

## Minimum Ignition Temperature of Hybrid Mixtures

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The minimum ignition temperature is an important safety value for handling gases, liquids and dust. The European regulation only provides standards to measure single substances and single-phase values. That poses a problem to industries where materials in different phases occur at the same time, as there is no way to prove that the mixture does not have an ignition temperature that is not below the single values.

Aim of a research project is to provide an extension to the standard for the minimum ignition temperature of dusts (IEC 80079-20-2). Therefore, the Godbert-Greenwald oven is modified to allow testing dust, liquid and gas alone and in mixture with each other.

The proposed experimental setup is only a slight modification to the furnace mentioned in the standard. Main changes are the solvent reservoir and the additional gas supply. First results are already published as a proof of concept (Addai 2016a,b) and now, in a second step, extended by dusts premixed with solvents.

To provide a reliable base for a standardization the influence of these changes according to the ignition behavior will be tested in detail. This includes various size parameters as well as the arrangement of the parts, that will follow.

First the minimum temperature of the single phases – gas, steam of evaporated liquids, dust – are tested and compared to the respective values obtained with the standard to be usually be applied. Subsequently the mixtures of phases are tested.

The proposed setup would further provide the possibility to make tests to check the influence of other oxidizing atmospheres or the humidity in the gas phase.

### 1. Introduction

Hybrid mixture explosions are usually encountered in systems where combustible dusts, gases, or solvents are produced, processed or handled. When two or more combustible substances with different states of aggregation are mixed, hybrid mixture explosions can occur. Some examples of the facilities where such mixtures could occur are amongst others paint factories (pigments and solvents), mining (dusts and gas), grain elevators (small grains and fermentation gases) and pharmaceutical industries. In order to prevent or mitigate the risk associated with these mixtures, knowledge of the explosion behaviour of hybrid mixtures is required. As it is known from recent research works (Addai, 2015a; Addai 2015b; Addai 2015c; Agreda 2011; Amyotte 2010; Dufaud 2009; Khalili 2012; Nifuku 2006), the explosion behaviour can be more critical for the mixture than for the single substance. In general, it is expected that

- the Minimum Ignition Energy is lower than that of the pure dust,
- the Minimum Ignition Temperature is lower than that of the pure dust,
- the mixture is ignitable at concentrations below the Lower Explosion Limit of the single substance and
- the Maximum Explosion Rise Velocity is higher than that of the pure dust.

Performing a risk assessment, it is the operator's responsibility to ensure safety for the workers in his facility. Therefore, the critical safety relevant values have to be determined. At the moment, this is practically impossible wherever hybrid mixtures occur, as they are not covered by the relevant standards, IEC 80079-20-2 or VDI 2263 for dusts and DIN EN 14522:2005 or DIN EN ISO/IEC 80079-20-1:2016-02 for liquids and

gases. As part of a project of several German institutions to introduce such procedures for testing hybrid mixtures into the relevant standards, the Otto-von-Guericke-University primarily focuses on the MIT. An experimental investigation of the Minimum Ignition Temperature and the Lower Explosion Limit hybrid mixtures in air were performed in a modified Godbert-Greenwald (GG) furnace.

## 2. Minimum Ignition Temperature

### 2.1 Definition and procedures

The Minimum Ignition Temperature is generally defined as the lowest temperature of a surface that is able to ignite a substance. Practically, it is only a valid – a comparable – value, if it is determined according to a specific standard. This includes the apparatus, the atmosphere and the procedure to measure.

The DIN EN 14522:2005 defines the procedure for gases and liquids. The MIT is determined in a preheated 200 ml Erlenmeyer flask (Figure 1) in air in at least three test series.

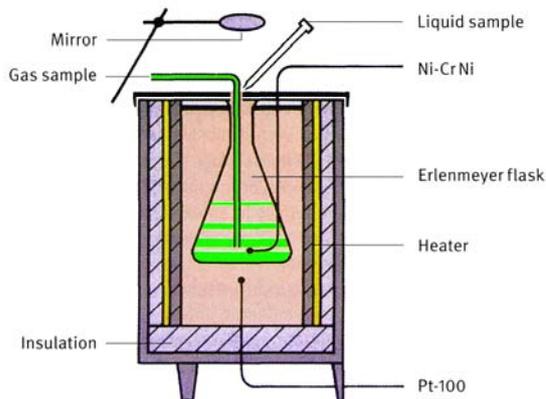


Figure 1: Principle setup of the determination of ignition temperature according to DIN EN 14522

Due to the experimental setup, it is obvious, that this cannot be used for hybrid mixture including dust, as the solid particles would have to be removed with much labour after each single experiment.

For dusts, there are two different methods defined that are both mentioned in IEC 80079-20-2: the BAM oven (Figure 2a) and the Godbert-Greenwald oven (Figure 2b)

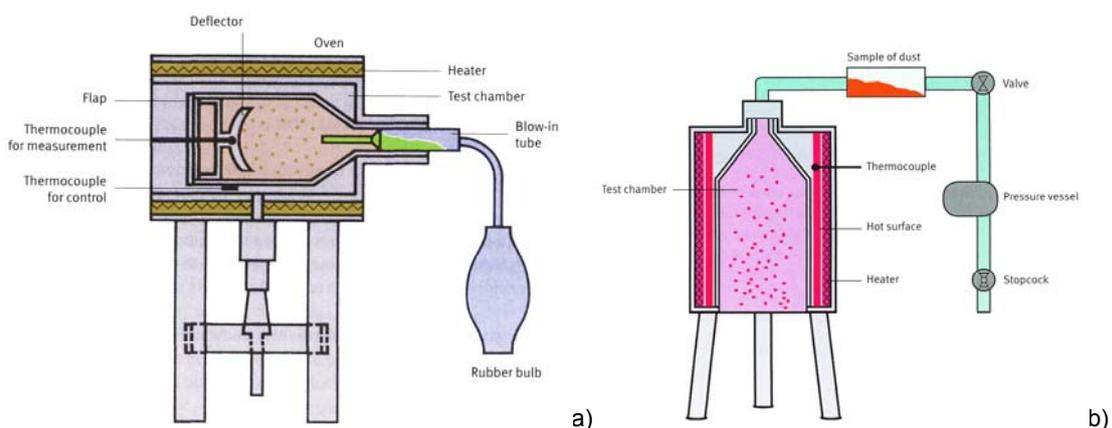
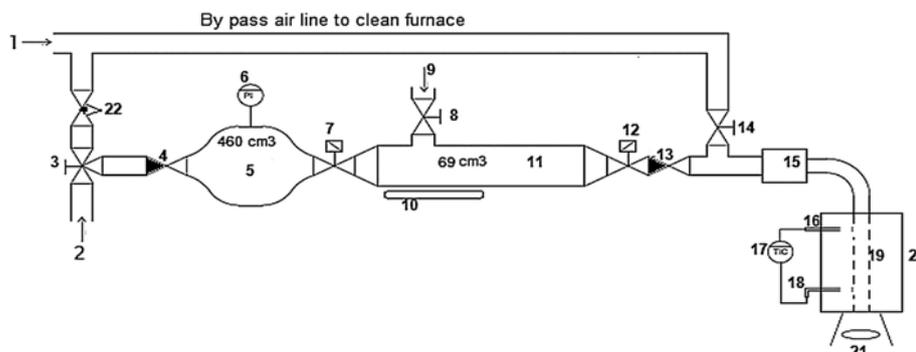


Figure 2: Principle setup of the a) BAM oven and the b) Godbert-Greenwald oven

Due to handling reason and the defined and controllable amount of air in the pressure vessel used in the GG oven this setup was chosen for further modification.

## 2.2 Modifications and adopted procedures

Based on an apparatus according to Figure 2b the setup was modified. A general difference is the length of the oven that is approximately twice as long as stated in the standard. The modifications are shown in Figure 3.



1. Air supply	12. Solenoid valve
2. Gas supply	13. Check valve
3. T- shape ball valve	14. L- shape ball valve
4. Check valve	15. Dust Chamber / reservoir
5. Air / gas reservoir or gas.	16. Thermocouple
6. Digital pressure gauge	17. Temperature controller
7. Solenoid valve	18. Electric power supply
8. L- shape ball valve (two port)	19. Steel furnace tube
9. Solvent or liquid supply	20. Furnace shell and insulation materials
10. Heating filament.	21. Mirror
11. Solvent reservoir	

Figure 3: Modified Godbert-Greenwald oven

To allow gas mixing, the inlet of the pressure vessel (5) is extended by a gas supply (2). Additionally a heated pipe, which is separated by solenoid valves, is added before the dust chamber (15). This solvent reservoir (11) is used to evaporated the liquids before the experiment and generate mixtures with vapor to test for the MIT of the hybrid mixtures as well as for the pure substance.

The general procedure for testing the MIT did not have to be changed. Only the possible variations of fuels in gas, vapor and solid phase has to be taken into account. The modified version of the GG oven still can be used for normal pure dust testing without limitation.

## 3. Results

For the first part of this study different combustible dusts, gases and vapours were used. To specify the dusts their particle size distribution was measured using a CAMSIZER. Scanning Electron Microscopy (SEM) images with a magnification of 1000, to view the surface structure, were taken and the Carbon, Hydrogen, Nitrogen, Sulphur and Oxygen contents was determined. The preparatory analysis of the dusts such as the, median value, volatiles content, moisture content and heat of combustion are shown in Table 1.

Table 1: Preparatory analysis of the dusts

Dust sample	median value [ $\mu\text{m}$ ]	volatile content [mass %]	moisture content [mass %]	heat of combustion [kJ/kg]
Wood	61	84.4	0.20	16,446
HD-PE	18	99.8	0.01	42,740
Starch	14	93.8	0.50	15,302

The gases and liquids were standard chemicals of at least 99% purity.

### 3.1 Minimum ignition temperature of single substances

The MIT of the tested pure materials are listed in Table 2. The values of the gases and vapours do not differ significantly from values published in Brandes and Möller (2003).

Table 2: Single MIT values

dust	MIT [°C]	gas	MIT [°C]	solvent	MIT [°C]
Wood	460	Hydrogen	540	Toluene	535
HD-PE	340	Methane	600	Hexane	225
Starch	380				

### 3.2 Minimum ignition temperature of two component mixtures

The influence of small amounts of an added secondary substance is given in following Tables 3 and 4 as reduction of the MIT. For the tests only concentrations below the respective LEL of the single substance were used. This means that these amounts would not lead to an ignition alone.

Table 3: Reduction of the MIT of dust due to the admixture of gas or solvent

dust	$\Delta$ MIT by adding 0.8 vol% Hexane [K]	$\Delta$ MIT by adding 0.6 vol% Toluene [K]	$\Delta$ MIT by adding 3.0 vol% Hydrogen [K]	$\Delta$ MIT by adding 2.0 vol% Methane [K]
Wood	20	0	0	0
HD-PE	15	0	0	0
Starch	5	0	0	0

The influence of gases and vapours is almost not measurable. Even for Hexane that has a lower MIT than the dusts, the reduction does not exceed 20 K.

Table 4: Reduction of the MIT of gas or solvent due to the admixture of dust

dust	$\Delta$ MIT by adding 87 g/m <sup>3</sup> HD-PE [K]	$\Delta$ MIT by adding 82 g/m <sup>3</sup> Starch [K]	$\Delta$ MIT by adding 97 g/m <sup>3</sup> Wood [K]
Methane	30	15	10
Hydrogen	35	30	0
Hexane	0	0	0
Toluene	25	15	0

None of the effects is drastically, but in several cases, at least a significant reduction in the MIT can be shown. Hexane, which has the lowest MIT of all substances, is not influenced by the presents of the additions.

### 3.3 Minimum ignition temperature of dust premixed with solvents

In order to figure out how the MIT might be further influenced, some dusts were premixed with solvents and then tested for their MIT. As this work is part from another project that was done in parallel, different substances were used. The total project includes the determination of the Minimum Ignition Energy and the explosion severity (Maximum Explosion Pressure, dp/dt) as well as the Minimum Explosible Concentration. It is at this point omitted to list the specific material data. An overview of the substances is given in Table 5. The liquids were added to the dried dusts and then mixed overnight in an overhead rotary mixer. Glycerin as liquid phase was harder to apply to the dusts. Therefore, a spray was used to avoid droplet formation.

Table 5: Tested materials.

dusts	solvents
Lycopodium	Acetone
Wheat flour	2-Propanol
Graphite	Glycerin
Sand	Water

For sand as an inert material, as well as for graphite no ignition could be achieved, as the possible amount of solvent was too small to influence the ignition temperature.

For water as liquid phase, the expected behavior is well known and can be reproduced. The MIT increases the wetter the dust are.

Due to the high evaporation rate, no reproducible result for Acetone and flour could be achieved. With Lycopodium, there was an increase in the MIT of 10 K with Acetone contents between 2.5 % and 3.5 %.

The influence of Glycerin is small and of opposite direction for the two dusts, as shown in Figure 4.

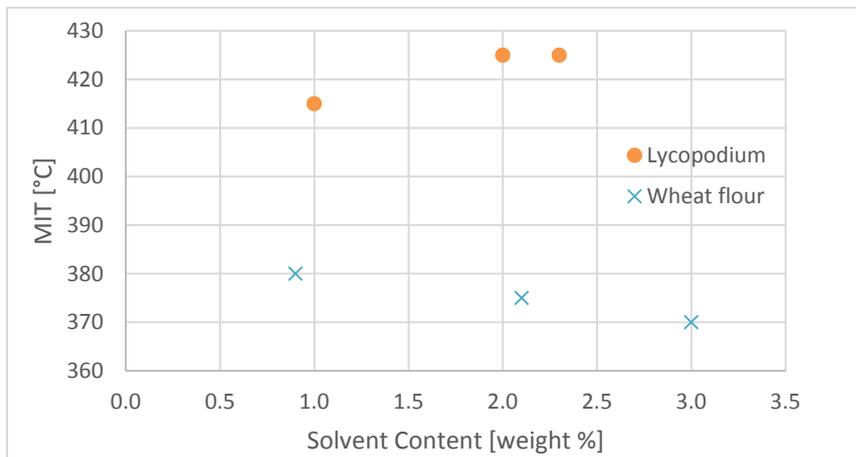


Figure 4: MIT of dust with different Glycerin contents

Results of the dusts tested with 2-Propanol are presented in Figure 5. The influence is small and no clear trend is visible. For flour, the temperature changes lie within the measurement uncertainty.

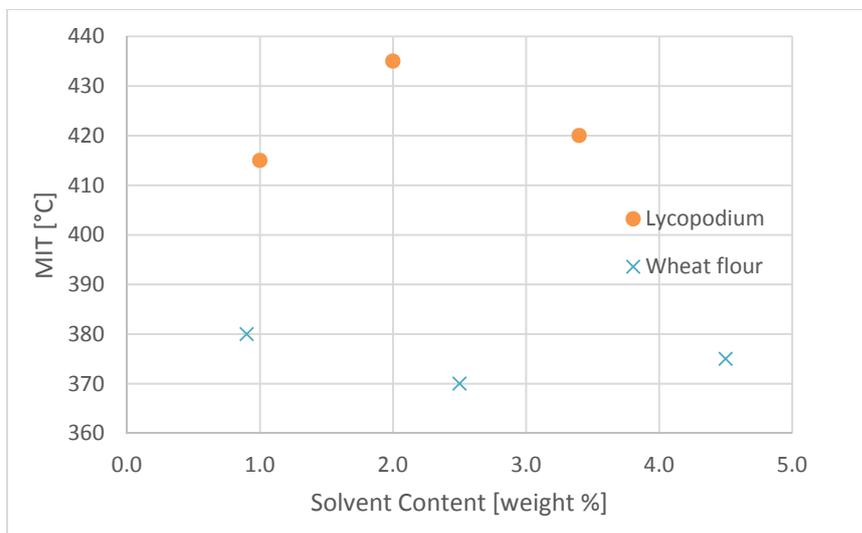


Figure 5: MIT of dust with different 2-Propanol contents

#### 4. Conclusions

As preliminary work for a more extensive project to introduce hybrid mixtures into the standards to determine safety relevant values, a procedure for the MIT is presented. First results prove that there can be an influence on the MIT when substances of different phases are mixed. Up to now, the results shown rather weak effects and not all findings can totally be explained. Therefore, more combinations need to be tested and the underlying reaction processes need to be identified. For the premixed dusts, this includes the description of the desorption process. On the other hand, adsorption might play a role in the two phase mixtures as well.

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