Final report on round robin test RoRo93

Dear RoRo colleagues,

As you will remember, we started a worldwide round robin test in May '93. By the end of 1993, a total of 39 of the 65 labs we wrote to had complied with our request and sent us their test results. Our thanks to all RoRo participants for their help. We were very pleased with the result and our initial high hopes were exceeded.
The round robin test RoRo93 is now at an end and we can report the results to you.

Contents:

Birsfelden, January 19, 1994 Kühner AG

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1. Test dust RoRo93

The test dust for this round robin test was specially prepared for us by the Ciba company in a batch of 150 kg and provided free of charge. The reference values given in the Tables for the 1-m^3 vessel and the 20-L apparatus were determined before the start of the round robin test by the Ciba specialist team "Explosion technology" (FETES). The other reference values were determined by the Ciba safety testing lab (SPS) . We would like to thank Ciba, FETES and the SPS for their generous help without which the round robin test could not have been performed.

Owing to its low median value, its high Kmax and its very low minimum ignition energy, the following dust was selected for the round robin test:

RoRo93 = 2, 2, 6, 6 - tetramethylpiperidine derivative (light stabilizer)

2. Results determined in the 20-L apparatus

Table 2 summarizes all results determined in the 20-L apparatus in chronological order. It also shows the reference results of the 1-m^3 vessel and the 20-L apparatus.
The characteristics are shown individually in graphical form in the following sections accompanied by a brief comment. The individual values are always plotted in relation to the **arithmetic mean** of **all** measured values in the 20-L apparatus and compared with the reference results.
The standard deviations published earlier for the 1-m^3 vessel and the 20-L apparatus were used for the tolerance limits.

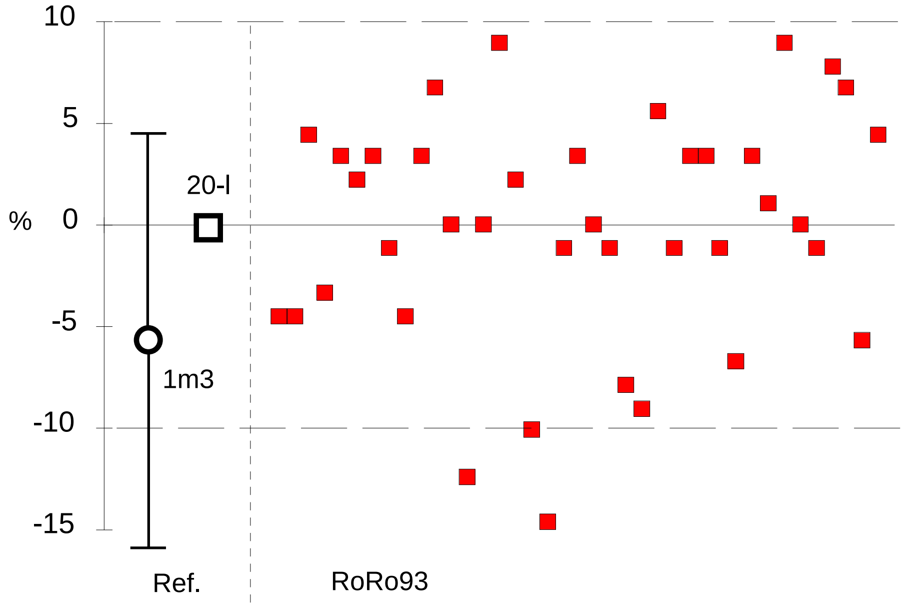
Table 1: Standard deviations

Kmax (m·bar/s)	Deviation for Kmax (± %)	Deviation for Pmax (± %)
50	30	10
100	20	10
200	12	10
300	10	10
≥ 400	5	10

Table 2: Summary of the results determined in the 20-L apparatus

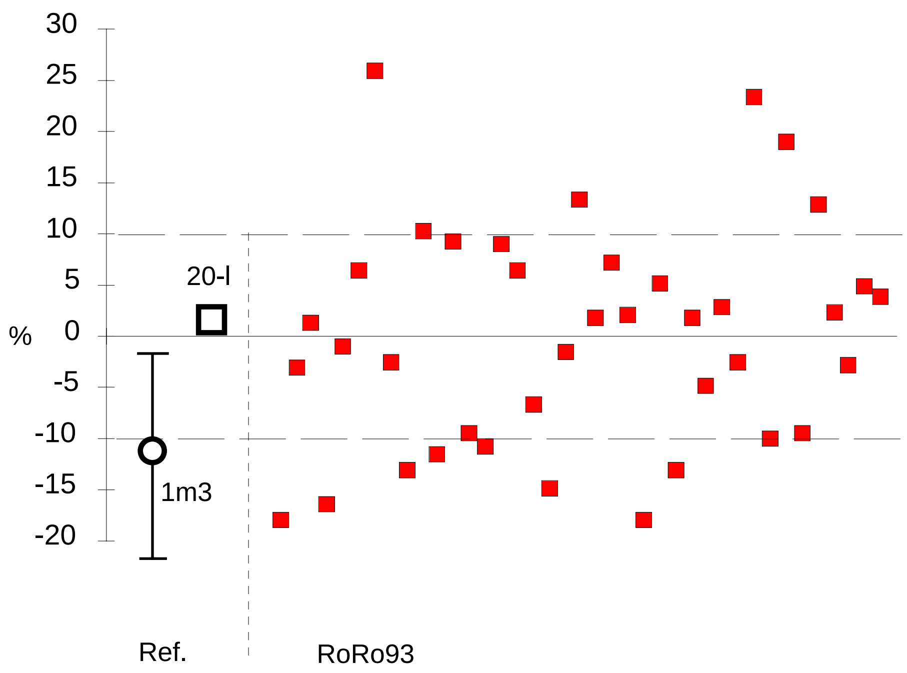
#	LEL (g/m3)	Pmax (bar)	C (Pmax) (g/m3)	Kmax (m·bar/s)	C (Kmax) (g/m3)	LOC (Vol%)	MIE estimated	Comments
1m3	**30**	**8.4**	250	**255**	500	**9.5**	< 10 mJ	Ref.
20-l	**20**	**8.9**	250	**293**	500	**9**	< 10 mJ	Ref.
1	**20**	**8.5**	250	**237**	500	**-**	< 10 mJ	not Sobbe
2	**20**	**8.5**	250	**280**	250 - 500	**9.8**	< 10 mJ	
3	**20**	**9.3**	250	**293**	250 - 500	**-**	< 10 mJ	
4	**20**	**8.6**	250	**242**	250	**-**	< 10 mJ	
5	**10**	**9.2**	250	**286**	250 - 500	**9.8**	< 10 mJ	
6	**20**	**9.1**	-	**308**	-	**10.6**	-	
7	**10**	**9.2**	250	**364**	250	**-**	-	
8	**10**	**8.8**	250	**282**	250	**8.2**	-	2 series
9	**-**	**8.5**	250	**251**	250 - 375	**-**	< 10 mJ	
10	**-**	**9.2**	250	**319**	250 - 500	**-**	-	
11	**20**	**9.5**	-	**256**	-	**6.6**	-	not Sobbe
12	**10**	**8.9**	250 -375	**316**	250	**9.8**	< 10 mJ	
13	**20**	**7.8**	-	**262**	-	**-**	-	
14	**15**	**8.9**	250	**258**	500	**-**	< 10 mJ	
15	**10**	**9.7**	250	**315**	250	**-**	-	
16	**10**	**9.1**	250	**308**	250	**9.8**	-	
17	**10**	**8**	250	**270**	500	**-**	-	
18	**10**	**7.6**	250	**246**	250	**-**	< 10 mJ	
19	**-**	**8.8**	250	**285**	250 - 500	**-**	-	
20	**10**	**9.2**	250	**328**	250	**-**	-	
21	**-**	**8.9**	250	**294**	250	**-**	-	1 series
22	**10**	**8.8**	250	**310**	250	**-**	-	
23	**10**	**8.2**	-	**295**	-	**11.5**	-	
24	**10**	**8.1**	250	**237**	500	**-**	< 10 mJ	
25	**20**	**9.4**	250	**304**	250 - 500	**-**	-	
26	**20**	**8.8**	250	**251**	250 - 500	**< 10 ***	-	* not on graph
27	**20**	**9.2**	-	**294**	-	**< 13 ***	-	* not on graph
28	**10**	**9.2**	250	**275**	250 - 500	**-**	-	
29	**10**	**8.8**	250	**297**	500	**-**	< 1 mJ	
30	**25**	**8.3**	-	**282**	-	**-**	< 10 mJ	
31	**10**	**9.2**	250	**357**	250	**9**	< 10 mJ	
32	**20**	**9**	250	**260**	250 - 500	**-**	-	
33	**30**	**9.7**	300	**344**	300	**-**	-	1 series
34	**-**	**8.9**	250	**262**	500	**-**	-	
35	**-**	**8.8**	250	**326**	500	**-**	-	2 series
36	**-**	**9.6**	500 - 750	**296**	500 - 750	**-**	-	
37	**-**	**9.5**	250	**281**	500	**-**	-	
38	**15**	**8.4**	250	**303**	250 - 500	**-**	-	
39	**30 ***	**9.3**	-	**300**	-	**-**	< 10 mJ	* 2 kJ

2.1 Maximum explosion overpressure Pmax



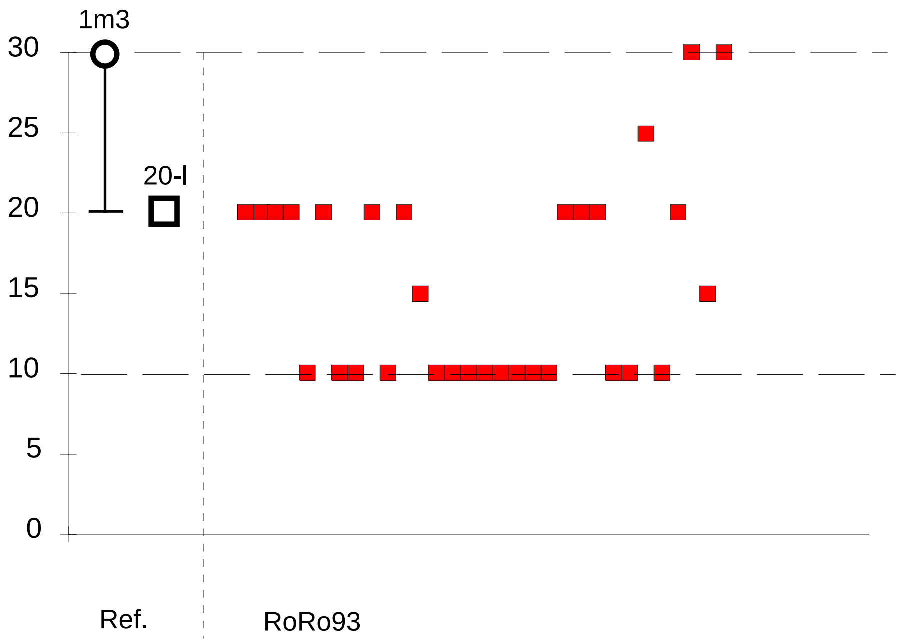
The arithmetic mean of the 39 RoRo results is: **Pmax = 8.9 bar** and thus corresponds exactly to the reference value for the 20-L apparatus. The value Pmax = 8.4 bar for the 1-m^3 vessel is somewhat lower. The reason for this lies in the estimated and linear correction for Pmax compared with the large vessel (cf. directions for use: Pmax > 5.5 bar).

2.2 Maximum material-specific constant Kmax



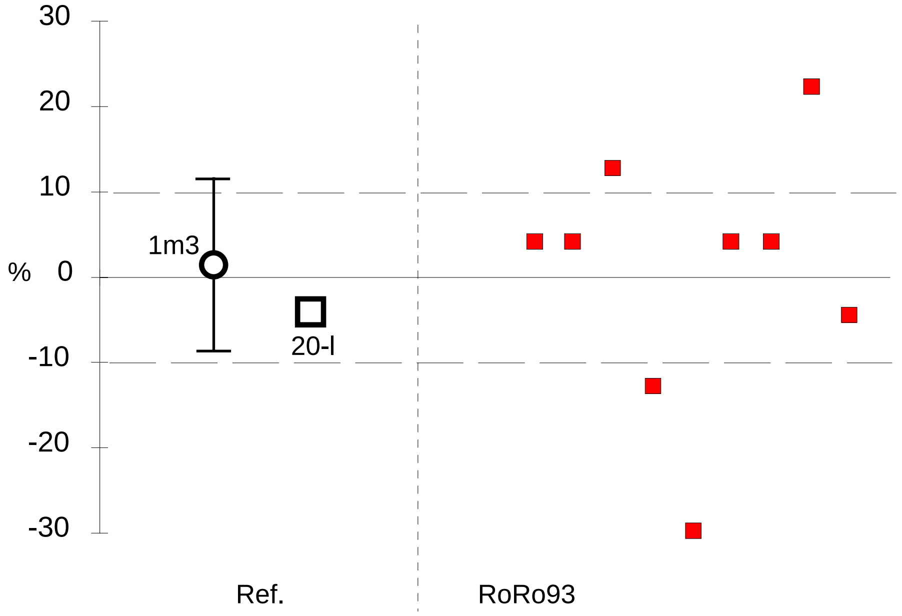
The arithmetic mean of the 39 RoRo results is: **Kmax = 289 m·bar/s.** The value Kmax = 255 m·bar/s for the 1-m^3 vessel is somewhat lower, but is within the usual spread.

2.3 Lower explosibility limit LEL



The arithmetic mean of 31 RoRo results is**: LEL = 16 g/m^3**.
This mean value is not ideal for a comparison as most participants correctly performed measurements in accordance with the test procedure in concentration steps of 10 g/m^3. A tolerance of ± 10 g/m^3 must therefore be accepted for both vessel sizes.

2.4 Limiting oxygen concentration LOC



The arithmetic mean of the 9 RoRo results is: **LOC = 9.4 vol%** and corresponds to the reference value for the 20-L apparatus and the 1-m^3 vessel. The two results which deviate greatly from the rest are due to imperfect testing.

2.5 Notes on the tests in the 20-L apparatus

Lower explosibility limit LEL

This determination is naturally very sensitive to product residues from previous tests. It has thus proved advisable to insert a blank test (igniters only, no dust) between the individual tests with dust to remove the residues following cleaning.

Pmax, Kmax

Please always specify the mean of the maximum from **3** series.

Limiting oxygen concentration LOC

With decreasing oxygen concentration, the optimum dust concentration is shifted to lower values. The tests must therefore also be performed in this concentration range (e.g. 30, 60 g/m^3).

Igniters

The type and number of the chemical igniters used has a particularly large influence on the characteristics of the present RoRo dust. The 1•10 kJ igniter from the Sobbe company has proved unsatisfactory compared with the recommended 2•5 kJ igniters (also from Sobbe).
If igniters of other manufacturers are used, their suitability must be tested by comparison tests and with dusts with very different Kmax values.

Compressed air

For the 20-L apparatus only normal compressor compressed air may be used. With the use of, e.g. synthetic compressed air Kmax values which are clearly too low (-30%) were obtained.

3. Determination of the minimum ignition energy

With one exception, in the determination of the minimum ignition energy all participating testing labs obtained the same result within the limits of the measurement accuracy and the measurement range of the apparatus.

Table 3: Test results for the minimum ignition energy

#	**MIE** with Induct.	**MIE** without Induct.	apparatus
FETES	< 1 mJ	< 1 mJ	MIKE 3
Kühner AG	1 mJ < MIE < 3 mJ	1 mJ < MIE < 3 mJ	MIKE 3
1	< 1.5 mJ	-	*
2	< 1 mJ	1 mJ < MIE < 3 mJ	MIKE 3
3	< 10 mJ	< 10 mJ	KSEP 360
4	> 4 mJ	?	*
5	< 4 mJ	< 4 mJ	*
6	< 1 mJ	-	*
7	-	< 1 mJ	MIKE 3
8	< 2 mJ	< 2 mJ	*
9	1 mJ < MIE < 3 mJ	1 mJ < MIE < 3 mJ	MIKE 3
10	1 mJ < MIE < 3 mJ	1 mJ < MIE < 3 mJ	MIKE 3
11	1 mJ < MIE < 3 mJ	1 mJ < MIE < 3 mJ	MIKE 3
12	2 mJ < MIE < 3 mJ	-	*
13	< 12 mJ	-	*
14	1 mJ < MIE < 3 mJ	1 mJ < MIE < 3 mJ	MIKE 3
15	1 mJ < MIE < 3 mJ	1 mJ < MIE < 3 mJ	MIKE 3
16	1 mJ < MIE < 3 mJ	1 mJ < MIE < 3 mJ	MIKE 3

* this equipment was not manufactured by Kühner AG, apparatus names are not given as per agreement.

4. Results of the screening tests

Table 4 below summarizes all results determined with the screening test equipment in chronological order.

1. Modified Hartmann tube, dust explosion test in tube with hinged cover
2. Burning test (BC) with glowing platinum wire at room temperature and 100°C
3. Drop hammer, testing for impact sensitivity
4. BAM furnace, ignition test for dispersed dust, horizontal
5. Godbert-Greenwald furnace (GG), ignition test for dispersed dust, vertical
6. Lütolf furnace, determination of the relative self-decomposition temperature
7. Grewer furnace, determination of the relative self-ignition temperature

Table 4: Screening Tests

#	mod. Hartmann	BC 20 °C	BC 100 °C	Drop Hammer	BAM MIT (°C)	GG MIT (°C)	Lütolf RSDT (°C)	Grewer RSIT (°C)	Comments
Ref.1	2	2	2	negative	310	360	> 350	> 150*	* melting
Ref.2					330				
1	-	-	-	-	-	280	-	-	
2	2	1	-	-	-	-	-	-	
3	2	2	2	negative	300	-	> 300		
4	-	2	2	negative	330	-	> 350	> 150 *	* melting
5	-	-	-	-	310	-	-	-	
6	-	2 / 5 *	2 / 5 *	-	-	-	> 350	-	* with kieselgur
7	-	2	2	positive*	320	-	> 120	-	* with quartz sand
8	-	2	2	negative	330 - 340	-	-	-	
9	-	-	-	-	300	-	-	-	
10	-	1	1	-	330	-	142*, 325	> 140	* endothermic
11	-	2 / 5 *	2 / 5 *	-	-	-	-	-	* with kieselgur
12	-	2	2	negative	310	-	> 350	> 160 *	* melting
13	-	1	1	negative	310	-	> 350	> 150 *	* melting
14	-	2	2	negative	320	-	> 240	> 150 *	* melting
15	-	2	2	-	-	-	-	-	
16	-	2	2	negative	> 280	-	> 350*	> 145	gas flammable
17	-	2 / 5 *	2 / 5 *	negative	-	-	-	-	* with kieselgur
18	-	2 / 3*	2 / 3*	1 positive 9 negative	330	-	-	> 150	* with kieselgur
19	-	-	-	-	-	300	-	-	

4.1 Notes on the screening tests

Burning test (BC)
In the testing of substances with a low melting point, it must be noted that these products melt on contact with the ignition source and hence are usually found in burning class 2. In such instances the test must be repeated with admixture of kieselgur. In most cases, the mixture substance - kieselgur is then classified in burning class 5.

BAM furnace
As the outer parts of the BAM furnace cool down faster than the test plate in the interior of the furnace, the test plate is the hottest part of the furnace after a short time. The test must therefore be performed with a **falling** furnace temperature. When the tests are carried out with a controlled furnace temperature, approx. 10% lower MIT values are measured as the wall of the furnace and not the test plate is the hottest part of the furnace.

5. Additional results

Table 5 below summarizes additional results in chronological order. It contains the following:

1. Ignition test for dust layer (MIT)
2. Differential Scanning Calorimetry (DSC), decomposition (deco)
3. Differential Scanning Calorimetry (DSC), exothermicity (exo)
4. Testing for exothermic decomposition and pressure buildup in closed vessel (mini-autoclave)
5. Melting point

Table 5: Further results

#	MIT layers	DSC deco. (°C)	DSC exo. (°C)	Mini-autoclave	Melting point (°C)
1	-	-	-	-	150
2	-	290	410	-	153
3	> 365	-	-	130 bar at 400°C	-
4	-	-	> 350	-	-
5	-	-	-	60 bar at 400°C	-
6	-	-	-	35 bar at 350°C	140 approx.
7	-	-	-	32 bar at 350°C	-