Final Report

Calibration-Round-Robin

CaRo96

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Conclusion

According to international standards, e.g., ISO 9000, GLP, test equipment for the determination of the explosion indices must be calibrated in certain intervals. This calibration also applies to the 20-l-apparatus and the 1-m^3-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 no internationally recognized reference samples nor reference equipment available for the determination of the explosion indices. Therefore the following calibration method has been carried out successfully:
A dust has been selected, prepared and supplied to **42** 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.

CaRo96 - Reference values for the Explosion Indices Pmax and Kmax

Pmax (bar)	**8.3** ± 10% (7.5 ... 9.1)
Kmax (bar·m/s)	**199** ± 12% (175 ... 223)

CaRo96 - Reference values for the Minimum Ignition Energy MIE

13 mJ < **MIE** < **40** mJ

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Birsfelden, 27. Feb. 97

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Participants

Some participants wish to remain anonymous. Therefore the following list shows only the nationality and type equipment's used.

Country	20-l apparatus	1 m3 vessel	2.4 m3 vessel	minimum ignition energy
Australia	1	-	-	1
Belgium	1	-	-	-
China	1	-	-	-
Germany	13	3	-	8
England	3	-	-	1
Finland	1	-	-	-
Holland	2	1	-	1
Italy	2	-	-	-
Canada	2	-	-	1
Norway	1	-	-	1
Austria	1	-	-	1
Poland	1	-	-	-
Switzerland	5	1	1	3
Spain	1	-	-	-
South Africa	1	-	-	-
Taiwan	1	-	-	1
Hungary	-	-	-	1
USA	3	1	-	2
	40	**6**	**1**	**21**

This calibration round robin test „CaRo96" 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 CaRo96 test sample has been milled, homogenized and shipped in an air tight package. Therefore the sample has to be tested „as delivered".

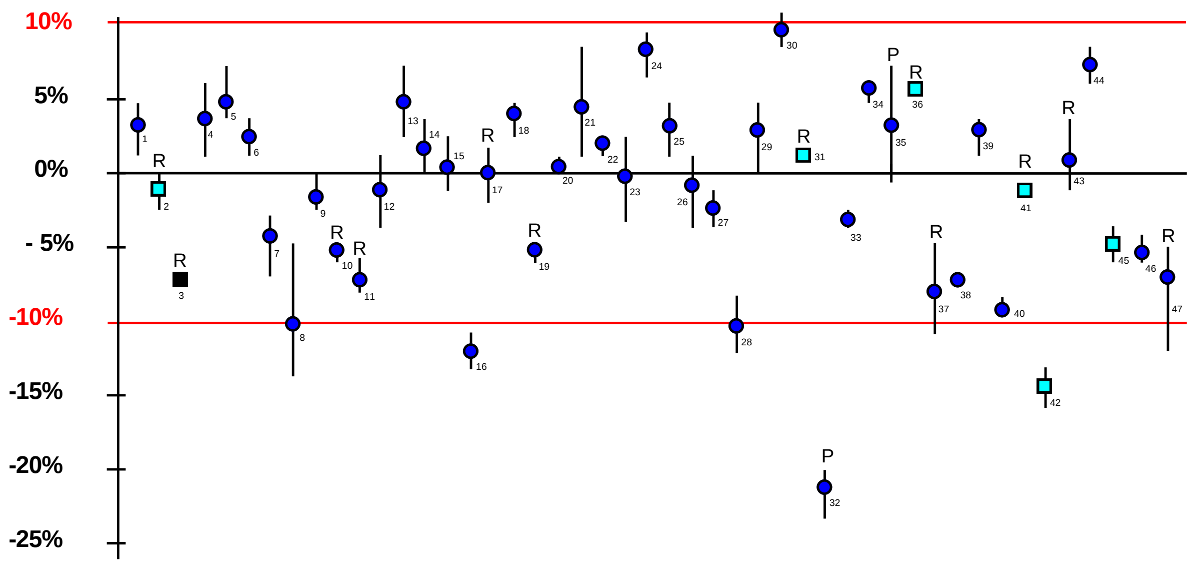
CaRo96 = Irganox 1222 (Antioxidant)

(Phosphonic acid, 3,5-di-tert.butyl-4-hydroxy-benzyl-, diethylester)

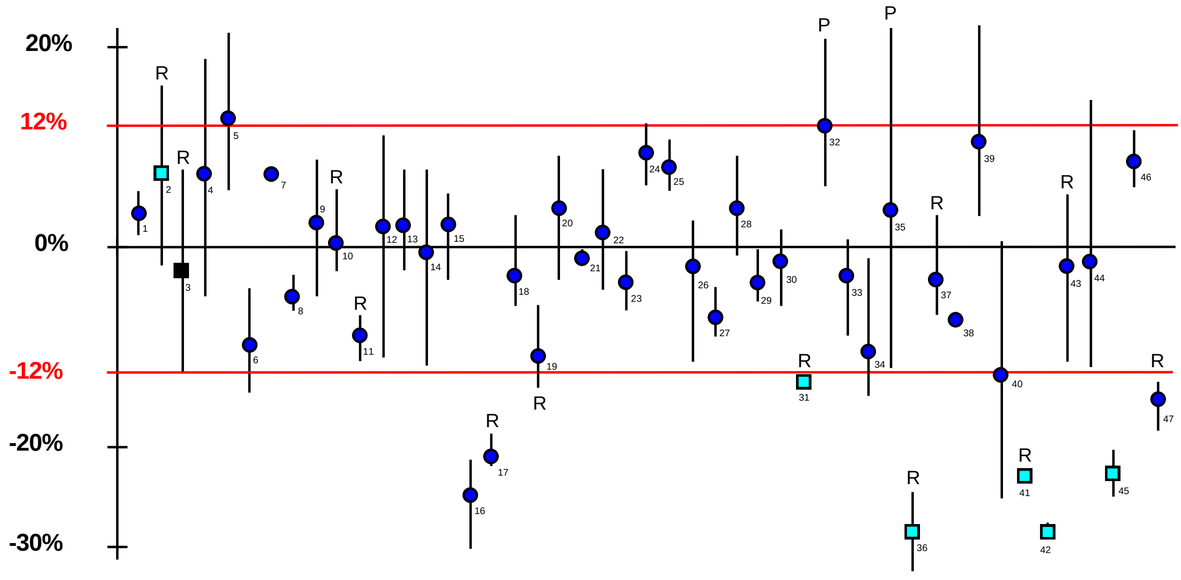
The median value was determined with the method „SYMPATEC": **M (D_{50}) = 32µm**

Explosion Indices Pmax, Kmax

Pmax = 8.3 bar ± 10% (7.5 ... 9.1)



Kmax = 199 bar·m/s ± 12% (175 ... 223)



● 20-l ■ $1m^3$ ■ $2.4m^3$

R ring nozzle P mushroom no letter: rebound nozzle

scatter → 46 certificate no.

Test procedure:

The method for determination of Pmax, Kmax is described in the „Manual CaRo96“.

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, by use of the Cubic Law.

Scatter of Pmax:

The maxima of each series must not deviate by more than **10**% of Pmax.

Otherwise this series must be repeated !

Scatter of (dP/dt)max:

The maxima of each series must not deviate more than the values given in the table below. Otherwise this series must be repeated !

(dP/dt)max	Kmax	Deviation (%)
371 - 740	101 - 200	**+/- 12**
> 740	> 200	**+/- 10**

Calculation of the reference values:

First the mean values of all test results (47) 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.

This procedure of iteration has been repeated until the mean value remains constant:

Reference values, determined	with 37 of 47 equipment’s	with all 47 equipment’s
Pmax (bar)	**8.3** ± 10% (7.5 ... 9.1)	8.2 ± 10% (7.4 ... 9.0)
Kmax (bar·m/s)	**199** ± 12% (175 ... 223)	192 ± 12% (169 ... 215)

Due to the large number of participants, the deviations in the mean values are very small.

Cause of errors:

On our recommendation, some laboratories had to repeat the tests. The reasons are:

a) The scatter of individual series has exceeded the limits given in the CaRo96 - manual.
b) Faulty gauge (vacuum, pressure of dust storage chamber)
c) The protective Silicon-layer on the pressure sensor was too old, hard or even missing.
d) The additional delay of the electrical fuses in the chemical ignitors was not taken into consideration.

Dust distribution in the 20-l-apparatus:

The CaRo96 shows, once more, that the rebound nozzle and the older ring nozzle give the same results. But the rebound nozzle is much easier to clean, therefore we recommend to use in future the **rebound nozzle** only.

The very old mushroom shaped nozzle is less suitable for testing powders (scatter) and should also be replaced with the rebound nozzle.

Large vessels:

The difference in the Kmax-values between different large vessels ($1m^3$, $2.4m^3$) are far too high (up to 40%). The CaRo96-dust has been classified too low by some laboratories.

The reason is a too low turbulence, caused by not optimized dust distribution system valves.

Minimum Ignition Energy MIE

Test procedure:

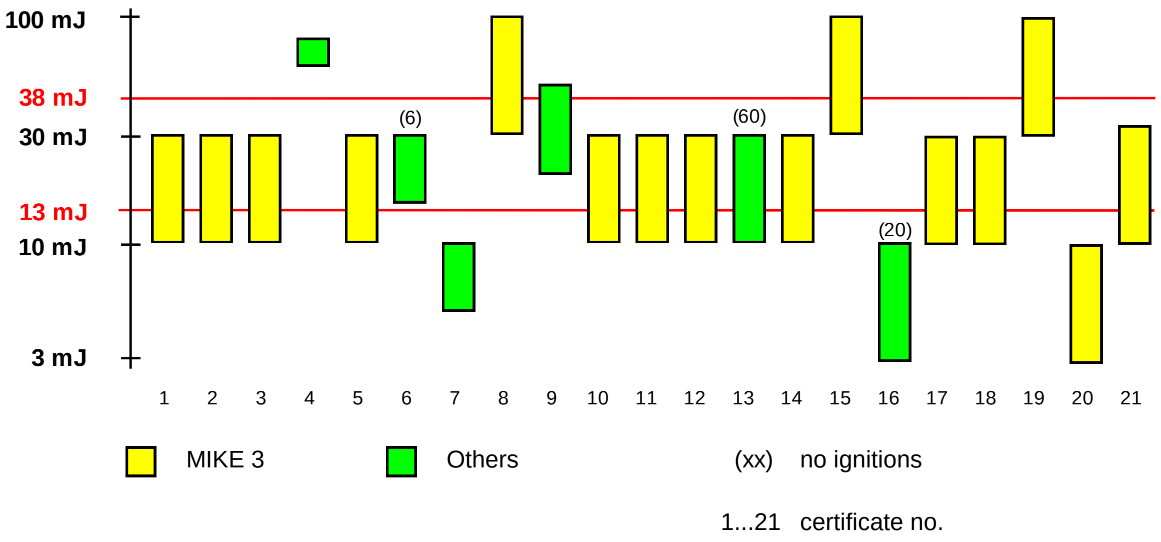
The method for determination of the MIE is described in the „Manual CaRo96“.

MIE - Calculation of the reference values, method 1:

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

E1 < MIE < E2

For each energy value E1 and E2 the following procedure has been used separately:
First the mean values of all test results (21) has been calculated. In a 2nd step all results with both values (E1, E2) outside of the tolerance band are excluded for the subsequent calculation of the mean value.
This procedure of iteration has been repeated until the mean values remain constant:



References, determined	with 17 of 21 equipment’s	with all 21 equipment’s
E1, E2	**13** mJ < MIE < **38** mJ	12 mJ < MIE < 33 mJ

Due to the large number of participants, the deviations in the mean values are very small.

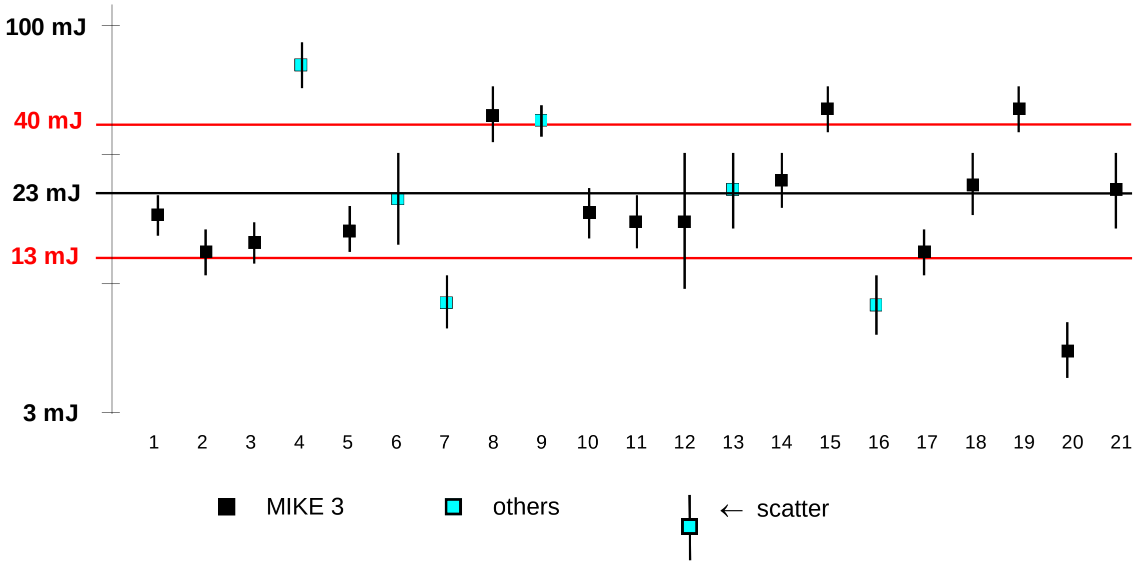
MIE - Calculation of the reference values, method 2:

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

E1 < MIE < E2

In addition to E1 and E2, this method considers the probability of ignition. The number of dust concentrations **with** ignition, divided by the total number of dust concentrations tested (at same ignition energy) gives the probability of ignition and allows to estimate the position of the MIE in the E1-E2 range.

This „statistical“ energy value „Es“ simplifies the comparison between individual test results and has no meaning for safety measures.



References, determined	with 17 of 21 equipment’s	with all 21 equipment’s
E1, Es, E2	**13** mJ < **23** mJ < **40** mJ	11 mJ < 20 mJ < 36 mJ

The number of dust concentrations to be tested has not been specified in CaRo96 and was therefore varying. We considered this matter with the introduction of a scatter. All test results which hit with their scatter the energy band E1-E2 can be accepted for the subsequent calculation of the mean value.

an example:

	IE/mg	300	600	900	1200	1500		probability
E2 =	30mJ	(10)	7	1	2	(10)	→	**3** of **5**
E1 =	10mJ		(10)	(10)	(10)			

Es = l0^ (log E2 - **3** • (log E2 - log E1) / (**5** + 1)) = 17mJ

Note: both methods show roughly the same result.