

Calibration-Round-Robin CaRo 17

Final Report

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1. Conclusion

According to international standards, test equipment must be calibrated at intervals by comparison with a standard or a calibrated testing apparatus. This calibration also applies to 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. 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 the following calibration method has been carried out successfully:

A dust has been selected, prepared and supplied to **74** test laboratories all over the world. The mean values of the explosion indices, measured by the participating laboratories, has 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 procedures.

CaRo 17 – Reference values for the Explosion Indices Pmax and Kmax

Pmax (bar)	8.2 ± 10% (7.4 ... 9.0)
Kmax (bar·m/s)	243 ± 10% (219 ... 268)

CaRo 17 – Reference values for the Minimum Ignition Energy MIE

Es / 3	Es	Es · 3
0.5 mJ	1.6 mJ	4.7 mJ

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1.1 Participants

For details see section "list of participants".

	Pmax, Kmax (73)		MIE (73)	
	20-l	1 m ³	MIKE	others
Australia	1		1	
Austria	2		1	
Belgium	2		2	
Canada	2		1	1
China	1		2	
Czech Republic	1		1	
France	4		5	
Germany	14	5	16	1
Hungary	1			
India			2	
Italy	2		2	
Japan	1		3	
Norway	1		1	
Poland	1			1
Romania	1			
Serbia	1			
South Africa	1			
Spain	1		1	
Switzerland	4		5	
The Netherlands	1		1	
United Kingdom	5		4	2
USA	19	2	17	3
Total:	66	7	65	8

1.2 Test substance

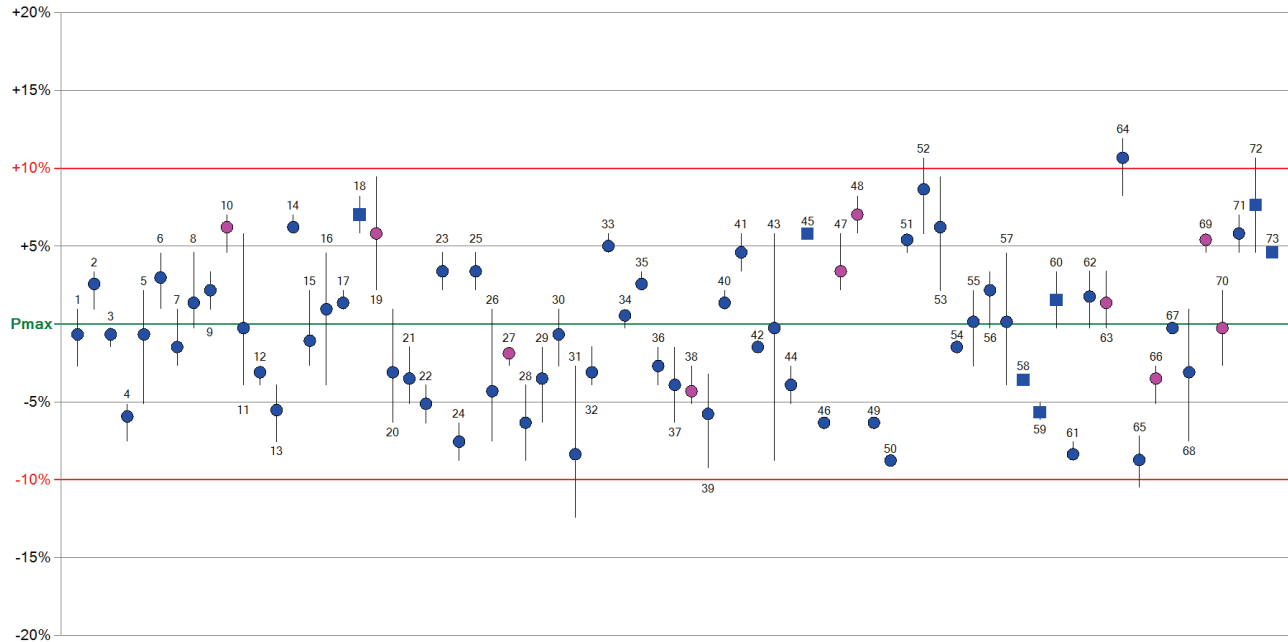
For correct calibration the CaRo 17 test sample has been milled, homogenized and shipped in an air tight package. Therefore the sample has to be tested „as delivered“.

CaRo 17 = Niacin USP special (Nicotinic acid)

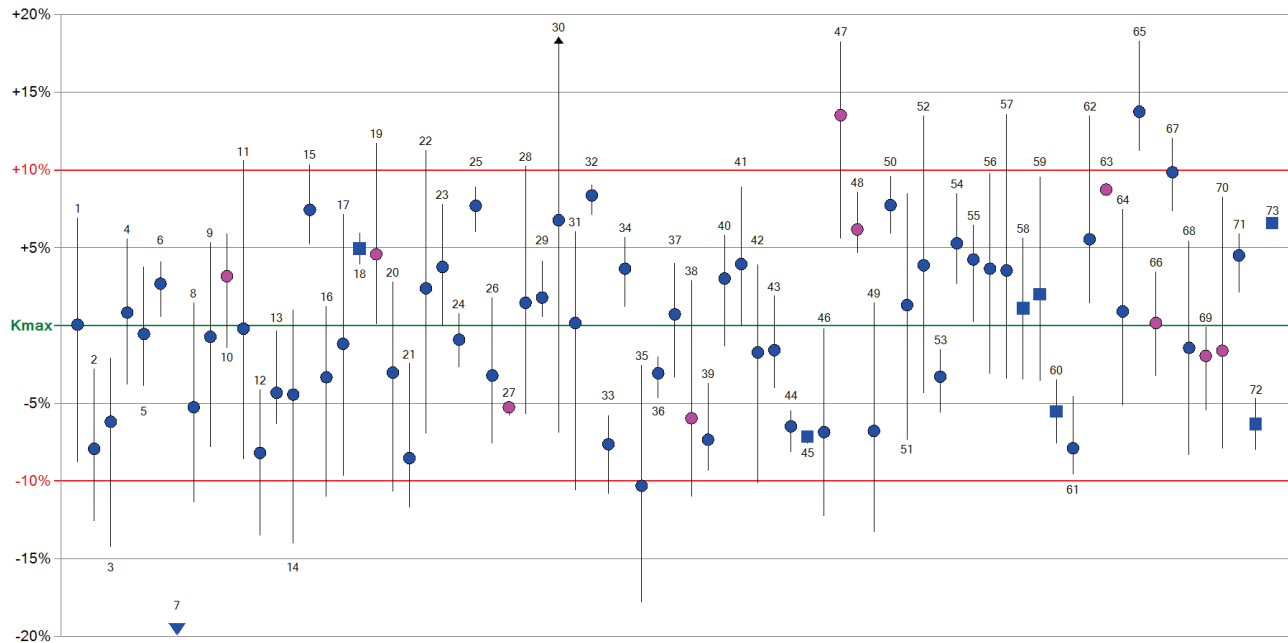
Particle size:	d 10 [µm]	d 50 = median [µm]	d 90 [µm]
Sample 1	3.7	18.8	65.8
Sample 2	3.8	19.1	69.1
Sample 3	3.7	18.5	64.3
Sample 4	3.7	18.1	65.0

2. Explosion Indices Pmax, Kmax

Pmax = 8.2 bar ± 10% (7.4 ... 9.0) at 505 g/m³



Kmax = 243 bar·m/s ± 10% (219 ... 268) at 677 g/m³



● 20L Sobbe ■ 1m3 Sobbe ● 20L Simex ■ 1m3 Simex

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 „Manual CaRo 17“

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:

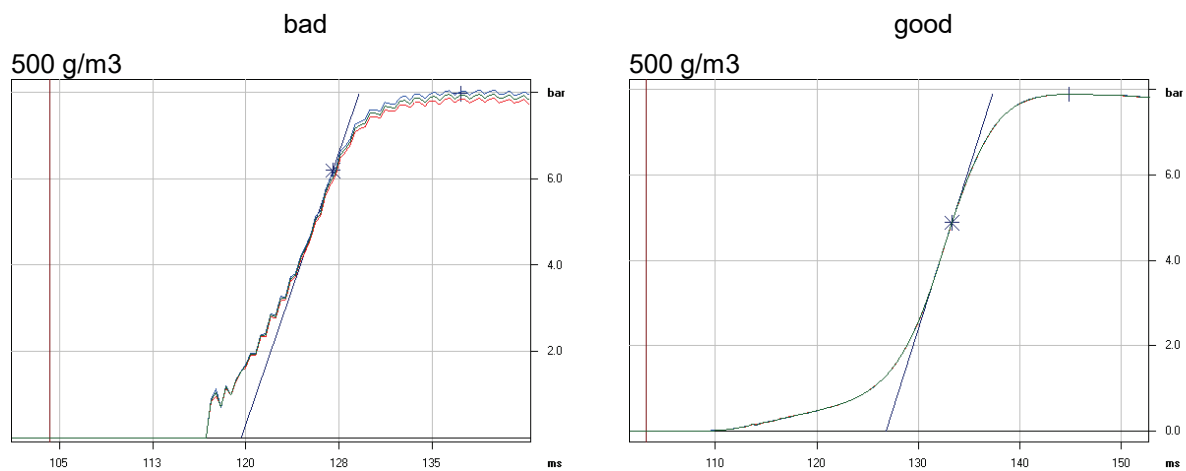
The maxima of each series must not deviate by more than **10%** of Pmax resp. Kmax. Otherwise this series must be repeated!

2.4 Calculation of the reference values:

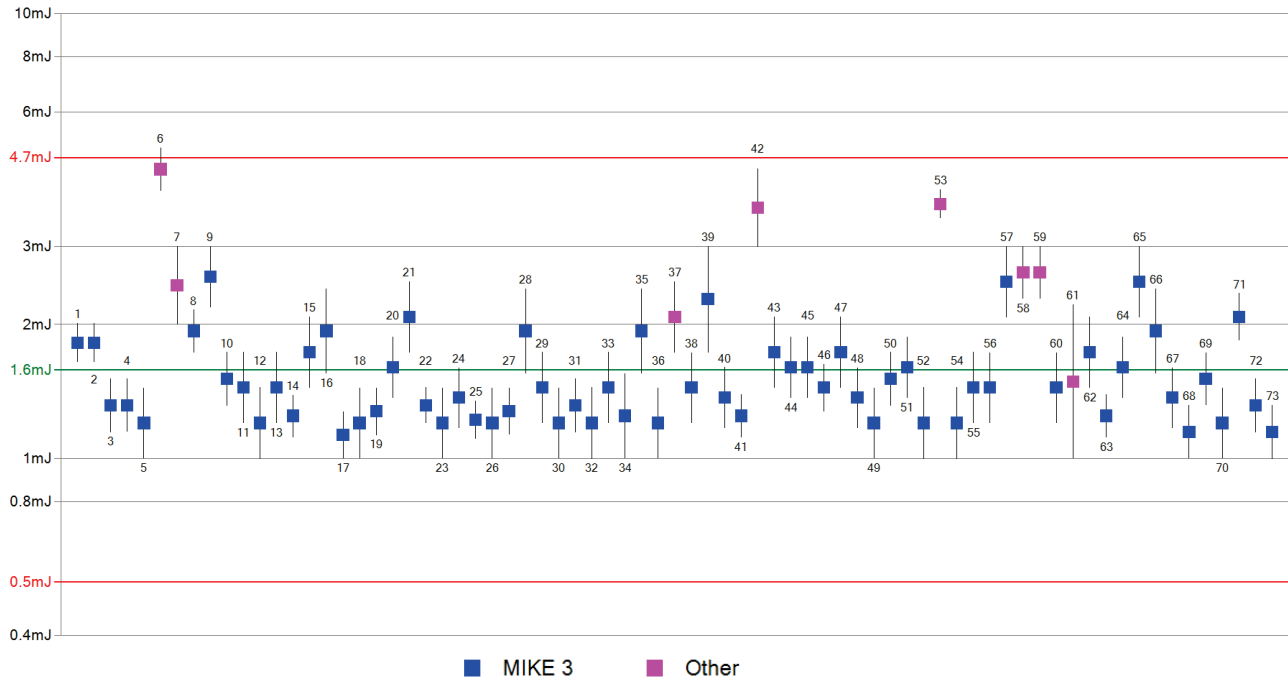
First the mean values of all test results (73) has been calculated. In a 2nd step all results outside of the tolerance band are excluded prior to the subsequent calculation of the mean value. Due to the large number of participants the mean values did not change.

2.5 Troubles with Simex-Igniters

Simex sent some faulty igniters with pressure oscillations. Pressure oscillations increase the turbulence and therefore the Kmax-value. Through the superimposed oscillation, the automatic evaluation is influenced or even wrong, which means the tangent has to be evaluated manually. The behavior of the igniters is strongly variable from one production lot to another. Igniters with superimposed oscillations will give false results. In that case contact the manufacturer of the igniters.



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 „Manual CaRo 17“.

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 apparatus, 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):

Provided that for the energy E_2 a minimum of 5 dust concentrations evenly distributed are tested, the position of the MIE in the E_1 - E_2 range can be estimated. At ignition energy E_2 , the number of dust concentrations with ignition, is divided by the total number of dust concentrations tested.

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

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 between two equipment (a, b) is given, when the E_s -values differ less than a factor of 3 (EN 13821): $1/3 < E_s(a) / E_s(b) < 3$

Accordingly: Conformity in the CaRo 17 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	$E_s \cdot 3$
0.5 mJ	1.6 mJ	4.7 mJ

4. List of Participants

Country	Company Laboratory	E-Mail	Pmax Kmax	MIE
Australia	Simtars	negar.fasihiani@simtars.com.au	✓	✓
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