Final Report

Calibration-Round-Robin

CaRo98

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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 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 **38** 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.

CaRo98 - Reference values for the Explosion Indices Pmax and Kmax

Pmax (bar)	**8.3** ± 10% (7.5 ... 9.1)
Kmax (bar·m/s)	**236** ± 10% (212 ... 260)

CaRo98 - Reference values for the Minimum Ignition Energy MIE

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

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Participants

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

country	20-l apparatus	1 m3 vessel	2.4 m3 vessel	minimum ignition energy
Australia	1	1		1
Belgium				1
Germany	9	1		7
England	3			3
Finland	1			
France	4			2
Holland	1			
India	1			
Italy				1
Japan	1			2
Norway	1			1
Switzerland	4	1	1	2
Spain	1			1
South Africa	1			
Taiwan	1			1
Hungary				1
USA	4	1		2
total	**33**	**4**	**1**	**25**

This calibration round robin test „CaRo98“ 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 CaRo98 test sample has been milled, homogenized and shipped in an air tight package. Therefore the sample has to be tested „as delivered“.

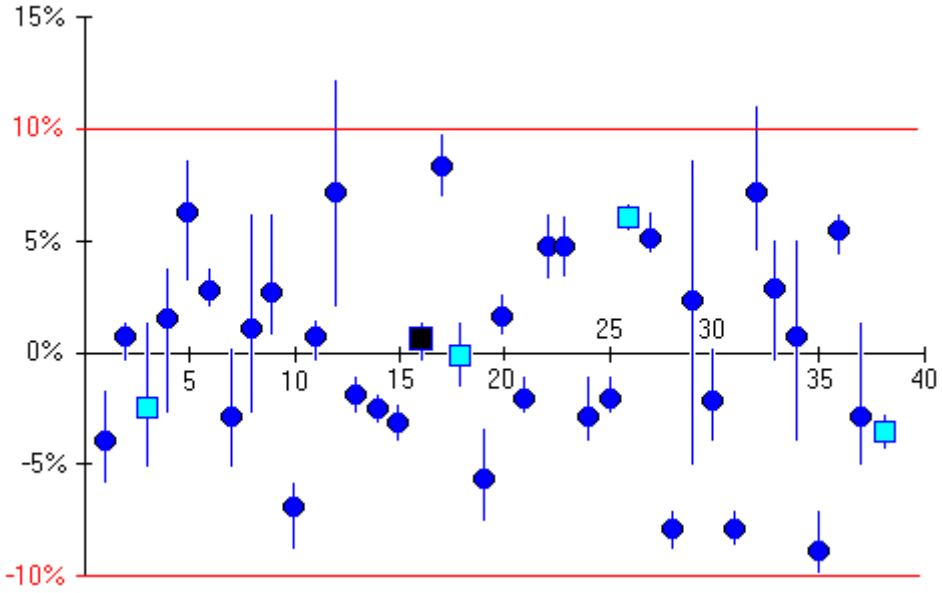
CaRo98 = Niacin USP (Pyridine-3-carboxylic acid)

Particle size distribution:

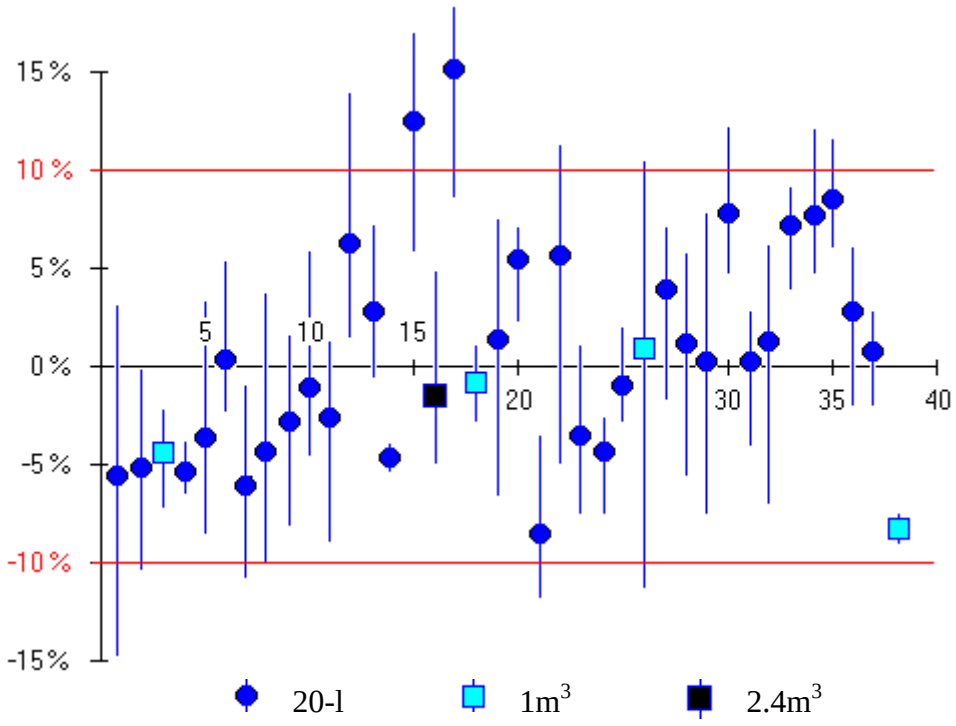
d (0.1)	d (0.5)	d (0.9)
10% of particles	50% of particles	90% of particles
< 9 µm	**< 25 µm**	**< 65 µm**

Explosion Indices Pmax, Kmax

Pmax = 8.3 bar ± 10% (7.5 ... 9.1)



Kmax = 236 bar·m/s ± 10% (212 ... 260)



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 Pmax, Kmax is described in the „Manual CaRo98“.

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.

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 !

Calculation of the reference values:

First the mean values of all test results (38) has been calculated. In a 2nd step all results outside of the tolerance band (2) are excluded prior to the subsequent calculation of the mean value.

Reference values, determined	with 36 of 38 equipment	with all 38 equipment
Pmax (bar)	**8.3** ± 10% (7.5 ... 9.1)	8.3 ± 10% (7.5 ... 9.1)
Kmax (bar·m/s)	**236** ± 10% (212 ... 260)	238 ± 10% (214 ... 262)

Cause of errors:

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

a) Faulty gauge (vacuum, pressure of dust storage chamber)
b) Leaky apparatus (o-ring, ball valve)
c) The protective Silicon-layer on the pressure sensor’s was too old and too hard.
d) Missing cooling on the 20-l-apparatus.

Dust distribution:

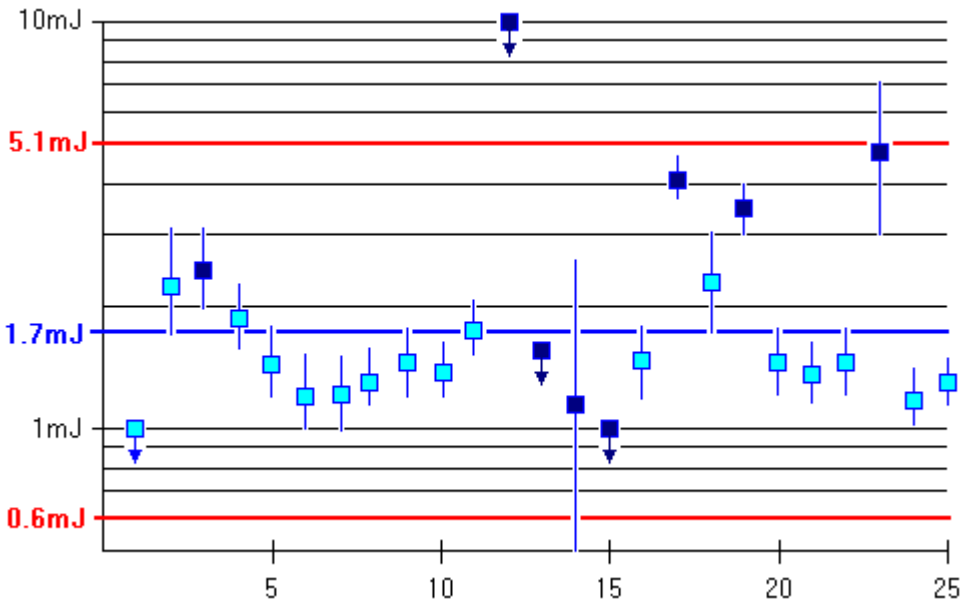
20-l-apparatus with rebound nozzle: 29
20-l-apparatus with ring nozzle: 4
Large vessels with ring nozzle: 5

Large vessels:

Also with large vessels ($1m^3$, $2.4m^3$) a good correspondence of test results has been achieved. Remarkable are the different ignition delay times (tv) dependent from the type of valve:

Valve with blasting cap activation: tv = 600ms (2 apparatus)
Valve with electro pneumatic drive: tv = 550ms (3 apparatus)

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 CaRo98“.

Estimation of the statistical energy (Es):

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

$$E1 < MIE < E2$$

Fur 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:

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.

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

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

Dependent from the data received, the following methods has been applied:

no	data	method of Es-estimation:
17	E1, E2, I, NI	Es = 10^ (log E2 - I[E2] • (log E2 - log E1) / ((NI+I)[E2]+1))
4	E1, E2	Es = 10^ ((log E2 + log E1) / 2)
4	E2	an estimation is impossible

Criteria for conformity:

Conformity between two equipment (a, b) is given, when the Es-values differ less than a factor of 3 (CEN 305 WG 1, MIE-draft).

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

Accordingly:

Conformity in the CaRo98 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**

An estimation of (Es) is impossible with test results indicating a single energy (E2), but the classification of the test dust was correct.

The criteria for conformity has been fulfilled by all laboratories.