

THE EFFECT OF REDUCED OXYGEN LEVELS ON THE ELECTROSTATIC IGNITION SENSITIVITY OF DUSTS

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When handling powders with low values of minimum ignition energy (MIE), the reliable control of potential electrostatic ignition hazards can become difficult. In such situations 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 ~50% of the minimum required for combustion, typically resulting in an operating level of 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, to determine whether "partial" inerting would allow potential electrostatic ignition sources to be controlled with a more moderate reduction in the oxygen level.

In addition, current commercial equipment can only measure dust cloud MIEs down to a minimum of 1 mJ. In the past this has not been a major issue as very few materials gave such low results. However, increasing numbers of materials are giving results of <1 mJ, and other research has shown that actual values can be much lower (even <0.1 mJ). Therefore, 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.

KEYWORDS: dust cloud explosion; minimum ignition energy (MIE); inerting

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 values of MIE, it may not be possible to control all potential electrostatic ignition sources reliably during handling and processing operations. For example, even if all fixed metallic parts are earthed and insulating plastics are excluded, small pieces of tramp metal (eg 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], this 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.

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 handling / 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)^[3] 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, as shown in Figure 1. The equipment comprises a cylindrical glass tube, with two radially mounted electrodes, which fits on top of a dispersion cup. A

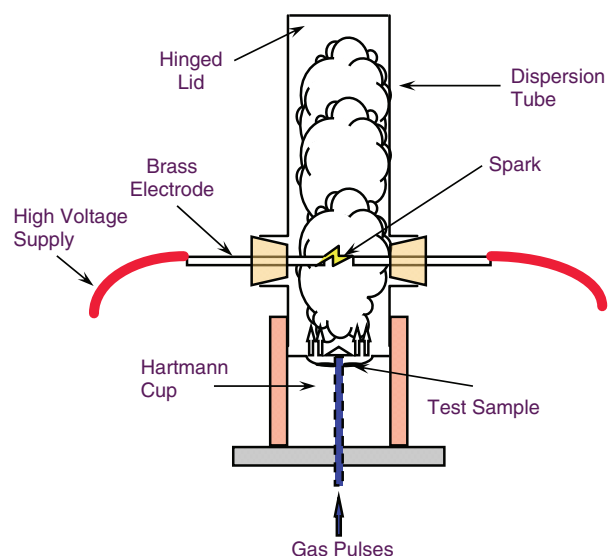


Figure 1. Standard Kühner MIKE3 apparatus for MIE measurement

weighed sample of the powder is placed in the cup, and a pulse of compressed air from a reservoir is then released to disperse the powder. After a preset time delay, a capacitive spark of known energy (1000, 300, 100, 30, 10, 3, or 1 mJ) is passed through the dust/air atmosphere to attempt to initiate ignition. If ignition occurs, the test is repeated with a lower spark energy. For a nonignition to be recorded at a given spark energy there must be no flame during a series of ten tests at each of four nominal dust concentrations (900, 1500, 2400, and 3600 mg/l), and three ignition delays (60, 120 and 180 ms); a total of 120 tests.

In order to determine the MIE of the powder in question in 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. This meant that the standard computer control system could continue to be used. To establish the required oxygen concentration in the tube before dispersion, the flushing facility around the electrodes was employed, which enabled the atmosphere to be purged without disturbing the powder in the dispersion cup. Measurements with an oxygen analyser were carried out

prior to each series of tests and showed that the oxygen concentration in the equipment was consistently $<0.5\%$ v/v above the nominal concentration of the cylinder.

INITIAL RESULTS

The results on the original powder examined are presented in Table 1. They illustrate that, rather surprisingly, ignitions still occurred at 1 mJ with as low as 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. It is also interesting to compare these results to those of some small scale thermal stability screening tests under similar reduced oxygen atmospheres, which exhibit a relatively similar trend.

Previous work with propane/oxygen/nitrogen mixtures^[6] indicates that reducing the oxygen concentration from 21% to 14% v/v increases the MIE of that atmosphere by an order of magnitude. If a similar effect were occurring with the powder under evaluation, then an MIE still <1 mJ at 14% oxygen would suggest an MIE of around 0.1 mJ at normal atmospheric oxygen levels. This value of MIE is similar to, if not slightly lower than, most common flammable gases and vapours, and so would clearly present an unusual degree of hazard compared to most powders.

Although an MIE of 0.1 mJ would be far lower than previously considered feasible for even sensitive powders, recent research by others in the field^{[5],[9]} using specialised equipment has indicated that some powders can have MIEs in this region. The fact that the circuit used in that work was different to that used in the standard MIKE3 apparatus will mean that the figures are not directly equivalent to those determined in the MIKE3, however, these factors were not considered critical with regard to the current work and are not evaluated here in detail.

ADDITIONAL WORK

The initial results highlighted one of the limitations of the standard MIE equipment, in that differentiation of MIEs <1 mJ is not possible. In the past, very few materials had such low MIEs so this was not a major concern, and it was generally assumed that they would only be marginally below 1 mJ. However, measurements of <1 mJ are becoming increasingly common, and a different degree of

Table 1. Results of reduced oxygen MIE tests on original organic dust "Powder T"

Atmosphere	Minimum ignition energy	Small scale thermal stability – aerated cell ^[8]
21% O ₂	<1 mJ	Onset 170°C, very rapid 275°C, max >500 °C
14% O ₂	<1 mJ	Onset 170°C, rapid from 255°C, max >500 °C
12% O ₂	30–100 mJ	Onset 175°C, marked from 255°C, max 475°C
10% O ₂	>1000 mJ	Onset 180°C, marked from 260°C, max 460°C
8% O ₂	>1000 mJ	Onset 185°C, progressing slowly to max 440°C
6% O ₂	N/A - below LOC	Onset 200°C, progressing slowly to max 445°C

Table 2. Measured MIEs with reduced oxygen, with atmospheric MIEs from^{[5],[9]} and LOC data

Oxygen (% v/v)	21	18	16	14	12	10	8	6	LOC
Ti Grade E	0.012					<1	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						3–10		4–7%
CARO 03	0.54		100–300	300–1000					12.6–14.9%
Powder T				<1	30–100	>1000			7–9%

hazard and/or risk may be presented by a material with an MIE marginally below 1 mJ and one at 0.1 mJ.

Therefore, subsequent work was carried out to determine whether it was possible to use MIE values obtained in the standard equipment but with a reduced oxygen atmosphere to obtain a more accurate atmospheric MIE value for powders which gave a result of <1 mJ in the standard test. At the same time, consideration was still given to the level of partial inerting required with sensitive powders to allow potential electrostatic ignition sources to be controlled at oxygen levels above the LOC.

To carry out the work, additional samples of powders with MIEs <1 mJ in the standard test were obtained. Most of these were the same as tested in [5] & [9], and were obtained directly from the relevant workers to avoid any differences (eg particle size, purity, moisture content) between nominally similar materials. The results of the tests are given in Table 2, together with those for Powder T.

DISCUSSION

The results obtained clearly show a general trend of increasing MIE with reducing oxygen concentration. However, the change at first does not appear consistent across the powders. For example, the two different Ti grades have very different MIEs at 21% oxygen, but at 8% oxygen they are the same. Compared to this, the Sulphur (which has the second lowest MIE at 21% oxygen) increases much more rapidly (>1000 mJ at 10%, compared to <1 mJ for the Ti Grade E).

In order to determine whether differences in the LOCs of the powders might be responsible for these observations, appropriate measurements were carried out according to EN 14034-4^[4]. The results of these tests did indeed appear to account for some of the apparent anomalies in the observed MIE values at reduced oxygen concentrations. This is in agreement with [10], which suggested the general relationship shown in Figure 2. 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.

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 $IE = MIE * e^{23.2 - (23.2 - 21) * O_2}$. 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 m³ 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 power 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 $[O_2 - LOC]^x$ rather than $[O_2]^x$) 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]/[13] respectively, as shown in Figure 3, though with a modified exponent of 2.5.

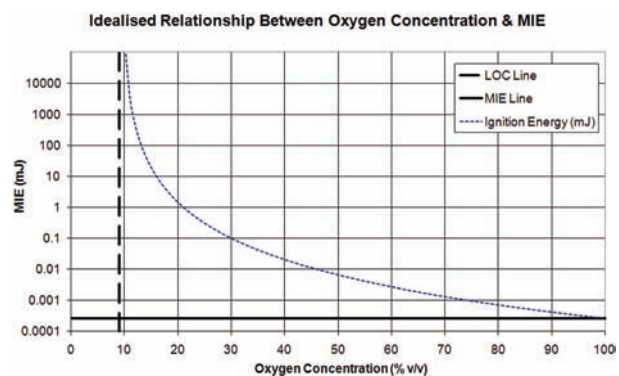


Figure 2. General form of relationship between oxygen concentration and MIE as given in [10]

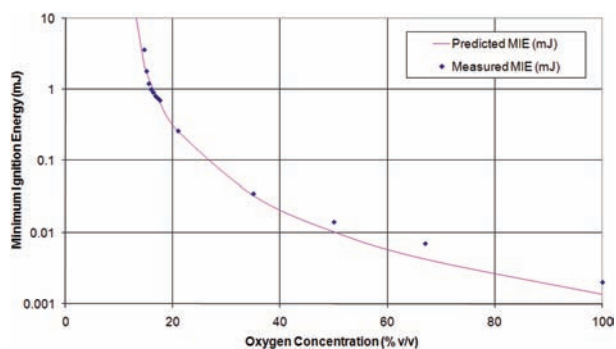


Figure 3. MIE of propane in oxygen/nitrogen atmospheres

DATA ANALYSIS

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:

$$MIE_{[O_2]} = MIE_{21} * ((21 - LOC) / ([O_2] - LOC))^c \quad (1)$$

where MIE_{21} is the MIE measured under standard conditions of 21% oxygen (mJ, according to [1]); $MIE_{[O_2]}$ is the MIE at a particular oxygen concentration (mJ); $[O_2]$ is the oxygen concentration of the atmosphere (% v/v); LOC is the limiting oxygen concentration for combustion (% v/v, according to [4]) c is a constant (a value of 3.75 gave the best fit across all of the powders examined).

This equation gives a general curve of the same form as proposed in [10], but in addition it provides an improved fit to the LOC data (insufficient data are available to determine how good the correlation is for MIEs at elevated oxygen levels). The data obtained on the various powders, together with the calculated data from this equation, are shown in Figures 4 to 11.

These results illustrate that, although there is a certain degree of scatter, the equation proposed does appear to provide a reasonable 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

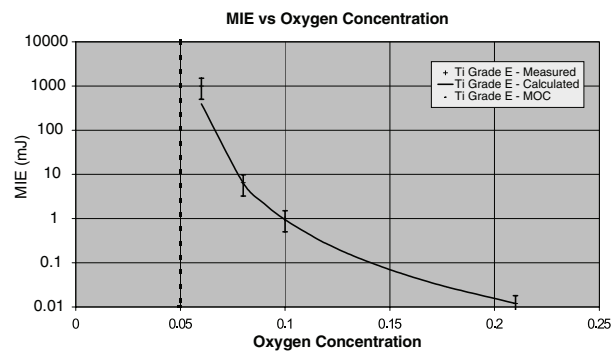


Figure 4. MIE vs O₂ concentration for Ti Grade E

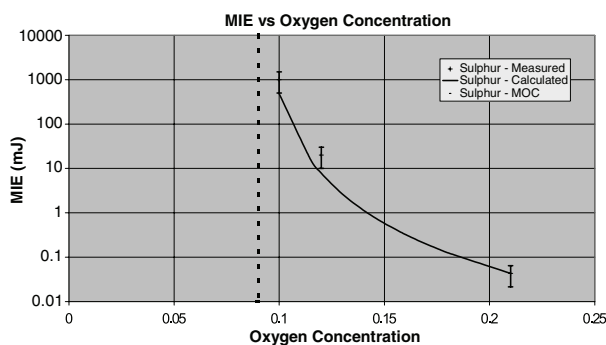


Figure 5. MIE vs O₂ concentration for sulphur

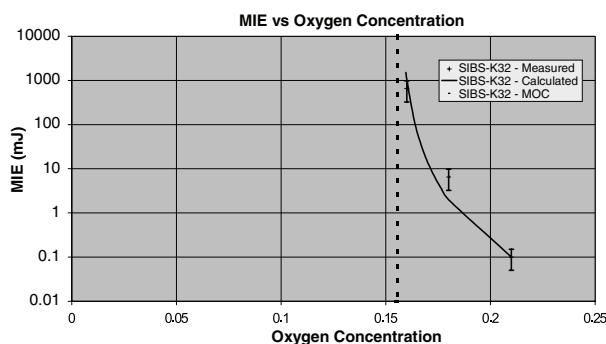


Figure 6. MIE vs O₂ concentration for SIBS-K32

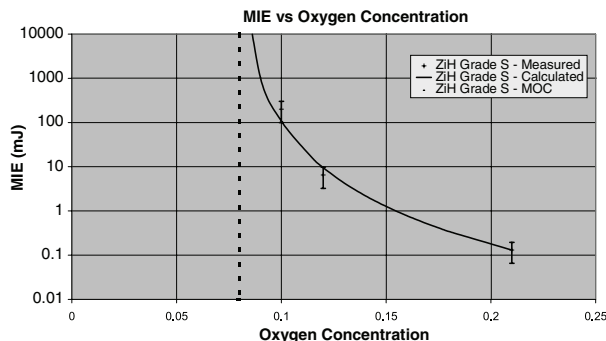


Figure 7. MIE vs O₂ concentration for ZiH Grade S

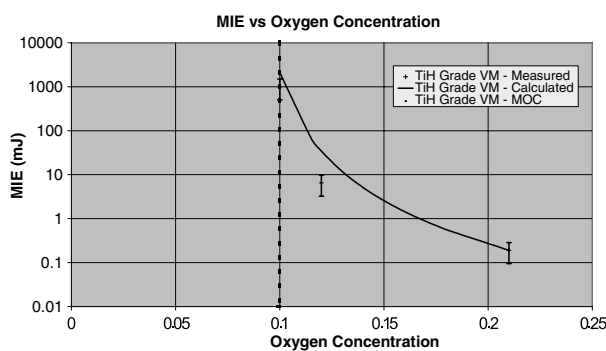


Figure 8. MIE vs O₂ concentration for TiH Grade VM

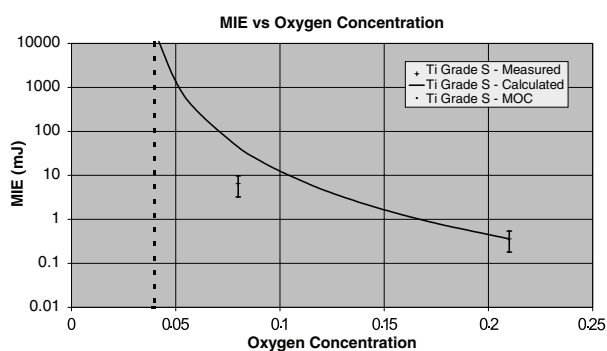


Figure 9. MIE vs O₂ concentration for Ti Grade S

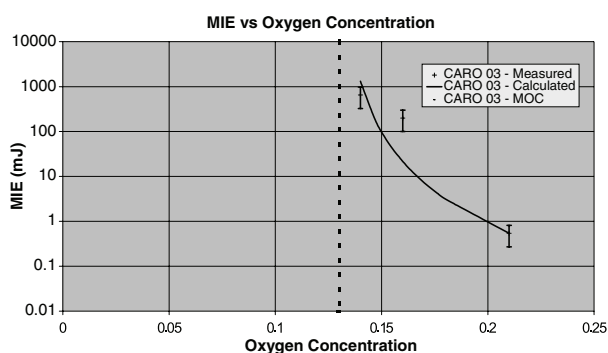


Figure 10. MIE vs O₂ concentration for CARO 03

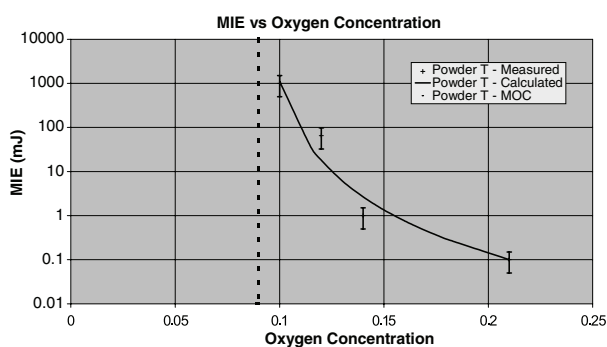


Figure 11. MIE vs O₂ concentration for powder T

0.1 mJ under normal atmospheric oxygen conditions, and so presents an unusual degree of hazard.

The form of the equation also means that it is possible to obtain an estimate of the LOC of a powder by carrying out a series of MIE measurements at different oxygen concentrations, as was the stated intention of [10]. The results of the predictions according to the correlation presented here are given in Table 3, and are in general quite good. Using the same form of equation with propane, but with an exponent of 2.5 as discussed previously, also gives a good prediction of its LOC, perhaps suggesting that a slightly different approach is required for gases and vapours compared to powders. A reasonable correlation with ethane, using solely data at elevated oxygen concentrations from [13], is also obtained.

The main exception is with the Ti Grade S, where only two MIE data points were available. In view of this, the lack of an accurate prediction of the LOC is not particularly surprising. However, it is worth noting that in this case, and in fact all the cases both for powders and gases where the predicted value is significantly different from the measured value, the LOC is underestimated. This at least suggests that if this approach is used it will err on the side of safety, though it is not considered that it is likely to become a routine method for determining LOC values.

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 is 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. Although the MIE in pure oxygen is a relatively theoretical parameter,

Table 3. Predicted LOC data based on MIEs at reduced oxygen concentrations

	MIE data points	Measured LOC	Predicted LOC
Ti Grade E	4	4–7%	5.2%
Sulphur	3	9%	9.2%
SIBS-K32	3	16.0–16.5%	15.5%
ZiH Grade S	4	8%	7.8%
TiH Grade VM	3	10%	8.5%
Ti Grade S	2	4–7%	1.0%
CARO 03	3	12.6–14.9%	13.1%
Powder T	4	7–9%	9.0%
Ethane	5	8.8%	7.6%
Propane	13	9.8%	9.7%

the LOC is of important practical use and the fact that it is present in the correlation means that the equation can also be used to estimate the LOC if sufficient MIE data at varying oxygen concentrations are known. Furthermore, this correlation also appears to be valid for simple hydrocarbon gases, though a different exponential constant is required.

Finally, the work also confirms that at atmospheric oxygen conditions, the organic powder originally examined does 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.

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