

Final Report
Calibration-Round-Robin
CaRo 03

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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 P_{max} and K_{max} 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 **53** 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 03 - Reference values for the Explosion Indices P_{max} and K_{max}

P_{max} (bar)	8.1 ± 10% (7.3 ... 8.9)
K_{max} (bar·m/s)	232 ± 10% (209 ... 256)

CaRo 03 - Reference values for the Minimum Ignition Energy MIE

E_s / 3	E_s	E_s • 3
0.6 mJ	1.7 mJ	5.1 mJ



Birsfelden, January 2004

Adolf Kühner AG
Christoph Cesana

Participants

For details see section "list of participants".

	Pmax, Kmax (49)		MIE (44)	
	20-l	1 m ³	MIKE	others
Austria	1		1	
Australia	1		1	
Belgium	3		2	
Germany	12	3	11	2
England	5		2	2
Finland	1			
France	6		4	1
Holland	2		1	
Hungary				1
India			1	
Italy			1	
Japan			1	
Canada	1		1	
Norway	1			1
Poland	1			
Switzerland	6		5	
Spain	1		1	
U.S.A.	5		5	
total:	46	3	37	7

This calibration round robin test „CaRo 03“ has been accepted world wide as the best and most reliable calibration method for this type of test equipment.

Test substance

For correct calibration the CaRo 03 test sample has been milled, homogenized and shipped in an air tight package. Therefore the sample has to be tested „as delivered“.

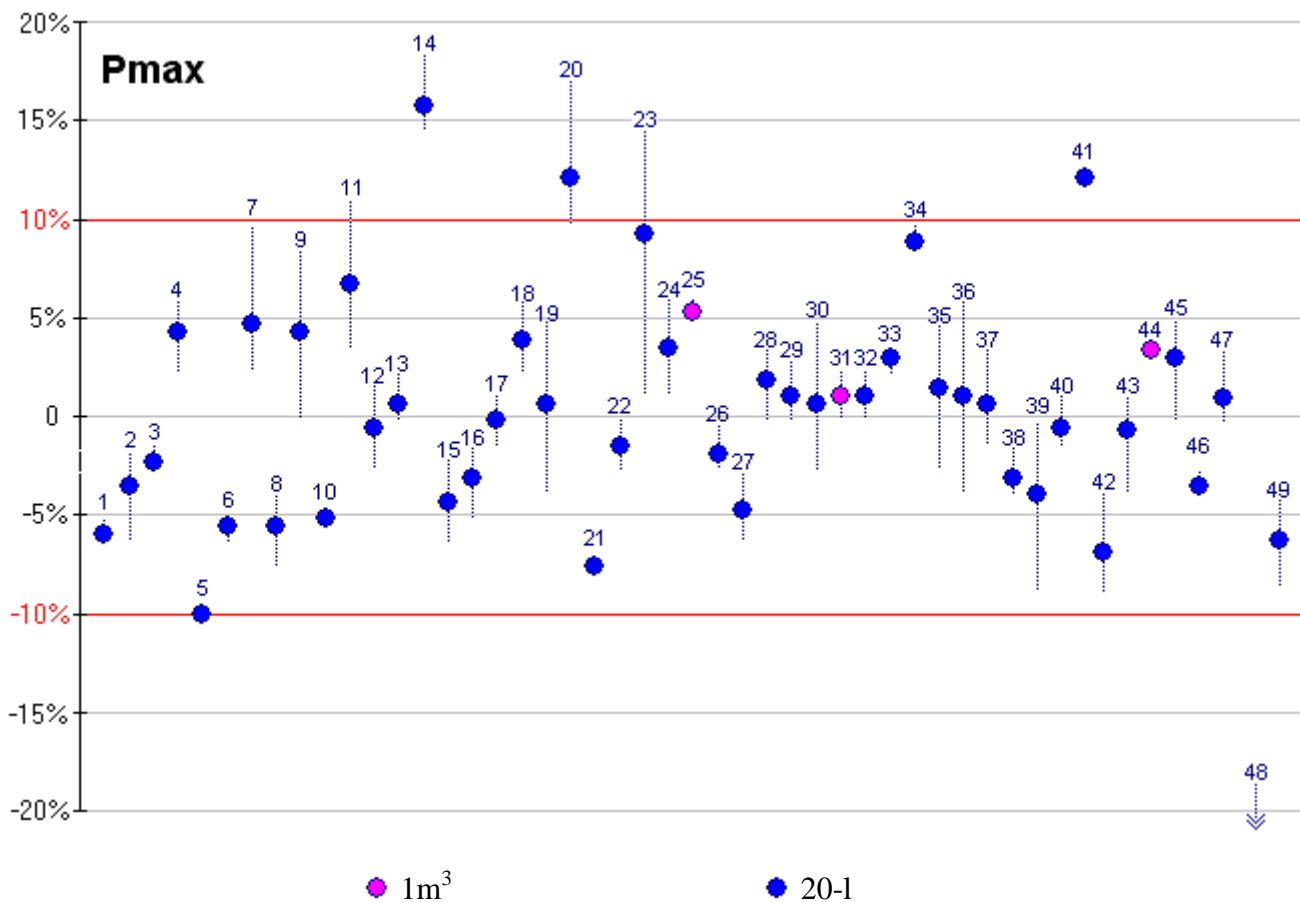
CaRo 03 = Niacin USP (Nicotinic acid)

Particle size:

	d 10	d 50 = median	d 90
Sample 1	5.6 µm	21.0 µm	62.5 µm
Sample 2	5.8 µm	21.2 µm	66.5 µm
Sample 3	6.4 µm	21.8 µm	62.5 µm
Sample 4	5.9 µm	22.0 µm	67.0 µm

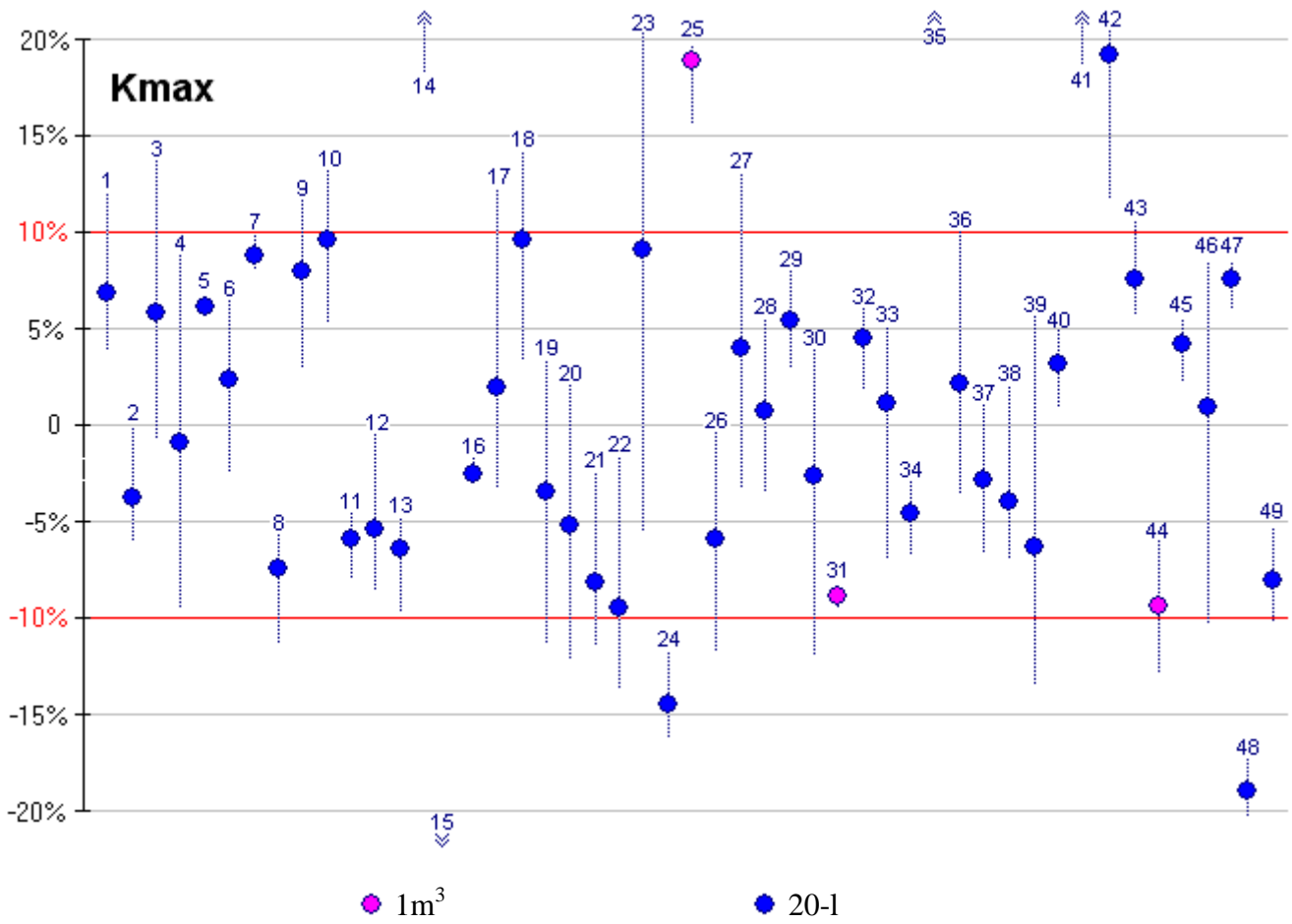
Explosion Indices Pmax, Kmax

Pmax = 8.1 bar ± 10% (7.3 ... 8.9) at 680 g/m³



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

Kmax = 232 bar·m/s ± 10% (209 ... 256) at 880 g/m³



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

Test procedure:

The method for determination of P_{max} , K_{max} is described in the „Manual CaRo 03“.

Evaluation:

The explosion indices P_{max} and $(dP/dt)_{max}$ are defined as the mean values of the maximum values of each series. Subsequently, the explosion index K_{max} is calculated from the mean value $(dP/dt)_{max}$.

Scatter of P_{max} and K_{max}

The maxima of each series must not deviate by more than **10%** of P_{max} resp. K_{max} . Otherwise this series must be repeated !

Calculation of the reference values:

First the mean values of all test results (49) 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.

Cause of errors:

Some laboratories has to repeat the tests. The reasons are:

- Faulty chemical ignitors (Sobbe - white)
- Faulty gauge (vacuum, pressure of dust storage chamber)
- Leaky apparatus (o-ring, ball valve)

Dust distribution:

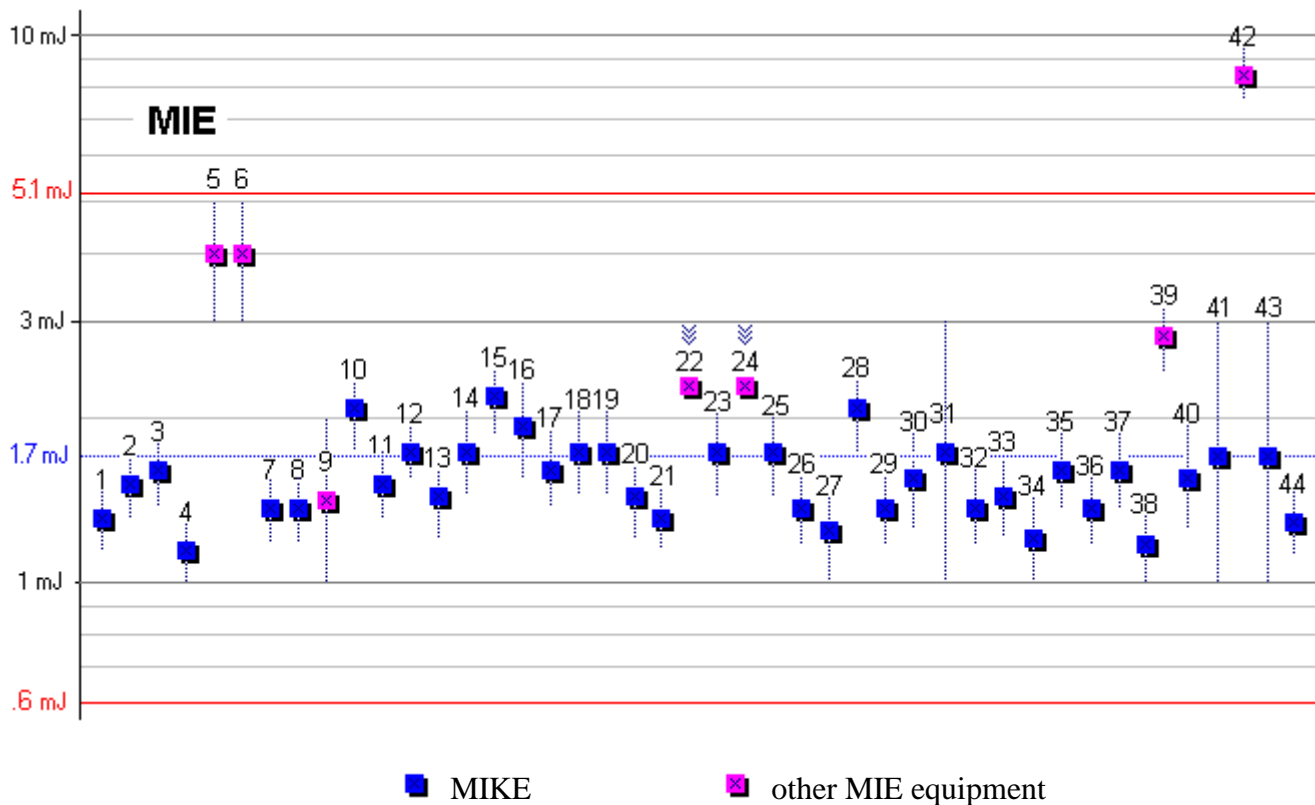
20-l-apparatus with rebound nozzle: 45
 20-l-apparatus with ring nozzle: 1
 Large vessels with ring nozzle: 3 (all)

Large vessels:

Remarkable are the different ignition delay times (t_v) dependent from the type of valve and volume of the vessel:

	t_v (ms)	volume	apparatus
Valve with electro pneumatic drive:	550	1m ³	1
Valve with electro pneumatic drive:	600	1m ³	2

Minimum Ignition Energy MIE



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

Test procedure:

The method for determination of the MIE is described in the „Manual CaRo 03“.

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 (prEN 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.

$$Es = 10^{(\log E2 - I[E2] \cdot (\log E2 - \log E1) / ((NI+I)[E2] + 1))}$$

where is:

$I[E2]$ = number of tests with ignition at the energy E2.

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

Criteria for conformity:

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

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

Accordingly:

Conformity in the CaRo 03 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

The criteria for conformity has been fulfilled by all laboratories (with 1 exception)

List of Participants

Country	Company Laboratory	E-Mail	Pmax Kmax	MIE
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Belgium	Janssen Pharmaceutica NV	cfannes@janbe.jnj.com	✓	✓
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