EXPERIMENTAL DETERMINATION OF MINIMUM IGNITION CURRENT (MIC) FOR HYDROGEN /METHANE MIXTURES FOR THE DETERMINATION OF THE EXPLOSION GROUP CORRESPONDING TO IEC 60079-20-1 STANDARD

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ABSTRACT

Power to gas could get an important issue in future, permitting the valorisation of green electric excess energy by producing hydrogen, mixing it with natural gas (NG) and use the NG grid as temporary storage. NG grid stakeholders expect that blends up to 20% seem to be a realistic scenario. The knowledge of the explosion group for these hydrogen/NG (H2NG) mixtures is a necessary information for the choice of equipment and protective systems intended for the use in potentially explosive atmospheres of these mixtures. Therefore, we determined experimentally the minimum ignition current (MIC), the MIC ratios referenced on MIC of pure methane, corresponding to IEC 60079-20-1 standard. The results are compared to those obtained by maximum experimental safe gap (MESG), the second standardized method. The tested gas mixtures started from 2 vol.% volume admixture in methane rising in 2% steps up to 20 vol.% of hydrogen. The interpretation of these results could conduct to consider methane/hydrogen mixtures containing more than 14 vol.% of hydrogen as Group IIB gases.

1.0 INTRODUCTION AND CONTEXT

The development of intermittent energies has got the goal to reduce CO_2 emissions and to reduce energy dependency. Consequently, the electricity system will not only continue to face varying electricity demand throughout the day, but increasingly experience generation-driven fluctuations related to the intermittent energy sources. The conversion of excess electricity to hydrogen by electrolysis could be a solution to integrate these new energy sources by using the existing natural gas grid as buffer. The stored hydrogen can then be transformed in electric or heat energy. In this context, the network design for natural gas cannot be used for pure hydrogen without significant modifications. Blending hydrogen into the existing natural gas pipelines with relatively low concentrations has been proposed therefore by European gas distribution operators. The benefits of this addition of hydrogen to NG were extensively reported in [1] in terms of economical, technical and safety issues and form a US point of view.

Similar work was conducted in the European Union, in the EC funded project NATURALHY, described in detail in [2] and [3] focused on the safety related to the transmission, the distribution and use of gas, the integrity of pipelines, the gas quality management and the performance of end user appliances. Demonstration was implemented from 2004 to 2009, to figure out the conditions under which hydrogen could be added. It was concluded that for domestic appliances, up to 20 % of hydrogen could be added to NG. Other projects like the "Sustainable Ameland" in the Netherlands focused on the gain of knowledge regarding the electrolysis, the mixing process and the behaviour of the gas network and gas appliances [3].

More recently, the experimental project GRHYD [4] supported by the French government started in 2013 in the urban area of Dunkerque in France. It is intended to evaluate the economic and technical feasibility of the use of H2NG blends up to 20% of hydrogen for urban buses and for urban gas distribution network providing about 200 households. Regarding the addition of hydrogen in the transportation network, the Jupiter 1000 project supported by European Union and the French government is slated to come into service in 2018 in Marseille [5]. H2NG will be delivered to industrial installations.

Since flammability and explosive characteristics of hydrogen are quite far from those of methane,

which is considered as representative of natural gas, this technical issue needs to be completed by detailed risk assessments focused on the potential increase of accidental risks linked to the transport of blends of natural gas and hydrogen through the existing NG pipeline networks and use in domestic appliances.

Therefore, the goal of the HYDROMEL project, conducted from 2006 to 2009, was to reach recommendations for the safe deployment of hydrogen and H2NG transport. Within this project, large-scale jet fires were studied experimentally and numerically and the main characteristics of the flame including visible length, radiation fluxes and blow-out have been assessed [6].

An extended literature review of the potential effects of hydrogen enriched natural gas up to 20 vol.% on safety was recently provides by HSL [7]. It was established that the flammable limits, as well as limiting oxygen concentration can be reasonably calculated using Le Chatelier's law. Experimental data obtained by Askar et al. in 2016 on methane/hydrogen mixtures containing 10, 25, 50 and 75 vol.% of hydrogen moderate this last point concerning upper flammability limit [16].

Another critical point is the ignition sensitivity of the methane/hydrogen mixtures, which was also reviewed by HSL [7]. Proust et al. shown hydrogen is significantly more sensitive to friction than methane in [8]. Similarly, the sensitivity to electric and electrostatic ignition sources is strongly influenced by the addition of hydrogen in methane: the minimum ignition energy (MIE) decreases as the hydrogen content increases. Hankinson et al. established experimentally a MIE of 0.1 mJ in the case of a mixture containing 75 % methane and 25 % hydrogen [17]. This was compared to the maximum equivalent energy of Corona discharges, approximately 0.1 mJ. Consequently, mixtures containing more than 25 % hydrogen could be considered sensitive to this electrostatic discharge [7].

Ma et al. stated that the MIE of multi-component gas mixtures can be approximately calculated as the linear weighted sum of the MIE of each component, the weighting factor is respectively the volume fraction of each component [9].

They also mention that, although the attempt to measure MIE by integrating the current/voltage is justified, the result still significantly depends on the choice of pulse shape, discharge gap, and even electrode material [9]. The experimental determination of this parameter for gases is not very usual, and is described only in one American standard [10]. On the contrary, in the case of dusts, the MIE determination is very common, and three reference documents [11], [12] and [15] provide several electric circuit descriptions and prescribe the different elements of the experimental device (e.g. modified Hartman tube, electrodes…) which should be used to obtain MIE values. That was extensively described and discussed by Janès et al. [23]. As these MIEs are obtained by the mean of standardized apparatuses and protocols, data can be compared and measurements can be reproduced.

The characterization of the sensitivity of gases to electric or electrostatic sources can also be evaluated by the determination of minimum ignition current (MIC), which correspond to the IEC 60079-11 standard [14] method. The existence of this standardized method can be considered as an advantage, comparing to the MIE determination of gases.

The sensitivity to the electric and electrostatic sources is one of the two aspects of gas grouping system prescribed in IEC 60079-20-1 standard [13], by means of determining the MIC ratio. This MIC ratio is calculated by dividing the MIC of the tested gas by the MIC of pure methane concentrated at 8.3 vol. % in air, as it is defined in IEC 60079-20-1 and IEC 60079-11 standards.

The second aspect is the maximum experimental safe gap (MESG). Concerning this last parameter, the HSL review [7], states that no data were available in the literature concerning MESG of methane/hydrogen mixtures. Experimental MESG data on these blends were published very recently by Askar et al. [16]. They found that similarly to the explosion regions with 10% hydrogen the MESG decreases only very slightly. However, the dependency is more linear for higher hydrogen fractions. If more than 25 % hydrogen is added, the MESG falls below 0.9 mm, which could lead to classify such a mixture in gas group IIB.

This classification system by group is intended to define, in the European Union, the specifications on electrical equipment implemented or used in the areas where potentially explosive atmosphere can be present, as it is required since 1999 by the European directive 1999/92/CE [21] and [22]. It

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is also a way to estimate the probability of ignition of pure gases or gases mixtures, by classifying their reactivity. The MIC ratio and MESG criteria prescribed in the IEC 60079-20-1 standard to assign a gas group are reported in Table 1.

Table 1: Criteria used for classification of gases in gas groups, from IEC 60079-20-1 standard [13].

Classification based on MESG	Group IIA: MESG ≥ 0.9 mm
	Group IIB: 0.5 mm < MESG < 0.9 mm
	Group IIC: MESG ≤ 0.5 mm
Classification based on MIC	Group IIA: MIC ratio > 0.8
	Group IIB: 0.45 ≤ MIC ratio ≤ 0.8
	Group IIC: MIC ratio < 0.45
Cases for which the determination both MESG and MIC values is required	Group IIA: 0.8 ≤ MIC ratio ≤ 0.9
	Group IIB: 0.45 ≤ MIC ratio ≤ 0.5
	Group IIC: 0.5 mm ≤ MESG < 0.55 mm

These criteria indicate that only MIC ratio value is sufficient to assign a gas mixture into a gas group in these cases:

•   Group IIA: MIC ratio > 0.9

•   Group IIB: 0.5 ≤ MIC ratio ≤ 0.8

•   Group IIC: MIC ratio < 0.45.

Concerning the case of methane/hydrogen mixtures, the IEC 60079-20-1 standard [13] gives the following statement to help assigning a gas to a group: industrial methane, such as natural gas, is classified as Group IIA, provided it does not contain more than 25 % by volume of hydrogen.

However, this 25 vol.% threshold seems relatively high, and the experimental measurement of parameters allowing methane/hydrogen blends (i.e. CMI ratio and MESG) was considered as relevant, to characterize more precisely the ignition sensitivity of these mixtures. As some data concerning MESG were recently provided [16], the main aim of the work reported in this paper is the experimental determination of MIC and MIC ratios, with respect to the MIC of pure methane, of methane/hydrogen mixtures up to 20 vol.% of hydrogen.

2.0   EXPERIMENTAL

2.1   Spark test apparatus and additional elements

The experimental device used consists of a spark test apparatus and an explosion chamber that is filled with an explosive test mixture, as showed in Figure 1. The experimental apparatus is compliant to Annex B of IEC 60079-11 standard [14].

The spark test apparatus consists of a contact arrangement in an explosion chamber. It is arranged to create sparks in the prescribed explosive test mixture. One of the electrodes consists of a rotating cadmium contact disc with two slots. The other contact electrode consists of four tungsten wires with a diameter of 0.2 mm clamped on a circle of 50 mm diameter to an electrode holder. The electrode holder rotates so that the tungsten contact wires slide over the slotted cadmium disc.

The distance between the electrode holder and the cadmium disc is 10 mm. The free length of the contact wires is 11 mm. the contact wires are straight and fitted to be perpendicular to the surface of the cadmium disc when not in contact with it. The electrode holder rotates at 80 r/min by an electric motor, while the cadmium disc is turned more slowly in the opposite direction.

A counting device allows recording the number of revolutions of the motor-driven shaft of the electrode holder. The driving motor is stopped automatically after an ignition of the explosive mixture, using a pressure switch.

The electric circuit connected to the spark test apparatus consists of a stabilized 24 V power supply, a 92.8 mH calibrated inductance and an adjustable resistance, which allows obtaining a current in the range of sought MIC values. The equivalent inductance of the adjustable resistance was insignificant compared to the main inductance.

The explosion chamber volume is 250 cm^3. It is continuously fed with the explosive test mixture, using two mass flowmeters. One flowmeter, calibrated to air, is used to control the air flow, whereas the other one controls the methane/hydrogen mixture flow. The methane/hydrogen mixture is directly provided by a cylinder of gas. That second flowmeter has been calibrated independently to methane and to hydrogen. The correction factor corresponding to each tested methane/hydrogen mixture has been adjusted by weighted linear interpolation, using the content of each of the two gases, from these two independent calibrations. The total gas flow feeding the explosion chamber was kept the same for all tests performed (i.e. 1.6 to 1.7 l/min).

A homogeneous mixture at the selected methane/hydrogen blend concentration in the explosive methane/hydrogen blend - air test mixture is obtained by applying a 3 min purge time before starting a test, which corresponds to about 20 times the volume of the gas chamber. An orifice on the explosion chamber is used to circulate the gas mixture, avoiding the pressure increase into the gas chamber during the test mixture feeding before the ignition.

Figure 1: View of the spark test apparatus.

2.2   Procedure for determination of the minimum ignition current

The IEC 60079-11 standard [14] is intended for the certification of electrical circuits by ignition tests using pure reference gases. Consequently, this standard doesn't describe a MIC measurement procedure for explosive test mixtures, but explains how the reference calibration spark test apparatus is implemented and the reference electric circuit. Concerning calibration tests intended for group I gases, the reference explosive test mixture identified in the IEC 60079-11 standard is methane/air at a concentration of 8.3 (±0.3) vol.% methane, considering that this concentration leads to the minimum ignition current. To be compliant to this standard, an ignition should occur in these conditions using an ignition current of 110 to 111 mA.

The current interval between the highest value at which no ignition was observed and the lowest value at which an ignition was observed is considered as the range of MIC. To be sure to be conservative, the MIC value was taken as the highest current for which no ignition was observed. By this way, no ignition could be observed with a current lower or equal to the MIC value obtained. This protocol was repeated for different concentrations of the explosive test mixtures, by 0.1 vol.% steps. This allows determining the optimal explosive gas concentration.

2.3   Material tested

The tests were carried out on test explosive mixtures constituted of methane/air, hydrogen/air or methane/hydrogen/air.

The oxidizing gas was provided by:

- Compressed air supply network operated at INERIS. Gas composite analysis conducted on a sample showed a moisture content less than 0.77 mol.%, an oxygen concentration of 20.4 (±0.9) vol.% and a hydrocarbon content less than 0.5 mg/L. This oxidizing gas was used during MIC measurements of pure methane only.

- Compressed air cylinder supplied by Air Liquide company, reference ALFAGAZ 1. Gas composite analysis conducted on a sample showed a moisture content less than 0.77 mol.% and a hydrocarbon content less than 0.5 mg/L. The oxygen content is guaranteed at 20 (± 1) vol.% by the supplier. This oxidizing gas was used during MIC measurements of methane and methane/hydrogen mixture containing 8 vol.% of hydrogen.

- Compressed air cylinder supplied by Air Liquide company, reference ALFAGAZ 2. Gas composite analysis conducted on a sample showed a moisture content less than 0.77 mol.% and an hydrocarbon content less than 0.5 mg/L. The oxygen content is guaranteed at 20.9 (± 0.2) vol.% by the supplier. This oxidizing gas was used during MIC measurements of methane and methane/hydrogen mixtures, excepted that containing 8 vol.% of hydrogen.

The analytical methods used were Karl-Fisher [19] for moisture content, X 43-290 standard [18] for hydrocarbon content and micro-chromatography for oxygen concentration in the case of compressed air supply network operated at INERIS.

The flammable gases were provided by:

- Compressed methane cylinder supplied by Air Liquide company, reference CH4 N35, with a purity greater than 99.5 vol.%. This gas was used for calibration of the spark test apparatus on methane and MIC measurements on methane.

- Compressed methane/hydrogen mixtures cylinders supplied by Air Liquide company, reference CRYSTAL, containing 2, 6, 8, 10, 12, 16, 18 and 20 vol.% of hydrogen. These gases were used for MIC measurements on methane/hydrogen mixtures.

2.4   Ambient conditions

All tests were performed in our laboratory, sheltered from sunlight and at room temperature which was recorded but not controlled. Its range was from 19 to 23°C, varying by no more than 2 K during the same day. It was experimentally verified that the temperature of the mixture tested in the explosion chamber is equal the room temperature. Concerning the pressure into the explosion chamber, considering the low tested mixture flow on one hand and the presence of an orifice on the explosion chamber on the other, it is not different to the atmospheric pressure.

The relative humidity was also recorded and not controlled. Its range was from 45 to 80 %, varying by no more than 10 % during the same day.

3.0   RESULTS AND DISCUSSION

3.1   Minimum Ignition Current of pure methane

Firstly, the optimal concentration of pure methane, which provides the lowest MIC value, was verified during tests performed the same day at 7.8, 8.3 and 8.8 vol.%. The results obtained demonstrate that the optimal concentration is equal to 8.3 vol.%, as it is indicated in the IEC 60079-11 standard [14].

These tests were carried out using as oxidizing gases 1) compressed air from INERIS internal network (containing 20.4 (±0.9) vol.% of oxygen) and 2) compressed air from ALFAGAZ 1 cylinder (containing 20 (±1) vol.% of oxygen). The results show a higher ignition sensitivity of methane in the case of compressed air from INERIS internal network.

The proposed hypothesis was the presence of compressor oil mist in the compressed air from INERIS internal network which could promote the ignition sensitivity of the explosive test mixture. Nevertheless, the analysis of hydrocarbons contained in the three oxidizing gases shows in all cases that hydrocarbon concentration doesn't reach the detection threshold of the test method used. Thus, a significant difference between the samples tested could not be determined.

As a second step, the variability of MIC of pure methane at the optimal concentration of 8.3 vol.% in air was studied, based on measurements performed on several days, and using three different oxidizing gases: 1) compressed air from INERIS internal network (containing 20.4 (±0.9) vol.% of oxygen) ; 2) compressed air from ALFAGAZ 1 cylinder (containing 20 (±1) vol.% of oxygen) and 3) compressed air from ALFAGAZ 2 cylinder (containing 20.9 (±0.2) vol.% of oxygen).

It demonstrates some variability in the MIC range obtained under the same test conditions, as well as an influence of the oxidizing gas used.

The evaluation of uncertainty was performed by dividing the standard deviation by the square root of the number of values. The multiplication factor was adjusted to the number of values according to the Student's t-distribution, as a function of the confidence interval.

Besides the oxidizing gas used, many other factors may influence the result of MIC measurement. The following could be mentioned: atmospheric pressure, attrition of the cadmium disc, attrition and surface oxidation of the tungsten electrodes. The influence of these parameters was not assessed in this work but will require further attention.

3.2   Minimum Ignition Current of methane/hydrogen mixtures

The MIC measurements were performed on each of the 10 methane/hydrogen mixtures containing from 2 to 20 vol.% of hydrogen. All tests were performed with compressed air from ALFAGAZ 2 cylinder (containing 20.9 (±0.2) vol.% of oxygen), except in the case of the mixture containing 8 vol.% of hydrogen, which was tested with compressed air from ALFAGAZ 1 cylinder (containing 20 (±1) vol.% of oxygen).

The MIC ranges obtained for all mixtures tested are represented in Figure 2.
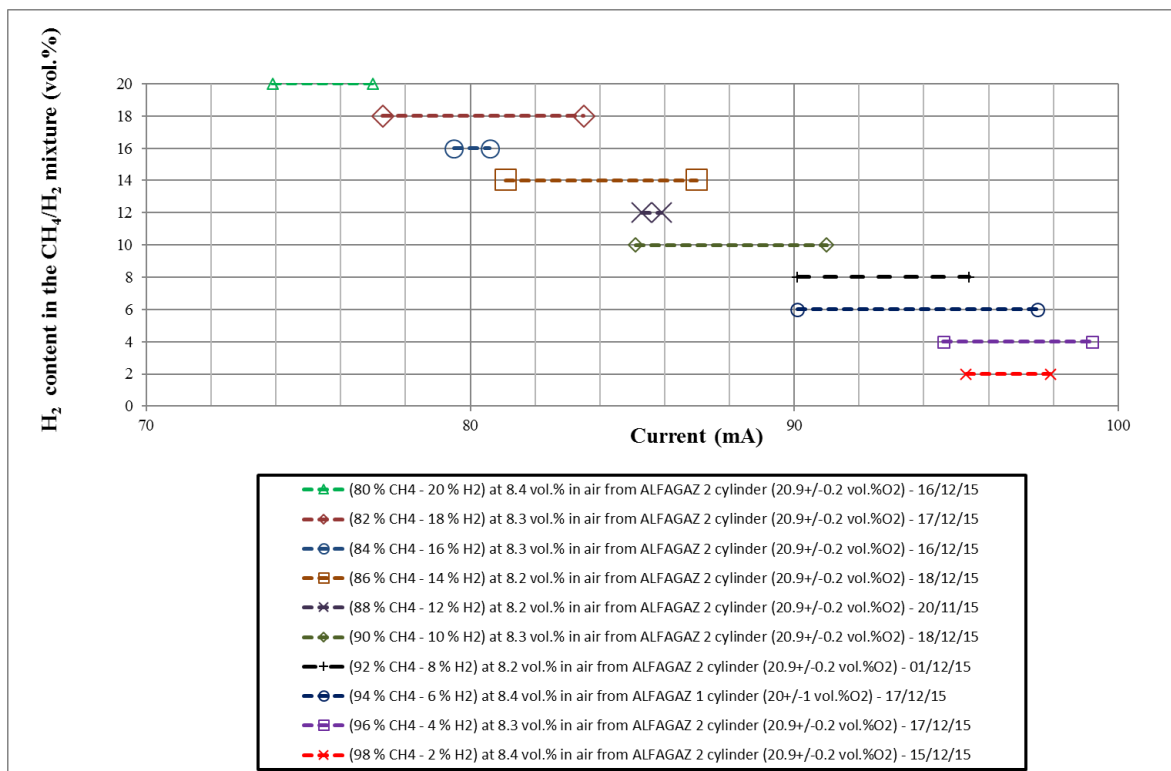
Figure 2: MIC ranges obtained on methane/hydrogen mixtures tested.

The optimal concentration, which leads to the lower ignition current, was reached by 0.1 vol.% steps. This value is close but lower than the accuracy associated to the mass flowmeters used, quantified at 0.25 vol.% for the methane/air mixture containing 8.3 vol.% of methane. Insofar, the accuracy of the concentration of flammable methane/hydrogen mixture in the explosive test mixture should be considered as 0.25 vol.%.

A linear least squares regression shows a very good correlation between the lower limit of the range of MIC of methane/hydrogen mixtures and the hydrogen concentration in the methane/hydrogen mixture.

This result appears to be in accordance with the fact that the MIE of a gas mixture can be approximately evaluated as the linear weighted sum of the MIE of each component, using volume fraction of each component as weighting factors [9]. The dependency of the MIC from the hydrogen fraction seems like that of MESG values, as was expected in [16]. Moreover, an evaluation of the MIE corresponding to the experimental MIC obtained during this work, using the theoretical energy amount stored in the inductance of the electric circuit of the spark apparatus used, was performed. It conduces, in the case of the mixture containing 20 % hydrogen, to theoretical ignition energy of 136 µJ. This appears in accordance with the MIE obtained by Hankinson [17], who reports a 0.1 mJ MIE in the case of a CH_4/H_2 mixture containing 25 % H_2.

This could be related to the fact that the MIC is linked to the activation energy of the explosive test mixture, which appears varying linearly with the hydrogen content.

Nevertheless, it should be mentioned that the current delivered by the spark apparatus isn't transferred completely to the test mixture, due to losses phenomenon during the sparking process. The fraction of theoretical spark energy consumed in test mixture ignition should be studied in more details. Uncertainty on the lower limit of the MIC range was considered equal to that on the MIC value on pure methane, obtained on the same day. This was assessed by dividing the standard deviation by the square root of the number measurement, i.e. 2. In this case, the multiplication factor corresponding to an 80 % confidence interval, according to the Student's law, it is equal to 3.08.

3.3   Minimum Ignition Current Ratio of methane/hydrogen mixtures

The MIC ratios were calculated for each mixture tested, obtained by dividing the MIC of each mixture tested by the mean value of two determinations of MIC of pure methane at 8.3 vol.% in air,

performed on the same day. The first MIC of pure methane was determined before testing the methane/hydrogen mixture(s), and the second after.

Indeed, many factors such as atmospheric pressure and ambient temperature, attrition of the cadmium disc, attrition and surface oxidation of the tungsten electrodes could influence the result of MIC measurement. As all these parameters are not controlled and their daily variability is expected lower than during all the test campaign, using a daily mean of MIC of pure methane is considered more accurate than an overall mean.
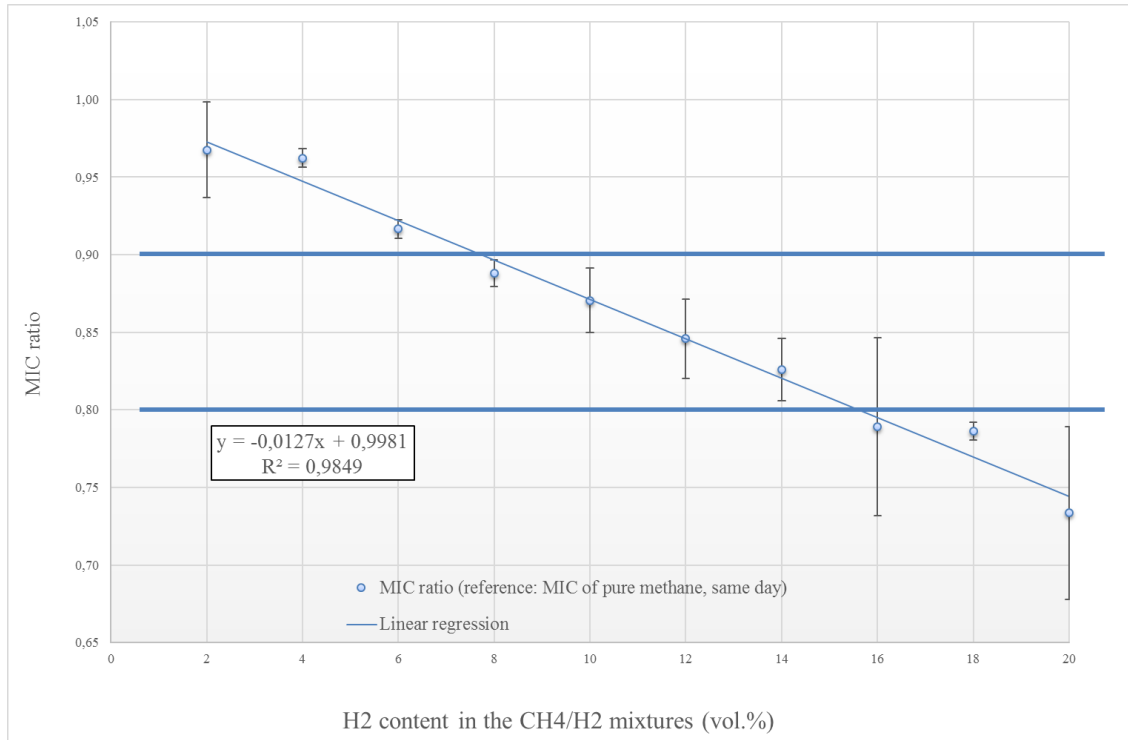
The results are shown in the Figure 3.

Figure 3: MIC ratio, as a function of hydrogen content in the methane/hydrogen mixtures.

A linear least squares regression shows again a very good correlation between the MIC ratio and the hydrogen content in the methane/hydrogen mixture.

Uncertainty on the lower limit of the MIC ratio was calculated using the logarithmic method. The partial uncertainty on MIC ratio was considered as the same that the partial uncertainty on pure methane obtained during the same day.

4. CLASSIFICATION IN GAS GROUPS AND CONCLUSION

The application of gas classification criteria in terms of MIC ratio from IEC 60079-20-1 standard [13] reproduced in Table 1 on results reported in this paper leads to the following classification:

- Methane/hydrogen mixtures containing 2, 4 and 6 vol.% of hydrogen: Group IIA, as MIC ratio is greater than 0.9,

- Methane/hydrogen mixtures containing 8, 10, 12 and 14 vol.% of hydrogen: the determination of MESG is necessary to conclude if these mixtures are classified in Group IIA or IIB, as MIC ratio is comprised between 0.8 and 0.9

- Methane/hydrogen mixtures containing 16, 18 and 20 vol.% of hydrogen: Group IIB, as MIC ratio is lower than 0.8.

This last result appears not to agree with the statement of IEC 60079-20-1 standard [13], which provides a classification in Group IIA up to 25 vol.% of hydrogen in a methane/hydrogen mixtures.

The same conclusion can be drawn by comparison to the recent results obtained by Askar et al., as they found that for less than 25 % added hydrogen, the methane/hydrogen mixture remains in explosion group IIA. They also established that at hydrogen fractions of more than 75 %, the mixture is allocated to explosion group IIC [16]

Thus, despite the results obtained in this work would conduce to consider methane/hydrogen mixtures containing 16 vol.% of hydrogen or more as Group IIB gases, other results lead to the conclusion that these mixtures would remain in Group IIA classification.

It should be added that all recent MIC and MESG experimental results on methane/hydrogen mixtures were discussed at the beginning of 2016 in the IEC 60079-20-1 standard maintenance group. These discussions lead to a last modification of the revision draft of the IEC 60079-20-1 standard which was in progress [20]. Thus, the range of MIC ratio values implying both determination of MESG and MIC ratio will be enlarged in the case of Group IIA classification. As it seems justified as the previous range was defined on the bases of pure substances behaviour, this adaptation avoids the mismatch highlighted in this paper, which appears during the data interpretation under the current criteria of the standard IEC 60079-20-1 [13]. Nevertheless, complementary experimentally work on MIC of methane/hydrogen mixtures, implying in possible different laboratories, is planned in a near future.

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