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Problems in experimental measurements of dust explosions

K.J. Mintz*

Canadian Explosives Research Laboratory, CANMET, 555 Booth St., Ottawa, ON K1A 0G1, Canada

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Abstract

The experimental method employed for measuring the parameters of dust explosions was critically examined using methane/air as a model system. Because of the rapid rate that air enters the test vessel (in order to disperse the dust), the system is not at thermodynamic equilibrium which causes the actual concentration of air to be about 15% less than indicated by the measured pressure at the time of ignition, thus creating a significant error in the determination of explosion pressures. For measuring the lower explosible limit, the ignitor must be of optimum strength, otherwise too high or low limit values are obtained. Pyrotechnic ignitors, which are usually used for igniting dusts, produce a significant pressure by themselves; a method for accurately taking this effect into account is given. Some unusual problems encountered with particular dusts are discussed.

1. Introduction

The parameters of dust explosions are commonly carried out in vessels of the order of 0.020 m³. Although the exact method of creating the dust cloud varies from vessel to vessel, the fundamental principle is that a strong pulse of air disperses the dust (which is usually placed in another smaller vessel outside the test vessel). Thus, the dust explosion testing is carried out under turbulent, dynamic conditions. In order to ensure the meaningfulness of the results, a detailed study was carried out, using methane–air explosions to compare dynamic with static conditions.

2. Experimental

The 0.020 m³ vessel was essentially the same type as used by Cashdollar and Hertzberg [1]. The dust was placed in a chamber underneath the vessel and the vessel

* Fax: (+1-613) 995-1230. Tel.: (+1-613) 995-1413.

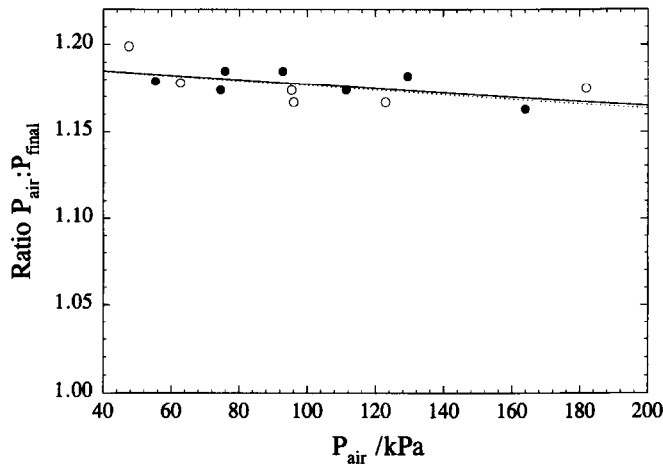


Fig. 1. The effect of different tank pressures (P_{tank}) on the $P_{\text{air}}/P_{\text{final}}$ ratio, where P_{air} is the peak pressure and P_{final} is the pressure at equilibrium: (○) $P_{\text{tank}} = 1.1 \text{ MPa}$; (●) $P_{\text{tank}} = 1.3 \text{ MPa}$.

evacuated. A 0.016 m^3 pressure vessel containing air, usually at 1.1–1.3 MPa, was connected to the dust chamber through a solenoid valve. A control panel allowed the selection of time that the solenoid valve remained open as well as the selection of delay time before firing (and, for ignition by an electric arc, the length of time that the arc discharges). Generally, the aim was to have the explosion test carried out at as close to normal atmospheric pressure as possible, so as to be applicable to most industrial applications. A CEC1000 pressure transducer, connected to a Nicolet 4094 digital oscilloscope, was used to measure the pressure throughout the experimental sequence. Methane–air mixtures were prepared in a binary mixer from technical grade methane and dry air, and an infrared analyzer was used to verify the methane concentration. Also, a paramagnetic oxygen analyzer was used to measure the concentration of oxygen remaining in the vessel after the trial. Two types of ignition were used: an electric arc and Sobbe pyrotechnic ignitors of nominal energy from 250 to 5000 J.

3. Results and discussion

3.1. Calibration of air concentration

These tests were carried out without dust and an ignition source, i.e. only air was involved. After the solenoid valve was opened, the pressure inside the vessel increased rapidly (from 0 kPa absolute) until the valve was closed, then slowly decreased to reach a constant value approximately 15% smaller after about 15–70 s. The initial pressure rise was close to linear because only a small fraction of the air in the pressure tank was required to pressurize the test vessel to 1 bar. Fig. 1 shows that the ratio of the peak pressure, P_{air} to the final pressure P_{final} is nearly independent of normal

variations of the pressure in the pressure vessel (which provide different flow rates) and solenoid valve open times (to provide different final pressures). In addition, different initial vessel pressures (which are used by other workers) do not substantially affect this ratio.

Because of the rapid rate at which the air enters the vessel, it is not at thermodynamic equilibrium (i.e. does not follow the Boltzmann distribution law) and therefore does not have a definable temperature. Although the pressure in the explosion vessel decreases after the solenoid valve closes, the *concentration*, which is a more fundamental quantity, must remain constant. The final pressure, therefore, must be used as the actual 'pressure' at which the explosion test is carried out, even though ignition of the dust cloud is usually done about 0.1–1 s after the solenoid valve is closed, i.e. when the measured pressure is still near P_{air} . In an explosion test, it is impossible, of course, to measure a P_{final} free from the effects of the explosion; however, the fact that the ratio $P_{\text{air}}/P_{\text{final}}$ is quite constant means that it is valid to divide the measured value of P_{air} by 1.17 ± 0.01 (95% confidence limit) to yield an 'equivalent pressure', the pressure that would be measured for that concentration of air if the air were at equilibrium.

A large body of evidence exists that for gases and dusts the explosion pressure is directly proportional to the initial pressure (at constant initial temperature). In addition, the explosion pressure of dusts has been found to be directly proportional to the reciprocal absolute temperature at constant initial pressure [2]. These two relationships can be combined to state that the explosion pressure is directly proportional to the initial concentration of gas. This conclusion is quite reasonable because it implies a reaction which is first order in oxygen concentration.

The implication of the above is that explosion tests carried out (in 0.02 m³ vessels) at an apparent (measured) pressure of 1 bar are actually carried out at the equivalent of 0.85 bar, and therefore a correction factor of 1.17 must be applied to the measured explosion pressures to yield the correct values for 1 bar.

3.2. CH₄-air explosion tests: electric arc ignition

First, tests were carried out under quiescent conditions by preparing a mixture in the 0.020 m³ vessel by partial pressures and measuring the methane concentration by the infrared analyzer, to an accuracy of $\pm 0.1\%$ absolute. (The total pressure in all tests was 1 bar.) Fig. 2 shows that the explosion pressure of this system reaches a broad maximum of 700 ± 10 kPa when an electric arc is used, close to the value of 720 kPa obtained by Hertzberg et al. [3]. Fig. 3 shows that the peak rate of pressure rise also rises smoothly to a maximum of 24.8 ± 0.6 MPa s⁻¹, close to the value of 25 MPa s⁻¹ of Hertzberg et al. [3].

Second, tests were carried out under turbulent conditions similar to those when dusts are tested. A quantity of methane to give an approximately stoichiometric mixture was metered into the vessel; the solenoid was opened so that air could flow quickly from the pressure tank into the vessel to raise the total *equivalent* pressure to 1 bar, and the arc fired at various delay times. Fig. 4 shows that the explosion pressure decreases slightly with delay time; the extrapolated value to infinite delay time, which should be a quiescent state, is 710 ± 20 kPa, close to the value obtained under true

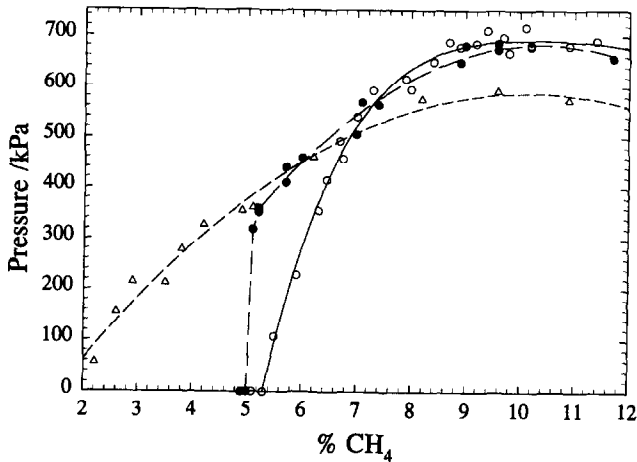


Fig. 2. Explosion pressure as a function of methane concentration under quiescent conditions, for different ignition sources: (○) arc ignitor; (●) 250-J Sobbe ignitor; (△) 5000-J Sobbe ignitor.

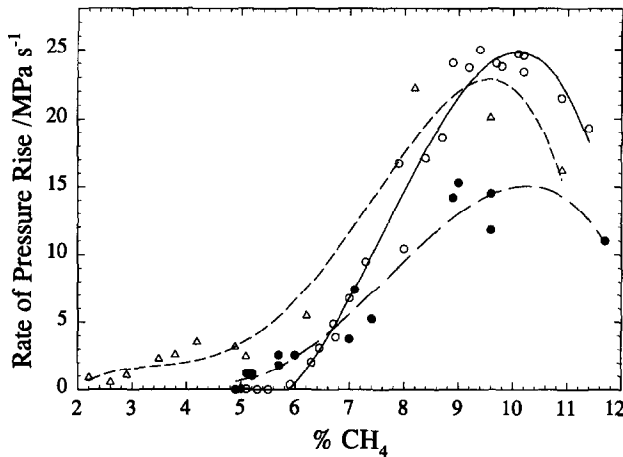


Fig. 3. Rate of pressure rise as a function of methane concentration under quiescent conditions, for different ignition sources: (○) arc ignitor; (●) 250-J Sobbe ignitor; (△) 5000-J Sobbe ignitor.

quiescent state. If the apparent (measured) pressure were used instead of the equivalent pressure, then the explosion pressure would be only about 600 kPa, which would clearly be in error. With the rate of pressure rise, the scatter is much greater, as can be seen in Fig. 5. More work would have to be carried out to establish the functional dependence of rate on delay time. Since the physical processes governing the effects of turbulence on dust-air flames are mostly the same as those involved in gas flames, an estimate of the zero-turbulence parameters of dust-air explosions can be made by extrapolation from the values obtained at the usual delay time of 100 ms. For the

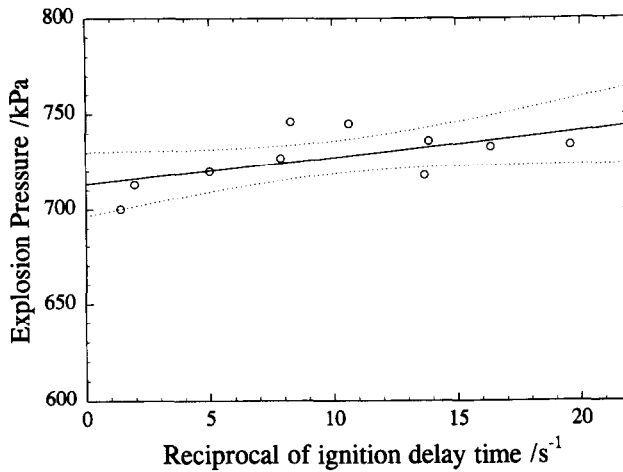


Fig. 4. Peak explosion pressure for stoichiometric methane–air mixtures as a function of turbulence, with the 95% confidence limits shown as dotted lines.

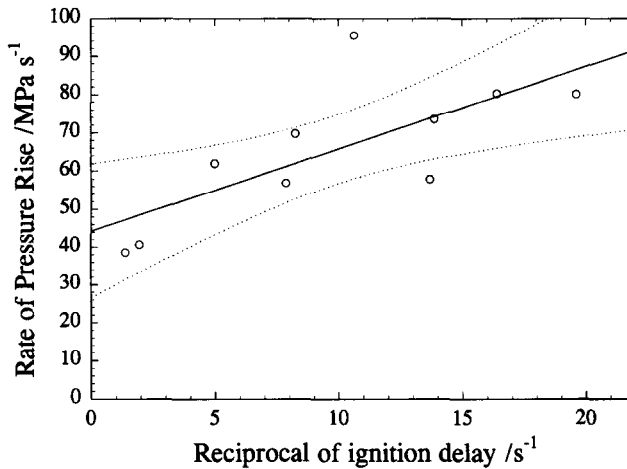


Fig. 5. Peak rate of pressure rise for stoichiometric methane–air mixtures as a function of turbulence, with the 95% confidence limits shown as dotted lines.

explosion pressure, that value would be about 2–4% lower than that measured; for the rate of pressure rise, the value would be of the order of half.

3.3. CH_4 –air explosion tests: Sobbe pyrotechnic ignitors

The data in Figs. 2 and 3 show that the electric arc tests yield an apparent lower flammability limit (LFL) of about 5.9%, well above the established value of 5.0%.

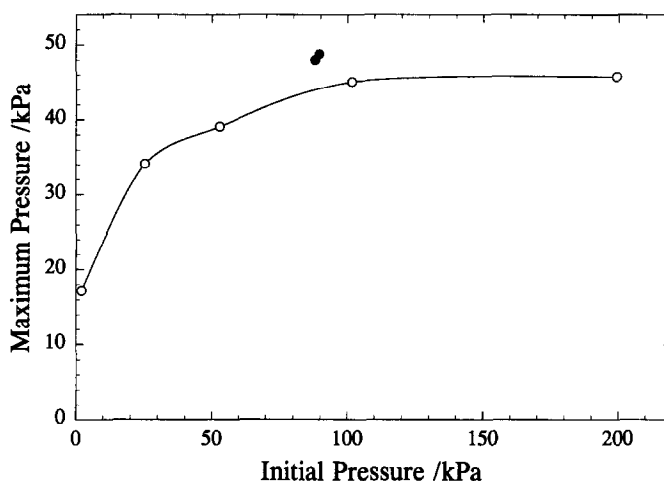


Fig 6. Peak explosion pressure from the 5000-J Sobbe pyrotechnic ignitor in 20 l vessel at different initial pressures: (○) quiescent mixtures; (●) turbulent mixtures.

Therefore, a stronger ignitor is required to measure the LFL. Tests were carried out with two strengths of pyrotechnic ignitors: 250-J and 5000-J, as shown in Figs. 2 and 3. The problem in using these types of ignitors is allowing for their effects on the system. When ignited in air at 1 bar, the 250-J ignitor produces 9 kPa maximum overpressure. Since this value is of the same order as the precision of the results, it does not affect significantly the accuracy of the explosion pressure measurements. (Nevertheless, 9 kPa was subtracted from the apparent explosion pressures to yield the best estimate of the 'true' explosion pressure.) On the other hand, as shown in Fig. 6, the 5000-J ignitor produces a significant pressure, which is a function of total pressure, particularly at low pressures. Turbulence increases the peak pressure, but also increases the rate at which the pressure decreases after the maximum, probably because of faster heat transfer to the walls of the vessel. The usual way of accounting for the 5000-J ignitor is simply to subtract its maximum pressure from the total overpressure. There is a logical problem with that method: the maximum pressure of the ignitor occurs very soon after ignition, well before the maximum pressure due to the dust or gas, thus, that method overcorrects for the ignitor. Furthermore, the time to maximum explosion pressure varies with the concentration of ignitor and also varies from dust to dust, so that even relative values of explosion pressure will be in error due to this effect. In order to resolve that problem, the pressure trace for the ignitor by itself was subtracted from the experimental pressure trace to yield the net pressure trace in each trial. The latter was used to obtain the maximum overpressure and the maximum rate of pressure rise. However, it should be realized that the ignitor also causes an increase in temperature of the gas, but likely not uniformly. Therefore, use of the Sobbe ignitors increases the uncertainty of explosion tests; to reduce this uncertainty, it is best to use as small an ignitor as possible.

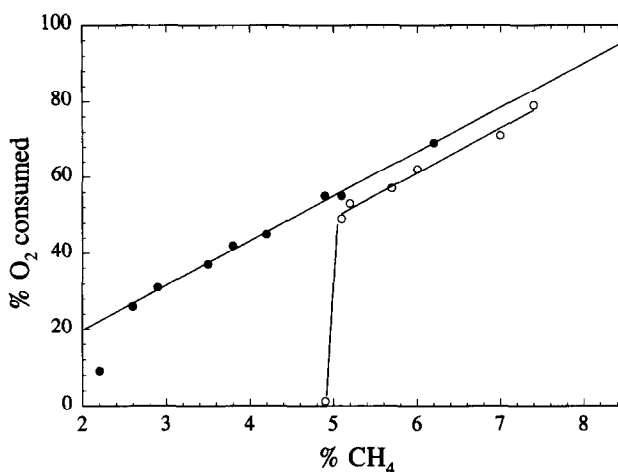


Fig. 7. Percentage oxygen consumed as a function of percent methane in methane–air mixtures: (○) tests with a 250-J Sobbe ignitor; (●) tests with a 5000-J Sobbe ignitor.

The results from tests under quiescent conditions with the 250-J and 5000-J ignitors (Fig. 2) yield completely different results for the LFL, with the former yielding a definitive value of $5.1 \pm 0.1\%$, close to the accepted value of 5.0%, whereas the latter does not yield a sharp cut-off, but appears to be at less than 3%. The shape of the curve for the 5000-J ignitor tests is indicative of overdriving, i.e. the system does not reach a steady state in the limited vessel size, but still ‘sees’ the effect of the ignitor. Hertzberg et al. [3] have attributed the cause of the lower peak explosion pressure obtained with a pyrotechnic ignitor (compared to an electric discharge) to it being a nonpoint source and thus causing a less adiabatic reaction. Alternatively, it can be considered that a fraction of the methane reacts during the time that the ignitor is active (due to the overdriving) and thus does not contribute to the ‘normal’ explosion pressure. From the difference in peak explosion pressures, this hypothesis leads to an estimate of the percentage thus reacted: 3% for the 250-J ignitor, 10% for the 1000-J ignitor and 16% for the 5000-J ignitor. (These values, of course, are specific to a particular set of ingredients.) The peak maximum rate of pressure rise using 250-J ignitors is distinctly less than that using either electric arc ignition or 5000-J ignitors. This parameter is measured earlier in the process than the maximum pressure, thus is more susceptible to effects from the ignitor. Furthermore, the time to ignition is much shorter for tests with the 5000-J ignitor; therefore, the pressure peaks from the ignitor and the dust explosion will overlap more causing a greater uncertainty in the results. Further work would have to be carried out to determine if the apparent decrease in rate of pressure rise is in fact real.

Fig. 7 shows the percentage of oxygen consumed for different methane–air mixtures using 250-J and 5000-J ignitors. In both cases, the result is a straight line which extrapolates to between 0% and 1% CH₄ at one end and close to the stoichiometric

mixture at the other end. The break-point, i.e. the point at which the points deviate from the straight line, may be indicative of a nonself-sustaining explosion. Finding the break-point on the oxygen consumption curve may be a more decisive method than using the peak pressures (cf. Fig. 2). The methane concentration in the vessel measured after the test was close to zero for all tests that fall on the line. The data in Fig. 2 show that the quantity of oxygen consumed was twice that of methane (within 5%), which is consistent with the simple reaction: $\text{CH}_4 + 2\text{O}_2 \rightarrow \text{CO}_2 + 2\text{H}_2\text{O}$.

The LFL was also measured under low-turbulent and high-turbulent conditions (570 and 20 ms delay times, respectively), using a 250-J ignitor. The results were $4.8 \pm 0.1\%$ and $4.9 \pm 0.1\%$, respectively, which are slightly lower than those obtained in the quiescent mixture (5.1%) and close to the accepted value (5.0%).

3.4. Dust tests: blank runs

In order to separate the physical effects of dust from the chemical, tests were carried out using finely powdered CaCO_3 and a 5000-J ignitor in exactly the same way as trials with explosible dusts were carried out. The peak pressure decreased significantly with increasing dust concentration (49 kPa with no dust to 41 kPa with 100 g/m^3 and 35 kPa with 500 g/m^3). The decrease is probably caused by the dust absorbing energy that would otherwise go to the air; thus, the gas does not heat up as much and produces less pressure. A second factor is the backpressure caused by the dust which decreases the rate of air entering the vessel; the resultant lower level of turbulence affects the shape of the pressure trace after the peak pressure, because the transfer of heat to the walls decreases. (At higher loadings of dust, the solenoid valve must be left open longer to achieve the same 1 bar pressure in the explosion vessel.) Therefore, for accurate work, the pressure trace of a trial using the same concentration of an inert dust as that used for the explosible dust and with the same ignitor must be used for subtraction from the experimental pressure trace before further analysis is carried out. In principle, the physical size of the inert dust should be matched to the combustible dust; however, that is a second-order effect and is much smaller than normal trial-to-trial variability.

3.5. Effects of dust dispersion

Considerable work has shown that dust particles above 0.5 mm are nonexplosible [4]. Tests were carried out in the standard way (using 5000-J Sobbe ignitors) on a sample of petroleum coke that had particles of between 1.6 and 9.5 mm diameter. The minimum explosible concentration was found to be 410 g/m^3 and the maximum explosion pressure to be 380 kPa, which would seem to contradict previous knowledge. However, dispersing the sample under the same conditions as the explosion tests, but without any ignition source, produced a fine powder. Therefore, the necessity of applying a high-pressure air blast to disperse a dust sample can significantly affect the physical characteristics of the dust and thus its explosibility characteristics. This material had actually been involved in an explosion in a factory. Later tests proved that normal handling of the material (e.g. dropping a bag) could generate

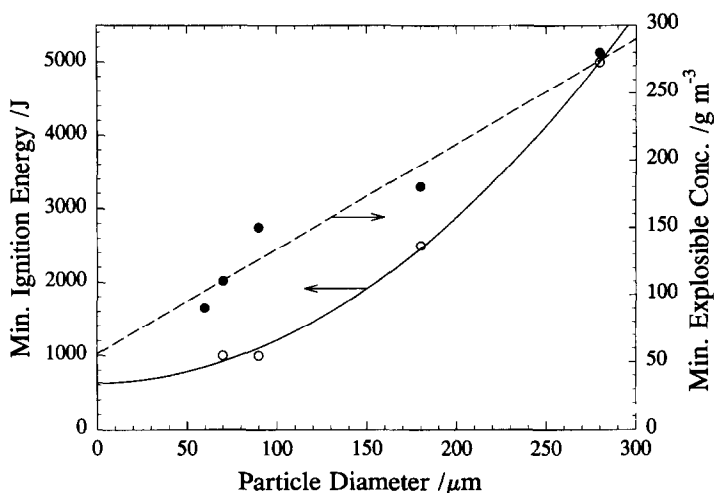


Fig. 8. Minimum ignition energy of pyrite dust (○) and minimum explosible concentration using a 5000-J Sobbe ignitor (●) as a function of particle diameter.

powder similar to that observed in the test without an ignition source. Therefore, knowing the friability of a dust sample is important both for accurate measurement and interpretation of its explosibility characteristics as well as for its hazard in use.

3.6. Sulphide dusts

These dusts are of interest because explosions of sulphide dust clouds occur in mines only when initiated by a blast, i.e. an extremely strong ignition source. In laboratory-scale apparatuses, electric arc ignition does not ignite an iron disulphide (pyrite) dust cloud and does not even produce any reaction, unless artificially stimulated by operating in pure oxygen. However, use of the Sobbe ignitors does produce explosions when ordinary air is used. Sulphide dusts are much denser than other explosible dusts, hence, a higher dispersion pressure (13 bar rather than 11 bar) and a shorter ignition delay time (30 ms rather than 100 ms) must be used to produce a reasonably homogeneous cloud. Use of different ignitor strengths allowed measurement of the minimum ignition energy as a function of particle size for pyrite (Fig. 8), showing the increase in energy required to initiate pyrite of larger sizes. Fig. 8 also shows that the minimum explosible concentration increases with particle size. The explosions are rather weak compared to other explosible dusts: the explosion pressure varies from 200 to 300 kPa, as the particle size decreases from 280 to 60 μm .

The mechanism of the reaction was explored by examining the solid products produced and by analysing the gas after the trials by infrared spectrophotometry. When the explosion is weaker (lower concentration of dust or less oxygen in the 'atmosphere'), Fe_2O_3 (haematite) is the predominant product. When the explosion is stronger, then Fe_3O_4 (magnetite) is produced. The reason is the thermodynamics of

the system: the net energy release is slightly greater for the haematite reaction, which means that the equilibrium shifts towards the magnetite reaction at higher temperatures, which are produced during stronger explosions. The quantity of SO₂ produced (measured by using the IR band at 9 μm) was approximately twice the quantity of oxygen consumed, which is consistent with simple oxidation. With lead sulphide, lead sulphate was formed predominantly; therefore, the amount of SO₂ produced was much less. When tested in pure oxygen, no SO₂ was produced, presumably because the excess oxygen favoured the sulphate production.

A particular sulphide ore dust from a mine produced a small overpressure at concentrations as low as 500 g/m³, but its lower explosible concentration was 3500 g/m³ (using 5000-J ignitors). Since this is much greater than for other dusts, it can be easily missed using standard techniques, yet is important because such a concentration and possible explosion can occur in the mine. An indication of a sample's possible explosibility is the observance of some overpressure at lower concentrations.

4. Conclusions

This work has shown that the characteristics of the dust dispersal system and the ignition system must be studied in detail for any particular apparatus before it is possible to generate accurate data. The methane–air model system was effective for this purpose. In particular, explosion pressures generated in 0.020 m³ vessels may be underestimated by 17%. (For hazard quantification studies in deep mines, the increased air pressure must also be taken into account.) The effects due to the pressure generated by pyrotechnic ignitors and the heat absorbance of the dust must also be properly taken into account.

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