

**Final Report**  
**Calibration-Round-Robin**  
**CaRo 03**

**Adolf Kühner AG**  
**Dinkelbergstr. 1**  
**CH-4127 Birsfelden**  
**Switzerland**

**Tel. +41 (0)61 319 93 93**  
**Fax. +41 (0)61 319 93 94**  
**E-mail office @ kuhner.com**  
**Internet www.kuhner.com**

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## Conclusion

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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-m<sup>3</sup>-vessel for the determination of P<sub>max</sub> and K<sub>max</sub> 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<sub>max</sub> and K<sub>max</sub>

<b>P<sub>max</sub></b> (bar)	<b>8.1 ± 10%</b> (7.3 ... 8.9)
<b>K<sub>max</sub></b> (bar·m/s)	<b>232 ± 10%</b> (209 ... 256)

### CaRo 03 - Reference values for the Minimum Ignition Energy MIE

<b>E<sub>s</sub> / 3</b>	<b>E<sub>s</sub></b>	<b>E<sub>s</sub> • 3</b>
<b>0.6 mJ</b>	<b>1.7 mJ</b>	<b>5.1 mJ</b>



Birsfelden, January 2004

Adolf Kühner AG  
Christoph Cesana

## Participants

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For details see section "list of participants".

	Pmax, Kmax (49)		MIE (44)	
	20-l	1 m <sup>3</sup>	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	
<b>total:</b>	<b>46</b>	<b>3</b>	<b>37</b>	<b>7</b>

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“.

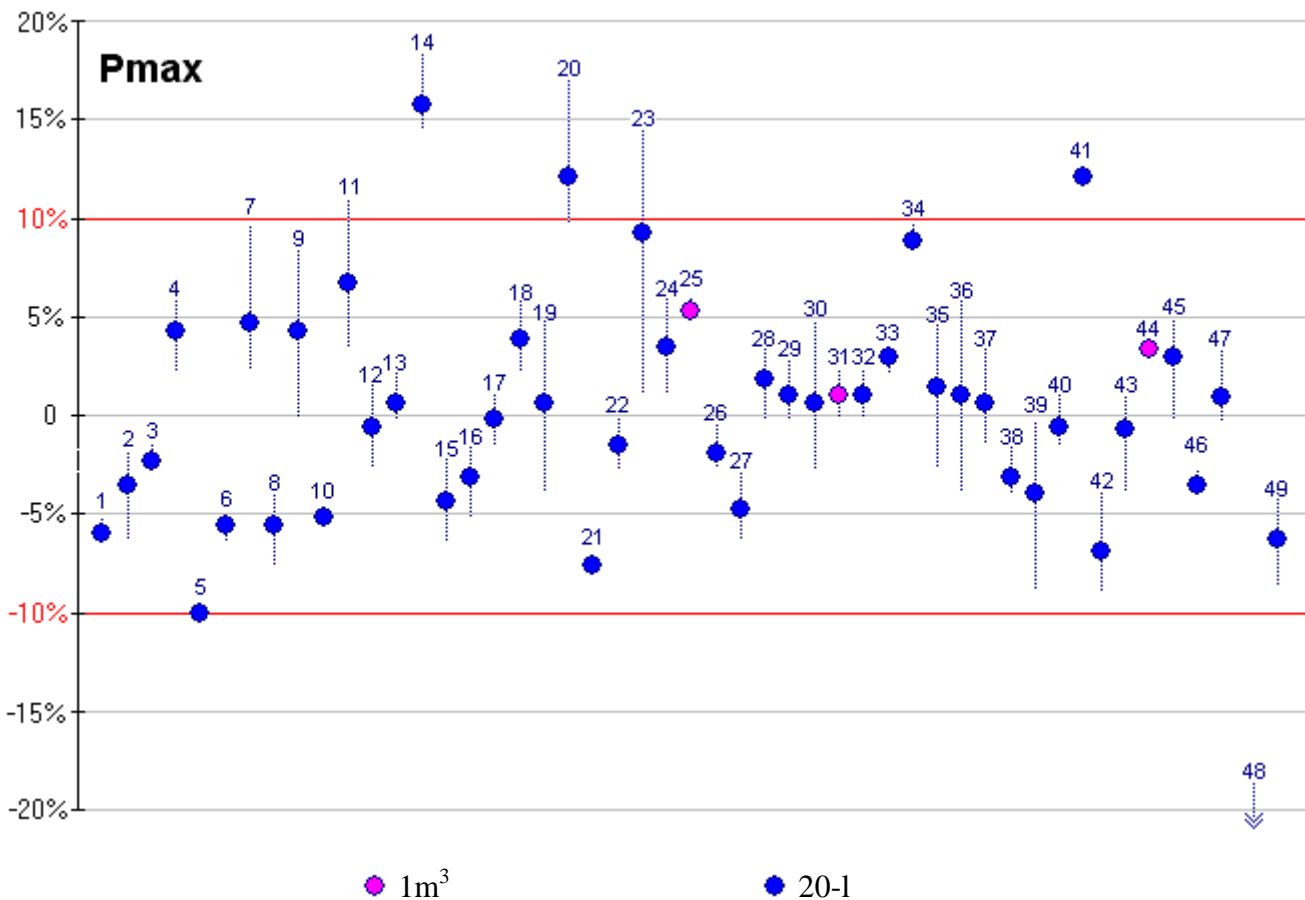
**CaRo 03 = Niacin USP (Nicotinic acid)**

#### Particle size:

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

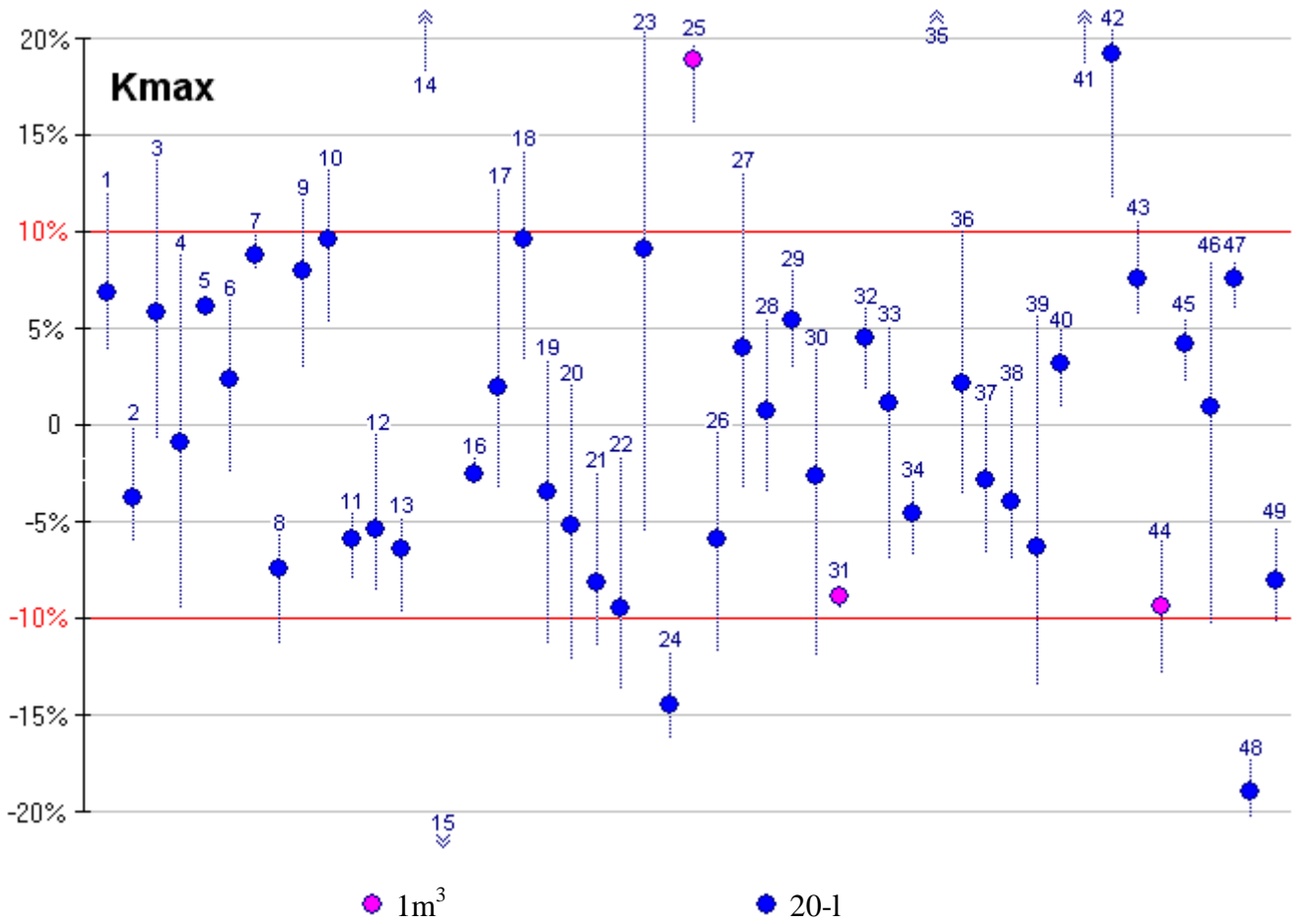
### Explosion Indices Pmax, Kmax

**Pmax = 8.1 bar ± 10% (7.3 ... 8.9) at 680 g/m<sup>3</sup>**



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/m<sup>3</sup>**



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:

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

**Dust distribution:**

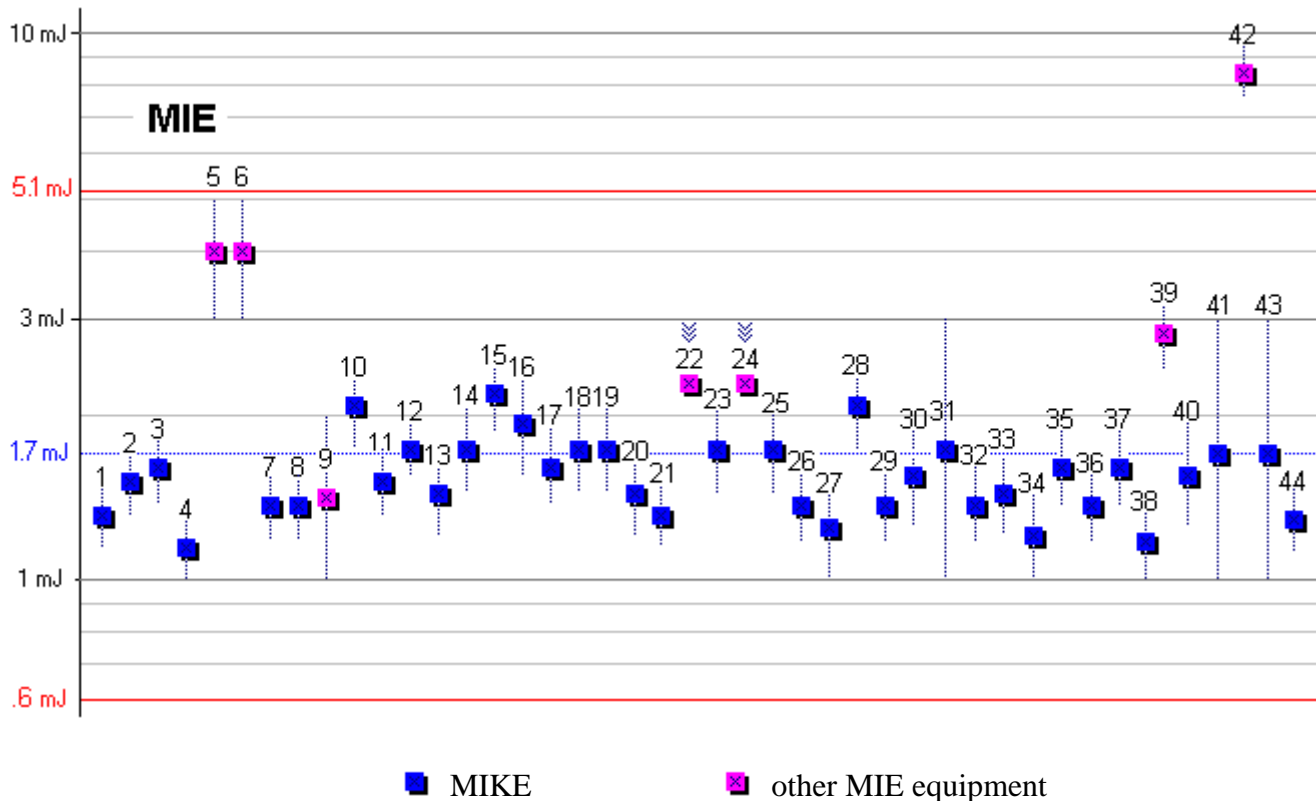
20-l-apparatus with rebound nozzle: 45  
 20-l-apparatus with ring nozzle: 1  
 Large vessels with ring nozzle: 3 (all)

**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 <sup>3</sup>	1
Valve with electro pneumatic drive:	600	1m <sup>3</sup>	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:

<b>Es / 3</b>	<b>Es</b>	<b>Es • 3</b>
<b>0.6 mJ</b>	<b>1.7 mJ</b>	<b>5.1 mJ</b>

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

## List of Participants

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Country	Company Laboratory	E-Mail	Pmax Kmax	MIE
Austria	AUVA Allg. Unfallversicherungsanstalt	karin.schmidt@auva.sozvers.at	✓	✓
Australia	Simtars Mining Research Center	ray.davis@nrm.qld.gov.au	✓	✓
Belgium	D.D. Engineering	magda.lebleu@ddeng.be	✓	
Belgium	Janssen Pharmaceutica NV	cfannes@janbe.jnj.com	✓	✓
Belgium	Katholieke Universiteit Leuven	filip.verplaetsen@mech.kuleuven.ac.be	✓	✓
Germany	BAM Bundesanstalt für Materialforschung und -prüfung	fachgruppe-II.2@bam.de	✓	✓
Germany	BASF Aktiengesellschaft	sabine.schenk@basf-ag.de	✓	✓
Germany	Bayer Cropscience Physikalische Analytik G813	paul.bittner@bayercropscience.com	✓	✓
Germany	Bayer AG Bayer Industry Services BIS-SUA Verfahrens- und Anlagensicherheit	uwe.heinz.uh@bayerindustry.de claus- dieter.walther.cw@bayerindustry.de	✓	✓
Germany	B I A Staubexplosionslabor	h.beck@hvbv.de	✓	✓
Germany	Boehringer Ingelheim Pharma KG AVE-Sicherheits- und Materialprüfstelle	noack@ing.boehringer-ingelheim.com	✓	✓
Germany	EXAM BBG Prüf- +Zertifizier GmbH	woersdoerfer@bg-exam.de	✓	✓
Germany	Forschungszentrum Karlsruhe GmbH	andrey.denkevits@iket.fzk.de	✓	
Germany	Henkel KGaA VTA-Sicherheitstechnik	michael.schrieber@henkel.com	✓	✓
Germany	IBExU GmbH Staublabor	post@ibexu.de	✓	✓
Germany	Anonym		✓	✓
Germany	Wacker Chemie GmbH Stabex, Zentrale Analytik	jean-michel.dien@wacker.com michael.stepp@wacker.com		✓
Germany	Wilhelm-Jost-Institut e.V. Explo- sions- und Brandschutz	birnbacher@wji.de	✓	✓
England	Building Research Establishment	manchesters@bre.co.uk	✓	✓
England	Chilworth Technology Limited	mmerrit@chilworth.co.uk	✓	✓
England	Ciba Specialty Chemicals Safety Testing Laboratory	greg.rogers@cibasc.com	✓	✓
England	GlaxoSmithKline Hazard Determination Laboratory	michael.i.gilmore@gsk.com	✓	
Finland	VTT Building and Transport, Fire Technology	johan.mangs@vtt.fi	✓	
France	ATOFINA Centre de Recherche Rhône-Alpes	damien.drutel@atofina.com huguette.lagarigue@atofina.com	✓	✓
France	Aventis Pharma Département Sécurité des Procédés	andre.guilland@aventis.com	✓	✓
France	Anonym		✓	

Country	Company Laboratory	E-Mail	Pmax Kmax	MIE
France	ENSIC CNRS, LSGC	perrin@ensic.inpl-nancy.fr	✓	
France	INERIS	mohamed.boudalaa@ineris.fr	✓	✓
France	Rhoditech Process Safety Laboratory	herve.vaudrey@eu.rhodia.com	✓	✓
France	SNPE, DFP/TCFC	f.tissandier@snpe.com		✓
Holland	DSM, DSM Research	eddy.oost-van-t@dsm.com	✓	
Holland	TNO Prins Maurits Laboratory Explosion Prevention & Protection	bruijn@pml.tno.nl	✓	✓
Hungary	Gedeon Richter Ltd Safety Laboratory	gy.negyesei@richter.hu		✓
India	CIBA India Private Limited Research & Technology	vijay.bhujle@cibasc.com		✓
Italy	Stazione sperimentale Combustibili Sicurezza	mazzei@ssc.it		✓
Japan	TIIS, The Technology Institution of Industrial Safety Asaka Safety Test Laboratory	matsuda@ankyoo.or.jp		✓
Canada	Dalhousie University Department of Chemical Engineering	paul.amyotte@dal.ca	✓	✓
Norway	GexCon AS	cta@gexcon.com	✓	✓
Poland	Central Mining Institute Experimental Mine "Barbara"	z.dyduch@gig.katowice.pl	✓	
Switzerland	CIMO SA Laboratoire de sécurité	patrice.sciboz@cimo-sa.ch	✓	✓
Switzerland	Firmenich SA Safety Laboratory - DCDC	franco.ferregutti@firmenich.com	✓	
Switzerland	Lonza AG Sicherheitslabor	eberhard.irle@lonza.com	✓	
Switzerland	F. Hoffmann-La Roche AG PSSU-SL	thomas.glarner@roche.ch		✓
Switzerland	Sicherheitsinstitut, Basel Prüflabor	abisel@swissi.ch	✓	✓
Spain	Laboratorio Oficial J.M. Madariaga (LOM)	jgtorrent@qyc.upm.es	✓	✓
U.S.A.	Abott Laboratories Process Safety Lab D-R45R, R-8	zhenglong.xiao@abbott.com	✓	✓
U.S.A.	BASF Corporation	boicoug@basf-corp.com		✓
U.S.A.	CIBA CSTL	larry.floyd@cibasc.com	✓	✓
U.S.A.	Kidde-Fenwal inc. Combustion Research Center	joseph.senecal@kidde-fenwal.com	✓	
U.S.A.	Dupont Explosion Hazards Lab	robert.gravell@usa.dupont.com		✓
U.S.A.	Merck & Co., Inc. Process Safety Lab.	michael_toth@merck.com	✓	✓
U.S.A.	Anonym		✓	