The effect of reduced oxygen levels on the electrostatic ignition sensitivity of dusts

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Abstract. When handling powders with low values of minimum ignition energy (MIE), it is often necessary to employ additional protective measures such as explosion venting, suppression, containment or inerting. Inerting generally involves reducing the oxygen concentration to around 5% v/v; however, it has been shown with gases that more modest reductions in the oxygen content can still have a significant effect on the MIE. Therefore, a test program was carried out to assess the impact of reduced oxygen levels on the MIE of a series of sensitive powders. In addition, this work was also used to investigate whether testing of such sensitive materials in the standard equipment but with reduced oxygen levels could enable the prediction of MIEs <1 mJ at standard atmospheric oxygen levels.

1. Introduction

The sensitivity of flammable dust clouds to ignition by electrostatic discharges is characterised by the minimum ignition energy (MIE), commonly determined according to EN 13821 [1]. The lower the MIE, the more potential electrostatic ignition hazards that require addressing and, for powders that have very low MIEs, it may not be possible to control all potential electrostatic ignition sources reliably. For example, even if all fixed metallic parts are earthed and insulating plastics are excluded, small pieces of tramp metal (e.g. bolts) carried in an insulating powder may still be capable of producing spark discharges that can ignite such sensitive powders [5]. As a consequence, when handling low MIE powders, CLC/TR 50404 [2] advises that it may be necessary to implement explosion protection measures such as venting, suppression or containment. In situations where explosion protection measures are not feasible, either due to operational or financial constraints, inerting is often employed to prevent the formation of a flammable atmosphere.

According to CEN/TR 15281 [3], inerting generally requires the oxygen concentration to be reduced to <60% of the Limiting Oxygen Concentration (LOC), which in most cases results in an operating level of around 5 - 6% v/v. For some equipment and operations, it may not be possible to achieve such low oxygen levels. However, previous work [6] has shown that more modest reductions in the oxygen content can still have a significant effect on the MIE of flammable gas / air atmospheres. Assuming that a similar effect occurs with dust / air atmospheres, if the MIE can be raised above the igniting power of the largest electrostatic ignition source present, then this may present a possible basis for safe operation.

2. Initial Work

The original phase of work was designed to assess the electrostatic hazards of a particular organic powder (referred to as “Powder T”) in a specific handing / processing operation. Using the commercially available Kühner MIKE3 apparatus and the standard test method [1], the MIE of the powder in air was already known to be <1 mJ. At this level, the reliable control of electrostatic ignition sources in the system was not considered possible. The powder also had an unusually low limiting oxygen concentration (LOC) [5] of 7 - 9% v/v, which meant that basing safety on inerting was also not possible due to constraints within the system. As a result, explosion protection measures had been put in place previously. However, in order to reduce the probability of the protective systems being called upon, it was decided to investigate whether a slight reduction in the oxygen concentration, or partial inerting [6], would have a beneficial effect upon the dust cloud MIE, and what oxygen level might be required.
The test program was carried out using the standard test procedure of EN 13821\(^1\) and the Kühner MIKE3 equipment. In order to determine the MIE an oxygen depleted atmosphere, the equipment was simply fed from a cylinder of compressed gas with the required nitrogen / oxygen composition, rather than the normal air compressor. The flushing facility around the electrodes was used to establish the required oxygen concentration in the tube before dispersion, which enabled the atmosphere to be purged without disturbing the powder in the dispersion cup. Oxygen measurements were carried out prior to each series of tests and showed that the concentration in the equipment was consistently within 0.5 % v/v of the nominal concentration of the cylinder.

The results on Powder T showed that ignitions still occurred at 1 mJ with 14% v/v oxygen in the atmosphere. Below this level the change in MIE was rather rapid, increasing to >1000 mJ (the maximum range of the equipment) with only a further 4% change in oxygen concentration. Published work with propane / oxygen / nitrogen mixtures\(^6\) indicates that reducing the oxygen concentration from 21% to 14% v/v increases the MIE by an order of magnitude. If a similar effect were occurring with Powder T, then an MIE <1 mJ at 14% oxygen suggests an atmospheric MIE of around 0.1 mJ, which would clearly present an unusual degree of hazard.

### 3. Additional Work

These results highlight a limitation of the standard MIE equipment, that differentiation of MIEs <1 mJ is not possible. In the past this was not a major concern as few materials had MIEs <1 mJ, and it was generally assumed that they would only be slightly below 1 mJ. However, results <1 mJ are becoming more common, and different hazards / risks may be presented by a material with an MIE just below 1 mJ and one at 0.1 mJ. Therefore, the work was extended to see if more precise MIE values could be determined from values measured in the standard equipment with a reduced oxygen atmosphere. Additional samples of powders with MIEs <1 mJ were obtained, most being the same as tested in [5] & [9], obtained directly from the relevant workers to minimize any differences. The results of the tests are given in Table 1, together with those for Powder T.

| Oxygen (% v/v) | 21 | 18 | 16 | 14 | 12 | 10 | 8 | 6 | LOC |
|---------------|----|----|----|----|----|----|---|---|-----|
| Ti Grade E    | 0.012 | 3-10 | >1000 | 4-7% |
| Sulphur       | 0.043 | 10-30 | >1000 | 9% |
| SIBS-K32      | 0.1 | 3-10 | 300-1000 | 16.0-16.5% |
| ZiH Grade S   | 0.13 | 3-10 | 100-300 | >1000 | 8% |
| TiH Grade VM  | 0.19 | 3-10 | >1000 | 10% |
| Ti Grade S    | 0.36 | 100-300 | 300-1000 | 4-7% |
| CARO 03       | 0.54 | 30-100 | >1000 | 7-9% |
| Powder T      | <1 | 30-100 | >1000 | 7-9% |

Table 1 - Measured MIEs with Reduced Oxygen, with Atmospheric MIEs from [5] & [9], and LOC data

The results clearly show a general trend of increasing MIE with reducing oxygen, though with some apparent inconsistencies. For example, the two Ti grades have very different MIEs at 21% oxygen, but at 8% oxygen they are the same; and Sulphur has the second lowest MIE at 21% oxygen, but increases rapidly to >1000 mJ at 10%. To determine whether differences in the LOCs might be responsible, measurements were carried out according to EN 14034-4\(^4\), and these did appear to account for some of the apparent anomalies. This agrees with [10], which suggested the general relationship shown in Figure 1. This is simply an exponential curve, with asymptotes at the LOC and MIE in pure oxygen. However, the precise form of the curve warrants further discussion.

[Figure 1 - General form of Relationship between Oxygen Concentration and MIE as given in [10]](image)

[Figure 2 - MIE of Propane in Oxygen / Nitrogen Atmospheres](image)
4. Data Analysis

In [10], where testing was carried out on a number of powders with both reduced and elevated oxygen levels, the form of the equation was proposed as being $IE = MIE \times e^{23.2-23.2-21 \times O_2}$. This was reported to give a good fit at reduced oxygen levels for some of the dust examined in that paper, though not with others, and also no agreement was found with the LOC or with elevated oxygen concentrations. Some of the issues may have occurred as not all of the measurements were carried out using the standard MIE apparatus; at higher ignition energies a 20 litre sphere was used with both spark (5 to 50 J) and chemical igniters (100 J to 10 kJ), and a 1 m$^3$ vessel was also used with 100 J to 10 kJ chemical igniters.

In [11], where testing was carried out with hydrocarbon gases at elevated oxygen levels, a different form of equation was proposed, with the ignition energy being proportional to the oxygen concentration raised to a powder of 4 or 5 (depending whether conditions were quiescent or flowing respectively). The LOC was not used as a factor in that study, presumably as only elevated oxygen levels were of interest. However, incorporating the LOC into the equation (ie using $[O_2 - LOC]$ rather than $[O_2]$) results in this type of correlation giving a good fit to the data on propane in reduced and enriched oxygen atmospheres presented in [7] & [12]/[12] respectively, as shown in Figure 2, though with a modified exponent of 2.5. In view of these factors, the following correlation was proposed for the relationship between the MIE of the dusts tested and the oxygen concentration in the atmosphere:

$$MIE_{[O_2]} = MIE_{21} \times ((21 - LOC)/([O_2] - LOC))^{3.75}$$

{[1]}

Figure 3 - MIE vs $O_2$ Concentration for Ti Grade E

Figure 4 - MIE vs $O_2$ Concentration for Sulphur

Figure 5 - MIE vs $O_2$ Concentration for SIBS-K32

Figure 6 - MIE vs $O_2$ Concentration for ZiH Grade S

Figure 7 - MIE vs $O_2$ Concentration for TiH Grade VM

Figure 8 - MIE vs $O_2$ Concentration for Ti Grade S
The experimental and predicted data are shown in Figures 3 to 10 and illustrate that, although there is some scatter, the equation does provide a good correlation with the MIE and LOC values measured. Furthermore, it does appear to confirm that the original organic powder under investigation, Powder T, may indeed have an MIE in the region of 0.1 mJ under normal atmospheric oxygen conditions, and so presents an unusual degree of hazard.

5. Conclusions
The experimental technique devised enabled the reliable determination of the MIE of dust clouds at reduced oxygen concentrations, with only minor changes to the standard method and equipment. The results of such tests may enable partial inerting to be applied with more confidence in situations where either standard inerting or control of ignition sources if not feasible or, as in the case initially investigated, to reduce the possible demands placed on explosion protection systems.

In addition, a correlation was devised to describe the relationship between the MIE of a dust cloud and the oxygen concentration of the atmosphere. This is based on an exponential relationship between two asymptotes, one at the LOC and the other at the MIE in pure oxygen. Finally, the work also confirmed that at atmospheric oxygen conditions, the organic powder originally examined may have an MIE in the region of 0.1 mJ. This clearly means that it may present increased hazards and risks compared to powders with MIEs only slightly below 1 mJ.

In view of the increased frequency at which measurements of <1 mJ are being obtained for dust clouds, the ability to obtain a more precise MIE value for sensitive powders may become increasingly important. In particular, it may be relevant when evaluating the relative merits of different bases of safety, as lower values of MIE are likely to mean an increased ignition risk, which may result in a preventative measure, such as inerting, being preferred to protective measures, such as venting or suppression.

References
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