Final Report

Calibration-Round-Robin

CaRo 15

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1. Conclusion

According to international standards (e.g. ISO 9000, GLP), 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 **62** 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. It also demonstrates that with this method it was possible to discover and rectify the cause of any errors with installations producing results differing widely from the reference values.

CaRo 15 - Reference values for the Explosion Indices Pmax and Kmax

Pmax (bar)	**8.2** ± 10% (7.3 ... 9.0)
Kmax (bar·m/s)	**245** ± 10% (220 ... 269)

CaRo 15 – Reference values for the Minimum Ignition Energy MIE

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

zertifiziertes Qualitätssystem BVQi ISO 9001/EN 29001

Birsfelden, January 2016

Adolf Kühner AG
Christoph Cesana

1.1 Participants

For details see section "list of participants".

	Pmax, Kmax (75)		MIE (56)	
	20-l	1 m^3	MIKE	Others
Australia	1		1	
Austria	3		2	
Belgium	4		2	
Bosnia	1			
Brazil	1			
China	1		1	
Czech Republic	1		1	
England	3		3	
France	5		3	
Germany	18	3	14	
Hungary	1			
India			2	
Italy	2		2	
Japan	1		3	
Norway	1		1	
Poland	1			
South Africa	1			
Spain	2		1	
Switzerland	4		5	
Taiwan	1		1	
U.S.A	20		12	2
Total:	**72**	**3**	**54**	**2**

This calibration round robin test „CaRo 15“ has been accepted worldwide as the best and most reliable calibration method for this type of test equipment.

1.2 Test substance

For correct calibration the CaRo 15 test sample has been milled, homogenized and shipped in an air tight package. Therefore the sample has to be tested „as delivered".

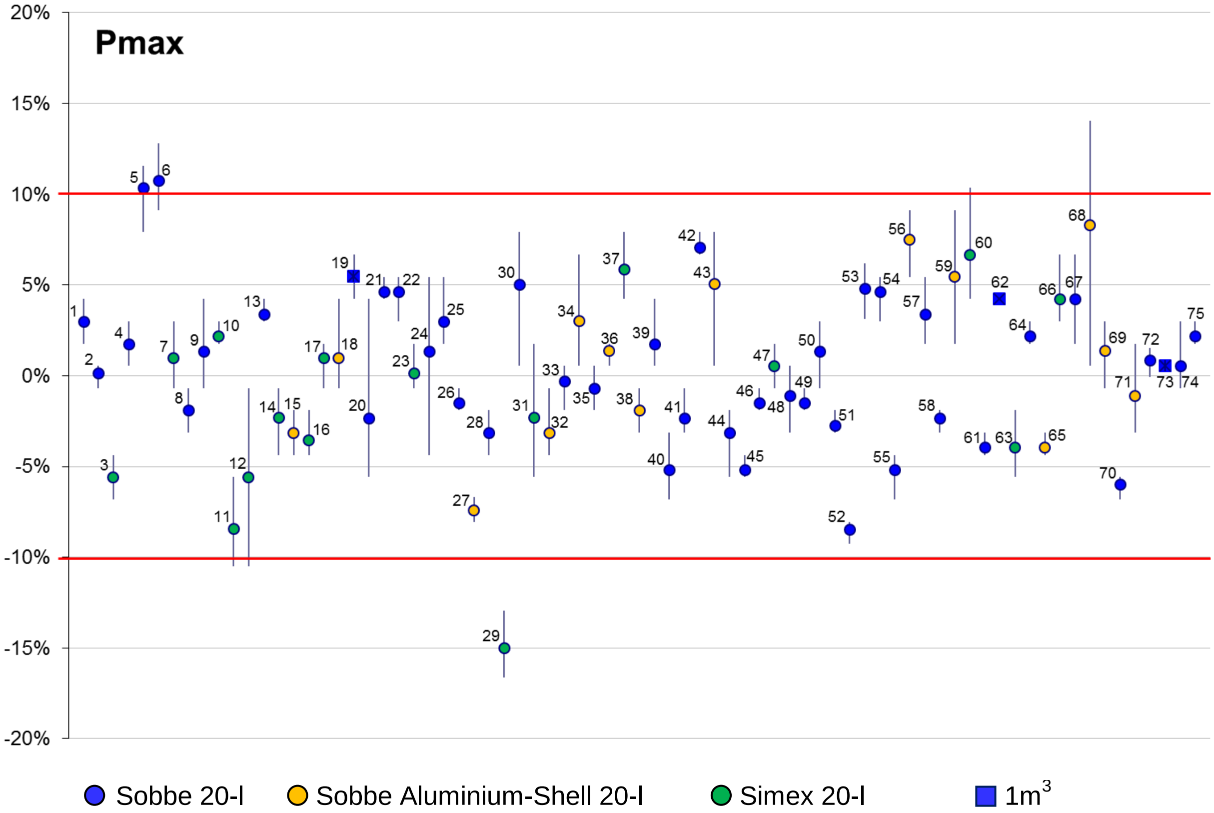
CaRo 15 = Niacin USP special (Nicotinic acid)

Particle size:

	d 10 [µm]	**d 50 = median [µm]**	d 90 [µm]
Sample 1	4.3	**18.7**	52.1
Sample 2	4.5	**19.2**	52.4
Sample 3	4.5	**19.0**	51.7
Sample 4	4.9	**20.6**	62.4

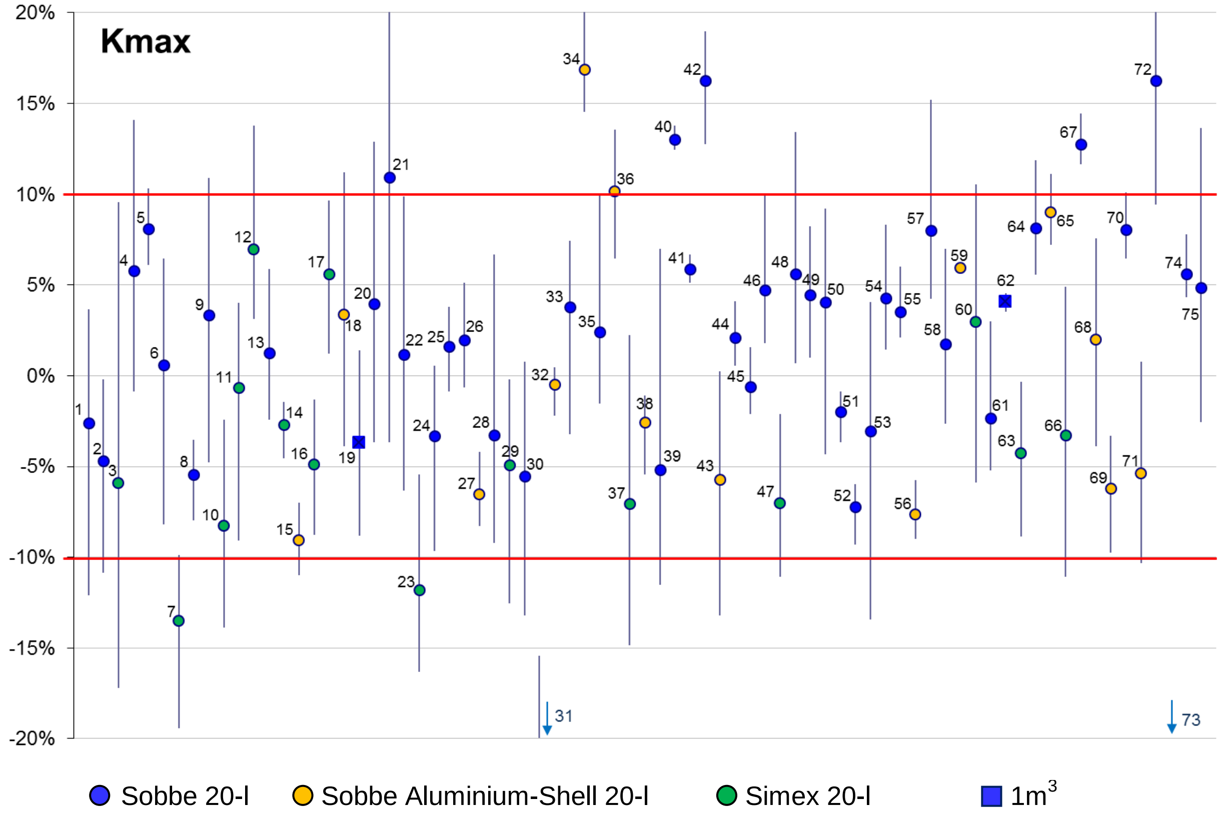
2. Explosion Indices Pmax, Kmax

Pmax = 8.2 bar ± 10% (7.3 ... 9.0) by 636 g/m3



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

Kmax = 245 bar·m/s ± 10% (220 ... 269) by 839 g/m3



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 15".

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.

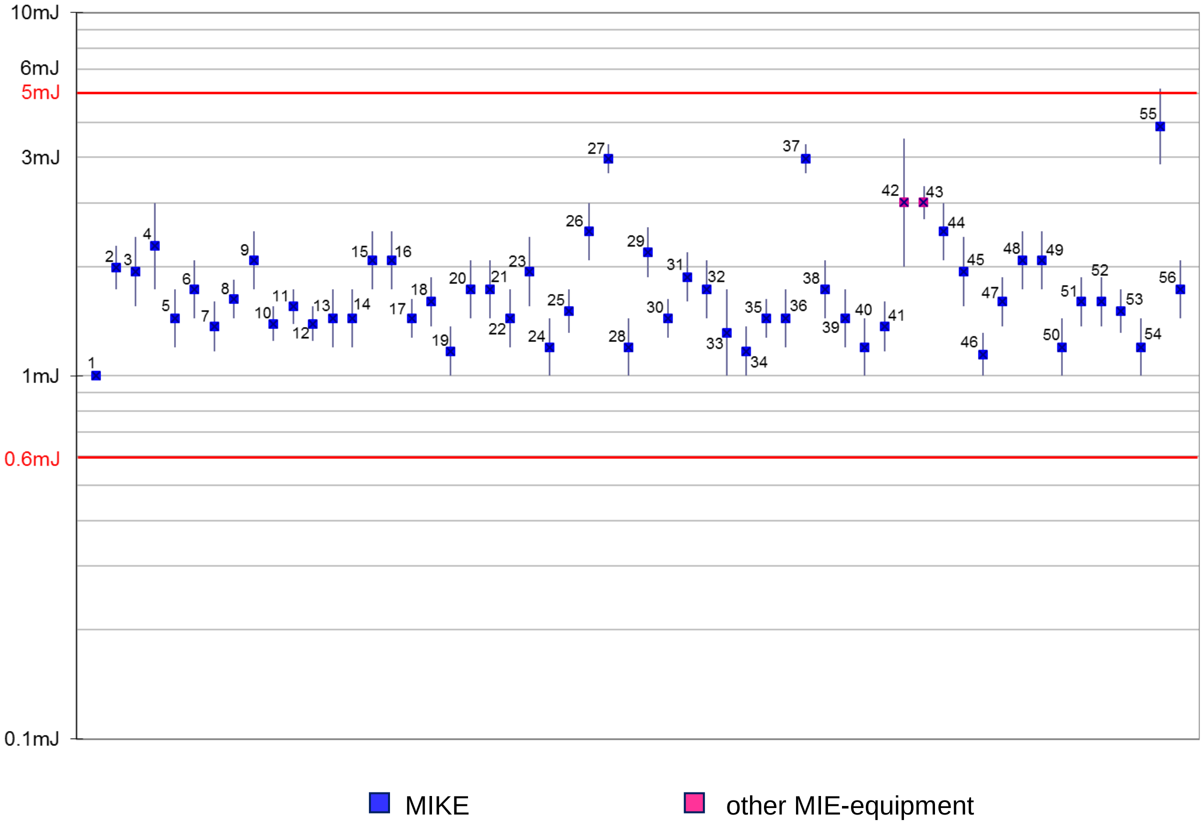
2.3 Scatter of Pmax and Kmax:

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 (75) 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.

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 15“.

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 apparatus, 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):

Provided that for the energy E2 a minimum of 5 dust concentrations evenly distributed are tested, the position of the MIE in the E1-E2 range can be estimated. At ignition energy E2, the number of dust concentrations with ignition, is divided by the total number of dust concentrations tested.

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

where is: I[E2] = number of tests with ignition at the energy E2.
(NI+I)[E2] = total number of tests at the energy E2.

3.3 Criteria for conformity:

Conformity between two equipment (a, b) is given, when the Es-values differ less than a factor of 3 (EN 13821).

1/3 < Es(a) / Es(b) < 3

Accordingly:

Conformity in the CaRo 15 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**

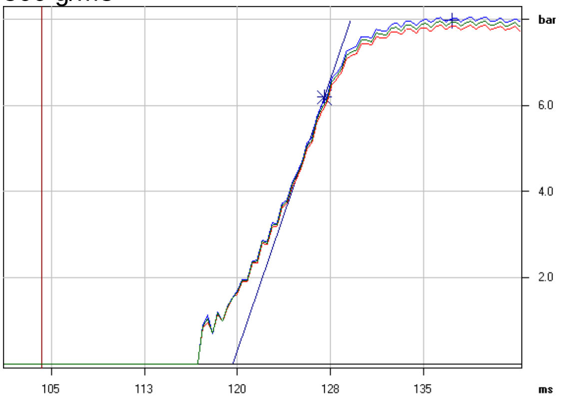
4. Troubles with chemical igniters

4.1 Simex-Igniters

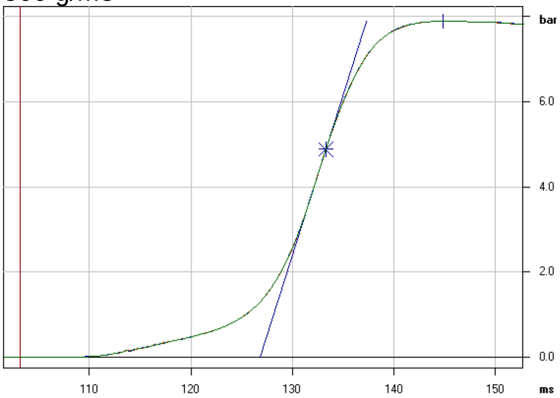
Simex sent some faulty igniters with pressure oscillations. Pressure oscillations increase the turbulence and therefore the Kmax-value. Through the superimposed oscillation, the automatic evaluation is influenced or even wrong, which means the tangent has to be evaluated manually. The behaviour of the 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.

bad **good**

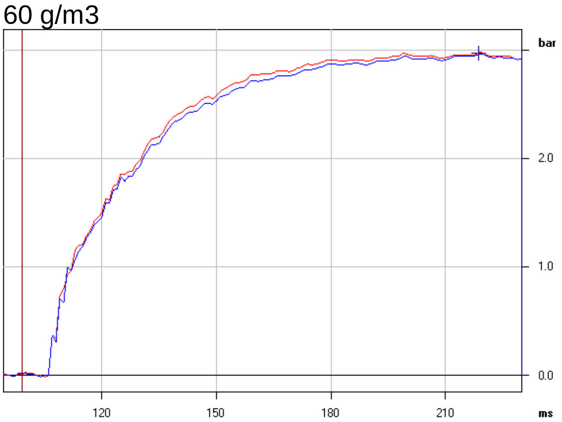
500 g/m3



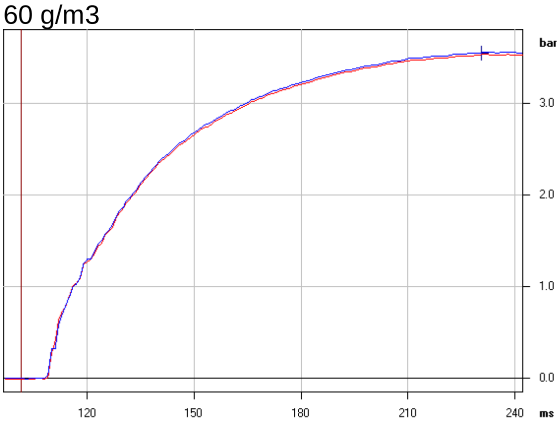
500 g/m3



60 g/m3



60 g/m3



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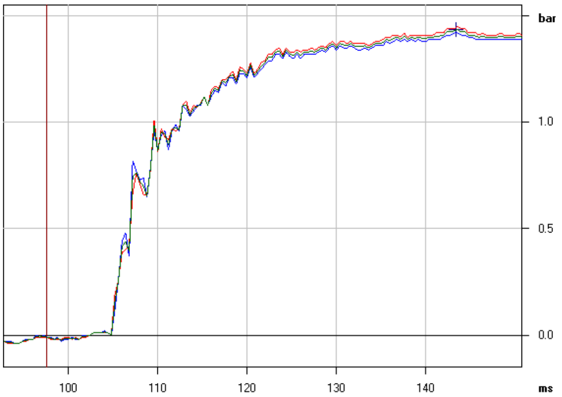

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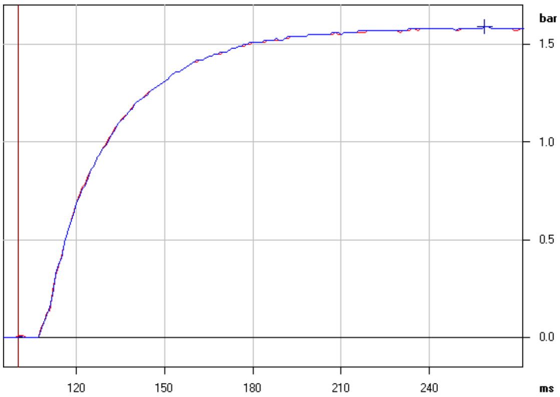

5. List of Participants

Country	Company Laboratory	E-Mail	Pmax Kmax	MIE
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Austria	AUVA	silvia.springer@auva.at	✓	✓
Austria	FireX Gresslehner	dietmar.gresslehner@firex.at	✓	✓
Austria	Montanuniversität Leoben	thomas.ehgartner@unileoben.ac.at	✓	✓
Belgium	ADINEX N.V	frederik.norman@adinex.be	✓	✓
Bosnia and Herzegovina	UNIVERSITY OF TUZLA	zvjezdan.karadzin@untz.ba	✓	
Brazil	IPT-Instituto de Pesquisas	ricalca@ipt.br	✓	
China	Shanghai Research Institute of Chemical Industry	xiao_srici@163.com	✓	✓
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Country	Company Laboratory	E-Mail	Pmax Kmax	MIE
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