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Quality assurance for the tests to determine explosive parameters of the combustible dust-air mixtures

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Abstract. For any type of combustible dust, several important explosive parameters must be taken into account when designing and using protection systems: namely the ease with which dust clouds ignite and their burning rate, maximum explosion pressure, maximum pressure rise speed. of explosion. The accuracy of the results obtained necessary for the design and use of protection systems depends on a number of factors but also on the accuracy of the application of the test method (s) by the personnel involved. Test quality assurance is a requirement of EN ISO / IEC 17025 for the accreditation of testing laboratories. The standard requires laboratories to have a procedure in place to monitor the validity of test results, which involves participation in interlaboratory comparisons. This paper presents some specific issues highlighted during the successful participation of INSEMEX-GLI in several rounds of interlaboratory comparisons for the tests to determine explosive parameters of the combustible dust-air mixtures.

1.Introduction

Many technological processes involving combustible dusts, suspended or accumulated, have the potential to lead to fire, explosion or decomposition in the presence of oxygen and an ignition source. In spite of extensive research and development to prevent and mitigate dust explosions in the process industries, this phenomenon continues to represent a constant hazard to industries including manufacturing, using and handling of combustible dust material. For any type of combustible dust, several important explosive parameters must be taken into account when designing and using protection systems: namely the ease with which dust clouds ignite and their burning rate, maximum explosion pressure, maximum pressure rise speed. of explosion. The accuracy of the results obtained necessary for the design and use of protection systems depends on a number of factors but also on the accuracy of the application of the test method (s) by the personnel involved.

Test quality assurance is a requirement of EN ISO / IEC 17025 [1] for the accreditation of testing laboratories. The standard requires laboratories to have a procedure in place to

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monitor the validity of test results, which involves participation in interlaboratory comparisons.

According to ISO/IEC 17025 a laboratory shall have quality control procedures for monitoring the validity of tests and calibrations undertaken. This monitoring may include the participation in interlaboratory comparisons or proficiency testing programs. The data obtained from the monitoring process must be recorded in such a way that trends can be identified, they must be analyzed and when they are found to be outside the pre-set criteria, actions must be taken to correct the situation and prevent the reporting of incorrect results. Interlaboratory comparisons represent the organization, performance and evaluation of measurements or tests on the same or similar articles by two or more laboratories in accordance with pre-established conditions. Other means may include the regular use of reference materials, or replicate tests or calibrations using the same or different methods. By these mechanisms a laboratory can provide evidence of its competence to its clients, interested parties and the accreditation body [2].

The test laboratory group INSEMEX GLI is the main supplier of laboratory testing services for the INSEMEX OEC product certification body, being accredited by RENAR (accreditation certificate no. LI 347) on its competence to perform tests based on SR ISO / IEC 17025: 2018 requirements and accreditation criteria established by the Romanian Accreditation Association - RENAR.

The most important combustion characteristics for combustible dusts are, according to the series of standards SR EN 14034-1,2,3: Determination of explosion characteristics of powder clouds, [2, 3, 4]:

- Maximum explosion pressure Pex - the highest overpressure that occurs during an explosion of a powder mixture in a closed vessel;
- Maximum pressure rise rate (dP / dt) max - the maximum value of the pressure increase per unit time during explosions, for all explosive atmospheres in the explosive range of combustible substances in a closed vessel under the specified test conditions and atmospheric conditions standard;
- Severity explosion factor - K_{max}, K_{st} volume independent characteristic that is calculated using the cubic equation:

$$(\mathrm{dP}/\mathrm{dt})_{\max} \times \sqrt[3]{\mathrm{V}} = const. = K_{st} = K_{max} \quad (1)$$

-where V represents the volume of the test vessel, in our case the volume of the sphere is 20 liters.

The explosion and combustion characteristics of powders have to be known, in order to develop and adopt adequate preventive and protective safety measures.

2.The interlaboratory comparisons scheme for the tests to determine explosive parameters of the combustible dust-air mixtures

2.1 Scope

Determination of the explosion characteristics using standard test methods:
-maximum explosion pressure Pmax - the highest overpressure that occurs during an explosion of a powder mixture in a closed vessel;
-maximum pressure rise rate (dP / dt) max - the maximum value of the pressure increase per unit time during explosions, for all explosive atmospheres in the explosive range of

combustible substances in a closed vessel under the specified test conditions and atmospheric conditions standard;
-severity explosion factor - K_{max}, and calibration of the relevant test apparatus.

Inter-laboratory comparisons - Organization, execution and evaluation of measurements or tests on the same or similar items by two or more laboratories in accordance with predetermined conditions.

The organizer of the interlaboratory comparisons scheme was Cesana AG and TÜV SÜD Schweiz AG from Switzerland. Cesana AG and TÜV SÜD Schweiz AG named the program: "Calibration-Round-Robin" and the results were presented online in CaRo Final Report.

2.2 Reason and calibration method

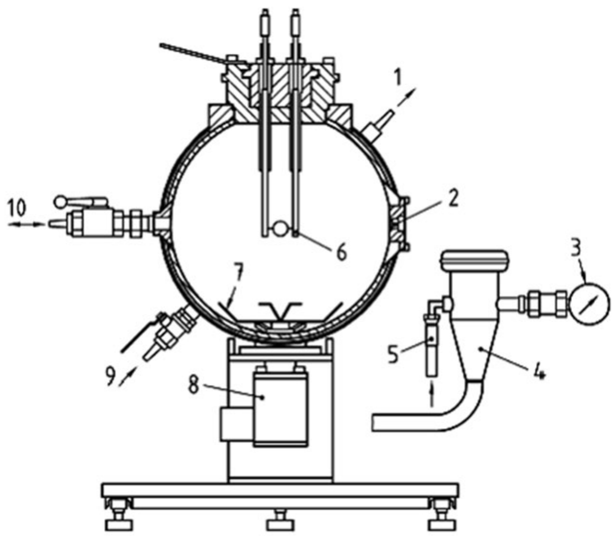
According to international standards, test equipment must be calibrated regularly by comparison with a standard or calibrated testing apparatus. This calibration also applies to the 20-l-apparatus for the determination of P_{max} and K_{max}. The test procedure is an important part of this calibration. A general check at the component level is incomplete and hence inadmissible. Unfortunately, there are neither internationally recognized reference samples nor reference equipment available for the determination of these explosion characteristics. Therefore, a calibration round robin test (CaRo) is carried out every two years.

The mean values of the explosion indices, measured by the participating laboratories, was calculated as reference values. All apparatuses, with results lying within the given tolerance range, meet the calibration requirements and will receive a calibration-certificate issued by the scheme organizer. Based on experience with previous round robin tests, the scheme organizer will be able to discover and rectify the cause of any errors with installations producing results deviating significantly from the reference values.

The test dust will be selected, homogenized, packed under nitrogen and mailed by us. Additionally, the scheme organizer supply with the test dust preliminary reference-results for the CaRo. Therefore, the participants will be able to pre-check their own test results. At the final, the scheme organizer will make a detailed overall evaluation with calculation of the standard values and the associated tolerance range. Those apparatuses which meet the calibration requirements will receive a certificate. All results will then be summarized in tabular form (anonymously) and mailed to the participants.

2.3 Testing installation – KSEP 20l and test substance

KSEP 20-l-sphere was used for measurement of the dust explosion and combustion characteristics. The test chamber is a hollow sphere made of stainless steel, with a volume of 20 liters. A water jacket serves to dissipate the heat of explosions or to maintain thermostatically controlled test temperatures. For testing, the dust is dispersed into the sphere from a pressurized dust container via the fast acting valve and a dispersing nozzle. The fast acting valve is pneumatically opened and closed by means of an auxiliary piston. The valves for the compressed air are activated electrically. The ignition source comprises two electrically activated pyrotechnical igniters, each having ignition energy of 5 kJ. This is the nominal calorimetric energy based on the mass of pyrotechnic powder in the igniters. When activated, the igniters release a dense cloud of very hot particles with little gas. The igniters are fired by electrical fuse heads. The power supply circuit for the igniters is capable of firing the fuse heads in less than 10 ms. The two igniters are placed at the center of the explosion vessel, firing in opposite directions. On the measuring flange two piezoelectric pressure sensors are installed [6]. The scheme of a chamber is shown in figure 1.



Keyword: 1-water outlet opening, 2-pressure transducers, 3-manometer, 4-dust recipient (0,6 dm^3), 5-air inlet opening, 6-ignition sources, 7-dispersing nozzle, 8-fast acting valve, 9-water inlet opening, 10-outlet opening (air, reaction products)

Fig. 1. - testing installation KSEP-20 type

Ignition of dust and dispersing of the dust is timed with dual digital timing relay. The relay has a fixed time interval between the opening of fast-opening valve and with connecting power to clamps of initiator. Time delay was set on 60 ms. **Niacin** USP special (Nicotinic acid) was used for the measured samples. Particle size of samples is shown in table 1.

Table 1. - Particle size of test substance

CaRo test substance - Niacin USP special (Nicotinic acid)			
Particle size	**d 10 [µm]**	**d 50=median [µm]**	**d 90 [µm]**
Sample 1	3,7	18,8	65,8
Sample 2	3,8	19,1	69,1
Sample 3	3,7	18,5	64,3
Sample 4	3,7	18,1	65,0

3. Results of inter-laboratory tests

Results obtained by INSEMEX GLI in 2017 (73 laboratories participated, 66 laboratories with a 20l test vessel and 7 laboratories with a 1m3 test vessel) CaRo17- for determination of the explosion characteristics of dusts are presented in the figure 2.

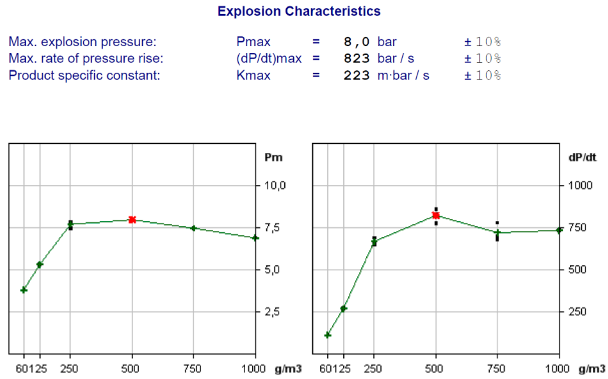


Fig. 2. – the results obtained by INSEMEX -GLI at CaRo17

The results of the inter-laboratory tests CaRo 17 [7] for Pmax are presented in figure 3 and for K_{max} in figure 4. The results of the laboratory within INSEMEX GLI are highlighted in yellow (number 12).

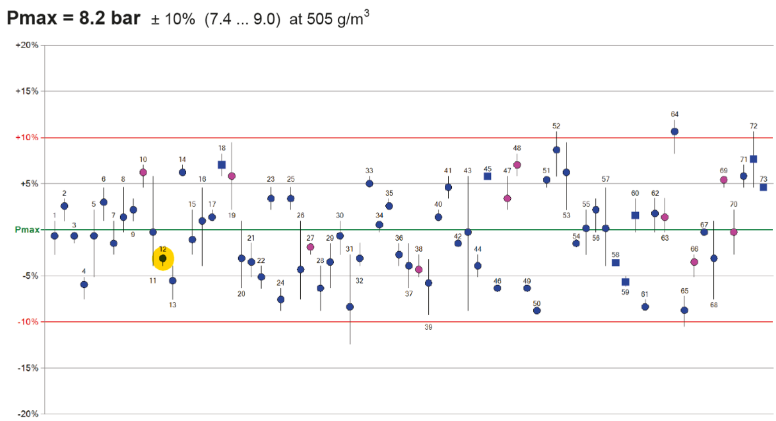


Fig. 3. - Results (as difference) for P_{max}. INSEMEX GLI laboratory at position 12

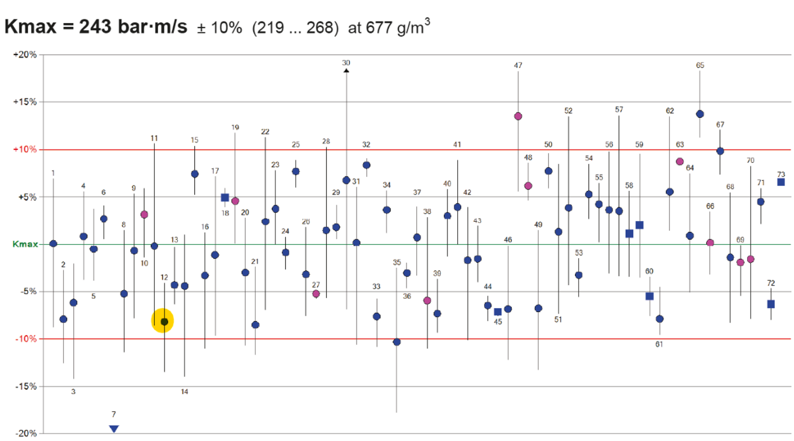


Fig. 4. - Results (as difference) for K_{max}. INSEMEX GLI laboratory at position 12

Results obtained by INSEMEX GLI in 2019 [8] (68 laboratories participated, 64 laboratories with a 20l test vessel and 4 laboratories with a 1m3 test vessel) CaRo19- for determination of the explosion characteristics of dusts are presented in the figure 5.

Explosion Characteristics

Max. explosion pressure:	Pmax	=	7,8 bar	± 10%
Max. rate of pressure rise:	(dP/dt)max	=	889 bar / s	± 10%
Product specific constant:	Kmax	=	241 m·bar / s	± 10%

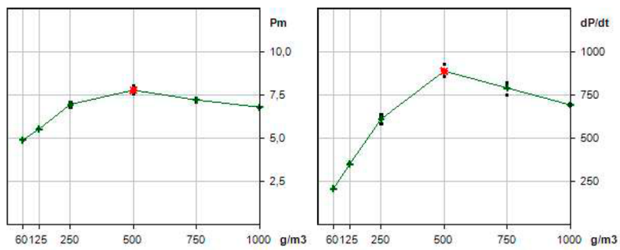


Fig. 5. – the results obtained by INSEMEX -GLI at CaRo19

The results of the inter-laboratory tests CaRo19 for Pmax are presented in figure 6 and for K_{max} in figure 7. The results of the laboratory within INSEMEX GLI are highlighted in yellow (number 35).

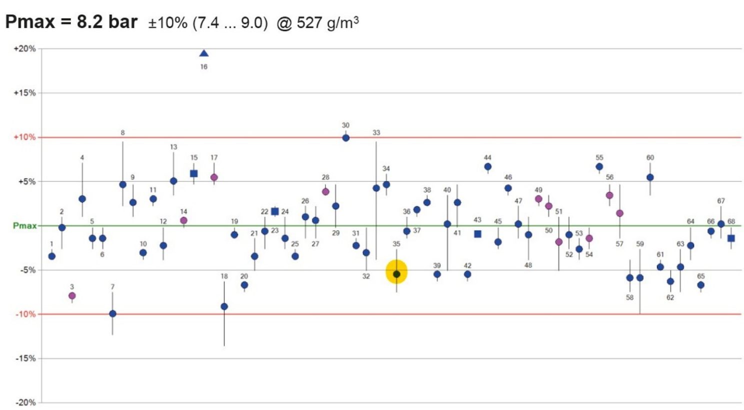


Fig. 6. - Results (as difference) for P_{max}. INSEMEX GLI laboratory at position 35

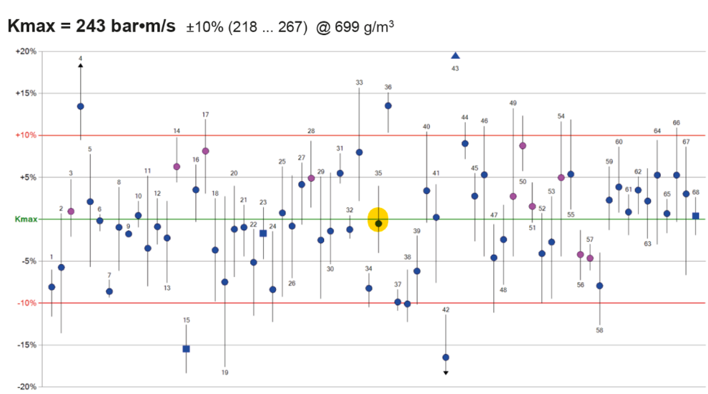


Fig. 7. - Results (as difference) for K_{max}. INSEMEX GLI laboratory at position 35

Results obtained by INSEMEX GLI in 2021 [9] (67 laboratories participated, 59 laboratories with a 20l test vessel and 8 laboratories with a 1m3 test vessel) CaRo21- for determination of the explosion characteristics of dusts are presented in the figure 8.

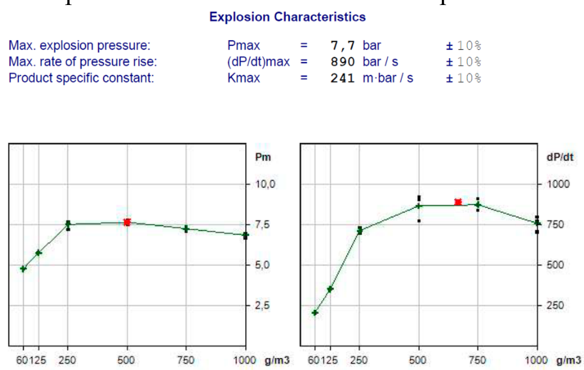


Fig. 8. – the results obtained by INSEMEX -GLI at CaRo21

The results of the inter-laboratory tests CaRo21 for Pmax are presented in figure 9 and for K_{max} in figure 10. The results of the laboratory within INSEMEX GLI are highlighted in yellow (number 36).

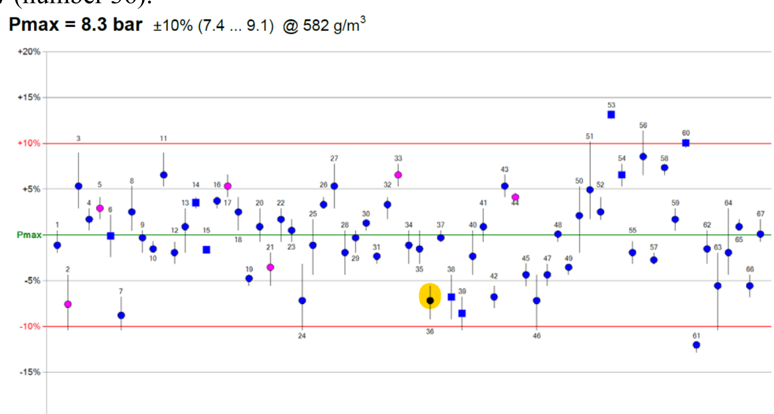


Fig.9 - Results (as difference) for P_{max}. INSEMEX GLI laboratory at position 36

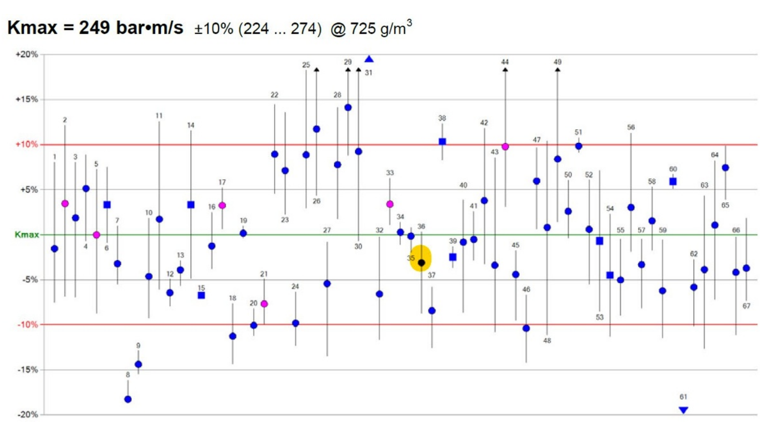


Fig.10 - Results (as difference) for K_{max}. INSEMEX GLI laboratory at position 36

4 Conclusion

Accurate determination of explosive parameters of the combustible dusts is of particular importance for the design and use of equipment and protective systems in environments with combustible dust. Therefore, testing laboratories must prove that they are correctly applying the standardised test methods. In this context, several laboratories participated in the calibration round organized by CESANA AG for determining the explosive parameters P_{max} and K_{max}.

After evaluating the results, there has been noticed that some of the laboratories do not fall within the reference range specified by the organizer of the interlaboratory tests. The most important thing is that INSEMEX GLI laboratory is not part of these laboratories. The results obtained by INSEMEX GLI for all three participations have fallen within the field of reference specified by the organizer of the rounds.

The various results obtained by the laboratories which have participated in the determination of the explosive parameters on the same type of dust, are due to the inaccurate application of the testing methods, and have been greatly influenced by the performances of the equipment used and by the competence of the involved personnel.

In order to ensure the quality of the test results for the determination of explosive parameters, it is essential to take into account all influencing factors such as: test atmosphere (temperature and relative humidity), human factor, measuring devices used, traceability of measurement, sampling and handling of the dust samples subject to testing.

Results obtained by INSEMEX GLI within the interlaboratory comparison tests have demonstrated the technical and professional competence of the laboratory, the capacity to ensure a quality control when monitoring the validity of the tests, an essential condition in maintaining the laboratory accreditation for performing tests to determine explosive parameters.

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