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Requirements for a Hybrid Dust-Gas-Standard: Influence of the Mixing Procedure on Safety Characteristics of Hybrid Mixtures

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Abstract: While developing a standard for the determination of safety characteristics for hybrid mixtures the authors discovered, that, beside the ignition source, the mixing procedure is the main difference between the single-phase standards for dusts and gases. The preparation of hybrid mixtures containing a flammable gas and a flammable dust in the 20 L-sphere can be realized in different ways. Either the flammable gas is filled only in the sphere or only in the dust container or in both. In previous works, almost always the first method is applied, without giving any information on the accuracy of the gas mixtures. In this work the accuracy of the gas mixtures and the results of the tests applying two methods of mixing were studied. No significant influence of the mixing method itself on the safety characteristics explosion pressure p_{ex} and the normalized rate of pressure rise (K-value) was found. Obviously, homogenization of the gas mixtures can be obtained sufficiently by the turbulence that is caused during the injection from the dust container into the explosion chamber within a short time. However, the mixing procedure has a great influence on the accuracy of the gas amount of the mixtures obtained. Without modifying the 20 L-sphere by installing precise pressure sensors, assuring its tightness and performing gas analysis, it must be expected, that the accuracy of the gas mixtures is very low. This has a significant influence on the measured safety characteristics and may lead to unsafe facilities or unnecessary expensive safety measures.

Keywords: hybrid mixtures; 20 L-sphere; pre-ignition pressure rise; post-injection pressure drop; safety characteristics; mixing procedure

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1. Introduction

While developing a new standard for the determination of the safety characteristics of hybrid dust gas mixtures the authors came across three main differences: The ignition source, the definitions and accuracies of the pressures and the mixing procedure. Since the commonly used and preferred method to test hybrid mixtures is a variation of the standard for the determination of safety characteristics of dusts, in this article we describe how the gas amount should be added to achieve a hybrid mixture.

The 20 L-sphere is a widely used test autoclave for the determination of safety characteristics of combustible dusts in a closed vessel. Due to its smaller volume and easier handling, it is much more frequently used compared to the 1 m^3, which is the traditional standard test vessel. Generally, it could also be used for the determination of safety characteristics of flammable gases. According to the European standards for the determination of safety characteristics of gases, such as explosion limits and limiting oxygen concentration [1] or maximum explosion pressures and maximum rates of pressure rise [2], a vessel of spherical or cylindrical shape (with height to width ratio of 1) larger than 0.005 m^3 shall be used. Larger differences between the procedures for determining safety characteristics of gases and dusts appear in the way of mixture preparation.

For the determination of the safety characteristics of gases, the gas mixtures are usually prepared by the method of mixing flows or according to the partial pressure method either directly in the explosion vessel or in a separate mixing vessel. The ignition is always triggered in quiescent mixtures, so that thermodynamic heating or cooling due to compression is already dissipated by the vessel wall before ignition. For the determination of the safety characteristics of dusts it is necessary to disperse the dust in the oxidating atmosphere (usually air). In the standard procedures this is realized by injecting a pressurized dust-air mixture into an explosion vessel (in case of the 20 L-sphere, it is previously partially evacuated). Shortly after injection the dust cloud gets ignited. For hybrid mixtures containing a combustible dust as well as a flammable gas there is no standard mixing procedure available yet, though three different mixing procedures were found in the available literature.

For hybrid mixtures the mixing procedure can have a great influence on the test result because it may affect the gas composition significantly or leads to inhomogeneities. The flammable gases are filled either in the 20 L-sphere or in the dust container and usually the flammable gas concentration is calculated with the partial pressure method.

Mixing Procedures

The mixing procedure for the determination of the safety characteristics of hybrid mixtures in the 20 L-sphere can be conducted in three different ways:

- Method I: A premixed gas-air mixture is used for both, the 20 L-sphere and the dust container before ignition (Figure 1, left).
- Method II: The flammable gas is directly injected into the 20 L-sphere and dust is injected by pressurized air from the dust container (Figure 1, middle).
- Method III: The 20 L-sphere is only filled with air and the dust container is pressurized by a mixture of flammable gas and air injecting the dust from the dust container (Figure 1, right).

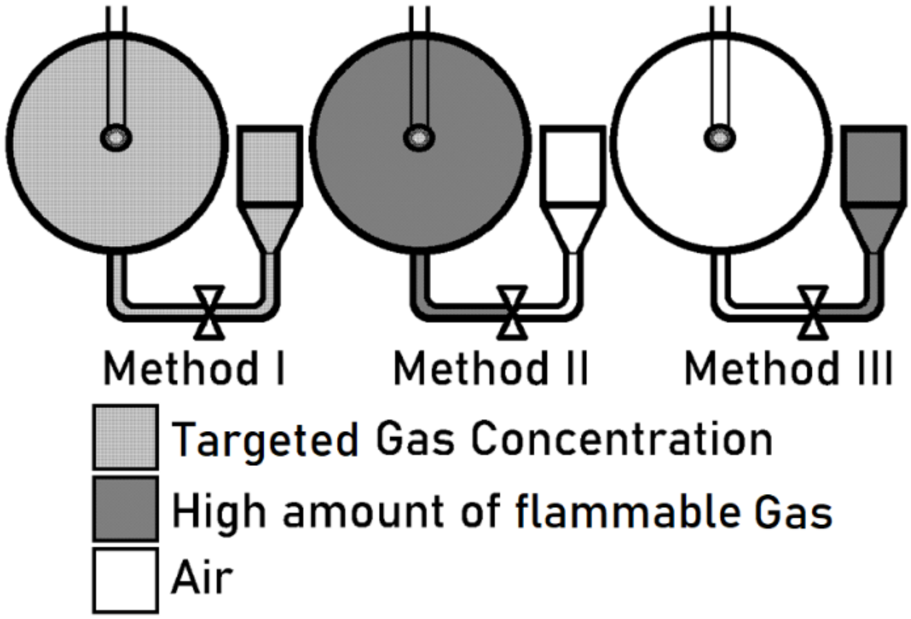

Figure 1. Three different ways of mixing; Gas concentration before opening the fast-acting valve between 20 L-sphere and dust container [3].

The three different mixing methods all have their benefits and disadvantages:

Method I: The most precise and homogeneous gas mixtures are obtained, if the explosion vessel and the dust container are both filled with the same premixture before injection of the dust. However, for hybrid mixtures of gas and dust this method is complicated and the common 20 L-sphere for dust must be modified slightly, because both, the sphere and the dust container, have to be evacuated to very low pressures before they are filled with the premixture. The latter is not possible, if dust is already in the dust container, but the residual air in the dust container can be considered for the determination of the final gas concentration. This method has the benefit, that the following potential sources of error are avoided:

- 1: A discrepancy between the calculated gas concentrations, derived from the measured partial pressures with the pressure sensors installed in the sphere and the real concentration of the gas;
- 2: Local concentration variations because of incomplete mixing at the ignition moment after 60 ms of ignition delay time (for 20 L-sphere, 600 ms for 1 m^3).

The other two mixing procedures assume, that the gas-phase is homogenous at the ignition moment and in none of the previous works the actual gas concentration is measured in the tests nor is the accuracy of the gas mixtures mentioned. The gas concentrations are calculated based on the partial pressures usually measured with sensors with a high measuring uncertainty. In most cases, other sources of errors are not mentioned at all and the accuracy of the gas preparation is hardly considered yet. The disadvantages of this mixing procedure are the dangerous explosive atmosphere in the dust container if gas concentrations above the lower explosible limit are tested and the previous preparation of these mixtures. Either it is prepared at an elevated pressure of more than 21 bar a, or the explosive mixture must be transferred by a compressor. Facilities that used this method so far are the Dortmund University, Germany [4] and the Dalhousie University, Canada [5] (with a 26 L-sphere) but it is not applied anymore [6]. The latest article applying this method of mixture preparation (not with flammable but with inert gases, so under rather safe conditions) was published in 2009 from the Northeastern University in Liaoning, China, but it seems it is not in use anymore, too [7].

Method II has the benefit that the common 20 L-sphere can be used as it is without any complicated modifications. There are no explosive gas-air mixtures at high pressures during mixture preparation, which makes this procedure the safest one. It is valid for hybrid mixtures with a gas fraction of up to 40%. However, this procedure is more prone to errors due to leakage of the 20 L-sphere resulting in larger deviations to the desired concentration of flammable gas. This method is recommended in the manufacturer's handbook [8] and applied by most of the research facilities such as the Otto von Guericke University, Germany [9], Karlsruhe Institute for Technology, Germany [10,11], the Dalhousie University in Halifax, Canada [12], Anhui University of Science and Technology Huainan, China [13], Henan Polytechnic University Jiaozuo, China [14], Texas A&M University, USA [15] (with a 36 L-sphere), the University of Witwatersrand, South Africa [16] (with a 40 L-sphere), the Italian National Research Council in Naples, Italy [17], Xi'an University of Science and Technology in Shaanxi, China [18] and the University of Pardubice, Technical University of Ostrava and OZM Research, all three in Czech Republic [19] (with 20 L-sphere and 1 m^3).

Method III has the disadvantage, that explosible gas-air mixtures are pressurized to 21 bar in the dust container, so if an explosion occurs an overpressure of up to 200 bar might occur inside the dust container which is not designed for these high pressures and might burst. Therefore, this mixing procedure is commonly applied with low amounts of flammable gas below the lower explosion limit (LEL) in the dust container. Thus, applying this method of mixture preparation, only hybrid mixtures with very little amounts of flammable gas can be tested. Usually, premixtures of flammable gas and air prepared in gas cylinders are used for pressurizing the dust container.

The benefits of this method are that there are no modifications needed on the standard dust sphere, the test procedure is comparably short, and the concentrations might be more precise, because it is not dependent on potential leakages of the 20 L-sphere. This method is applied by Dalian University, China [20] (without premixtures), Sichuan University, China [21] (with premixtures), North University in Taiyuan, China [22] (with premixtures) and the University of Newcastle, Australia [23] (only gases under turbulence in the 1 m^3).

It should be mentioned, that in some articles it is not mentioned at all, where the amount of gas is added first, even though, this is crucial for a later interpretation of the data. It can be assumed, that if it is not stated or simply said, that the gas fraction was filled in using the “partial pressure method” (which actually all three methods are) Method II was used [24].

A comparison between the three mixing procedures has not taken place so far and with that the sources of error mentioned above have not been investigated as well.

2. Materials and Methods

For all the experiments a standard 20 L-sphere was used. Two piezo-resistive pressure sensors (company: Keller, type: PA-10, linearity: better than 0.5 % full scale) were additionally installed for the tests, one with a resolution of 0.1 mbar and a range of 1 bar, the other with a resolution of 1 mbar and a range of 10 bar. The first one was used for the filling process and had to be closed before initiating each ignition. The second one was used for additional checking of the explosion pressures and the determination of the Post-injection pressure drop before the actual tests [3]. An additional highly sensitive low vacuum pressure sensor (pirani gauge) with a resolution of 0.1 mbar was installed to measure the absolute pressure during evacuation prior to each test. Mixture preparation was carried out either according to Method I or Method II. The sphere was evacuated to a pressure lower than 5 mbar, all the connections lower than 2 mbar prior to each test. Afterwards either the premixtures from the mixing vessel were filled (Method I) or the components of the mixture were filled one by one (Method II). The air that was used throughout all the tests had an oxygen content of 20.9 $\pm$ 0.2 Mol%, free of oil and a temperature of 21 °C $\pm$ 5 K before injection. The humidity was not checked. This might be beneficial for future work. In each test the pressure in the 20 L-sphere was 400 $\pm$ 2 mbar before dust-injection. The pressure development was recorded with the two piezo-electric pressure sensors that are installed in the 20 L-sphere in the default configuration of the manufacturer (internal resolution of 1 mbar, displayed in the software with 10 mbar and a measurement frequency of 5 kHz) and with the additional piezo-resistive sensor with a measuring range up to 10 bar. As igniters two 1 kJ pyrotechnical igniters or optionally two exploding wires with a net energy of 1 kJ each were used, pointing in opposite direction placed in the center of the sphere according to the standards for the determination of explosion characteristics of dust clouds [25–29]. The methane and carbon dioxide that was used for all tests had a purity of over 99.99%.

2.1. Method I

Prior to each test the mixture of flammable gas and air was prepared in a separate pressure resistant mixing-vessel that was connected to both the 20 L-sphere and the dust chamber (see Figure 2). The mixtures were prepared according to the partial pressure method in the mixing vessel, evacuating the mixing vessel to less than 5 mbar (abs) first and filling in the components of the mixture subsequently according to their partial pressures. The mixing vessel was equipped with a PC-fan for homogenizing the mixtures. The pressure in the mixing vessel was measured with a piezoresistive pressure sensor (company: Keller, type: PA-10, linearity: better than 0.5% full scale) with a resolution of 5 mbar and a range of 50 bar (PIR 6 in Figure 2). The mixtures were prepared at a final pressure of 26 bar (abs) for each test. For safety reasons, the set-up was installed in a safety room and operated by remote control. For the tests without dust the dust container was also evacuated prior to filling the gas mixture by opening the fast-acting valve between the dust container and the 20 L-sphere several times, until the pressure in the 20 L-sphere did not rise anymore.

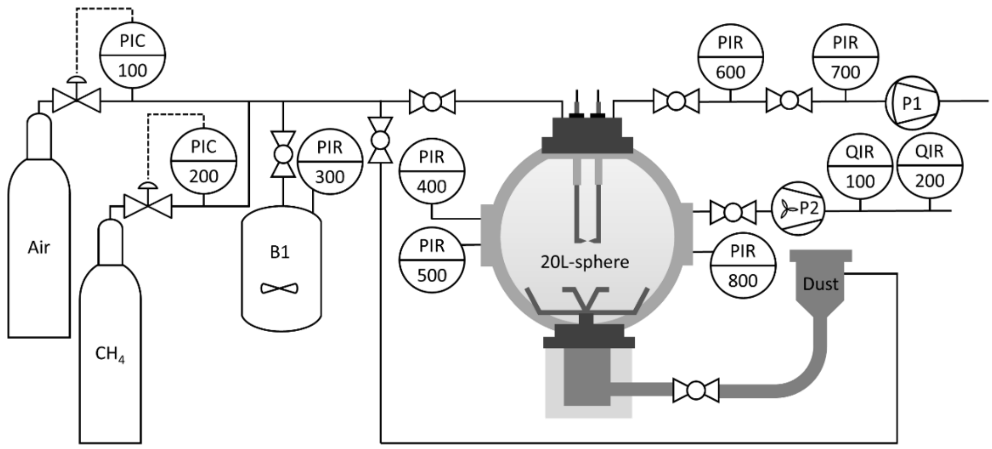

Figure 2. Schematic of the standard 20 L-sphere with adaptations for hybrid mixture testing, having premixtures inside a premixing vessel (B1) that can be filled to the 20 L-sphere and the dust container.

2.2. Method II

Filling the required amount of flammable gas for the mixture only into the 20 L-sphere is safer, faster and needs less modifications than method I (see Figure 3). The flammable gas was filled in the 20 L-sphere up to the desired partial pressure and air was added afterwards to 400 ± 2 mbar. With this method of mixing mixtures with a flammable gas fraction of maximum 40 Mol% can be tested. It is not appropriate for mixtures with air concentrations of less than 60 Mol% corresponding to an oxygen concentration of 12.6 Mol%.

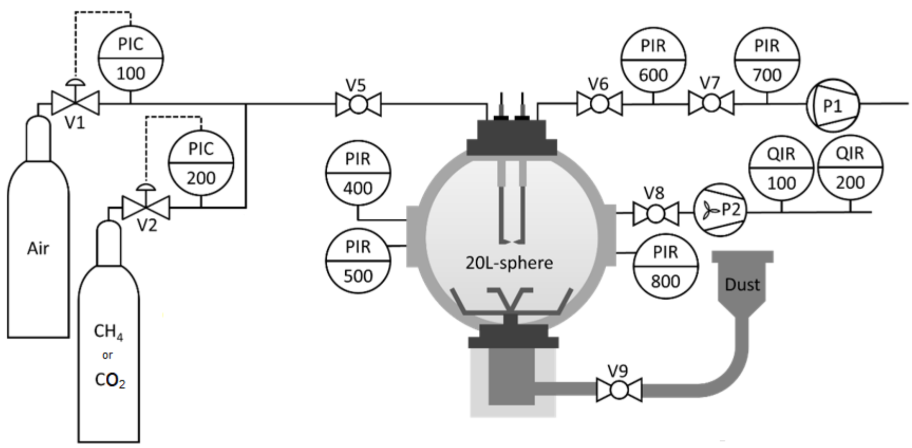

Figure 3. Schematic of the standard 20 L-sphere with adaptations for hybrid mixture testing, gas amount just inside the 20 L-sphere.

2.3. Gas Analysis

The accuracy of the composition of the gas mixtures was determined by gas analysis in separate tests without triggering the ignition. For these tests the flammable gas methane was replaced by carbon dioxide, which is easier to handle. A carbon dioxide-measuring system (Infralyt, Saxon Junkalor; Germany) with a range of 0% to 20% and a resolution of 0.01% was connected to the 20 L-sphere via a gas pump behind a valve (see Figures 2 and 3). The measuring system was calibrated with a reference gas.

An additional oxygen-measuring system (Mini MP, Servomex, United Kingdom) with a range of 0% to 100%, a resolution of 0.1%, a linearity of ±0.1% and an accuracy of ±0.1% was installed between the carbon dioxide-measuring system and the pump (see Figures 2 and 3).

First tests without igniters were conducted with the two mixing methods described with partial pressures equal to gas concentrations of 6 Mol%, 9 Mol% and 12 Mol% and the amount of gas was measured afterwards. Possible systematic deviations between the calculated and the actual gas fraction due to clearance volume, leakage, residual air or imprecise pressure sensors as well as the overall variation were determined in this way. Considering the low pressures and being far away from boiling temperatures ideal gas behavior was assumed for all the mixture components.

2.4. Comparison of Safety Characteristics

Two of the three mixing procedures were compared by conducting explosion tests with the same concentrations of methane and comparing the p_{ex} and K_G-value. The third method was not tested, because it is not very common and requires additional safety measures due to the compressed explosible atmosphere in the dust container. The values were also compared to literature values. For the comparison of the mixing methods hybrid tests with dust were not performed because the dust container could not have been evacuated resulting in an error of the gas amount for Method I.

3. Results and Discussion

3.1. Gas Analysis

Figure 4 illustrates the deviations of the measured partial pressure fractions from the mole fractions measured directly with the gas analyzer. The measured partial pressure fractions were always a bit higher than the directly measured mole fractions, no matter if the gas mixtures were prepared in a separate vessel or directly in the 20 L-sphere. This may be caused by residual air or by the linearity of the pressure sensor. When the mixtures were prepared in a separate mixing vessel, a mean deviation of 0.24 Mol% was determined that was subtracted from the measured partial pressure fractions in the following tests resulting in a variation of ±0.11 Mol%. When the gas mixtures were prepared directly in the 20 L-sphere the amount of the added gas measured with the gas analyzer was also lower than the measured partial pressure fraction. The variation was higher than the variation obtained for the mixtures prepared in the separate mixing vessel. The mean deviation was subtracted from the measured partial pressure fractions in the following tests as well. This method results in a decreasing variation of maximum ±0.15 Mol%, so comparable to the other method.

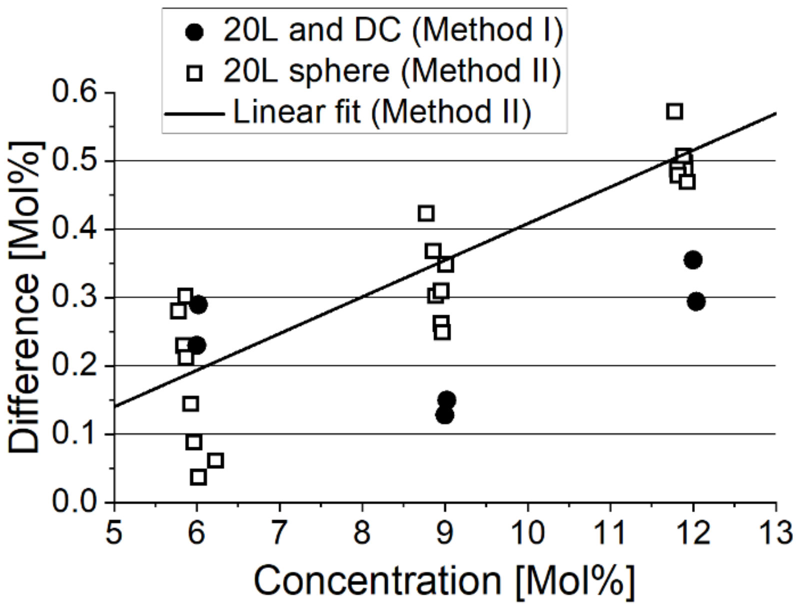

Figure 4. Difference between the calculated amount and the direct measured amount of gas against the calculated concentration.

It should be mentioned that the concentrations were calculated carefully after each test according to Euqation (1).

$$c_{gas} = p_{gas}/(PV + PIPR - PIPD) - Deviation \quad (1)$$

with p_{gas} being the partial pressure fraction of the flammable gas in bar, PV the partial vacuum before the injection of the dust cloud in bar, held between 400 ± 2 mbar, the pressure-increase due to the injection of the dust cloud, the so-called pre-ignition pressure rise (PIPR), held between 0.64 ± 0.01 bar. The pressure-drop after injecting the dust cloud due to the increased temperature because of the fast compression (Post-injection pressure drop, PIPD) was always recorded for at least three minutes for the gas analytic tests and for later explosion tests averaged (for PIPR and PIPD see [3]). For Method I the average deviation of 0.24 Mol% was subtracted for the three different gas concentrations, for Method II the values of 0.2 Mol%, 0.35 Mol% and 0.5 Mol% were subtracted (see Figure 4). It shall also be mentioned that other facilities had deviations of over 2 Mol%, which would lead to the determination of false safety characteristics.

3.2. Influence of the Mixing Procedure on the Explosion Characteristics

The explosion characteristics p_{ex} and K-value were measured with the two different mixing procedures and with two standard pyrotechnical ignitors with 1 kJ each. The values for p_{ex} are comparable with the two mixing procedures. The highest p_{ex} (average of the explosion overpressure values at the concentration with the highest values) is only slightly higher for the mixing procedure according to Method II (with 7.4 ± 0.2 bar), compared to the other mixing procedure (7.3 ± 0.2 bar). This difference is not significant (see Figure 5). When the data is compared to the explosion pressures from a reference database the values from the 20 L-sphere are slightly higher close to the stoichiometry. The difference increases at the methane lean side and might as well increase at the oxygen lean side. This is probably caused by the turbulence, that increases the values for p_{ex} especially for non-stoichiometric mixtures [30–33].

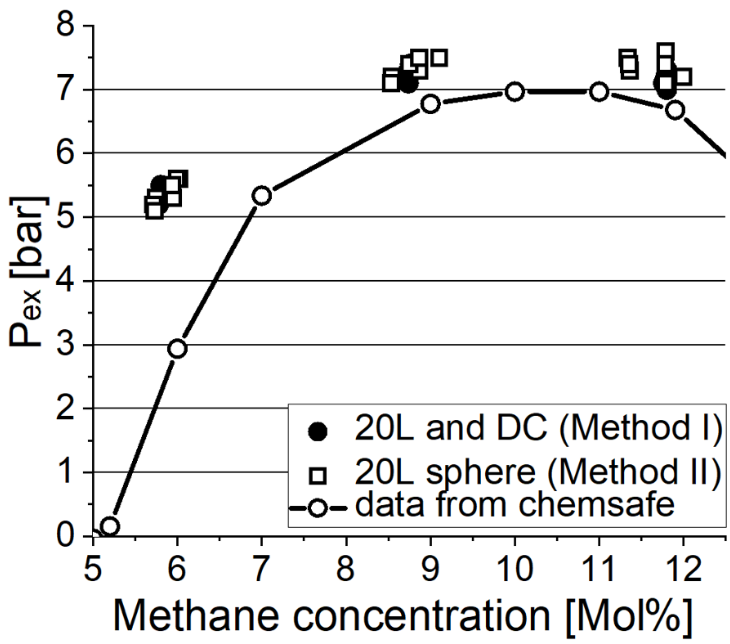

Figure 5. Explosion overpressure of methane; two different mixing procedures with gas inside the 20 L-sphere only and in the 20 L-sphere and the dust container (DC) and data from reference database [34].

The average K-values are also comparable for both mixing methods as shown in Figure 6, but the variation is higher when the mixtures are prepared according to Method

II. A comparison with the reference database is not possible, because gases are usually tested under quiescent conditions and in contrast to p_{ex} the K-values are highly affected by turbulence [30–33]. The literature values for the three tested concentrations are 10 bar/s, 240 bar/s and 140 bar/s and thus lower by a factor of 40, 6 and 6 compared to the values presented in this paper.

The highest K-value for the mixing procedure with separate mixing vessel (Method I) is 381 bar/s (±9%) and thus slightly lower, than for the other mixing procedure (Method II) with 390 bar/s (±14%), but the values are overlapping and can thereby be seen as comparable (see Figure 6). Following the standard procedure for the determination of the maximum rate of pressure rise of gases [2] a deviation of less than 10% between the two mixing procedures is not considered as significant. Consequently, it can be concluded that the mixing procedure has no influence on the explosion characteristics p_{ex} and K-value measured in the 20 L-sphere. Obviously, homogenization of the gas mixtures can be obtained sufficiently by the turbulence that is caused by the injection from the dust container within the 60 ms between injection of the dust and triggering the ignition source.

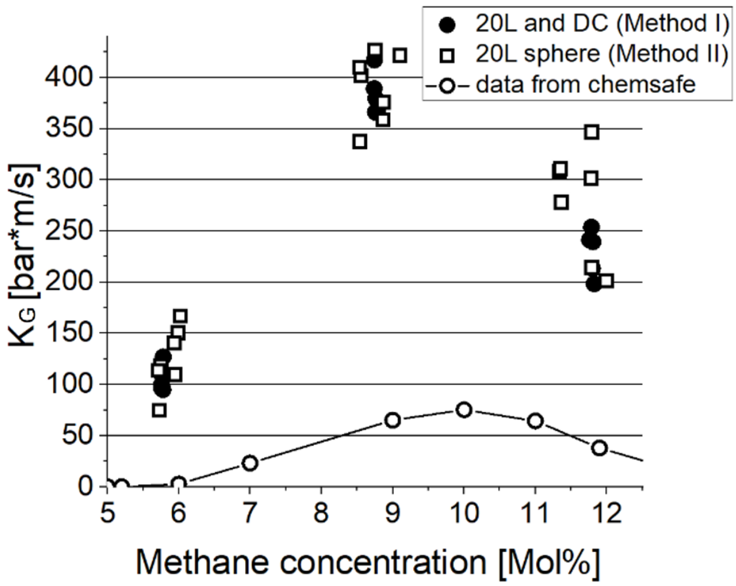

Figure 6. K-values of methane; two different mixing procedures: flammable gas inside the 20 L-sphere only and premixture in the 20 L-sphere and the dust container (DC).

4. Conclusions

The outcome of the presented work can be summarized with two main points:

- The three different mixing methods for hybrid mixtures can be expected to be interchangeable, a full mixing has taken place after the standard ignition delay time of 60 ms
- The amount of gas has to be measured and calibrated before starting the explosion tests since the real amount of gas is not equal to the partial pressure filled to the 20 L-sphere

The mixture preparation for hybrid mixtures can be conducted in three different ways, that were so far expected to be interchangeable but it was never tested before. Simple calculations of the gas amounts results in wrong gas concentrations and therefore in wrong safety characteristics. Regardless, if the tests are conducted with the same conditions and the same gas concentrations, the two presented methods, adding the gas amount only to the 20 L-sphere or having pre-mixed gases in the 20 L-sphere and the dust container, showed no difference concerning the determination of the explosion characteristics p_{ex} and K-value. It is assumed, that the mixing of the gas-phase is completed after the usual 60 ms ignition delay time. For testing hybrid mixtures, preparation of the gas mixtures is much easier directly in the 20 L-sphere, but the gas concentration should be validated before conducting explosion tests shown in this article. Moreover, the accuracy of the pressure sensors that are usually installed in the 20 L-sphere for dust explosion testing is too low for preparing gas mixtures. Consequently, for testing hybrid mixtures, the 20 L-sphere should be modified by installing a pressure sensor with a higher accuracy that can be blocked before triggering the ignition to avoid destruction. Moreover, a gas measuring system should be used and the tightness of the 20 L-sphere should be ensured by leak tests before conducting the explosion tests. For tests with flammable gas concentrations of more than 40 Mol% Method I or Method III (up to 60 Mol%) has to be applied.

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