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DOI

[10.1016/j.jlp.2022.104947](https://doi.org/10.1016/j.jlp.2022.104947)

Publication date

2023

Document Version

Final published version

Published in

Journal of Loss Prevention in the Process Industries

Citation (APA)

Spitzer, S. H., Askar, E., Benke, A., Cloney, C., D'Hyon, S., Dufaud, O., Dyduch, Z., Gabel, D., Taveau, J., & More Authors (2023). 1st international round robin test on safety characteristics of hybrid mixtures. *Journal of Loss Prevention in the Process Industries*, 81, Article 104947. <https://doi.org/10.1016/j.jlp.2022.104947>

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1st international round robin test on safety characteristics of hybrid mixtures

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ARTICLE INFO

Keywords:

Hybrid mixtures

20L-sphere

Round robin test

Turbulent combustion

ABSTRACT

There is no applicable existing standard for the determination of safety characteristics for hybrid mixtures. While developing a new standard in a joint research project in Germany first results from parameter studies led to a standard procedure that can be adopted by laboratories that are already testing dusts in the so called 20L-sphere with as little additional effort as necessary. In fact, one of the main objectives of this research project was to keep modifications and adjustments from the generally accepted dust testing procedures as easy and minimal as possible so as to limit potential deviations from one laboratory to another.

In this first round robin test on hybrid mixtures ever, with methane as gas component and a specific corn starch as dust sample, the practicality of the whole procedure, the scattering of the results and the deviation between the testing apparatuses is investigated. This paper summarizes the experimental procedure adopted and objectives of the first round-robin phase involving three of the four original German companies, plus volunteering laboratories from Australia, Belgium, Czech Republic, France, Poland and P.R. China. The results will have an impact on the new standard and may lead to robust data for later simulation purposes.

1. Introduction

Safety characteristics are used to plan processes in a way that they run under safe conditions. To obtain the safe handling of a plant it is either possible to avoid explosible mixtures or ignition or to mitigate the consequences of possible explosions. So far it was just possible to determine safety characteristics for single phase substances or mixtures (gaseous, liquid or solid)² but not if, for example, a process is run

with a combustible dust and a flammable gas component. The different standards were referring to the safety characteristics differently (lower explosion limit for gases, minimum explosible concentration for dusts) or they are characterized in different ways but called the same. The maximum explosion pressure p_{max} for dusts is the average value of three tests at the concentration with the highest obtained explosion pressures. The same characteristic for gases is the highest value of five tests at the

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¹ Facilities and all other authors in alphabetical order.

² ISO6184-3 (1985) is officially under revision since 2005 and with 4 pages too short to be applicable. None of the facilities, that were contacted referred to that standard and only few knew about it.



Fig. 1. Map with the facilities involved in the round robin test (CC BY-SA 3.0 <https://commons.wikimedia.org/wiki/User:Alexrk2>, image cut and marked by the corresponding author).

concentration with the highest obtained explosion pressures. The same is true for the maximum rate of pressure rise $(dp/dt)_{max}$: It is called the same for dusts and gases but it is stated once as an average, the other as the maximum of several tests. So, the confusion starts before even determining the safety characteristics. Other differences complicate both, a comparison between the different safety characteristics and a connection between the different ones, even further:

- The step-sizes are defined in different ways
- Gases are tested under quiescent conditions, dusts under turbulence
- The values for p_{max} for gases and vapors are stated in absolute pressures, the ones for dusts in relative pressure
- The values for p_{max} for dusts are further corrected when determined in the 20L-sphere but not for gases
- Different ignition sources are used
- The ignition energies differ with 2 J to 5 J for gases and liquids and 2 kJ or 10 kJ for dusts
- An “upper explosible concentration” is not determined for dusts

For all these reasons, safety characteristics of hybrid mixtures were so far just qualitative characteristics and a comparison between values from different sources was somewhere between difficult and impossible.

In the extensive literature survey about hybrid mixtures only in four of them the gas amount was verified but none of them stated how big the deviation and scattering was Schubert (1987), Ishihama et al. (1979), Pellmont (1979) and Singer (1964). Furthermore, none of them used the 20L-sphere or the 1 m³ so the impact of the fast

compression and the dynamic tests on the partial pressure method was not investigated so far as well.

On a national basis a new approach to a standard for hybrid mixtures was developed in Germany. Four institutions, Physikalisch Technische Bundesanstalt (PTB), Bundesanstalt für Materialforschung und -prüfung (BAM), Otto-von-Guericke University (OVGU) and Inburex Consulting, were investigating several important parameters that can be varied according to the different standards, aimed for precision while keeping the complexity at a feasible level and started an international round robin test to see, how applicable their new approach was.

The following test facilities submitted their data so far (see also Fig. 1)

- Physikalisch-Technische Bundesanstalt, Braunschweig, Germany
- Bundesanstalt für Materialforschung und -prüfung, Berlin, Germany
- Otto-von-Guericke University, Magdeburg, Germany
- VSB — Technical University of Ostrava, Ostrava, Czech Republic
- Adinex NV, Noorderwijk, Belgium
- The University of Lorraine, Nancy, France
- Ineris, Verneuil en Halatte, France
- GIG Research Institute, Mikolow, Poland
- Simtars Sponcom, Redbank, Australia
- The University of Shenyang, Shenyang, P. R. China
- Suzhou EnvSafe Test Co. Ltd, Suzhou, P. R. China

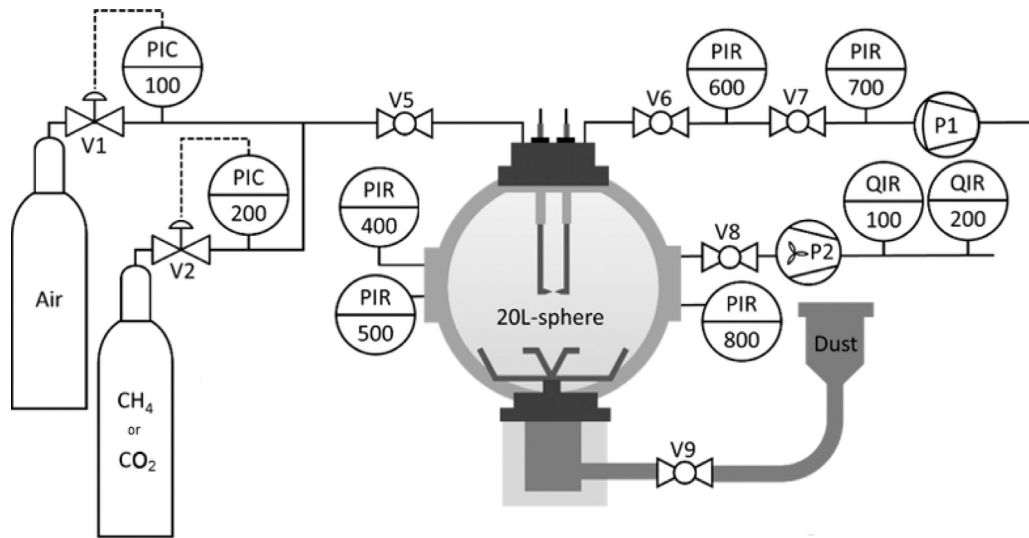


Fig. 2. 20L-sphere setup at BAM for hybrid dust gas mixtures with additional absolute pressure sensor (PIR 600 with one bar absolute or PIR 800 with 10 bar absolute resolution), gas analyzer (QIR 100 and QIR 200) and pump (P2).
Source: Taken from Spitzer et al. (2022b).

2. Experiments

An operating procedure for both the 20L-sphere can be found on the BAM-website (Spitzer, 2021). Before the experiments started an additional absolute pressure sensor had to be installed to the test vessel. This pressure sensor is used while filling the flammable gas into the sphere and the values are used to calculate the concentration of gas (partial pressure method). To verify, whether the calculated concentration of gas matches the real amount of gas added, a gas analyzer with a pump was also installed (see Fig. 2).

Two pre-test series are necessary before starting the hybrid explosion tests: First, a leakage test is conducted to see, how airtight the test vessel is. This step is crucial, if hybrid mixtures are tested because the accuracy of the fuel gas fraction depends on the accuracy of the absolute pressure sensor, the constant pre-ignition pressure rise just before the ignition is triggered and the tightness of the vessel avoiding additional introduction of atmospheric air into the test vessel during the filling procedure. A leakage-rate of lower than 1 mbar/minute was allowed in this test series.

Afterwards a test-series to evaluate the accuracy and scattering of the fuel gas fraction was conducted. For these tests, no ignition source was placed in the test vessel and no dust was added to the dust container. It was allowed to replace the flammable gas by another gas for safer handling. The tests were otherwise conducted as normal dust tests with the pressurizing of the dust container and the injection from the dust container to the test vessel. All the pressures during this process were recorded and after the injection the pressure was measured for another three minutes before the valve leading to the gas analyzer and the pump was opened. This is necessary because there is a pressure-drop after injecting the air due to the increased temperature because of the fast compression (post-injection pressure drop, PIPD, see Fig. 3). Normally the ignition takes place at the peak pressure. The pressure drop leads to wrongly calculated gas concentrations if it is not taken into account. Due to the explosion this pressure drop after equilibration cannot be recorded in the regular explosion tests. So the values obtained in the pre-tests were averaged. The average values were used to calculate the fuel gas fractions in the regular explosion tests.

If the measured amount of gas was more than 0.2 mol% lower than the calculated amount of gas the partial pressure of the gas was increased and vice versa.

When both, the vacuum test and the validation of the gas mixture, were successful a dust sample was sent to the facilities. The dust

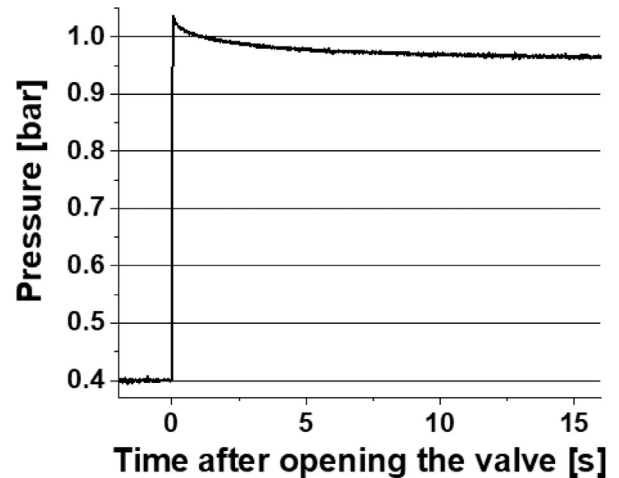


Fig. 3. Recorded pressure against time after opening the fast-acting valve without igniter and without dust.

Source: (Taken from Spitzer et al. (2022a)).

samples were checked on moisture content before dispatch. The particle size distribution was also checked occasionally.

The experimental procedure for the hybrid explosion tests was derived from the European standard for the determination of safety characteristics of dusts (EN14034-1 (2011), EN14034-2 (2011), EN14034-3 (2011) and EN14034-4 (2011)). The amount of gas was filled to the sphere and the molar fraction was calculated according to Eq. (1).

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

with c_{gas} as the fraction of the gas in mol%, p_{gas} the partial pressure of the gas filled to the sphere, PV (partial vacuum) as the pressure in the sphere before the injection of the dust, $PIPR$ (pre-ignition pressure rise) and the averaged post-injection pressure drop $PIPD$ from the pre-tests.

If the measured amount of gas in the pre-tests was differing from the calculated one systematically, Eq. (1) was adjusted to Eq. (2).

$$c_{gas} = p_{gas} / (PV + PIPR - PIPD) - Deviation \quad (2)$$

The following additional modifications and changes were made:

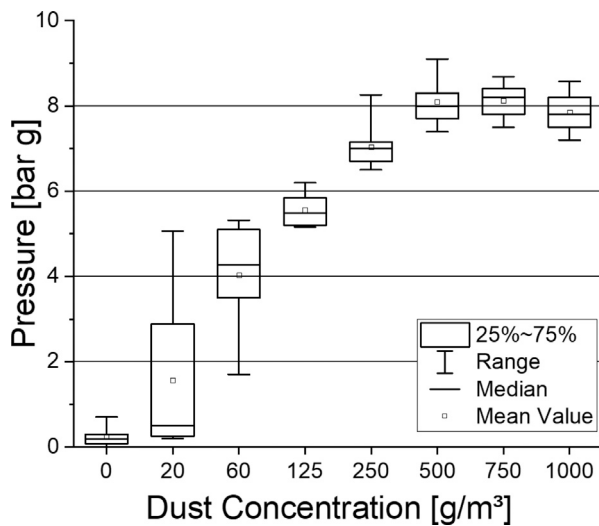


Fig. 4. Highest explosion pressures of corn starch with 3 mol% methane.

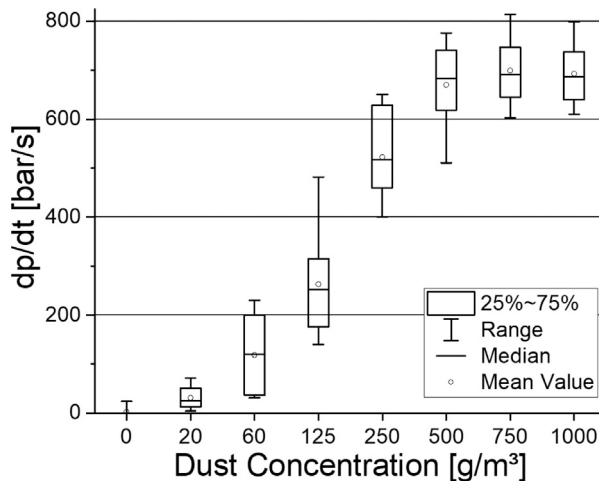


Fig. 5. Highest rates of pressure rise of corn starch with 3 mol% methane.

- For all experiments two 1 kJ pyrotechnical igniters were taken
- The pressure in the sphere before the dust injection starts was kept at 400 mbar abs $\pm$ 2 mbar
- The Pre-Ignition pressure rise was kept constant at 0.64 bar $\pm$ 0.02 bar

The methane was obtained by the facilities and it was required, that is has a purity of greater than 99 mol%.

The dust was tested in the concentrations of 0 g/m³, 20 g/m³, 60 g/m³, 125 g/m³, 250 g/m³, 500 g/m³, 750 g/m³ and 1000 g/m³ with the addition of 0 mol%, 3 mol% and 9 mol% of methane. At the points were the highest explosion pressures and pressure rises occurred the tests were repeated twice.

The following requirements for hybrid gas dust explosion tests were explicitly not defined:

- Gas analyzing system — The facilities used different kinds like Infrared, catalytic or gas chromatography with a coupled mass spectrometry, the resolution was demanded to be at least 0.1 mol%
- Amount of pressure sensors — Though in the dust standard two pressure sensors for measuring the explosion pressure are demanded, one research laboratory had just one sensor

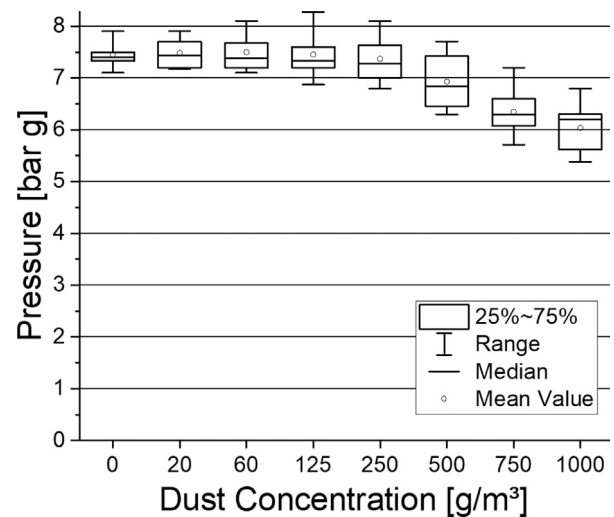


Fig. 6. Highest explosion pressures of corn starch with 9 mol% methane.

- Type of pressure sensor — Though in the standard test equipment piezo-electric sensors are used, one research laboratory used piezo-resistive pressure transmitters
- Measuring frequency — The measuring frequency should be defined in a later standard, most facilities had 20 kHz except for one with 5 kHz
- Pressurized air — Some laboratories had synthetic air, some fully dried compressed and some normal compressed, this should be specified in the later standard because this might have an impact
- Distribution system — Most facilities use a rebound nozzle today, but some use a mushroom shaped nozzle or a perforated dispersion ring

Shortly before the dispatch of the dust samples the moisture content was measured. Afterwards the dust was filled in bottles and sealed. The receiving facilities were asked to open the dust samples shortly before conducting the hybrid explosion tests and to check for the moisture content again. This way we could attest each other, that the samples were not damaged on the way. The moisture content we tested over the whole time was between 7 weight% and 9 weight%. The particle size distributions were tested every three months optically (MALVERN Mastersizer 2000). The 10th percentile ($d(0.1)$), the median value ($d(0.5)$) and the 90th percentile ($d(0.9)$) were $(9 \pm 1) \mu\text{m}$, $(13 \pm 1) \mu\text{m}$ and $(21 \pm 2) \mu\text{m}$.

The results from all the test facilities were averaged, if several results were sent for one point according to the dust standard. The presented values for the explosion pressure were corrected with the following Eqs. (3) and (4), derived from the standard for dusts, ASTM1226a (2012) and EN14034-1 (2011).

If the measured overpressure was below 5.5 bar g:

$$p_{\max} = 5.5 * (p_{\max,20L} - 0.32) / (5.18) [\text{bar g}] \quad (3)$$

If the measured overpressure was above 5.5 bar g:

$$p_{\max} = 0.775 * p_{\max,20L}^{1.15} [\text{bar g}] \quad (4)$$

The test was conducted as a single-blind test, the facilities were informed afterwards about the results from the other facilities.

3. Results and discussion

The results for $p_{\max}$ for 3 mol% of methane from the different facilities are shown in Fig. 4. It shall be mentioned, that only half of the facilities checked the value of 20 g/m³ because it is very unusual to perform dust tests with this little amount of dust. The scattering between the different facilities is small for the highest values but for the

determination of the LEL respectively the MEC this procedure seems to have too many degrees of freedom. For 3 mol% of methane and 20 g/m³ corn starch half of the institutions, that checked this amount, detected an explosion, one with an explosion pressure of 5 bar g, while the other half could not measure one (p_{Ex} being under 0.3 bar g). Two facilities even detected a very slight explosion with a p_{Ex} value of 0.7 bar g and 0.5 bar g with no dust at all. For the aimed characteristic p_{max} this method showed comparable results with a scattering of less than 10%. The results for $(dp/dt)_{max}$ for 3 mol% of methane from the different facilities are shown in Fig. 5. The scattering between the different facilities is less than 20% for the highest values. The highest values were also obtained by all facilities at the same concentration of 750 g/m³ or one step up or down. This could be an effect of different distribution systems, that were not specified in the operating procedure. The results for p_{max} for 9 mol% of methane from the different facilities are shown in Fig. 6. The scattering for all tested concentrations was 11% and thus lower than the ones for p_{max} tested at 3 mol% with an increasing tendency with increasing dust concentration (below 5% scattering with no dust, 9% at the highest point with 60 g/m³ and 11% at the end with 1000 g/m³). It should also be mentioned, that all the values are below the values of the dust tested alone. This may be caused by the fact, that with 9 mol% of methane the oxygen amount is already decreased to 19 mol% instead of 20.9 mol%. The results for $(dp/dt)_{max}$ for 9 mol% of methane from the different facilities are shown in Fig. 7. Except for three facilities all others measured the highest value of (dp/dt) with small amounts of dust added. This exception occurred at facilities with a self-written testing program and could be caused by one or more of the following reasons:

- The distribution system was not specified
- This facility took synthetic air
- The effect is with 10% about the same magnitude as the scattering and thereby hard to spot anyways
- The ignition delay time is defined on an event-basis³ in the different standards and could have an impact, if it is implemented otherwise

However, the highest values of p_{max} found by the different laboratories were still within two steps apart from each other at the very fine scale (it shall be pointed out, that the x-Axis in all plots is not linear).

The overall results are displayed in Table 1. The scattering for $(dp/dt)_{max}$ was 21%, for p_{max} it was 8%. This can be seen as reasonable considering, that it is higher than for gas testing alone where the scattering for methane is stated with 3.6% for p_{max} and 3.1% for $(dp/dt)_{max}$ (EN15967, 2011) but of the same magnitude as for dusts alone with 10% for p_{max} (ASTM1226a (2012) and EN14034-1 (2011)) and 20% for $(dp/dt)_{max}$ (ASTM1226a, 2012) even though more parameters must be considered for hybrid mixtures compared to dusts alone. In the European standard for the determination of $(dp/dt)_{max}$ for dusts the allowed scattering is depending on the value (EN14034-2, 2011). Above 200 bar/s a scattering of 10% is allowed which is lower than the one of this procedure, but still of a comparable magnitude. The decreasing value for p_{max} with increasing fuel gas fraction may be an effect of the decreasing amount of oxygen in the vessel. Some

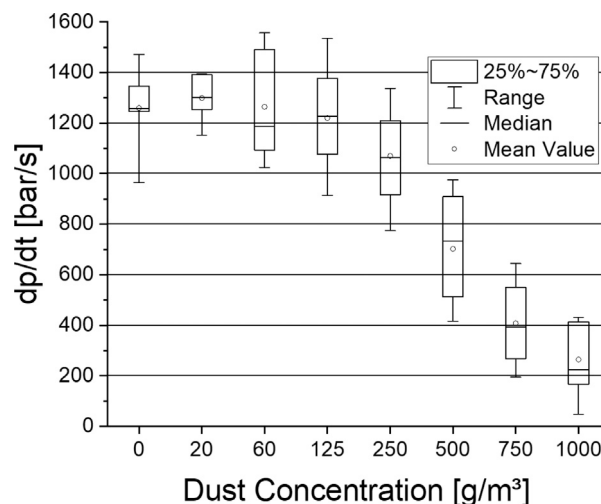


Fig. 7. Highest rates of pressure rise of corn starch with 9 mol% methane.

Table 1

Overall results for the different concentrations of methane and corn starch.

Methane mol%	$(dp/dt)_{max}$ bar/s		p_{max} bar g	
0	540 ± 120	±22%	8.5 ± 0.7	±8%
3	705 ± 108	±15%	8.2 ± 0.7	±8%
9	1291 ± 265	±21%	7.5 ± 0.6	±7%

Table 2

Values of p_{max} and $(dp/dt)_{max}$ for methane and hydrogen tested under turbulence with two 1 kJ chemical igniters, a partial vacuum of 400 mbar ±2 mbar, a PIPR of 0.64 bar to 0.68 bar and corrected with Eq. (4).

Gas	Conc. [mol%]	p_{max} [bar g]	$(dp/dt)_{max}$ [bar/s]	K_{st} -value [bar * m/s]
Methane	9.5	7.8 ± 0.5	1450 ± 150	394 ± 41
Hydrogen	30	6.6 ± 0.4		
	35		3750 ± 450	1018 ± 122

of the parameters that were not specified yet might be narrowed in the upcoming standard. However, taking into account that all the parameters for the determination of the safety characteristics of hybrid mixtures of gases and dusts have an impact the variation observed in the tests is reasonable. It is of the same magnitude than the variation that is defined in the standard for dusts.

An alternative solution for the fast implementation and validation of hybrid equipment is the comparison with well-known and often investigated values of gases tested under turbulence. A proposal are the following values for the verification of the pressures and the right amount of gas listed in Table 2.

The leakage-rate and the gas analysis would still have to be checked. To ensure the right handling and pre-tests a certificate like the following could be used. It would be either filled out by the company itself or checked from an external company (see Fig. 8).

4. Conclusions

A test method for the reliable, reproducible and simplified determination for the safety characteristics p_{max} and $(dp/dt)_{max}$ of hybrid dust gas mixtures was developed. Eleven facilities in seven countries were able to adjust their dust testing equipment and conduct hybrid explosion tests. The results showed, that the procedure works, leads

³ The ignition delay time is defined as the time between the first measurable pressure rise inside the sphere until activation of the ignition source (ASTM1226a (2012), EN14034-1 (2011), EN14034-2 (2011), EN14034-3 (2011) and EN14034-4 (2011)). This event-based definition is often misunderstood as the time between activation of the fast-acting valve and activation of the ignition source (descriptive definition). Since the ignition source takes 0–10 ms to ignite and the fast-acting valve takes 20 ms–50 ms to open this is the main source of error if equipment is self-built.

20L-sphere at the FACILITY in CITY, COUNTRY

Round Robin

Pre-Test 1: Leakage Check Target / Actual $< 1 \frac{\text{mbar}}{\text{minute}} / \frac{\text{mbar}}{\text{minute}}$	Pre-Test 2: Gas Analysis Target / Actual $< \pm 0,2 \text{ Vol.-%} / \pm \text{ Vol.-%}$
Explosion Test FIRST TESTED CONC. % GAS Overall Average (NUMBER OF facilities) / Your facility / Deviation $\frac{\text{bar}}{\text{s}} \pm \frac{\text{bar}}{\text{s}} / \frac{\text{bar}}{\text{s}} / \frac{\text{bar}}{\text{s}} (\text{ \% })$ $\text{bar g} \pm \text{bar g} / \text{bar g} / \text{bar} (\text{ \% })$ Second	
Explosion Test SECOND TESTED CONC. % GAS Overall Average (NUMBER OF facilities) / Your facility / Deviation $\frac{\text{bar}}{\text{s}} \pm \frac{\text{bar}}{\text{s}} / \frac{\text{bar}}{\text{s}} / \frac{\text{bar}}{\text{s}} (\text{ \% })$ $\text{bar g} \pm \text{bar g} / \text{bar g} / \text{bar} (\text{ \% })$	

Fig. 8. Proposal for a certificate that could be handed out with the test results.

to comparable results for $p_{\max}$ and $(dp/dt)_{\max}$ respectively the K_H -value. The variation between the different facilities for these safety characteristics was higher than for the safety characteristics of gases according to the standards, but of the same magnitude given in the standards for safety characteristics of dusts.

Especially the leakage-check of the test vessel before starting any test procedures and verification of the gas amount without ignitors and dust before conducting the actual explosion tests are crucial to obtain comparable results. Both was not conducted (or at least not stated) in the literature about hybrid mixture testing in the 20L-sphere. These two pre-tests will be inserted in the future standard and should be mandatory for everyone conducting hybrid tests.

The addition of very small amounts of dust is also a crucial step if the $(dp/dt)_{\max}$ is searched for and was not often performed before.

CRediT authorship contribution statement

Stefan H. Spitzer: Writing – original draft, Conceptualization, Investigation, Formal analysis, Validation, Software, Data curation. **Enis Askar:** Supervision, Writing – review & editing, Methodology. **Alexander Benke:** Investigation, Methodology. **Chris Cloney:** Supervision. **Sebastian D’Hyon:** Investigation. **Olivier Dufaud:** Investigation, Writing – review & editing. **Zdzislaw Dyduch:** Investigation. **Dieter Gabel:** Investigation, Resources. **Paul Geoerg:** Investigation,

Resources. **Vanessa Heilmann:** Investigation. **Vojtech Jankuj:** Investigation, Validation. **Wang Jian:** Investigation. **Ulrich Krause:** Supervision, Project administration, Funding acquisition. **Arne Krietsch:** Supervision, Project administration. **Miroslav Mynarz:** Investigation. **Frederik Norman:** Investigation. **Jan Skrinsky:** Investigation. **Jerome Taveau:** Supervision. **Alexis Vignes:** Investigation. **Sabine Zakel:** Supervision, Project administration. **Shengjun Zhong:** Investigation.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Stefan Spitzer reports financial support was provided by BAM Federal Institute for Materials Research and Testing.

Data availability

Data will be made available on request.

Acknowledgments

The authors gratefully acknowledge the corporation of our dust testing lab, Mike Wappler and Dr. Martin Schmidt, for their help and Sara Salih for conducting the tests in BAM. This work was funded by the Federal Ministry for Economic Affairs and Energy under the support code 03TNH006C.

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