

Report of the Round-Robin test on the Sustained Combustibility according to EN ISO 9038

1. Reason

This round robin test was initialized to improve the given verification data in the standard EN ISO 9038.

2. Organisation

The round robin was organized by PTB, AG 3.41. The participants agreed on the round robin on a voluntary basis, because till now the determination of sustained combustibility is not subject of international metrology organisations.

The samples and the forms (annex B) for reporting the results were prepared by PTB, AG 3.41. The samples were sent out in February and march 2006. The forms and the EN ISO 9038 (in some cases) were sent out via email at about the same time. The participants were asked to carry out the determination of the sustained combustibility within 6 weeks after receiving the samples.

3. Participants

The following 11 participants contributed to the round-robin:

Akzo Nobel Technology & Engeneering (The Nederlands)
Aqura GmbH, Marl (Germany)
BASF Coatings, Münster (Germany)
BASF AG, Ludwigshafen (German)
Gedeon Richter Ltd (Hungary)
National Research Institute of fire and Disaster (Japan)
PTB Braunschweig (Germany)
Siemens AG, Frankfurt (Germany)
Stanhope Seta (Great Britan)
Stazione sperimentale per i Combustibili (Italy)
The Government Laboratory of Hong Kong (Hong Kong)

The altitude of the locations was between sea level and 120 m. The ambient pressure varied between 100,2 kPa and 101,1 kPa.

4. Samples

Three different samples have been chosen:

n-Decane
n-Undecane
n-Dodecane

5. Method

The method is described in EN ISO 9038. Participants (with one exception) carried out the tests at least three times per substance and test condition.

6. Results

10 of the 11 participants carried out the number of tests asked for (3 or 5) at least for one heating time per test temperature. One participant carried out only one test per test condition. This data are reported in the annex B but not taken into account for the evaluation.

6.1 Raw data

The raw data are given in annex B table 1 and table 2. They were sent to PTB between March 2006 and August 2006.

6.2 Statistical treatment

As the result of the test is not a quantitative value but a yes or no answer, the statistical treatment according to ISO 4259 and ISO 5725 could not be used for the result.

6.3 Within-laboratory Outliers

With respect to the nature of the result – yes or no – it was checked whether in every test of the three respectively five tests the same answer was got (Table 1 and 2).

Table 1: Tests at 60°C

Participant	n-Decane 60°C		n-Undecane 60°C		n-Dodecane 60°C	
	Heating time		Heating time		Heating time	
	30 s	60 s	30 s	60 s	30 s	60 s
1	no	no	no	no	no	no
2	yes	-	no	-	no	-
3	yes	yes	no	no	no	no
4	yes	yes	no	no	no	no
5	yes	yes	no	no	no	no
6	yes	yes	no	no	no	no
7	yes (4 of 5)	yes	no	no	no	no
8	yes	yes	no	no	no	no
9	yes	yes	no	no	no	no
10	yes (4 of 5)	yes	no	no	no	no

Table 2: Tests at 75°C

Participant	n-Decane 75°C		n-Undecane 75°C		n-Dodecane 75°C	
	Heating time		Heating time		Heating time	
	30 s	60 s	30 s	60 s	30 s	60 s
1	yes	yes	no	no	no	no
2	yes	-	yes	-	no	-
3	yes	yes	yes	yes	no	no
4	yes	yes	yes	yes	no	no
5	yes	yes	yes	yes	no	no
6	yes	yes	yes	yes	no	no
7	yes	yes	yes (3 of 5)	yes (3 of 5)	no	no
8	yes	yes	yes	yes	no	no
9	yes	yes	yes	yes	no	no
10	yes	-	no(2 of 3)	yes	no	no

For n-Decane 2 within-laboratory outliers were reported (participant 7 and participant 10). However both reported only one of 5 tests of the same sample to be different. Therefore for the further evaluation this outliers are ignored and the remaining 4 single results are taken.

For n-Undecane 3 within-laboratory outliers were reported (participant 7 and participant 10). One of the participants reported twice 3 test of 5 with the same result (yes) therefore for the

further evaluation this results are taken as yes. The other participant reported 2 of 3 tests as no therefore for the further evaluation this results are taken as no.

For n-Dodecane no within-laboratory outliers were reported.

6.4 Between-Laboratory Outliers

With respect to the nature of the result – yes or no – it was checked whether for each sample the participant’s answer differed or not taking into account the results in 6.3 (Table 1 and 2).

Table 3: Laboratory comparison for n-Decane

n-Decane	Test condition	yes	no
	60°C, 30s	8 of 10	2 of 10
	60°C, 60s	7 of 9	2 of 9
	75°C, 30s	10 of 10	10 of 10
	75°C, 60s	9 of 9	8 of 8

For n-Decane one between-laboratory outlier was found for the test conditions 60°C, 30s respectively 60°C, 60s.

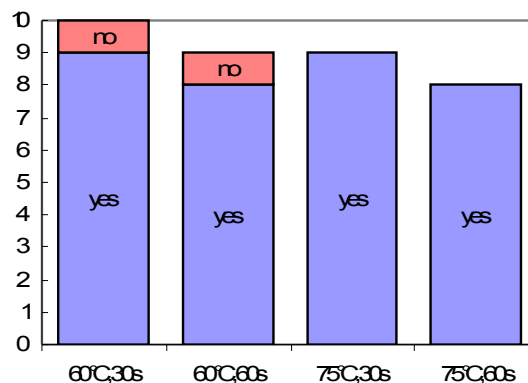


Table 4: Laboratory comparison for n-Undecane

n-Undecane	Test condition	yes	no
	60°C, 30s	0 of 10	10 of 10
	60°C, 60s	0 of 9	9 of 9
	75°C, 30 s	8 of 10	2 of 10
	75°C, 60s	8 of 9	9 of 9

For n-Undecane two between-laboratory outliers were found for the test condition 75°C, 30s.

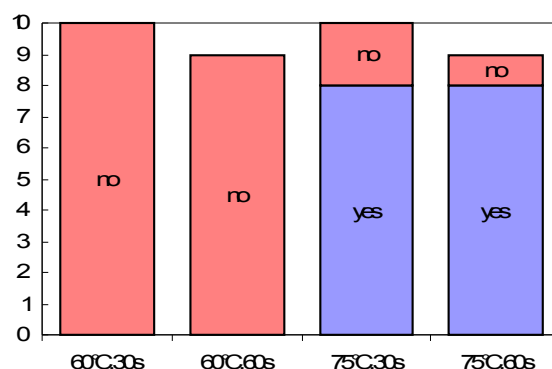
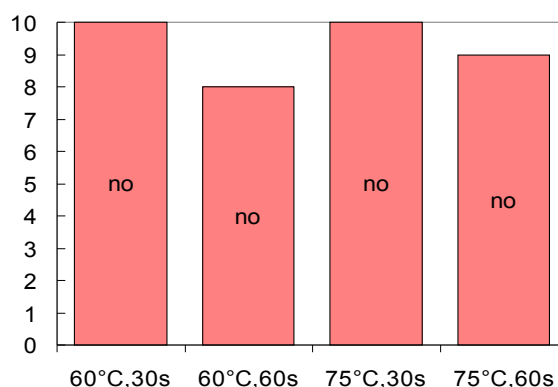


Table 5: Laboratory comparison for n-Dodecane

n-Dodecane	Test condition	yes	no
	60°C, 30s	0 of 10	10 of 10
	60°C, 60s	0 of 9	9 of 9
	75°C, 30 s	0 of 10	0 of 10
	75°C, 60s	0 of 9	9 of 9

For n-Dodecane no between-laboratory outlier was found



6.5 Evaluation

n-Dodecane

All 10 participants reported the same results: no sustained combustion ability regardless of the test conditions.

n-Undecane

All 10 participants reported the same results for the test conditions 60,0°C 30s respectively 60s: no sustained combustion

8 of the 10 participants reported regardless of the test conditions for the test conditions 75,0°C 30s respectively 60s: sustained combustion, whereas 2 reported no sustained combustion.

One of these two is the same who reported 'no sustained combustion' with n-Decane at 60°C, 30 s and 60°C, 60s. Looking in detail to the conditions reported by the participants which may influence the result (table 4) – humidity, ambient pressure, gas used for the test flame –, no reliable explanation could be found for the differences.

Table 6: Test conditions of the participants for n-Undecane

Participant	Result	rel. humidity %	ambient pressure kPa	gas for the test flame	Comment given by participant
1	no	20	100,6	methane	a flash extinguished the test flame (in the on position) after 2 ±4s. No ignition is observed.
2	yes	-	-	-	
3	yes	-	100,8	butane	
4	yes	40	101,0	natural gas	
5	yes	85	100,5	natural gas	
6	yes	56 – 63	100,8	butane	
7	yes (3 of 5)	59	100,8	town gas	
8	yes	60	100,2	natural gas	
9	yes	-	100,9	propane/butane	
10	no (2 of 3)	62	101,1	propane/butane	flame flashed and extinguished with pilot flame

n-Decane

All 10 participants reported the same results for the test conditions 75,0°C 30s re-spectively 60s: sustained combustion

8 of the 10 participants reported for the test conditions 60,0°C 30s respectively 60s: sustained combustion, whereas 2 reported no sustained combustion.

Looking in detail to the conditions reported by the participants which may influence the result (table 6) – humidity, ambient pressure, gas used for the test flame –, no reliable explanation could be found for the differences.

Table 6: Test conditions of the participants for n-Decane

Participant	Result	rel. humidity %	ambient pressure kPa	gas for the test flame	Comment given by participant
1	no	18	100,6	methane	.
2	yes	-	-	-	
3	yes	-	100,8	butane	
4	yes	40	101,0	natural gas	
5	yes	85	100,5	natural gas	
6	yes	56 – 63	100,8	butane	
7	yes (4 of 5) yes (5 of 5)	59	100,8	town gas	
8	yes	60	100,2	natural gas	
9	no	-	100,9	propane/butane	
10	yes (4 of 5) (3 of 3)	62	101,1	propane/butane	flash, expand and extinguish

Influence of humidity, ambient pressure and gas used for the test flame.

Ambient pressure:

As the ambient pressure varied between 100,2 kPa and 101,1 kPa, the corrected test temperatures varied from 59,7 °C to 60,0 °C respectively from 74,7 °C to 75,0 °C.

This is within the accuracy of the temperature measuring device required in ISO 9038 ($\pm 0,5^{\circ}\text{C}$). Therefore no effects can be discussed.

Humidity:

The humidity was reported only by part of the participants. It varied between 18 %RH and 85% RH. Although the participant having very dry atmosphere during the tests came to different results in 4 of 12 test conditions this seems not to be the reason as in case of n-Undecane the second different answer was reported by a participant having 62% RH. However, both reported the same effect – flame flashed and extinguished with pilot flame -.

Gas used for the test flame

There seems to be no influence of the gas used for the test flame.

Conclusion

Only for n-Dodecane the results are clear.

For n-Decane and especially n-Undecane the results need further discussion.

Annex A

Specification of the samples

n-Decane

Boiling point: 174 °C
Flash point: 46 °C
Viscosity: 0,9125 mPas

n-Undecane

Boiling point: 196 °C
Flash point: 61 °C
Viscosity: 1,1815 mPas

n-Dodecane

Boiling point: 216 °C
Flash point: 82 °C
Viscosity: 1,5144 mPas

Annex B

Raw Data n-Decane

Participant	Result				Comment
	60°C,30s	60°C,60s	75°C,30s	75°C,60s	
1	no	no			
	no	no			
	no	no			
			yes	yes	test portion ignites when the test flame is in the <i>off</i> position
		yes	yes		
		yes	yes		
2	yes				
	yes				
	yes				
			yes		
			yes		
			yes		
3	yes	yes			
	yes	yes			
	yes	yes			
	yes	yes			
	yes	yes			
			yes	yes	
			yes	yes	
			yes	yes	
			yes	yes	
4	yes	yes			
	yes	yes			
	yes	yes			
			yes	yes	
			yes	yes	
			yes	yes	
5	yes	yes			after ignition a part of the n-decane is flooded over the edge
	yes	yes			
	yes	yes			
			yes	yes	after ignition a part of the n-decane is flooded over the edge
		yes	yes		
		yes	yes		
6	yes	yes			
	yes	yes			
	yes	yes			
			yes	yes	
			yes	yes	
			yes	yes	

Participant	Result				Comment
	60°C,30s	60°C,60s	75°C,30s	75°C,60s	
7	no	yes			
	yes	yes			
	yes	yes			
	yes	yes			
	yes	yes			
	yes	yes			
			yes	yes	
			yes	yes	
			yes	yes	
			yes	yes	
8	yes	yes			
	yes	yes			
	yes	yes			
	yes	yes			
	yes	yes			
			yes	yes	
			yes	yes	
			yes	yes	
			yes	yes	
			yes	yes	
9	yes	yes			
	yes	yes			
	yes	yes			
			yes	yes	
			yes	yes	
10	yes	yes			
	no	yes			flame flashed, expanded and extinguished with pilot flame
	yes	yes			
	yes				
	yes		yes		
			yes		
			yes		
11	yes	no			
			yes	yes	

Raw Data n-Undecane

Participant	Result				Comment
	60°C,30s	60°C,60s	75°C,30s	75°C,60s	
1	no	no			
	no	no			
	no	no			
			no	no	a flash extinguishes the test flame (in the <i>on</i> position) after 2÷4s. No ignition is observed
			no	no	
			no	no	
2	no				
	no				
	no				
			yes		
			yes		
			yes		
3	no	no			
	no	no			
	no	no			
	no	no			
	no	no			
	no	no			
			yes	yes	
			yes	yes	
			yes	yes	
4	no	no			
	no	no			
	no	no			
			yes	yes	
			yes	yes	
			yes	yes	
5	no	no			
	no	no			
	no	no			
			yes	yes	part of the n-undecane is flooded over the edge
			yes	yes	
			yes	yes	
6	no	no			
	no	no			
	no	no			
			yes	yes	
			yes	yes	
			yes	yes	

Participant	Result				Comment
	60°C,30s	60°C,60s	75°C,30s	75°C,60s	
7	no	no			
	no	no			
	no	no			
	no	no			
	no	no			
			no	no	
			no	no	
			yes	yes	
			yes	yes	
			yes	yes	
8	no	no			
	no	no			
	no	no			
	no	no			
	no	no			
			yes	yes	
			yes	yes	
			yes	yes	
			yes	yes	
			yes	yes	
9	no	no			
	no	no			
	no	no			
			yes	yes	
			yes	yes	
			yes	yes	
10	no	no			
	no	no			
	no	no			
			no	yes	75°C, 30s: flame flashed, expanded and extinguished with pilot flame
			no	yes	
			yes	yes	
11	no	no			
			yes	no	

Raw Data n-Dodecane

Participant	Result				Comment
	60°C,30s	60°C,60s	75°C,30s	75°C,60s	
1	no	no			
	no	no			
	no	no			
			no	no	
			no	no	
			no	no	
2	no				
	no				
	no				
			no		
			no		
			no		
3	no	no			
	no	no			
	no	no			
	no	no			
	no	no			
			no	no	
			no	no	
			no	no	
			no	no	
			no	no	
4	no	no			
	no	no			
	no	no			
			no	no	
			no	no	
			no	no	
5	no	no			
	no	no			
	no	no			
			no	no	
			no	no	
			no	no	
6	no	no			
	no	no			
	no	no			
			no	no	
			no	no	
			no	no	

Participant	Result				Comment
	60°C,30s	60°C,60s	75°C,30s	75°C,60s	
7	no	no			
	no	no			
	no	no			
	no	no			
	no	no			
			no	no	
			no	no	
			no	no	
			no	no	
			no	no	
8	no	no			
	no	no			
	no	no			
	no	no			
	no	no			
			no	no	
			no	no	
			no	no	
			no	no	
			no	no	
9	no	no			
	no	no			
	no	no			
			no	no	
			no	no	
			no	no	
10	no	no			
	no	no			
	no	no			
			no	no	
			no	no	
			no	no	
11	no	no			
			no	no	