Calibration-Round-Robin CaRo23
Final Report

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 **53** 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.

CaRo 23 – Reference values for the Explosion Indices Pmax and Kmax

Pmax	(bar)	**8.0** ± 10%	(7.2 ... 8.8)
Kmax	(bar·m/s)	**248** ± 10%	(223 ... 273)

CaRo 23 – Reference values for the Minimum Ignition Energy MIE

Es / 3	Es	Es • 3
0.6 mJ	**1.7 mJ**	**5.1 mJ**

Cesana AG
Baiergasse 56
CH-4126 Bettingen
Switzerland

Phone +41 61 534 01 61
E-Mail caro@cesana-ag.ch
Internet www.cesana-ag.ch

1.1 Participants:

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

	Pmax, Kmax (62)		MZE (55)	
	20-l	1 m^3	MIKE	others
Australia	1		1	
Austria	1			
Belgium	1		1	
China	4		1	2
Czech Republic	1		1	
France	2		3	
Germany	12	2	12	
Italy	2		1	
Japan			1	
Norway	1		1	
Poland	1			1
Romania	1			1
South Africa	1			
Spain	1		1	
Switzerland	4		9	
Taiwan			1	
The Netherlands	1		1	
United Kingdom	6		4	1
USA	19	1	11	1
Total:	**59**	**3**	**49**	**6**

1.2 Test substance:

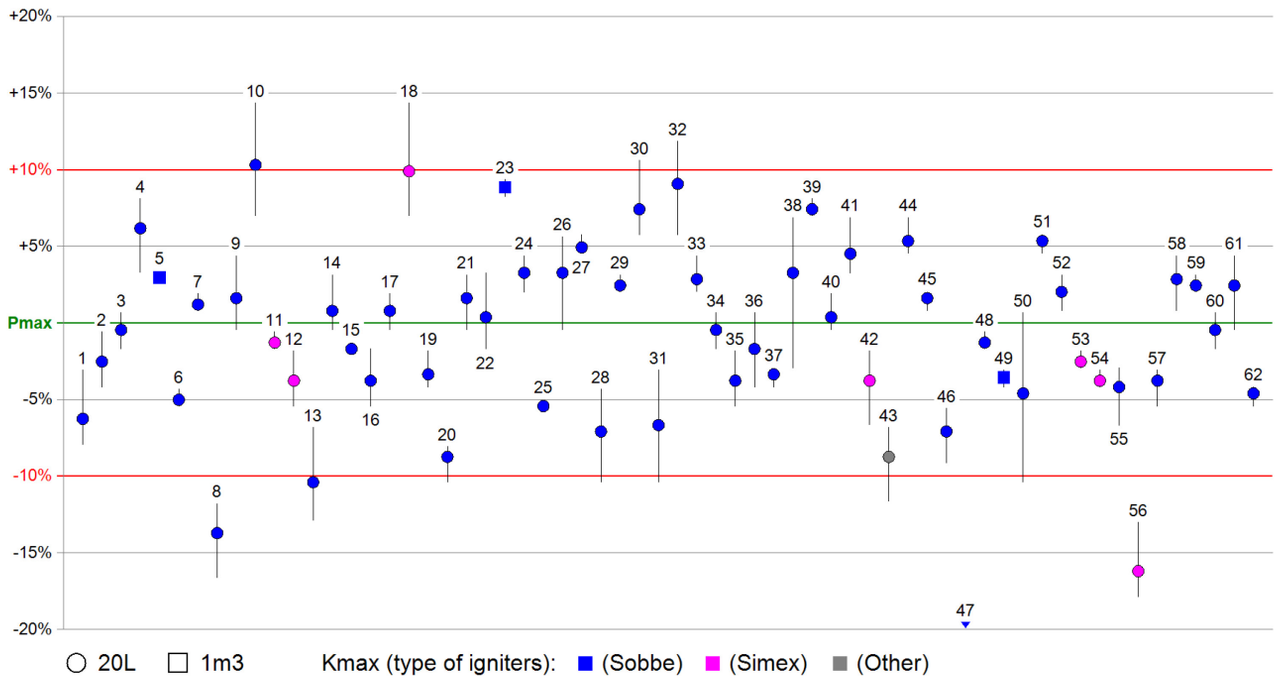
For correct calibration the CaRo 23 test sample has been milled, homogenized, packed under nitrogen and shipped in an air tight package. Therefore the sample has to be tested „as delivered“.

CaRo 23 = Niacin USP (Nicotinic acid)

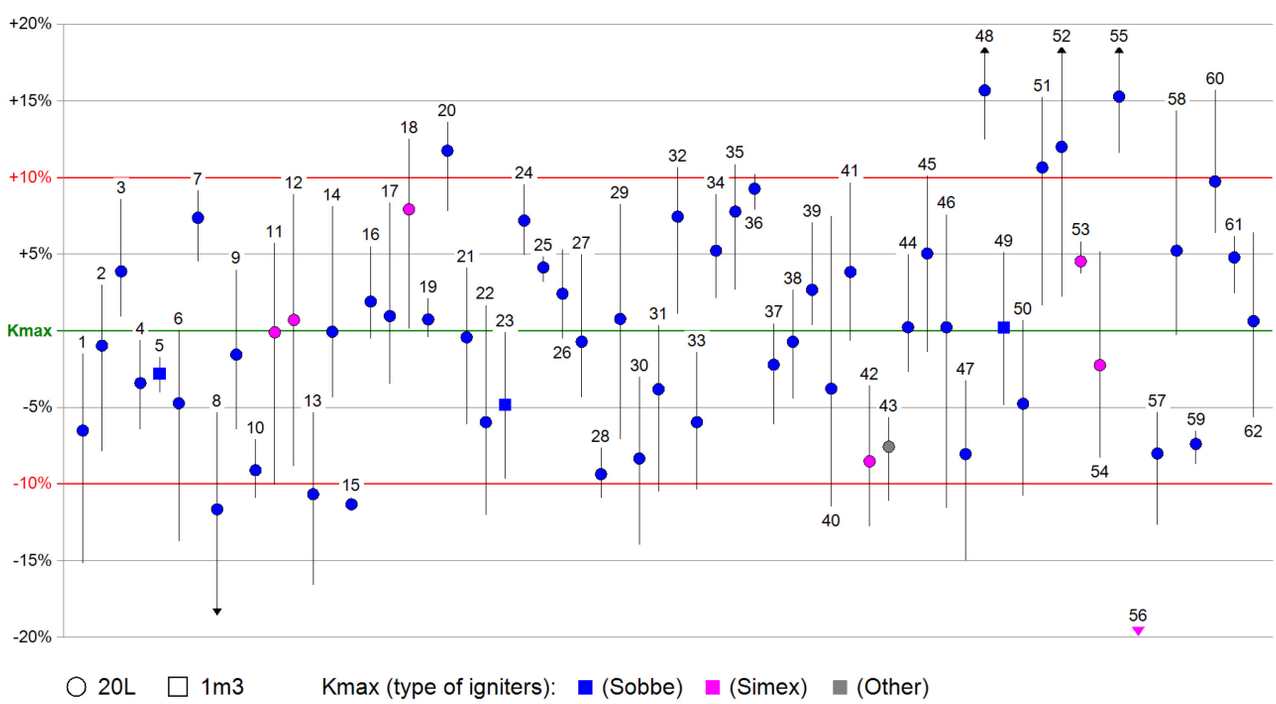
Particle size:	d 10 [µm]	**d 50 = median [µm]**	d 90 [µm]
Sample 1	5.28	**21.42**	83.50
Sample 2	5.24	**20.82**	78.21
Sample 3	5.45	**21.35**	78.75
Sample 4	5.35	**21.11**	78.31

2. Explosion Indices Pmax, Kmax

Pmax = 8.0 bar ±10% (7.2 ... 8.8) @ 525 g/m^3



Kmax = 248 bar•m/s ±10% (223 ... 273) @ 782 g/m^3



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 method for determination of Pmax, Kmax is described in the „Instructions CaRo 23“

2.2 Evaluation:

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.

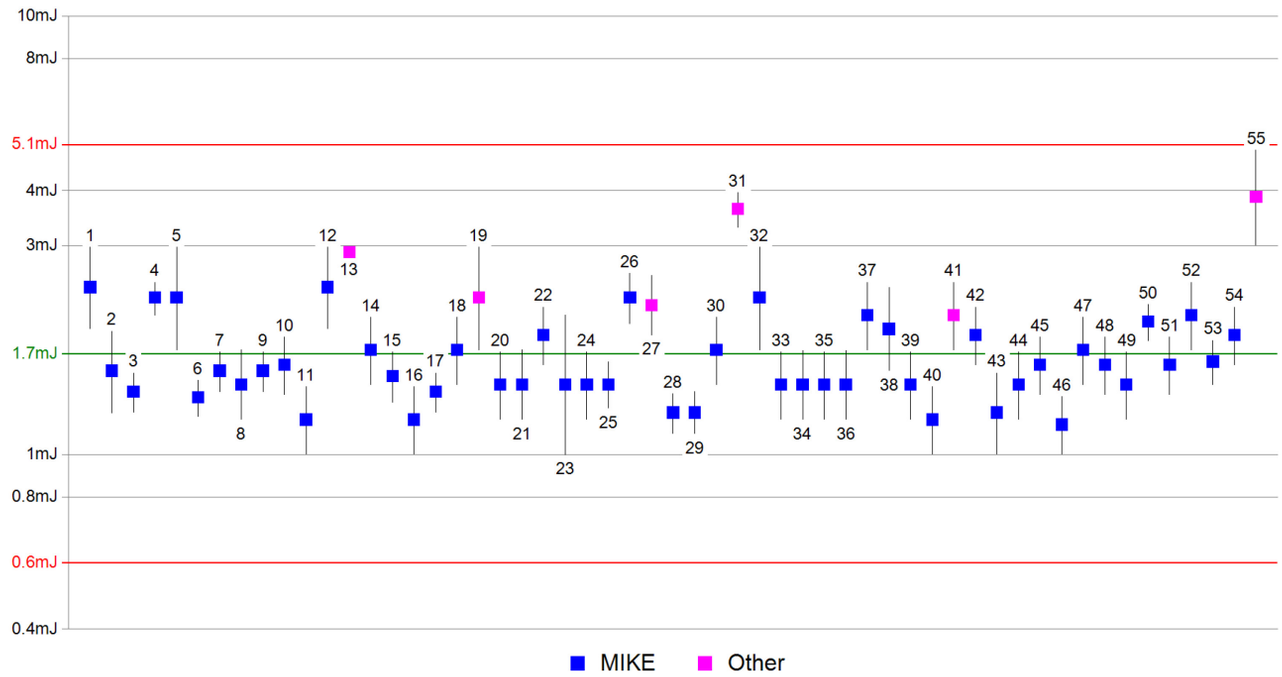
2.3 Scatter of Pmax and Kmax:

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

2.4 Calculation of the reference values:

First the mean value of all test results (62) 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.

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 CaRo 23“.

3.2 Estimation of the statistical energy (Es):

The minimum ignition energy MIE lies, by definition, between two energy values: E1 < MIE < E2

For the purpose of comparison between different apparatuses, only one MIE value (Es) instead of the energy range (E1, E2) shall be used. This single value (Es) can be estimated by use of the probability of ignition as follows (EN 13821):

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

where is: I[E2] = number of tests with ignition at energy E2

(NI+I) [E2] = total number of tests at energy E2

3.3 Criteria for conformity:

Conformity in the CaRo 23 is given, when the Es-value of each equipment differ less than a factor of 3 to the mean (Es) of all equipment:

Es / 3	Es	Es • 3
0.6 mJ	1.7 mJ	5.1 mJ

4. List of Participants

Country	Company Laboratory	Pmax Kmax	MIE
Australia	Simtars – Resources Safety & Health Queensland	✓	✓
Austria	FireX Greßlehner GmbH	✓	
Belgium	Adinex N.V.	✓	✓
China	BASF Advanced Chemicals Co., Ltd.		✓
China	DEKRA Testing and Certification Ltd.	✓	✓
China	TÜV SÜD Certification and Testing (China) Co., Ltd. Shanghai Branch	✓	✓
Czech Republic	VVUÚ, a.s.	✓	✓
France	INERIS	✓	✓
France	SOLVAY	✓	✓
Germany	BASF SE	✓	✓
Germany	Bayer AG Leverkusen	✓	✓
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	✓	✓
Italy	Politecnico di Torino	✓	
Italy	REDOX s.r.l.	✓	✓
Japan	Technology Institution of Industrial Safety		✓
Norway	Gexcon AS	✓	✓
Poland	GIG Research Institute	✓	✓

Country	Company Laboratory	Pmax Kmax	MIE
Romania	INCD INSEMEX Petrosani	✓	✓
South Africa	CSIR	✓	
Spain	LOM-AT. - Laboratorio Oficial J.M. Madariaga	✓	✓
Switzerland	DSM		✓
Switzerland	F. Hoffmann-La Roche AG		✓
Switzerland	Givaudan International SA	✓	✓
Switzerland	Syngenta Crop Protection AG		✓
Switzerland	TÜV SÜD Process Safety	✓	✓
Switzerland	TÜV SÜD Schweiz AG		✓
Taiwan	Yun Safety Tech		✓
The Netherlands	Nouryon	✓	✓
United Kingdom	BRE Global	✓	✓
United Kingdom	DEKRA Organisational & Process Safety	✓	✓
United Kingdom	Health and Safety Executive	✓	✓
United Kingdom	Sigma-HSE (UK) Ltd	✓	✓
United Kingdom	Syngenta Ltd.	✓	✓
USA	Ashland Inc.	✓	✓
USA	BASF Corporation	✓	✓
USA	Bristol Myers Squibb		✓
USA	DEKRA Services, Inc.	✓	✓
USA	EMSL Analytical, Inc.	✓	✓
USA	Exponent, Inc.	✓	✓
USA	Fauske & Associates, LLC	✓	✓
USA	FM Approvals	✓	
USA	IEP Technologies	✓	✓
USA	ioKinetic, LLC	✓	✓
USA	Merck & Co., Inc.	✓	✓

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 Pmax …
EN 14034-2: Determination of max. rate of explosion pressure rise (dp/dt)max …
ASTM E1226: Standard Test Method for Explosibility of 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	CaRo07: 77 apparatuses	CaRo18: 41 apparatuses
CaRo96: 68 apparatuses	CaRo09: 90 apparatuses	CaRo19: 127 apparatuses
CaRo98: 63 apparatuses	CaRo11: 122 apparatuses	CaRo20: 41 apparatuses
CaRo00: 69 apparatuses	CaRo13: 112 apparatuses	CaRo21: 125 apparatuses
CaRo03: 93 apparatuses	CaRo15: 131 apparatuses	CaRo22: 50 apparatuses
CaRo05: 98 apparatuses	CaRo17: 144 apparatuses	CaRo23: 117 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