

Calibration-Round-Robin CaRo25

Final Report

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1. Conclusion

According to international standards the 20-l-apparatus and the 1-m³-vessel for the determination of Pmax and Kmax and the apparatus for determination of the minimum ignition energy must be calibrated at regular intervals (at least every 12 months, or following any significant maintenance or repair).

For this purpose, an international calibration round test (calibration round robin = CaRo) has been carried out periodically since 1993.

A dust has been selected, prepared and supplied to **46** test laboratories all over the world.

The mean values of the explosion indices, measured by the participating laboratories, have been calculated as reference values. The testing laboratories have been informed about the evaluation with a certificate.

This report presents the results of this calibration method and describes the evaluation procedure.

CaRo25 – Reference values for the Explosion Indices Pmax, Kmax, LEL/MEC and LOC

Pmax	[bar]	8.2 ±10% (7.4 ... 9.0)
Kmax	[bar·m/s]	251 ±10% (226 ... 276)
LEL (CEN)	[g/m ³]	41 (30 ... 55)
MEC (ASTM)	[g/m ³]	43 (35 ... 48)
LOC	[O ₂ %]	11.0 (9 ... 12)

CaRo25 – Reference values for the Minimum Ignition Energy MIE

Es / 3	Es	Es • 3
0.6 mJ	1.7 mJ	5.1 mJ

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1.1 Participants:

Further details about participants who have agreed to a publication, can be found in section 4.

	Pmax, Kmax (71)		MZE (49)	
	20-l	1 m ³	MIKE	others
Australia	1		1	
Austria	1			
Belgium	2		1	
China	2			2
Czech Republic	1		1	
France	5		3	1
Germany	18	3	10	1
Hungary		1		1
India			1	
Italy	1		1	
Japan			1	
Norway	1		1	
Poland	2			1
Romania	2			1
Slovakia	1			1
Spain	2		1	
Sweden	1			1
Switzerland	1		4	
The Netherlands	1		1	
United Kingdom	4		1	1
USA	19	2	8	4
Total:	65	6	35	14

1.2 Test substance:

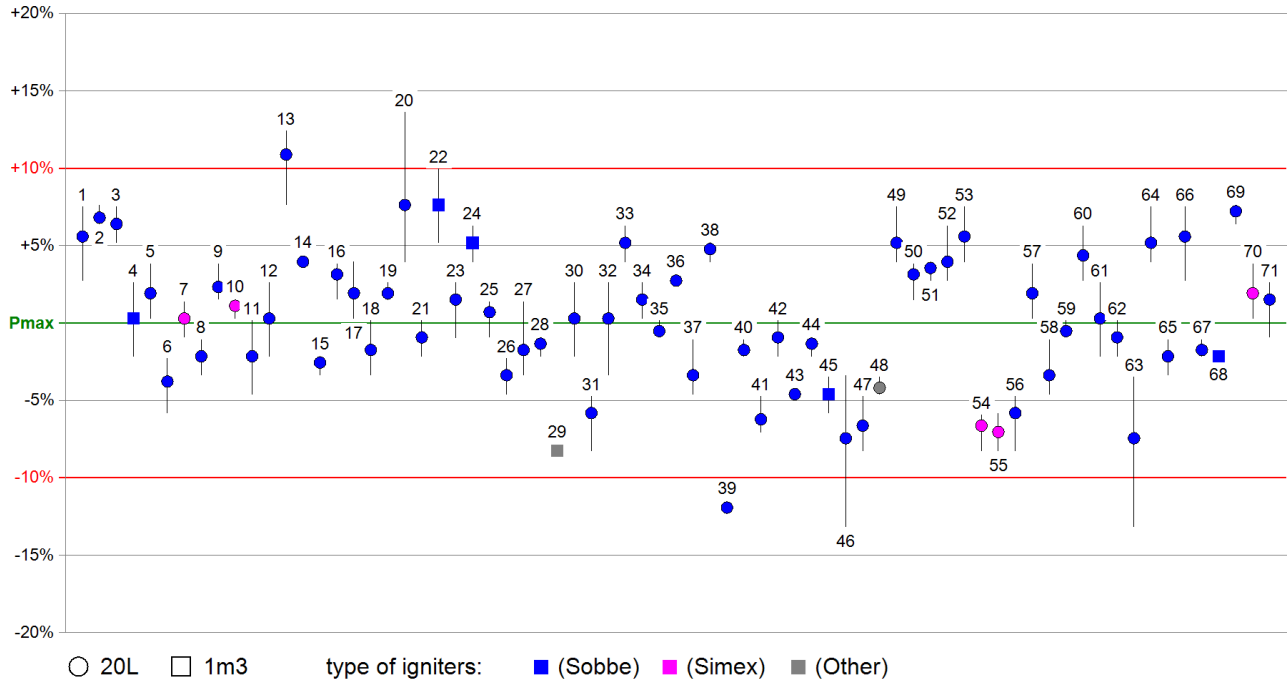
For correct calibration the CaRo25 test sample has been milled, homogenized, packed under argon and shipped in an air tight package. Therefore the sample has to be tested „as delivered“.

CaRo25 = Niacin USP (Nicotinic acid)

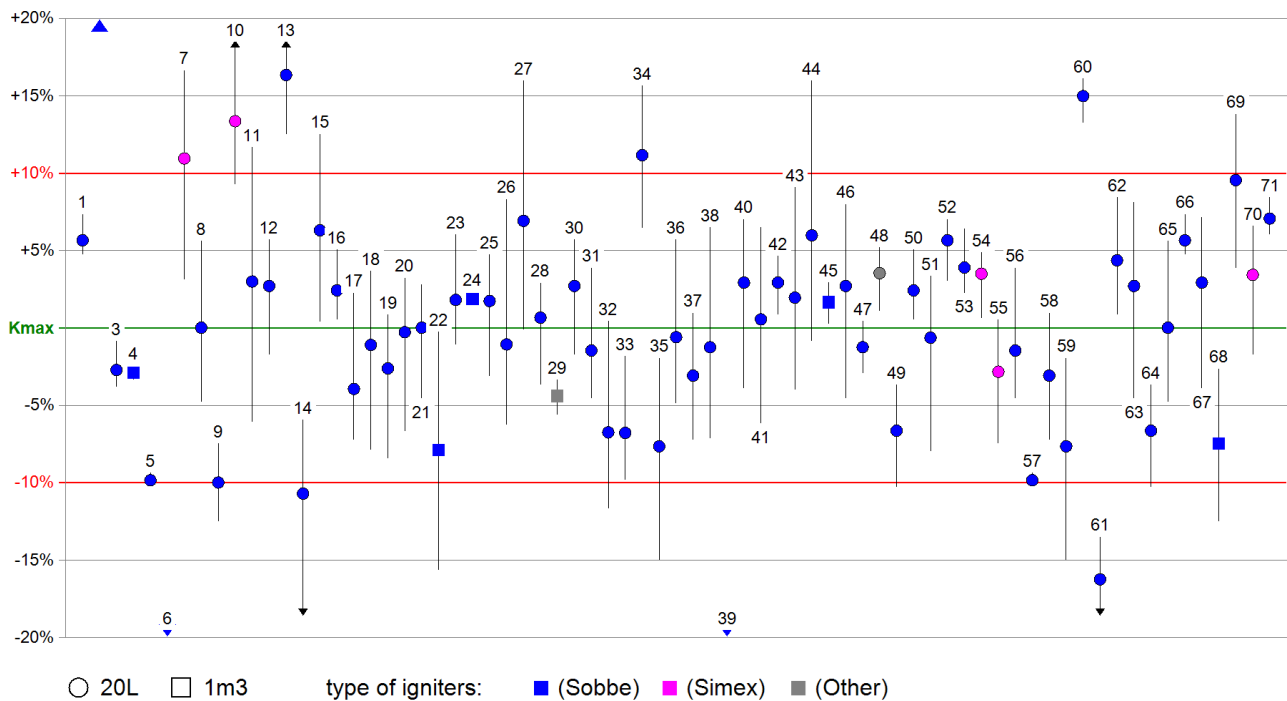
Particle size:	d 10 [µm]	d 50 = median [µm]	d 90 [µm]
Sample 1	4.22	17.52	65.37
Sample 2	4.22	17.54	66.44
Sample 3	4.20	17.56	66.53
Sample 4	4.07	17.33	66.10

2. Explosion Indices Pmax, Kmax, LEL/MEC, LOC

Pmax = 8.2 bar ±10% (7.4 ... 9.0) @ 546 g/m³

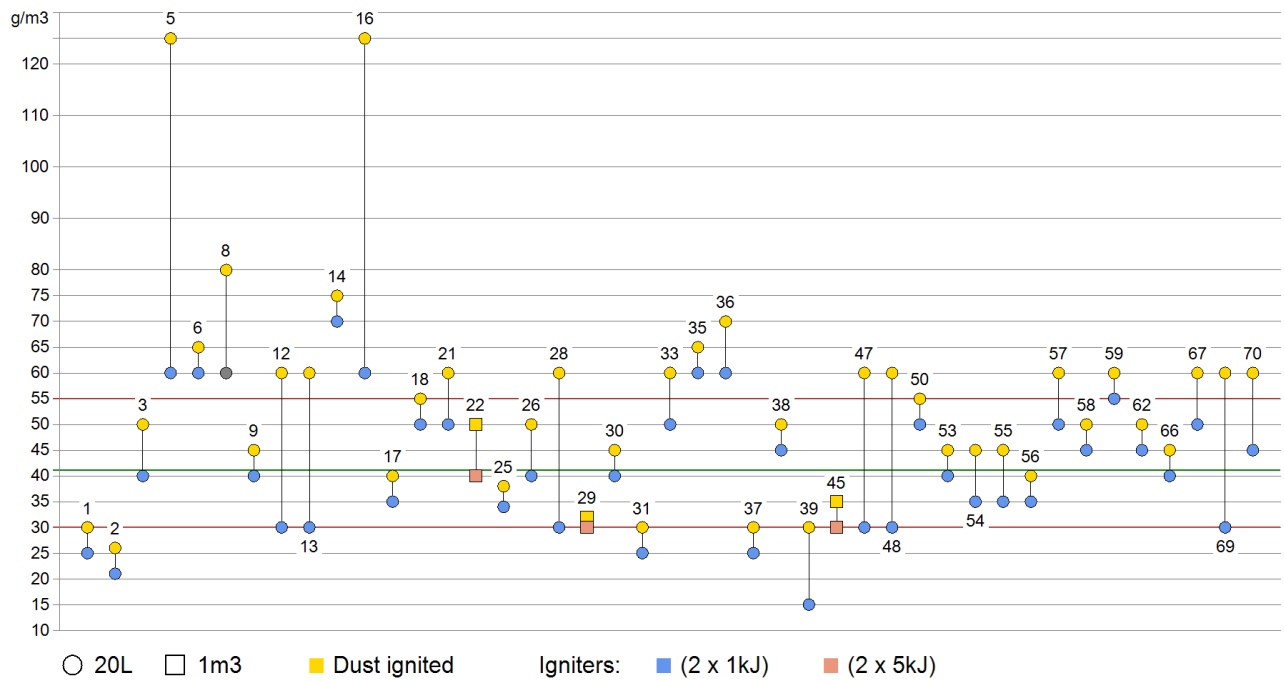


Kmax = 251 bar·m/s ±10% (226 ... 276) @ 732 g/m³

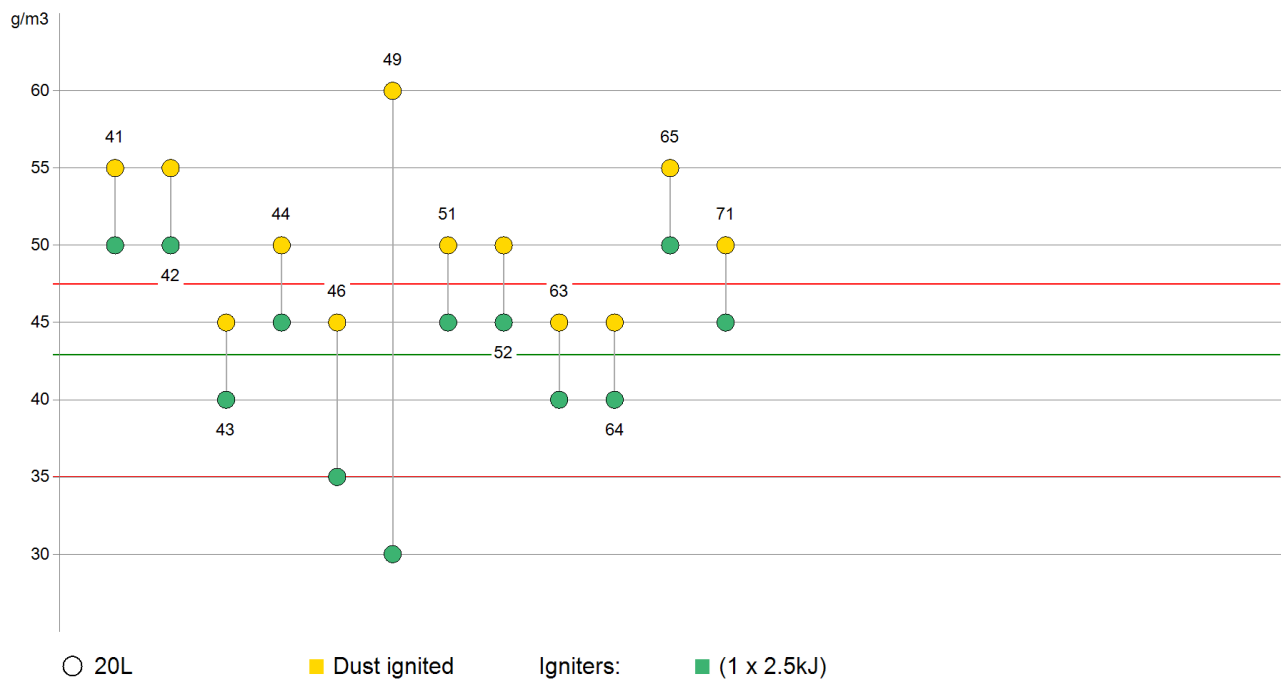


The individual results are drawn in relation to the arithmetic mean of all results and in chronological sequence (number of certificate).

LEL (CEN) = 41 g/m³ (30 ... 55)

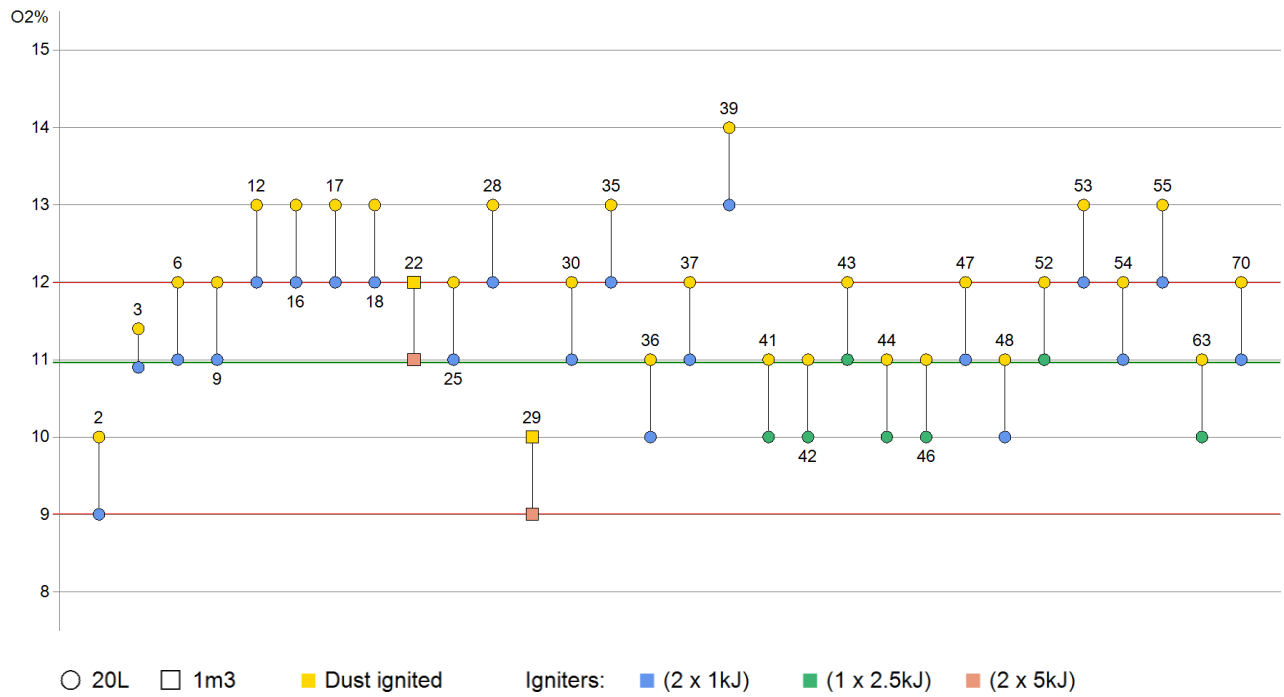


MEC (ASTM) = 43 g/m³ (35 ... 48)



The individual results are drawn in relation to the arithmetic mean of all results and in chronological sequence (number of certificate).

LOC = 11.0 O₂% (9 ... 12)



The individual results are drawn in relation to the arithmetic mean of all results and in chronological sequence (number of certificate).

2.1 Test procedure:

The determination methods are defined in the “Instructions CaRo25”

2.2 Evaluation of Pmax and Kmax:

The explosion indices Pmax and (dP/dt)max are defined as the mean values of the maximum values of each series. Subsequently, the explosion index Kmax is calculated from the mean value (dP/dt)max.

Scatter of Pmax and Kmax:

The maxima of each series must not deviate by more than **10%** of Pmax resp. Kmax. Otherwise this series must be repeated!

Calculation of the reference values:

First the mean value of all test results (71) has been calculated. In a second step all results outside of the tolerance band are excluded prior to the subsequent calculation of the mean value.

2.3 Evaluation of LEL/MEC:

The highest concentration of combustible dust at which no ignition occurs in three consecutive tests is given as the lower explosion limit (LEL/MEC). For the 1m³ vessel, only 1 test per concentration is sufficient.

The test specifications in the ASTM standard differ significantly from EN 14034-3.

ASTM E1515 states: “Direct comparisons between ASTM MEC and European CEN/CENELEC LEL determinations may not be possible.” We take this into account through separate evaluations.

For calibration, an increment of 50% of the previous concentration, as specified in EN 14034-3, is too imprecise. Therefore, a smaller increment had to be used for the CaRo. Unfortunately, some participants did not follow this CaRo test procedure.

Calculation of the reference values for LEL (CEN):

The mean value was calculated from all test results (43).

The limit values were set to match the 10g/m³ step size.

Calculation of the reference values for LEL (ASTM):

The mean value from all test results (12) was formed.

The limit values were set to match the 5g/m³ step size.

2.4 Evaluation of LOC:

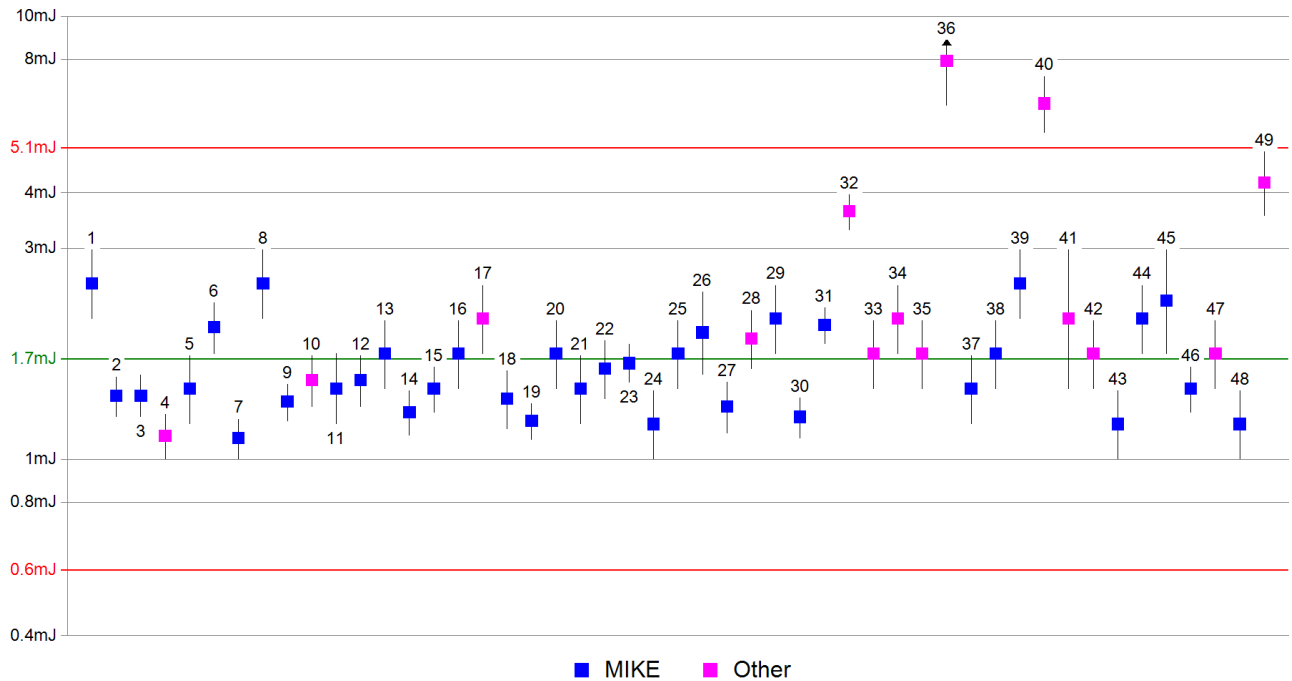
By gradually increasing the ratio of inert gas to air and changing the dust concentration, the oxygen concentration must be lowered to such an extent that an explosion no longer occurs at any given dust concentration. The value found in this way is the limiting oxygen concentration (LOC). For tests in the 20L sphere, no explosion may occur for three consecutive tests.

Calculation of the reference values:

The mean value from all test results (30) was calculated.

The limit values were set to match the 1% step size.

3. Minimum Ignition Energy MIE



The individual results are drawn in chronological sequence (number of certificate).

3.1 Test procedure:

The method for determination of the MIE is described in the „Instructions CaRo25“.

3.2 Estimation of the statistical energy (Es):

The minimum ignition energy MIE lies, by definition, between two energy values: $E_1 < MIE < E_2$

For the purpose of comparison between different apparatuses, only one MIE value (E_s) instead of the energy range (E_1, E_2) shall be used. This single value (E_s) can be estimated by use of the probability of ignition as follows (EN 13821):

$$E_s = 10^{\frac{\log E_2 - \frac{I[E_2] \cdot (\log E_2 - \log E_1)}{(NI + 1) \cdot [E_2] + 1}}{1}}$$

where is: $I[E_2]$ = number of tests with ignition at energy E_2
 $(NI+1) [E_2]$ = total number of tests at energy E_2

3.3 Criteria for conformity:

Conformity in the CaRo25 is given, when the E_s -value of each equipment differ less than a factor of 3 to the mean (E_s) of all equipment:

$E_s / 3$	E_s	$E_s \cdot 3$
0.6 mJ	1.7 mJ	5.1 mJ

4. List of Participants

Country	Company Laboratory	Pmax Kmax	LEL MEC	LOC	MIE
Australia	Simtars – Resources Safety & Health Queensland	✓	✓		✓
Austria	FireX Greßlehner GmbH	✓			
Belgium	Adinex N.V.	✓	✓	✓	✓
China	BASF Advanced Chemicals Co., Ltd.				✓
China	TÜV SÜD Certification and Testing (China) Co., Ltd. Shanghai Branch	✓	✓	✓	✓
Czech Republic	VVUÚ, a.s.	✓	✓		✓
France	EFFECTIS FRANCE	✓	✓	✓	✓
France	INERIS	✓	✓	✓	✓
France	SYENSQO	✓	✓		✓
Germany	BASF SE	✓	✓	✓	✓
Germany	BGN	✓	✓		✓
Germany	Boehringer Ingelheim Pharma GmbH & Co. KG	✓	✓		✓
Germany	consilab Gesellschaft für Anlagensicherheit mbH - Frankfurt	✓			✓
Germany	consilab Gesellschaft für Anlagensicherheit mbH - Leverkusen	✓			✓
Germany	DEKRA Testing and Certification GmbH	✓	✓		✓
Germany	EVONIK Operations GmbH	✓	✓	✓	✓
Germany	Henkel AG & Co. KGaA	✓	✓		✓
Germany	IFA - DGUV	✓	✓	✓	✓
Germany	Inburex Consulting GmbH	✓	✓	✓	
Germany	REMBE® Research+Technology Center GmbH	✓	✓		
Germany	Wacker Chemie AG	✓	✓	✓	✓
Hungary	ROBBSZIG-2023 Kft.	✓	✓	✓	✓
India	Laurus Labs Limited				✓
Italy	REDOX s.r.l.	✓			✓
Norway	Gexcon AS	✓	✓	✓	✓

Country	Company Laboratory	Pmax Kmax	LEL MEC	LOC	MIE
Poland	GIG Research Institute	✓	✓	✓	✓
Romania	INCD INSEMEX Petrosani	✓	✓		✓
Slovakia	Slovak University of Technology in Bratislava	✓	✓	✓	✓
Spain	LOM-AT. - Laboratorio Oficial J.M. Madariaga	✓	✓	✓	✓
Sweden	RISE Research Institutes of Sweden	✓	✓	✓	✓
Switzerland	DSM-Firmenich				✓
Switzerland	F. Hoffmann-La Roche AG				✓
Switzerland	Givaudan International SA	✓			✓
Switzerland	Syngenta Crop Protection AG				✓
The Netherlands	Nouryon	✓			✓
United Kingdom	DEKRA Organisational & Process Safety	✓	✓	✓	✓
United Kingdom	Sigma-HSE (UK) Ltd	✓	✓	✓	✓
USA	Ashland Inc.	✓			✓
USA	BASF Corporation	✓	✓		✓
USA	DEKRA Services, Inc.	✓	✓		✓
USA	DSM-Firmenich	✓			✓
USA	EMSL Analytical, Inc.	✓	✓		✓
USA	Fauske & Associates, LLC	✓	✓	✓	✓
USA	IEP Technologies	✓	✓	✓	✓
USA	Prime Process Safety Center	✓	✓	✓	✓

5. Standards, History

5.1 Standards:

The following Standards have been applied:

- EN 13821: Determination of minimum ignition energy of dust/air mixtures
- EN 14034-1: Determination of max. explosion pressure P_{max} ...
- EN 14034-2: Determination of max. rate of explosion pressure rise $(dp/dt)_{max}$...
- EN 14034-3: Determination of the lower explosion limit LEL ...
- EN 14034-4: Determination of the limiting oxygen concentration LOC ...
- ASTM E2019: Minimum Ignition Energy MIE of a Dust Cloud in Air
- ASTM E1226: Explosibility of Dust Clouds
- ASTM E1515: Minimum Explosible Concentration MEC of Combustible Dusts
- ASTM E2931: Limiting Oxygen Concentration LOC of Combustible Dust Clouds
- EN ISO/IEC 80079-20-2: ... Material characteristics. Combustible dust methods

5.2 History:

Our previous world-wide round robin tests:

CaRo93: 77 apparatuses	CaRo09: 90 apparatuses	CaRo20: 41 apparatuses
CaRo96: 68 apparatuses	CaRo11: 122 apparatuses	CaRo21: 125 apparatuses
CaRo98: 63 apparatuses	CaRo13: 112 apparatuses	CaRo22: 50 apparatuses
CaRo00: 69 apparatuses	CaRo15: 131 apparatuses	CaRo23: 130 apparatuses
CaRo03: 93 apparatuses	CaRo17: 144 apparatuses	CaRo24: 65 apparatuses
CaRo05: 98 apparatuses	CaRo18: 41 apparatuses	
CaRo07: 77 apparatuses	CaRo19: 127 apparatuses	

→ All final reports can be downloaded here:

<https://www.cesana-ag.ch/Calibration.shtml>

References

Cesana Ch., Eiche M., Schwaninger M., 2019,
Quality Management in the Determination of Safety Characteristics, CET-Paper