

Calibration-Round-Robin CaRo 19

Final Report

- 1. Conclusion..... 1
- 2. Explosion Indices Pmax, Kmax..... 3
- 3. Minimum Ignition Energy MIE..... 5
- 4. List of Participants 6
- 5. Standards, History..... 8

1. Conclusion

According to international standards 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 must be calibrated at regular intervals (at least every 12 months, or following any significant maintenance or repair). For this purpose, an international calibration round test (calibration round robin = CaRo) has been carried out periodically since 1993.

A dust has been selected, prepared and supplied to **58** test laboratories all over the world. The mean values of the explosion indices, measured by the participating laboratories, have 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 procedure.

CaRo 19 – Reference values for the Explosion Indices Pmax and Kmax

| | |
|-----------------------|--------------------------------|
| Pmax (bar) | 8.2 ± 10% (7.4 ... 9.0) |
| Kmax (bar·m/s) | 243 ± 10% (218 ... 267) |

CaRo 19 – Reference values for the Minimum Ignition Energy MIE

| | | |
|---------------|---------------|---------------|
| Es / 3 | Es | Es · 3 |
| 0.6 mJ | 1.7 mJ | 5.0 mJ |

This report shall not be published or reproduced other than in full.

Cesana AG

Baiergasse 56
 CH-4126 Bettingen
 Switzerland

Phone +41 61 534 01 61
 E-Mail caro@cesana-ag.ch
 Internet www.cesana-ag.ch

1.1 Participants:

Further details about participants who have agreed to a publication, can be found in section 4.

| | Pmax, Kmax (73) | | MZE (73) | |
|-----------------|-----------------|----------|-----------|----------|
| | 20-l | 1 m3 | MIKE | others |
| Australia | 1 | | 1 | |
| Austria | 2 | | 1 | |
| Belgium | 1 | | 1 | |
| Czech Republic | 1 | | 1 | |
| France | 5 | | 3 | |
| Germany | 18 | 3 | 16 | |
| India | | | 1 | |
| Italy | 2 | | 3 | |
| Japan | 1 | | 2 | |
| Norway | 2 | | 1 | |
| Poland | 2 | | | 2 |
| Romania | 1 | | | |
| South Africa | 1 | | | |
| Spain | 1 | | 1 | |
| Sweden | 1 | | | 1 |
| Switzerland | 3 | | 5 | |
| The Netherlands | 1 | | 1 | |
| United Kingdom | 4 | | 2 | 3 |
| USA | 17 | 1 | 11 | 3 |
| Total: | 64 | 4 | 50 | 9 |

1.2 Test substance:

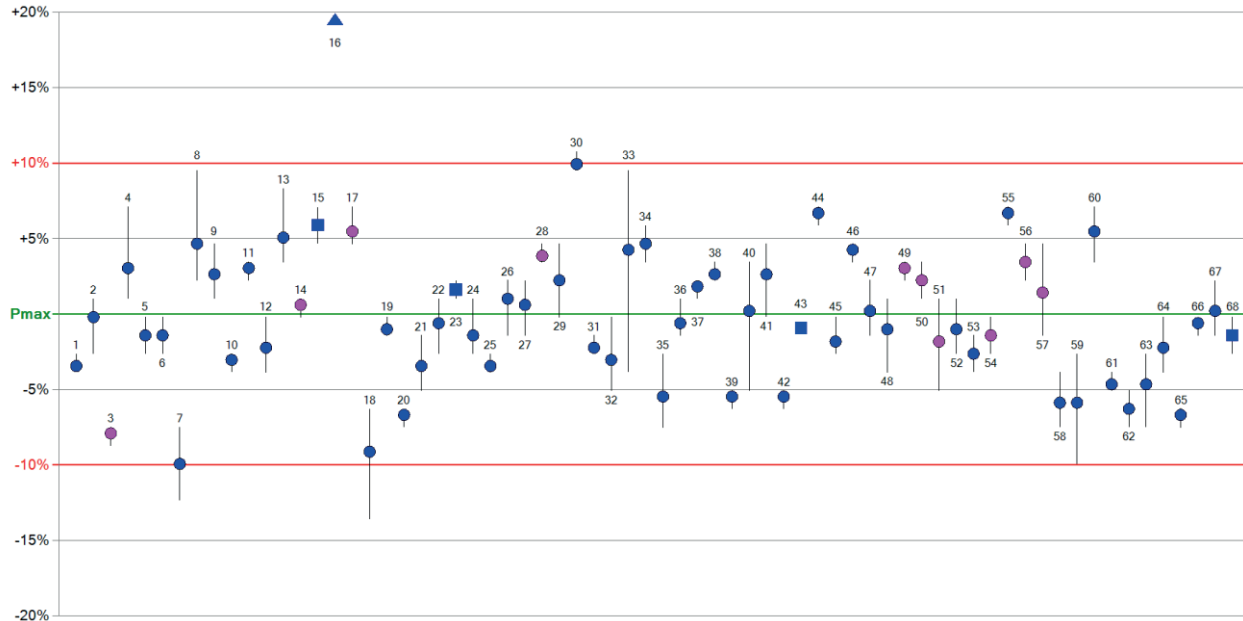
For correct calibration the CaRo 19 test sample has been milled, homogenized and shipped in an air tight package. Therefore the sample has to be tested „as delivered“.

CaRo 19 = Niacin CaRo Test Dust (Nicotinic acid)

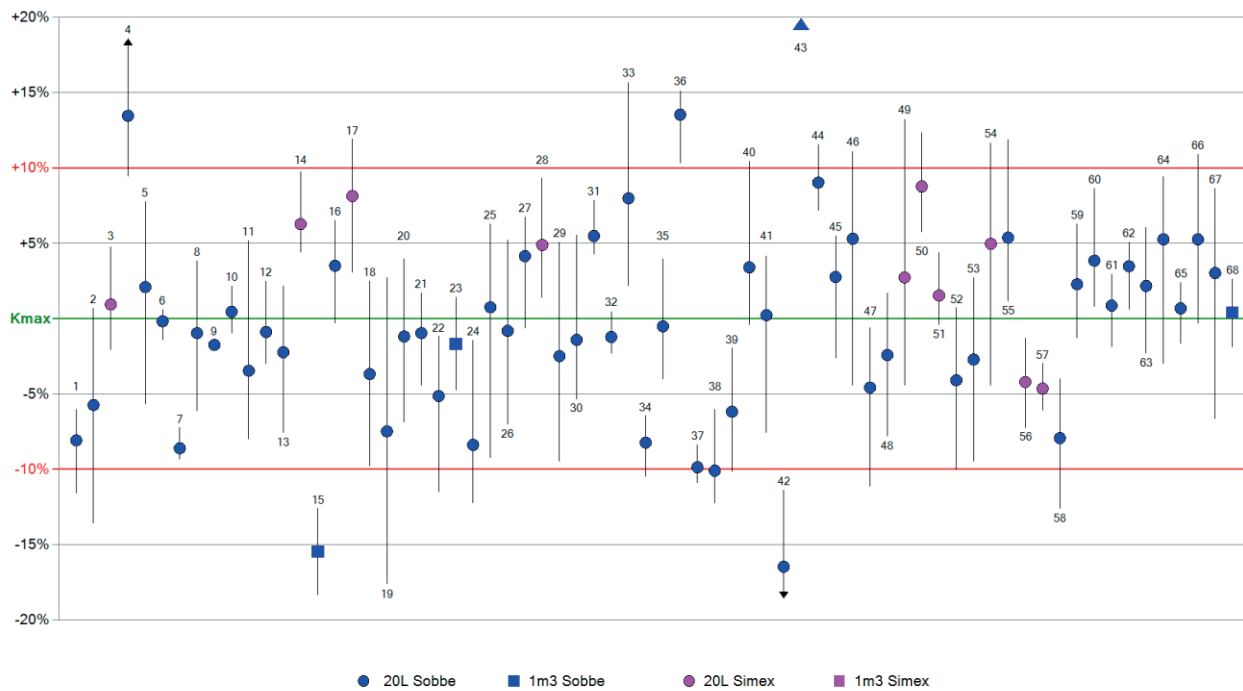
| Particle size: | d 10 [µm] | d 50 = median [µm] | d 90 [µm] |
|----------------|-----------|--------------------|-----------|
| Sample 1 | 4.6 | 20.5 | 77.7 |
| Sample 2 | 4.6 | 20.1 | 76.3 |
| Sample 3 | 4.6 | 20.2 | 76.4 |
| Sample 4 | 4.7 | 20.3 | 76.8 |

2. Explosion Indices Pmax, Kmax

Pmax = 8.2 bar ±10% (7.4 ... 9.0) @ 527 g/m³



Kmax = 243 bar·m/s ±10% (218 ... 267) @ 699 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 P_{max} , K_{max} is described in the „Manual CaRo 19“

2.2 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 $(dP/dt)_{max}$.

2.3 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!

2.4 Calculation of the reference values:

First the mean value of all test results (68) has been calculated.

In a second 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 Cause of errors:

Some laboratories had to repeat the tests.

The reasons were:

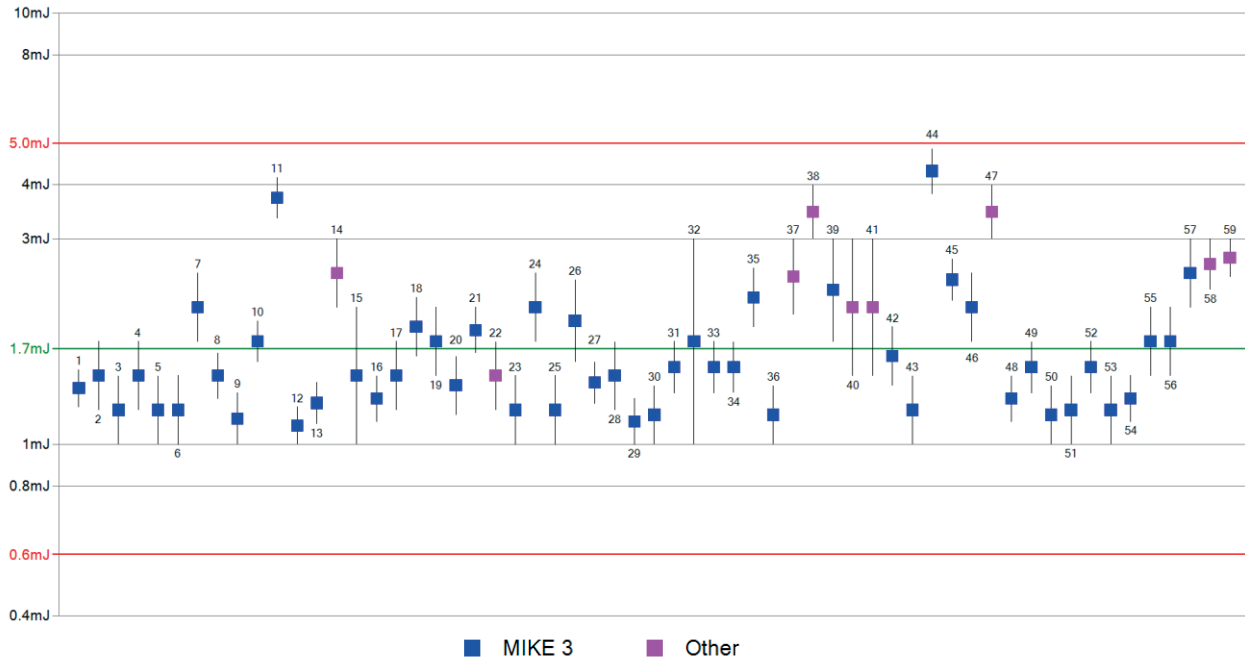
- a) Too high or too low initial pressure P_i .

The explosion indices P_{max} and K_{max} are direct proportional to the initial pressure P_i , the pressure in the sphere at the moment of ignition.

This relation is linear up to an initial pressure of approx. 3 bar.

- b) Too low K_{max} values due to additional ignition delay time caused by a bad contact between ignition rod and the leads of the ignitors (inadequate cleaning).
- c) Induced turbulences caused by pressure oscillations of the ignitors.

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 19“.

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 apparatuses, 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):

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

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 in the CaRo 19 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:

| | | |
|---------------|---------------|---------------|
| Es / 3 | Es | Es • 3 |
| 0.6 mJ | 1.8 mJ | 5.3 mJ |

3.4 Cause of errors:

- a) The use of synthetic air can increase the MIE.
- b) Broken wires in high voltage and ground cable.

4. List of Participants

| Country | Company Laboratory | E-Mail | Pmax Kmax | MIE |
|----------------|---|--|--------------|-----|
| Australia | Simtars | negar.fasihiani@simtars.com.au | ✓ | ✓ |
| Austria | AUVA | stp@auva.at | ✓ | ✓ |
| Austria | FireX Greßlehner GmbH | dietmar.gresslehner@firex.at | ✓ | |
| Belgium | Adinex N.V. | info@adinex.be | ✓ | ✓ |
| Czech Republic | VVUÚ, a.s. | mokosl@vvuu.cz | ✓ | ✓ |
| France | CNRS - LRGP | olivier.dufaud@univ-lorraine.fr | ✓ | |
| France | INERIS | ghislain.binotto@ineris.fr | ✓ | ✓ |
| France | SANOFI | cecile.masson-rojas@sanofi.com | ✓ | ✓ |
| France | SOLVAY | gilles.roman@solvay.com | ✓ | ✓ |
| Germany | BAM | mike.wappler@bam.de | ✓ | ✓ |
| Germany | BASF SE | johannes.a.fischer@basf.com | ✓ | ✓ |
| Germany | Bayer AG Leverkusen | verena.gramm@bayer.com | ✓ | ✓ |
| Germany | BGN | madlen.schoenherr@bgn.de | ✓ | ✓ |
| Germany | Boehringer Ingelheim Pharma GmbH & Co. KG | juergen.leininger@boehringer- ingelheim.com | ✓ | ✓ |
| Germany | consilab Gesellschaft für Anlagensicherheit mbH | jonas.kremer@consilab.de | ✓ | ✓ |
| Germany | DEKRA Testing and Certification GmbH | nina.thimm@dekra.com | ✓ | |
| Germany | EVONIK Technology & Infrastructure GmbH | matthias.vorwinkel@evonik.com | ✓ | ✓ |
| Germany | Henkel AG & Co. KGaA | michaela.berchter@henkel.com | ✓ | ✓ |
| Germany | IBExU | f.flemming@ibexu.de | ✓ | ✓ |
| Germany | IFA - DGUV | sascha.hohmann@dguv.de | ✓ | ✓ |
| Germany | Inburex Consulting GmbH | rene.dworschak@Inburex.com | ✓ | ✓ |
| Germany | Merck KGaA | thomas.keil@merckgroup.com | | ✓ |
| Germany | Siemens AG | michael.nau@siemens.com | ✓ | ✓ |
| Germany | Wacker Chemie AG | alfred.augsberger@wacker.com | ✓ | ✓ |
| India | GVS Cibatech Private Limited | vijay.bhujle@cibatech.com | | ✓ |
| Italy | INNOVHUB Stazioni sperimentali per l'industria S.r.l. | antonella.mazzei@mi.camcom.it | ✓ | ✓ |
| Italy | Redox s.r.l. | reactivity@labredox.com | ✓ | ✓ |

| Country | Company Laboratory | E-Mail | Pmax Kmax | MIE |
|-----------------|---|-----------------------------------|--------------|-----|
| Japan | Sumika Chemical Analysis Center | k.ito@scas.co.jp | ✓ | ✓ |
| Japan | Technology Institution of Industrial Safety | matsunaga@tiis.or.jp | | ✓ |
| Norway | Gexcon AS | terje.froystein.larsen@gexcon.com | ✓ | ✓ |
| Poland | Central Mining Institute | zdyduch@gig.eu | ✓ | ✓ |
| Poland | Research Center for Fire Protection (CNBOP-PIB) | dkbak@cnbop.pl | ✓ | ✓ |
| Romania | INCD INSEMEX Petrosani | insemex@insemex.ro | ✓ | |
| South Africa | CSIR | imthombe@csir.co.za | ✓ | |
| Spain | LOM-AT. | liliana.medic@upm.es | ✓ | ✓ |
| Sweden | RISE Research Institutes of Sweden | johanna.degrahl@ri.se | ✓ | ✓ |
| Switzerland | Dottikon Exclusive Synthesis AG | ralf.weingart@dottikon.com | | ✓ |
| Switzerland | DSM | romeo.isner@dsm.com | | ✓ |
| Switzerland | Firmenich S.A. | maria.fabbo@firmenich.com | ✓ | |
| Switzerland | Givaudan International SA | chantal.berchten@givaudan.com | ✓ | ✓ |
| Switzerland | Syngenta Crop Protection | julien.parra@syngenta.com | | ✓ |
| Switzerland | TÜV SÜD Process Safety | christina.mendelin@tuev-sued.ch | ✓ | ✓ |
| The Netherlands | Nouryon | annienke.kingma@nouryon.com | ✓ | ✓ |
| United Kingdom | Chilworth Technology / Dekra Insight | aidan.bushell@dekra.com | ✓ | ✓ |
| United Kingdom | Sigma-HSE (UK) Ltd | groggers@sigma-hse.com | ✓ | ✓ |
| United Kingdom | Syngenta | phil.robinson@syngenta.com | ✓ | ✓ |
| USA | Ashland LLC | tolechnowicz@ashland.com | ✓ | ✓ |
| USA | BASF Corporation | andrew.charlick@basf.com | ✓ | ✓ |
| USA | DEKRA Process Safety | don.churchwell@dekra.com | ✓ | ✓ |
| USA | EMSL Analytical, Inc. | emirica@emsl.com | ✓ | ✓ |
| USA | Exponent, Inc. | jvickery@exponent.com | ✓ | ✓ |
| USA | Fauske & Associates, LLC | dastidar@fauske.com | ✓ | ✓ |
| USA | Fike Corporation FPS | adam.morrison@fike.com | ✓ | ✓ |
| USA | Firmenich, Inc. | peter.de.rege@firmenich.com | ✓ | ✓ |
| USA | IEP Technologies | john.lussier@hoerbiger.com | ✓ | ✓ |
| USA | ioKinetic, LLC | barrett.c.iokinetic@iomosaic.com | ✓ | ✓ |

5. Standards, History

5.1 Standards:

The following Standards have been applied:

- EN 13821: Determination of minimum ignition energy of dust/air mixtures
- EN 14034-1: Determination of max. explosion pressure Pmax ...
- EN 14034-2: Determination of max. rate of explosion pressure rise (dp/dt)_{max} ...
- ASTM E1226: Standard Test Method for Explosibility of Dust Clouds
- EN ISO/IEC 80079-20-2: ... Material characteristics. Combustible dust methods

5.2 History:

Our previous world-wide round robin tests:

| | | |
|------------------------|-------------------------|-------------------------|
| CaRo93: 77 apparatuses | CaRo05: 98 apparatuses | CaRo15: 131 apparatuses |
| CaRo96: 68 apparatuses | CaRo07: 77 apparatuses | CaRo17: 144 apparatuses |
| CaRo98: 63 apparatuses | CaRo09: 90 apparatuses | CaRo18: 41 apparatuses |
| CaRo00: 69 apparatuses | CaRo11: 122 apparatuses | |
| CaRo03: 93 apparatuses | CaRo13: 112 apparatuses | |

→ All final reports can be downloaded here: <https://www.cesana-ag.ch/Calibration.shtml>

References

Cesana Ch., Eiche M., Schwaninger M., 2019,
Quality Management in the Determination of Safety Characteristics, CET-Paper