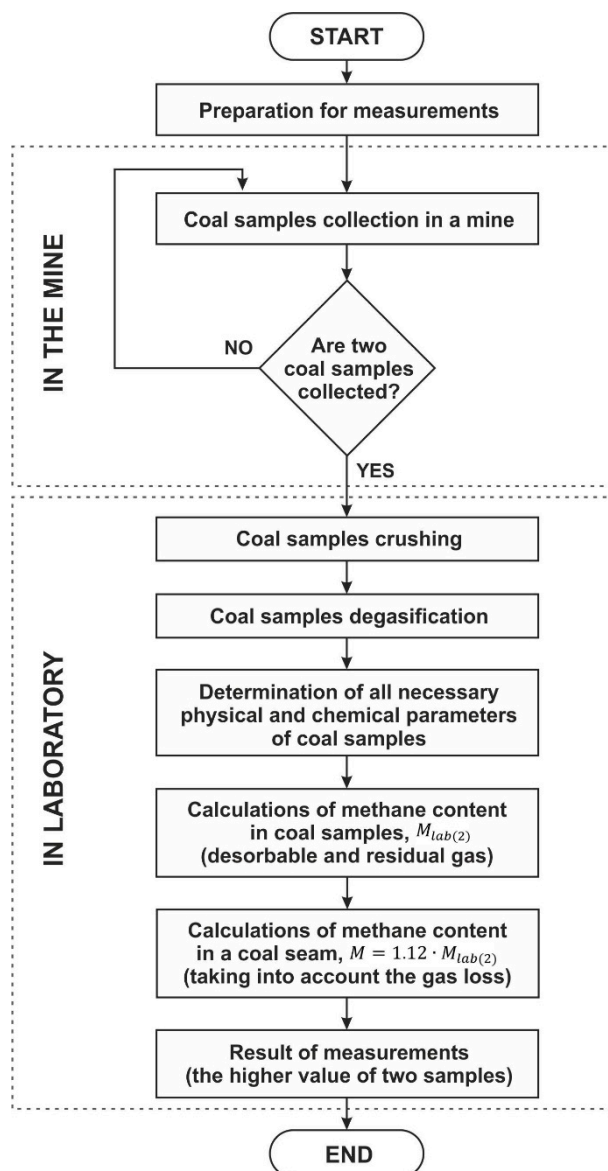


Figure 9. Flowchart of determining methane content.

The result of the determination is the higher value of gas content of both tested samples. According to the research into the sorption of methane in coal at various pressures, the result of determining methane content in the laboratory increases by 12%, which is gas loss related to the time of coal sampling.

The method has been validated in the Polish coal mining industry. The method covers all the stages of the determination, from taking a coal sample in the mine conditions to performing appropriate tests in the laboratory and calculating the determination result.

For the procedure of determining methane content in the coal sample the uncertainty of measurements was analysed [37]. The methodology developed was adapted and forms the basis for the development of the Polish standard PN-G-44200 [38].

5. Interlaboratory Studies on Determination of Methane Content in Coal Seams

After completing the research into a gas loss determination procedure, and developing a new method for the determination of methane content, comparative studies of methane content determination were performed. For the same coal samples, the results of determinations made by two laboratories M_{exp} were compared with the calculations made following the developed methodology M . The State Mining Authority authorizes laboratories in the field of research into methane content in coal seams. Methods of determining methane content by laboratories are expert methods. The analysis was performed for 67 methane content ascertainsments [33].

Figure 10 compares the results of methane content ascertainsments carried out by two laboratories with the results obtained on the basis of the method developed.

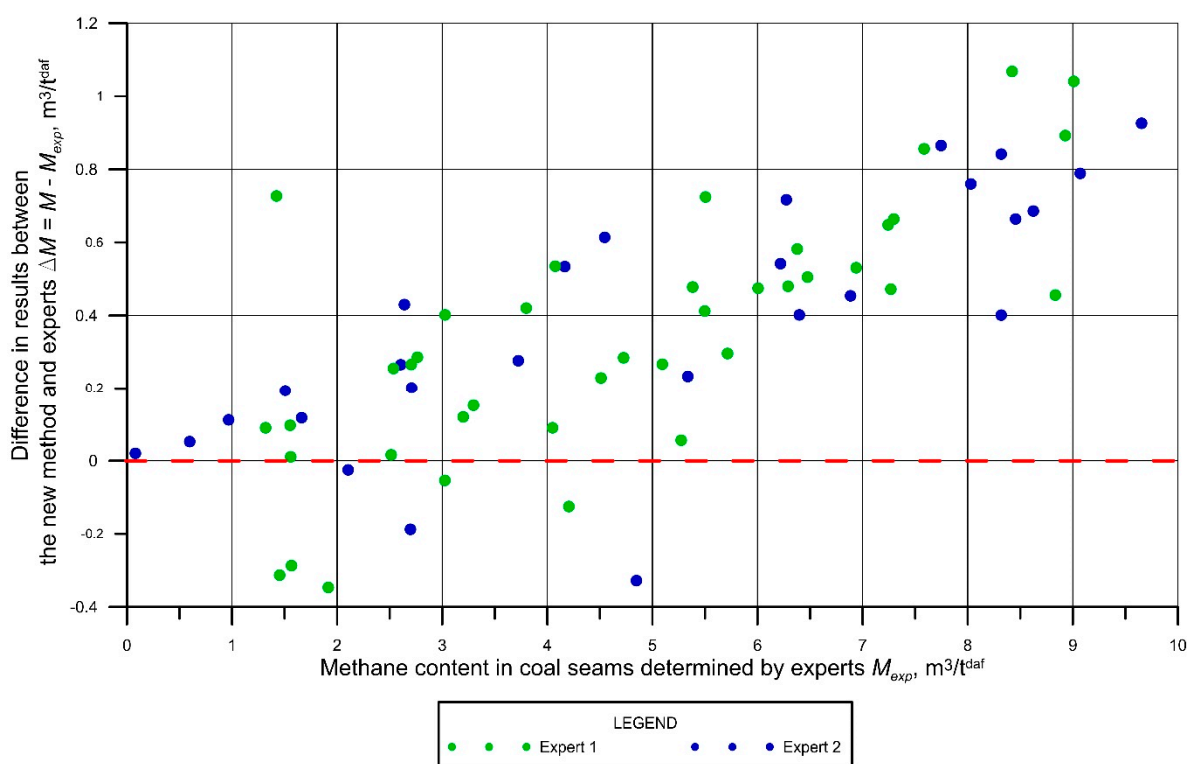


Figure 10. Comparison the results of the new method with the results of other experts/laboratories.

The analysis of Figure 10 shows that the results of ascertainsments made according to the new methodology are similar to experts' determinations for low methane content. Otherwise, for higher methane contents, the new methodology results are higher than experts' values. The vast majority of 67 determinations showed higher values than those provided by experts. Only in 8 cases, the experts' methane content showed a higher value than the result obtained using the new method. The difference between the performed determinations in 23 cases is greater than 10%. In other cases, it does not exceed 10%.

6. Conclusions

Gas loss during sampling in situ is a permanent problem when determining methane content in a coal seam while using various direct methods. Lost gas should be added to methane content in a coal sample.

Gas loss determination was carried out on the basis of research into methane desorption for three different saturation pressures. After analysing the desorption process, it can be concluded that gas loss should be estimated for the first two minutes. All the activities, from starting to collect samples to closing them in an airtight canister, should be completed within the first two minutes.

The estimation of desorption results allowed to determine the dependence between methane content in a coal sample and the initial methane content in coal. On the basis of the conducted research into sorption and desorption, gas loss was determined at 12%. This value of gas loss is taken into account when determining total gas content in a coal seam. The validated method for determining gas content allows for its wide use in Polish coal mining and was accepted as national standard PN-G-44200. The method can be used as a reference point for different procedures in world mining. The method for determining methane content, which is presented in the article, is a crucial step to standardising methane content procedures. The most important step is to unify gas loss calculations related to coal sampling for research purposes.

The advantage of this methodology and the proposed method of determining gas loss is the possibility of obtaining a quick result of the mathematical determination from the moment of coal sampling, which is especially important in mines with high methane emission.

Author Contributions: Conceptualization, N.S. and D.O.; methodology, N.S. and M.K.; software, M.K.; validation, N.S. and D.O.; formal analysis, D.O.; investigation, D.O. and M.K.; resources, M.K.; data curation, M.K.; writing—original draft preparation, D.O. and M.K.; writing—review and editing, D.O. and M.K.; visualization, M.K.; supervision, N.S.; project administration, N.S.; funding acquisition, N.S. All authors have read and agreed to the published version of the manuscript.

Funding: This work was supported by the National Centre for Research and Development—Project No. NR09002106.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Upon authors' request.

Conflicts of Interest: The authors declare no conflict of interest.

References

- Seidle, J. *Fundamentals of Coalbed Methane Reservoir Engineering*; PennWell Corporation: Tulsa, OK, USA, 2011.
- Saghafi, A. Discussion on determination of gas content of coal and uncertainties of measurement. *Int. J. Min. Sci. Technol.* **2017**, *27*, 741–748. [[CrossRef](#)]
- Creedy, D. Methods for the evaluation of seam gas content from measurements on coal samples. *Min. Sci. Technol.* **1986**, *3*, 141–160. [[CrossRef](#)]
- Bertard, C.; Bruyet, B.; Gunther, J. Determination of desorbable gas concentration of coal (direct method). *Int. J. Rock Mech. Min. Sci. Géoméch. Abstr.* **1970**, *7*, 43–65. [[CrossRef](#)]
- Smith, D.M.; Williams, F.L. A new technique for determining the methane content of coal. In Proceedings of the 16th Intersociety Energy Conversion Engineering Conference, American Society of Mechanical Engineers, Atlanta, GA, USA, 9–14 August 1981; pp. 1272–1277.
- Diamond, W.P.; Schatzel, S.J. Measuring the gas content of coal: A review. *Int. J. Coal Geol.* **1998**, *35*, 311–331. [[CrossRef](#)]
- Kissell, F.N.; McCulloch, C.M.; Elder, C.H. *The Direct Method of Determining Methane Content of Coalbeds for Ventilation Design*; Report of Investigations 7767; United States Department of the Interior, Bureau of Mines: Washington, DC, USA, 1973.
- Ulery, J.P.; Hyman, D.M. The modified direct method of gas content determination—Applications and results. In Proceedings of the Coalbed Methane Symposium Proceedings, Tuscaloosa, AL, USA, 13–17 May 1991; University of Alabama: Tuscaloosa, AL, USA, 1991. Paper 9163. pp. 489–500.
- Diamond, W.P.; Schatzel, S.J.; Garcia, F.; Ulery, J.P. The Modified Direct Method: A solution for obtaining accurate coal desorption measurements. In Proceedings of the International Coalbed Methane Symposium, Tuscaloosa, AL, USA, 14–18 May 2001; University of Alabama: Tuscaloosa, AL, USA, 2001. Paper 0128. pp. 331–342.
- Schatzel, S.J.; Hyman, D.M.; Sainato, A.; LaScola, J.C. *Methane Contents of Oil Shale from the Piceance Basin, CO. United States Department of the Interior, Bureau of Mines, Report of Investigations*; Paper 9063; United States Department of the Interior: Washington, DC, USA, 1987.

11. Mavor, M.J.; Pratt, T.J.; Britton, R.N. Improved Methodology for Determining Total Gas Content, Volume I. In *Canister Gas Desorption Data Summary*; Topical Report GRI-93/0410; Gas Research Institute: Chicago, IL, USA, 1994.
12. Mavor, M.J.; Pratt, T.J. Improved methodology for determining total gas Content, Volume II. In *Comparative Evaluation of the Accuracy of Gas-in-Place Estimates and Review of Lost Gas Models*; Topical Report GRI-94/0429; Gas Research Institute: Chicago, IL, USA, 1996.
13. McCulloch, C.M.; Levine, J.R.; Kissell, F.N.; Deul, M. *Measuring the Methane Content of Bituminous Coalbeds*; Report of Investigation 8515; United States Department of the Interior, Bureau of Mines: Washington, DC, USA, 1975.
14. Australian Standard AS 3980-1991. *Guide to the Determination of Desorbable Gas Content of Coal Seams—Direct Method*; Standards Association of Australia: Sydney, Australia, 1991.
15. Australian Standard AS 3980-1999. *Guide to the Determination of Gas Content of Coal—Direct Desorption Method*, 2nd ed.; Standards Association of Australia: Sydney, Australia, 1999.
16. Saghafi, A.; Williams, D.J.; Roberts, D.B. *Determination of Coal Gas Content by Quick Crushing Method*; CSIRO Investigation Report CETIR391R; CSIRO Research Publications Repository: Canberra, Australia, 1995; 9p.
17. Jianqing, Z. Study on the Gas Content of Coal Seam Based on the BP Neural Network. *Procedia Eng.* **2011**, *26*, 1554–1562. [\[CrossRef\]](#)
18. Li, N.; Peng, S.; Du, W.; Guo, Y. New method for predicting coal seam gas content. *Energy Sourcespart. A Recover. Util. Environ. Eff.* **2018**, *41*, 1272–1284. [\[CrossRef\]](#)
19. Wang, L.; Cheng, L.-B.; Cheng, Y.-P.; Liu, S.; Guo, P.-K.; Jin, K.; Jiang, H.-N. A new method for accurate and rapid measurement of underground coal seam gas content. *J. Nat. Gas. Sci. Eng.* **2015**, *26*, 1388–1398. [\[CrossRef\]](#)
20. Ryszk, M.; Sporysz, G. Verification of Direct Designation Method of Methane Content in Coal Seams. Part I—Review of Methane Content Designation Methods. In *Work Safety and Environmental Protection in Mining*; State Mining Authority: Katowice, Poland, 2008; pp. 3–8. (In Polish)
21. Ryszk, M.; Sporysz, G. Verification of direct designation method of methane content in coal seams. Part II—Comparison of methane designation results. In *Work Safety and Environmental Protection in Mining*; State Mining Authority: Katowice, Poland, 2008; pp. 31–38. (In Polish)
22. Zhao, Z.; Sui, F.; Yan, J. Prediction of methane content of deep coal seams in the Sunan mining area in Anhui Province, China. *Int. J. Oilgas. Coal Technol.* **2020**, *23*, 351. [\[CrossRef\]](#)
23. Chen, Y.; Yang, D.; Tang, J.; Li, X.; Jiang, C. Determination method of initial gas desorption law of coal based on flow characteristics of convergent nozzle. *J. Loss Prev. Process. Ind.* **2018**, *54*, 222–228. [\[CrossRef\]](#)
24. Wang, F.; Zhao, X.; Liang, Y.; Li, X.; Chen, Y. Calculation Model and Rapid Estimation Method for Coal Seam Gas Content. *Processes* **2018**, *6*, 223. [\[CrossRef\]](#)
25. Liu, Y.; Du, Y.; Li, Z.; Zhao, F.; Zuo, W.; Wei, J.; Mitri, H. A rapid and accurate direct measurement method of underground coal seam gas content based on dynamic diffusion theory. *Int. J. Min. Sci. Technol.* **2020**, *30*, 799–810. [\[CrossRef\]](#)
26. Xu, H.; Pan, Z.; Hu, B.; Liu, H.; Sun, G. A new approach to estimating coal gas content for deep core sample. *Fuel* **2020**, *277*, 118246. [\[CrossRef\]](#)
27. Gray, I. Reservoir Engineering in Coal Seams: Part 1-The Physical Process of Gas Storage and Movement in Coal Seams. *Spe Reserv. Eng.* **1987**, *2*, 28–34. [\[CrossRef\]](#)
28. Bakhshian, S.; Sahimi, M. Adsorption-induced swelling of porous media. *Int. J. Greenh. Gas. Control.* **2017**, *57*, 1–13. [\[CrossRef\]](#)
29. Bakhshian, S.; Shi, Z.; Sahimi, M.; Tsotsis, T.T.; Jessen, K. Image-based modeling of gas adsorption and deformation in porous media. *Sci. Rep.* **2018**, *8*, 1–12. [\[CrossRef\]](#)
30. Regulation of the Minister of Energy on Detailed Requirements for the Operation of Underground Mining Facilities. 2016. Available online: <http://isap.sejm.gov.pl/isap.nsf/download.xsp/WDU20170001118/O/D20171118.pdf> (accessed on 12 February 2021).
31. Kędzior, S. Methane contents and coal-rank variability in the Upper Silesian Coal Basin, Poland. *Int. J. Coal Geol.* **2015**, *139*, 152–164. [\[CrossRef\]](#)
32. Szlęzak, N.; Borowski, M.; Obracaj, D.; Swolkień, J.; Korzec, M. *The Method of Evaluation of Methane Content in Hard Coal Seams*; AGH University of Science and Technology Press: Kraków, Poland, 2011. (In Polish)
33. Korzec, M. Development the Method for Determining the Methane Content of Coal. Ph.D. Thesis, AGH University of Science and Technology, Krakow, Poland, 2013. (In Polish).
34. Langmuir, I. The adsorption of gases on plane surfaces of glass, mica and platinum. *J. Am. Chem. Soc.* **1918**, *40*, 1361–1403. [\[CrossRef\]](#)
35. Wierzbicki, M.; Skoczylas, N.; Kudasik, M. Methane pressure in coal seams of the USCB measured by direct method. *Pol. Min. Rev.* **2015**, *71*, 21–25. (In Polish)
36. Dutka, B.; Walaszczyk, J.; Wierzbicki, M. Estimation of the coal seam methane pressure on the base of the methane content and sorption properties of the coal from the “Krupiński” Coal Mine. *AGH J. Min. Geoengin.* **2009**, *33*, 145–152. (In Polish)
37. Szlęzak, N.; Korzec, M. Method for Determining the Coalbed Methane Content with Determination the Uncertainty of Measurements. *Arch. Min. Sci.* **2016**, *61*, 443–456. [\[CrossRef\]](#)
38. Polish Committee for Standardization PN-G-44200:2013-10. *Górnictwo—Oznaczanie Metanonośności w Pokładach Węgla Kamiennego—Metoda Zwiercinowa*; The Polish Committee for Standardization: Warszawa, Poland, 2013. (In Polish)