

Final Report
Calibration-Round-Robin
CaRo 07

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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 **41** 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 07 - Reference values for the Explosion Indices P_{max} and K_{max}

| | |
|----------------------------------|--------------------------------|
| P_{max} (bar) | 8.0 ± 10% (7.2 ... 8.8) |
| K_{max} (bar·m/s) | 241 ± 10% (217 ... 265) |

CaRo 07 - Reference values for the Minimum Ignition Energy MIE

| | | |
|--------------------------|----------------------|--------------------------|
| E_s / 3 | E_s | E_s • 3 |
| 0.6 mJ | 1.8 mJ | 5.4 mJ |



Birsfelden, January 2008

Adolf Kühner AG
Christoph Cesana

Participants

For details see section "list of participants".

| | Pmax, Kmax (41) | | MIE (36) | |
|----------------|-----------------|------------------|-----------|----------|
| | 20-1 | 1 m ³ | MIKE | others |
| Austria | 1 | | 1 | |
| Australia | 2 | | 1 | |
| Belgium | 3 | | 2 | |
| Canada | 1 | | 1 | |
| Czechoslovakia | 1 | | | |
| Germany | 6 | | 8 | 1 |
| England | 5 | | 2 | 2 |
| Finland | 1 | | | |
| France | 4 | | 3 | |
| Holland | 1 | | 1 | |
| Hungary | | | | 1 |
| Italy | 2 | | 1 | |
| Japan | | | 1 | |
| Norway | 1 | | | 1 |
| Romania | 1 | | | |
| Spain | 1 | | 1 | |
| Switzerland | 4 | | 3 | |
| U.S.A | 7 | | 6 | |
| total: | 41 | 0 | 31 | 5 |

This calibration round robin test „CaRo 07“ 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 07 test sample has been milled, homogenized and shipped in an air tight package. Therefore the sample has to be tested „as delivered“.

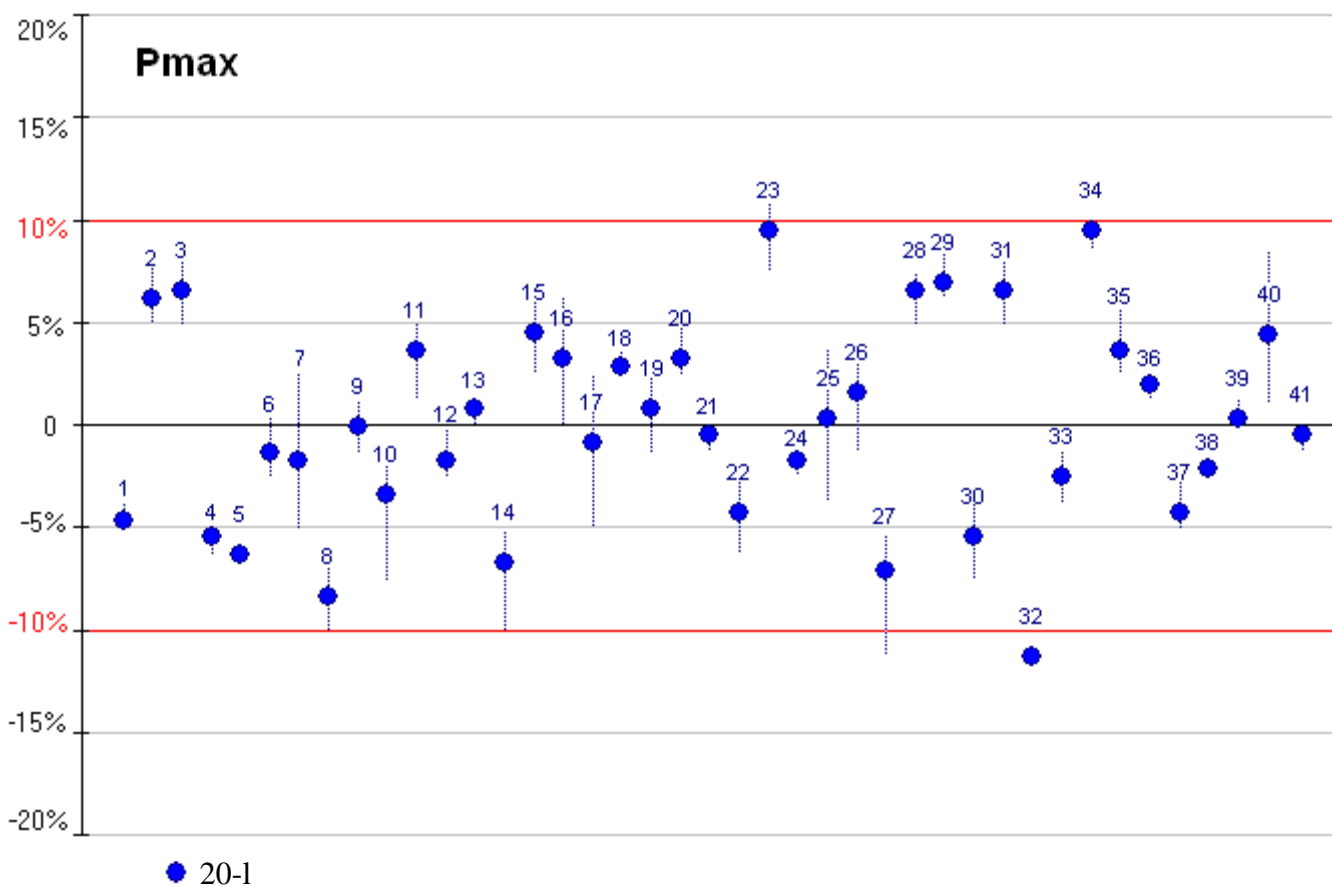
CaRo 07 = Niacin USP special (Nicotinic acid)

Particle size:

| | d 10 | d 50 = median | d 90 |
|----------|------|---------------|-------|
| Sample 1 | 7 µm | 28 µm | 74 µm |
| Sample 2 | 7 µm | 27 µm | 77 µm |
| Sample 3 | 7 µm | 27 µm | 75 µm |
| Sample 4 | 7 µm | 26 µm | 69 µm |

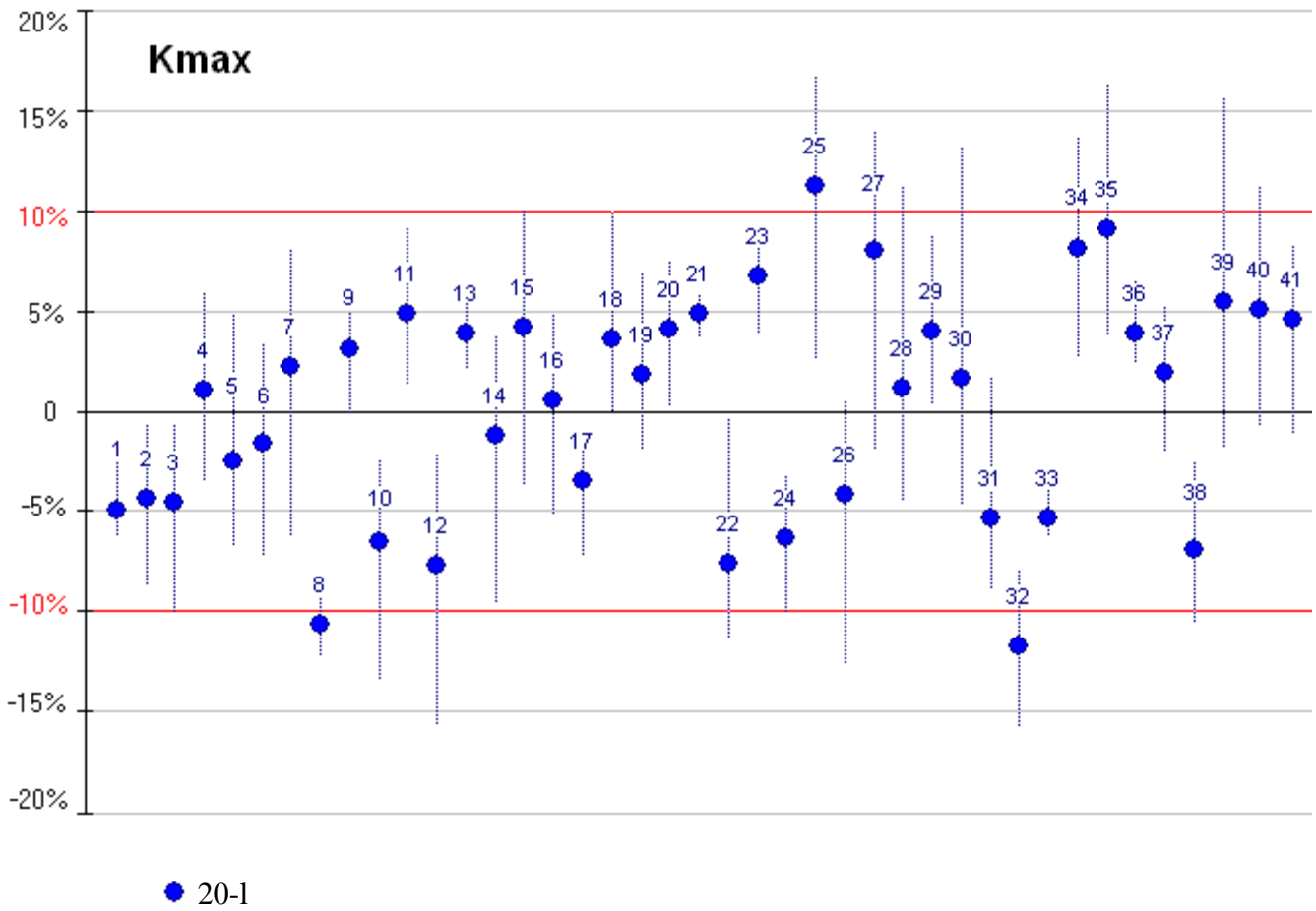
Explosion Indices Pmax, Kmax

Pmax = 8.0 bar ± 10% (7.2 ... 8.8) at 520 g/m³



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

K_{max} = 241 bar·m/s ± 10% (217 ... 265) at 690 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 07“.

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

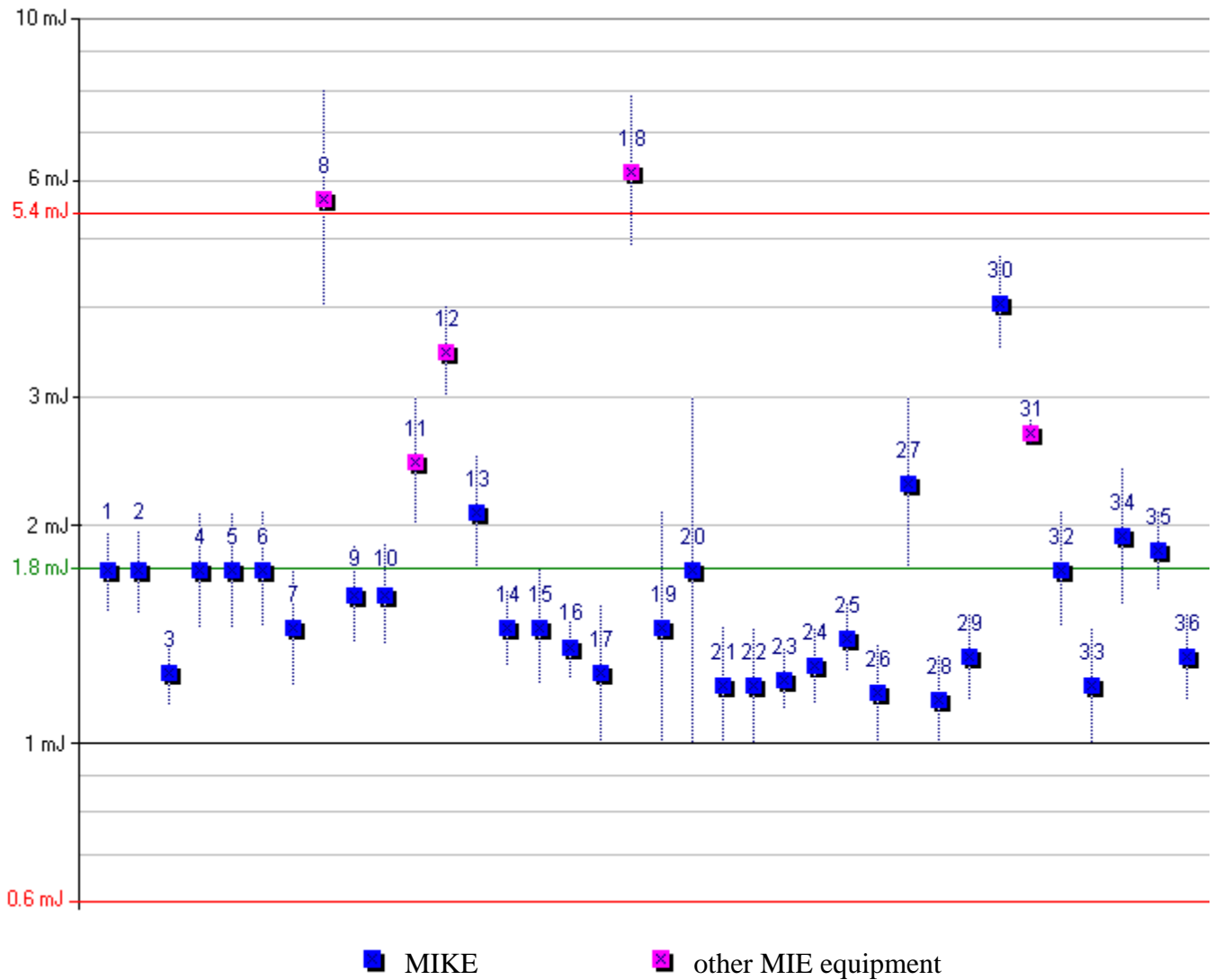
Scatter of P_{max} and K_{max}

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 (41) 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.

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 07“.

Estimation of the statistical energy (Es):

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

$$E1 < \text{MIE} < E2$$

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: $I[E_2]$ = number of tests with ignition at the energy E2.
 $(NI+I)[E_2]$ = 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 (EN 13821).

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

Accordingly:

Conformity in the CaRo 07 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 | 1.8 mJ | 5.4 mJ |

List of Participants

| Country | Company Laboratory | E-Mail | Pmax Kmax | MIE |
|----------------|---|--|-----------|-----|
| Austria | AUVA Sicherheitstechnische Prüfstelle der allgemeinen Unfallversicherungsanstalt | renate.waglechner@auva.at | ✓ | ✓ |
| Australia | SIMTARS, Dept. Of Natural Resources & Mines | ray.davis@nrm.gld.gov.au | ✓ | ✓ |
| Australia | TestSafe Fire & Explosion, Australia | david.pearson@workover.nsw.gov.au | ✓ | |
| Belgium | ADINEX N.V. | filip.verplaetsen@mech.kuleuven.be | ✓ | ✓ |
| Belgium | Anonym | | ✓ | ✓ |
| Belgium | D.D.ENGINEERING BVBA, Laboratory | magda.lebleu@ddeng.be | ✓ | |
| Canada | Dalhousie University, Laboratory Chemical eng. | paul.amyotte@dal.ca | ✓ | ✓ |
| Czechoslovakia | VVUU, a.s. vedouci scolichocentra Ostrava Czech Rep. | mokosl@vvuu.cz | ✓ | |
| Germany | BASF Aktiengesellschaft GCT/S Sicherheitstechnik | stefan.bitterlich@basf-ag.de | ✓ | ✓ |
| Germany | Bayer CropScience AG Formulation Technology | Juergen.Zindell@bayercropscience.com | | ✓ |
| Germany | Boehringer Ingelheim Pharma GmbH & Co. KG AVE/Sicherheits- und Materialprüfstelle | Beate.Podlech@boehringer-ingelheim.com | ✓ | ✓ |
| Germany | Currenta GmbH & Co. OHG, Sicherheitstechnisches Laboratorium | michael.schriewer@currenta.de | ✓ | ✓ |
| Germany | IBE-xU Institut für Sicherheitstechnik GmbH Staublabor | j.lucas@ibexu.de | ✓ | ✓ |
| Germany | Inburex Consulting GmbH | Martin-Gosewinkel@inburex.com | ✓ | ✓ |
| Germany | Nycomed GmbH, Sicherheitslabor | ralf.weingart@nycomed.com | | ✓ |

| Country | Company Laboratory | E-Mail | Pmax Kmax x | MIE |
|---------|--|------------------------------------|-------------------|-----|
| Germany | Wacker-Chemie GmbH Chemical Services/Chemical Safety | alfred.augsberger@wacker.com | | ✓ |
| England | BRE - Global Laboratory, Dust Test | amendt@bre.co.uk | ✓ | ✓ |
| England | Chilworth Technology Ltd. Laboratory I.E.H. | pbremble@chilworth.co.uk | ✓ | ✓ |
| England | Ciba Speciality Chemicals Laboratory CSTL | philip.robinson@ciba.com | ✓ | ✓ |
| England | Glaxo Smith Kline, Laboratory Fire & Explosion | Miahael.I.Gilmore@gsk.com | ✓ | |
| Finland | VTT Technical Research Centre of Finland | johan.mangs@vtt.fi | ✓ | |
| France | INERIS Laboratory DRA/PROC | Pierre-Alexandre.LE-LORE@ineris.fr | ✓ | ✓ |
| France | LSGC-CNRS-ENSIC | Laurent.Perrin@ensic.impl-nancy.fr | ✓ | ✓ |
| France | Sanofi - Chimie, Laboratoire Sécurité des Procédés | marc.huser@sanofi-aventis.com | ✓ | ✓ |
| Holland | DSM Laboratory Resolve | | ✓ | ✓ |
| Hungary | Chemical Works of Gedeon Richter Lt. Laboratory Safety of Chemical | gy.negyesei@richter.hu | | ✓ |
| Italy | Stazione Sperimentale Combustibili Laboratory Infiammabilità | mazzei@ssc.it | ✓ | ✓ |
| Japan | Sumitomo Chemical Co., LTD Safety Engineering Laboratory | takeuchif@sc.sumitomo-chem.co.jp | | ✓ |
| Norway | GexCon AS Dust Explosion Test Laboratory | cta@gexcon.com | ✓ | ✓ |
| Romania | SC D&D SAFE SRL Company | d&dsafe@comtrust.ro | ✓ | |

| Country | Company Laboratory | E-Mail | Pmax Kmax | MIE |
|-------------|---|---------------------------------|--------------|-----|
| Switzerland | Firmenich SA - Prozess Safety Laboratory | franco.ferregutti@wanadoo.fr | ✓ | |
| Switzerland | Hoffmann-La Roche AG, Basel Sicherheitslabor, Abt. PSUS-L | thomas.glarner@roche.com | | ✓ |
| Switzerland | Lonza AG Sicherheitslabor, Visp | ivo.doenni@lonza.com | ✓ | |
| Switzerland | Schweiz. Institut zur Förderung der Sicherheit | abisel@swissi.ch | ✓ | ✓ |
| Spain | Anonym | | ✓ | ✓ |
| U.S.A | BASF Corporation Laboratory Experimental Safety | chunyang.wie@basf.com | ✓ | ✓ |
| U.S.A | Ciba Speciality Chemicals Laboratory CSTL | larry.floyd@cibasc.com | ✓ | ✓ |
| U.S.A | Fauske & Associates, LLC | kfauske@fauske.com | ✓ | ✓ |
| U.S.A | International Speciality Products, Process Safety | jforman@ispxorm.com | ✓ | ✓ |
| U.S.A | KIDDE-FENWAL Combustion Research Center | kemi.sorinmade@kidde-fenwal.com | ✓ | |
| U.S.A | Merck & Co. Inc., Prozess Safety Laboratory | michael_toth@merck.com | ✓ | ✓ |