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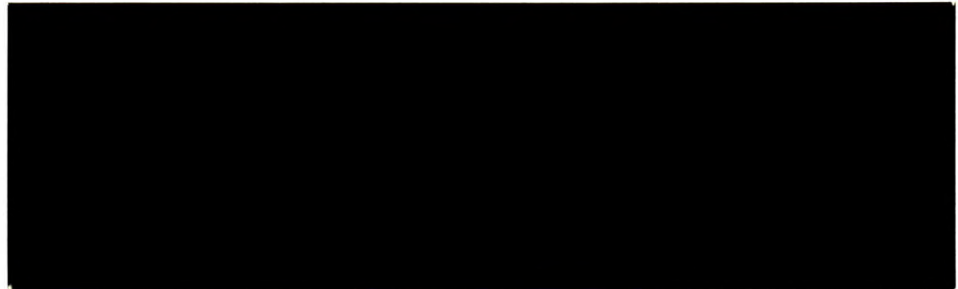
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**PROBLEMS IN LABORATORY MEASUREMENTS
OF DUST-AIR EXPLOSION PARAMETERS**

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by
K.J. Mintz*

ABSTRACT

Dust-air explosions can be serious safety hazards in chemical plants, grain elevators, mines and other locations. Explosion parameters to aid in identifying and remedying such hazards can be measured in laboratories. The various tests available at the Canadian Explosive Atmospheres Laboratory for servicing the needs of industry in this area will be described. Experiments have been carried out to determine the scale-up of results from laboratory measurements. Some dusts which are found to be "non-explosible" by certain standard methods are, in fact, capable of exploding. Some examples that have been studied in this laboratory are petroleum coke, fertilizer and sulphide ore dusts. The explosibility of a dust is well known to be a strong function of its particle size; the smaller the particle, the more explosible it is. However, a simple sieve analysis can be quite misleading and underestimate the explosibility, because particle adhesion and friability can be significant factors. The conclusion is that the explosibility of a dust should not be considered as a routine "black box" type of laboratory test, but should be carefully evaluated using both laboratory measurements and comparison with similar chemical species.

1001

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Dust/air explosions are generally recognized to be a serious potential safety hazard, not only in coal mines and grain elevators, but also in a wide variety of industries, such as pharmaceuticals, fertilizers, plastics, metal fabricating, etc. (1). Any plant that generates or handles dusts should be evaluated to determine if there is a potential dust explosion hazard and, if so, what mitigating actions should be carried out. Few plants never undergo change, therefore, it is wise to examine any changes that occur to determine the effect on explosion hazards. Nowadays, many plants are faced with the need to reduce the ambient dust concentration to satisfy various regulations promulgated to protect workers' health. In many cases, this is accomplished by installation of dust collectors. Unfortunately, this may involve trading a health problem for a safety problem, in that now, the dust is concentrated and confined in the dust collector. If there is an electrical spark or even an electrostatic discharge from the dust cloud itself an explosion may occur.

The first step in any hazard evaluation is obviously to determine if the dust in question is explosible, and if so, the required conditions for an explosion to occur. Clearly, it is impracticable to construct an actual plant and conduct the test there. However, some research groups have actually conducted full-scale explosion tests, for example, in a 500 m³ grain silo (2), and a 700 m long underground gallery (3). Other than the extremely high cost and difficult logistics in conducting such tests, it is difficult to vary the parameters sufficiently to study all possible conditions leading to an explosion. For these reasons, therefore, it is necessary to evaluate the explosibility of most dusts by means of laboratory-scale tests. This paper deals with some problems associated with the use of laboratory-scale tests for prediction of real-life hazards. Many chemical engineers are well aware of the difficulties in scaling-up a process from the laboratory to pilot plant to full-scale. For dust explosibility, an error in the hazard assessment may result in not just a loss of money, but also in injury or death. Having said that, it is my conviction that laboratory testing using up-to-date equipment and conducted and interpreted by capable people is sufficient to properly assess the explosion hazard of dusts.

The Canadian Explosive Atmospheres Laboratory conducts research into dust explosions as well as carrying out explosibility tests, on a cost-recovery basis, as a service to industry. The examples I will be discussing, come from studies carried out in this laboratory, during the past two years.

The properties of an explosible dust that can be of interest for a practical situation are shown on the first slide. The minimum explosible concentration is sometimes called the lower flammability limit, in analogy with gas explosions. The units usually used are g/m³, in contrast to those used for gas explosions which is often expressed in percent. The quantities required for explosions are much

higher than those required for deleterious health effects, which are usually in the mg/m^3 range. This parameter is usually not used for determining if a given plant is dangerous under normal conditions, but rather under abnormal conditions. For example, if all the dust on the floor of a factory were put into suspension, would the dust cloud be above the minimum explosible concentration? If so, then that factory should be regarded as hazardous.

The next two parameters measure the effects of an explosion, if one should occur. If it is decided to contain the explosion, then the structure must be designed to withstand the maximum explosion pressure. The maximum rate of pressure rise is used for the design of vents, if it is decided that it is impossible to exclude the possibility of explosions.

The minimum ignition temperatures of dust clouds and layers are useful for assessing the hazard of any hot surfaces that may be in the plant, whether under normal or abnormal conditions.

The minimum oxygen concentration is used for determining how much inert gas must be added to an area, if it is decided to prevent an explosion by inerting.

The minimum ignition energy has been used extensively in the past to decide on the maximum amount of electrical energy that is safe. It is usually expressed in mJ. This measurement, however, has fallen into some disfavour, because of the difficulty in measuring a "true" value. In addition, so many common sources of ignition are well above the range of energies available with electrical sparks, e.g. flames, welding torches, etc.

The classical apparatus for measuring most of these parameters has been the Hartmann apparatus [Slide 2]. Dust is placed in the tube and dispersed by an air blast, then ignited by an electrical spark. There are two versions of it, one made of steel and having a pressure transducer, used for measuring the maximum explosion pressure, and the other [slide 3], made of plastic, used for determining the minimum explosible concentration. Although the Hartmann is still the subject of an ASTM Standard and some testing laboratories still use it, it is regarded by experts in the field as being inadequate. For example, the most important dust explosion problem existent in Canada currently is that of metal sulphides in mines. If you take this material and test it all day in the Hartmann under the standard specified procedures, you will not obtain any explosion, not even any sign of reaction. Thus, a testing laboratory could then write a report saying that the dust is non-explosible, which could contradict the fact that it explodes frequently in a number of Canadian mines. The major cause of this discrepancy is the inadequate size of ignition source.

The leading laboratories now use an explosion vessel of 20-L or greater, and an ignitor of 5000 J. This size of vessel has been shown by research in Europe and the U.S. to correlate well with full-scale tests. The particular model we use is shown in the next slide [slide 4]. Dust is placed in a chamber below the vessel, a blast of air from a high pressure chamber [slide 5] disperses the dust and an ignitor is fired. The sequence of operations is controlled by a control panel designed and made in our laboratory [slide 6]. The pressure is measured by a pressure transducer, whose signal is fed into a conditioning unit, then into a storage digital oscilloscope. After the test, the gases in the vessel are pumped through a paramagnetic oxygen analyzer [slide 5], so that the percentage of oxygen reacted can be measured. An explosion test can be observed visually through the front window [slide 7].

Creation of a proper dust cloud in the vessel is essential. Each type of dust has different dispersibility characteristics; it is important to vary the dispersing pressure and the various timing events to optimize the dust cloud. Otherwise, there is a great risk of missing the explosion. For example, if you use the same conditions for sulphides as for coal, even using the 20-L vessel and a high-energy ignition, you will not get a proper explosion, the reason being the high density of the sulphide particles.

In the case of metal sulphides, the generation of the toxic gas SO_2 is at least as important as the blast effects from the explosion. Therefore, an infrared spectrophotometer was connected to the 20-L vessel and the gaseous contents after the explosion were expanded into the gas cell of the i.r.spectrophotometer. The concentrations of SO_2 were consistent with the mechanism proposed.

The Godbert-Greenwald furnace [slide 8] is used for measuring the minimum ignition temperature of dust clouds. A pulse of air passes through the sample chamber into the top of a furnace. If the furnace temperature is above the minimum ignition temperature, then a flame is emitted from the bottom of the furnace.

An image analyzer with an optical microscope [slide 9] is used to measure the diameters of the particles as well as examine the shapes of them. We have also examined the residues from explosion tests using a scanning electron microscope [slide 10]. After a strong explosion on pyrite (iron sulphide), we obtained mainly spherical particles, which were composed of iron oxide.

A typical experimental trace is shown on the next slide [slide 11]. Unlike other researchers, we evacuate the vessel completely before the test, so that all the air can be used to disperse the dust, plus ensuring that there are no contaminants in the vessel. Thus, the ordinate refers to absolute pressure. The pressure is brought up to about 1 atmosphere by the dispersing air, then the dust is

ignited. In this case, a continuous electric discharge was used. The actual explosion pressure is the peak value minus the pressure at the time of ignition. The derivative curve is generated so that the maximum rate of pressure rise can be determined.

When a chemical ignitor is used, it produces a significant pressure by itself. In the next slide [slide 12], a portion of a typical pressure trace is shown. In this case, atmospheric pressure has already been subtracted off. The initial shoulder is due to the ignitor. The procedure adopted by us is to subtract off the ignitor pressure trace to produce a corrected pressure trace. All results presented here are from this net pressure trace. Not everyone corrects for the ignitor pressure; that can cause some confusion.

If the explosion pressure is plotted against the concentration, typically a S-shaped curve is obtained [Slide 13]. At the low end, burning around the ignitor occurs, but it is insufficient to propagate the explosion throughout the dust cloud. But where does one draw the line between non-explosibility and explosibility? In the next slide [14], the abscissa is expanded at the low concentration end. Lee (4) has proposed that the limit should be the point at which the slope of the pressure-concentration curve changes, as indicated in the graph. Hertzberg (5) has proposed that two conditions should be satisfied simultaneously: (a) the pressure should be twice the original pressure, which in our case means that the overpressure should be 100 kPa, or 1 atmosphere, and (b) the K_{st}^1 value be 1.5 bar-m/s, which in our system means that the rate of pressure rise should be 560 kPa/s. The Hertzberg criterion yields a higher value of limit concentration than that of Lee. Throughout this paper, we will be using the Hertzberg criterion.

The majority of dusts are most explosible in the range of 500 - 1000 g/m³. A certain metal sulphide sample was sent to three laboratories for testing. We were the only one that found it to be explosible. The cause for the discrepancy can be seen in the slide [15]. The other laboratories tested up to only 2000 g/m³, at which concentration, the dust is not explosible. (Recall that the overpressure must be at least 100 kPa to have a true explosion.) In fact, this dust is not explosible until nearly 4000 g/m³. However, the fact that there was some reaction at lower concentrations should have indicated the possibility of explosions at higher concentrations. Another sample had no reaction at concentrations of 1000 g/m³, which indicated that the sample was truly non-explosible. Tests up to 5000 g/m³, which yielded no reaction or explosion, confirmed this expectation.

A respected reference (6) states that "low-volatile [petroleum] cokes ... do not present a dust

¹ $K_{st} = (dP/dt)_{\max} \cdot V^{1/3}$, where V is the volume of the vessel.

explosion hazard in industry under ordinary conditions". This conclusion was based on tests carried out in the Hartmann apparatus. We have carried out tests in the 20-L vessel on a coke containing 12% volatiles which produced full explosions, even at fairly low concentrations [slide 16]. The slide also shows that the minimum explosible concentration is decreased by drying the sample and, particularly, using only the finer fraction of the sample. An electrical spark did not ignite these samples in the 20-L vessel; only the use of the high-energy ignitor uncovered the explosibility of petroleum coke. This work was sparked by an explosion that occurred with the use of this dust.

Much research has been carried out on the effect of particle size on explosibility. It is generally agreed that particles of diameter above 0.5 mm are non-explosible. We have carried out tests on a sample that was screened by the supplier to be between 1.6 and 9.5 mm in diameter. Therefore, this product should not be explosible. The experimental results, however, show that it is explosible [slide 17]. The reason for this is that the dust that exploded was not the same as the dust placed in the vessel. The blast of air used to disperse the dust was strong enough to break up the particles, thus making them small enough that they would be explosible. The object of the lesson is clear. Friable dusts should not be tested as is, but rather they should be subjected to the same sort of treatment that they actually receive in the plant, for example, bags being dropped on the floor, loose material falling down chutes, etc.

In addition to the problem of friability, we have obtained some evidence that some large particles can contribute to the explosion. The reason for large particles generally being non-explosible is their small surface area to volume ratio relative to small particles. However, if the large particles happen to be very porous or fractured, they would have a much larger surface area than calculated on the basis of their diameter. It is prudent to use a microscope to examine the dust being tested for evidence of unusual particle shapes.

The parent material can sometimes yield misleading information on the explosibility of the dust. Ammonium nitrate is widely used as a fertilizer. Because of its tendency to cake, it is usually coated with a small amount of some material, often organic. Ammonium nitrate dust by itself is non-explosible. If the test dust contains the same proportion of the organic dust as is in the product, it is still non-explosible. However, the dust in the plant, especially in the dust collection system, is much more likely to be heavily enriched in the coating agent, and thus be explosible. Therefore, it is most important to evaluate the explosibility of the dust that is actually being generated.

I do not want to leave you with the impression that we make everything explode. Aluminum dust is known to be capable of strong explosions and vigorous fires. A fabricating plant sent a sample

of their aluminum dust for evaluation. Even after varying all the parameters, we could not obtain any explosion or even any evidence of reaction in the 20-L vessel. The minimum ignition temperature of a dust cloud of this material was found to be 700 °C. A dust layer of the material did not show any sign of exothermic behaviour up to the maximum temperature of 720 °C. The primary reason for the inertness is the coarseness of the particles; 99% by weight did not pass a 300 μ sieve. In addition, the plant was using a sticky coolant which coated the particles, preventing dispersion of the finest particles as well as possibly inerting the surfaces of the particles. Possibly if the coolant were changed to something less sticky or more volatile, the explosibility and flammability characteristics of the dust would increase.

In this paper, I have given a number of examples taken from our work, where we have obtained unexpected results. They can all be understood by going back to the fundamentals of explosions. I trust that I have made it clear that explosibility testing should not be considered as a "black box", but that the testing program for a dust should be carefully planned, taking into consideration the particular operation for which the tests are to be performed. Only in that way can reliable results for hazard analysis be obtained.

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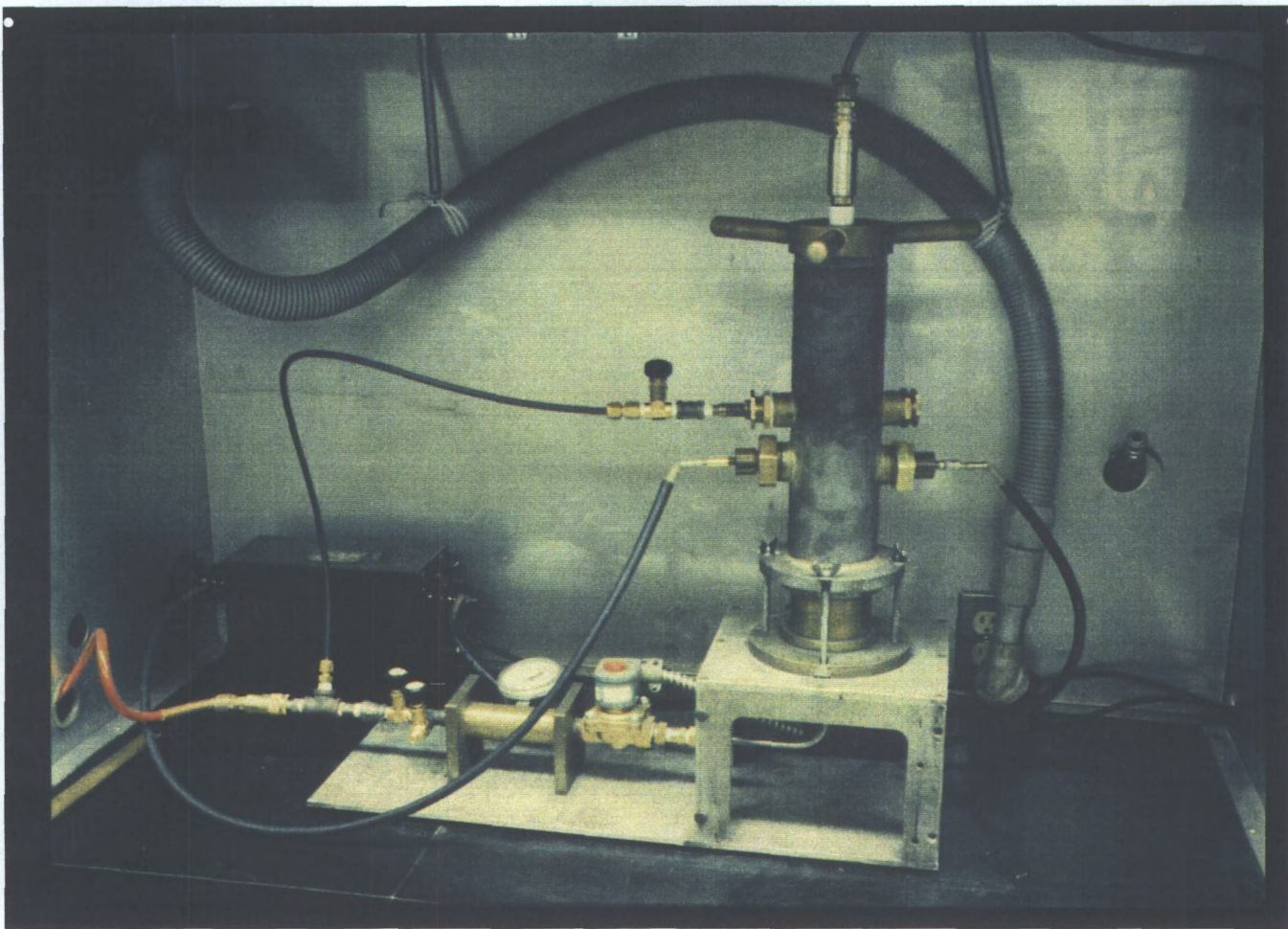


Fig. 2. Hartmann apparatus for explosion pressure tests

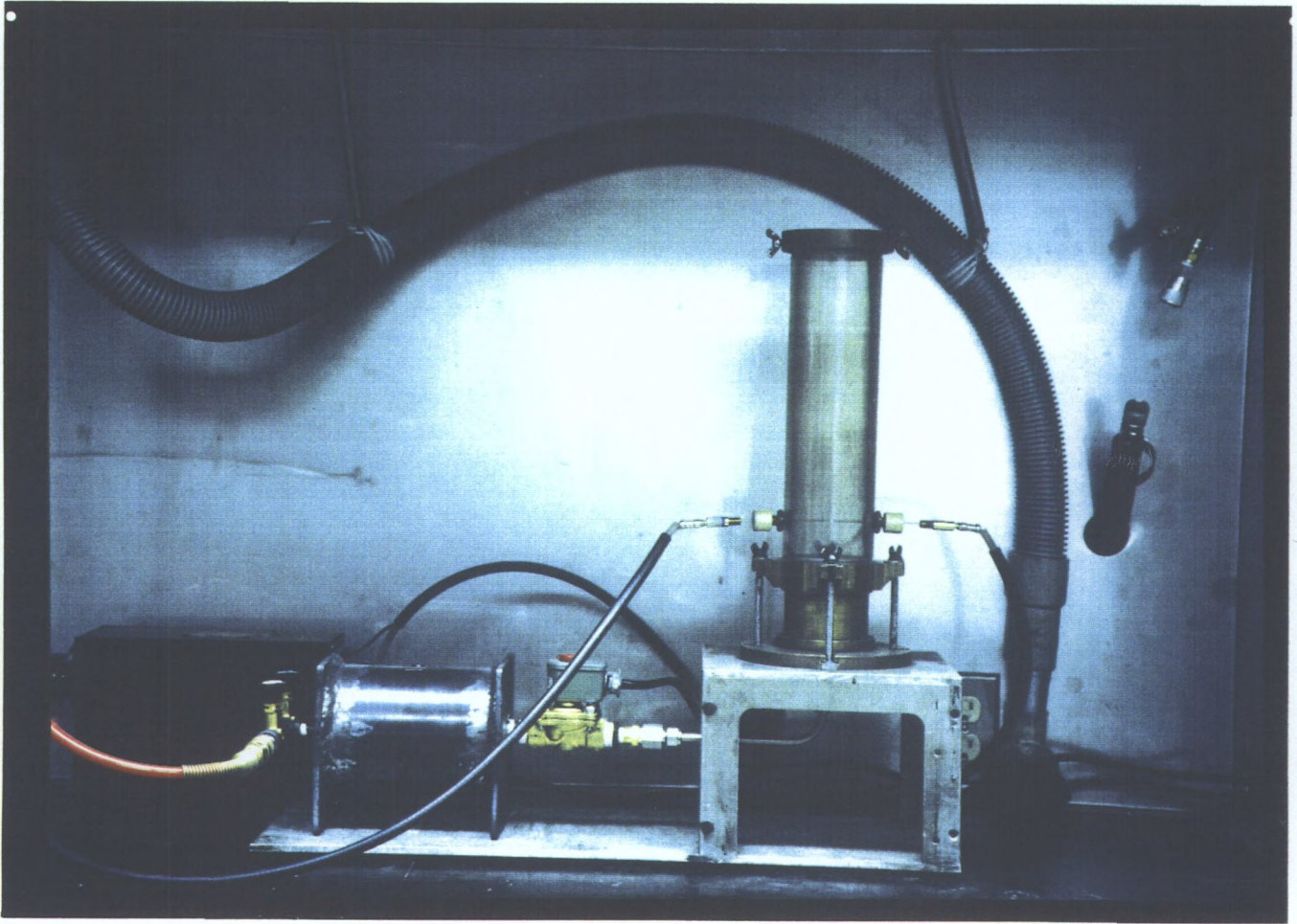


Fig. 3. Hartmann apparatus for minimum explosible concentration tests

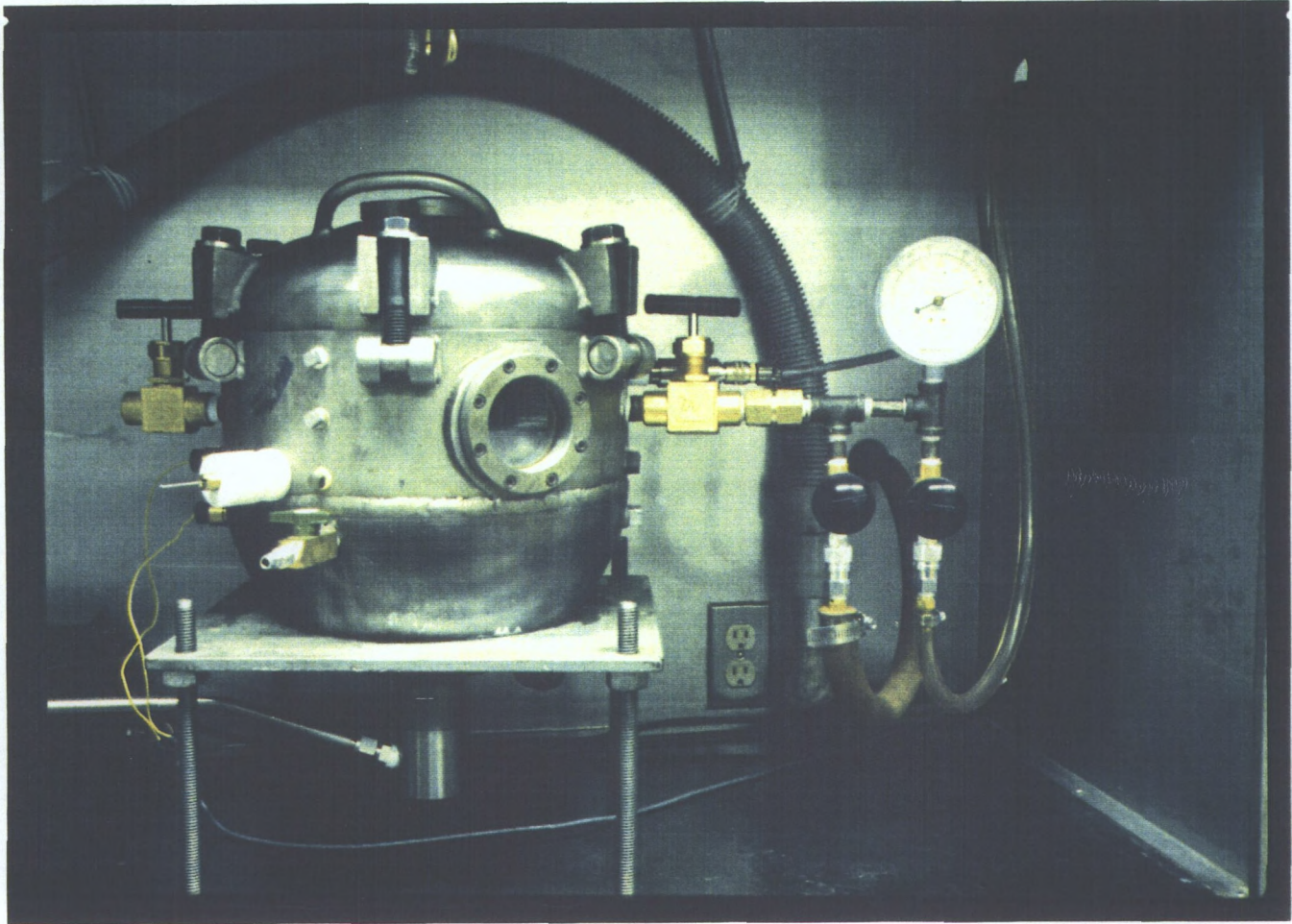


Fig. 4. 20-L Vessel for dust explosion testing

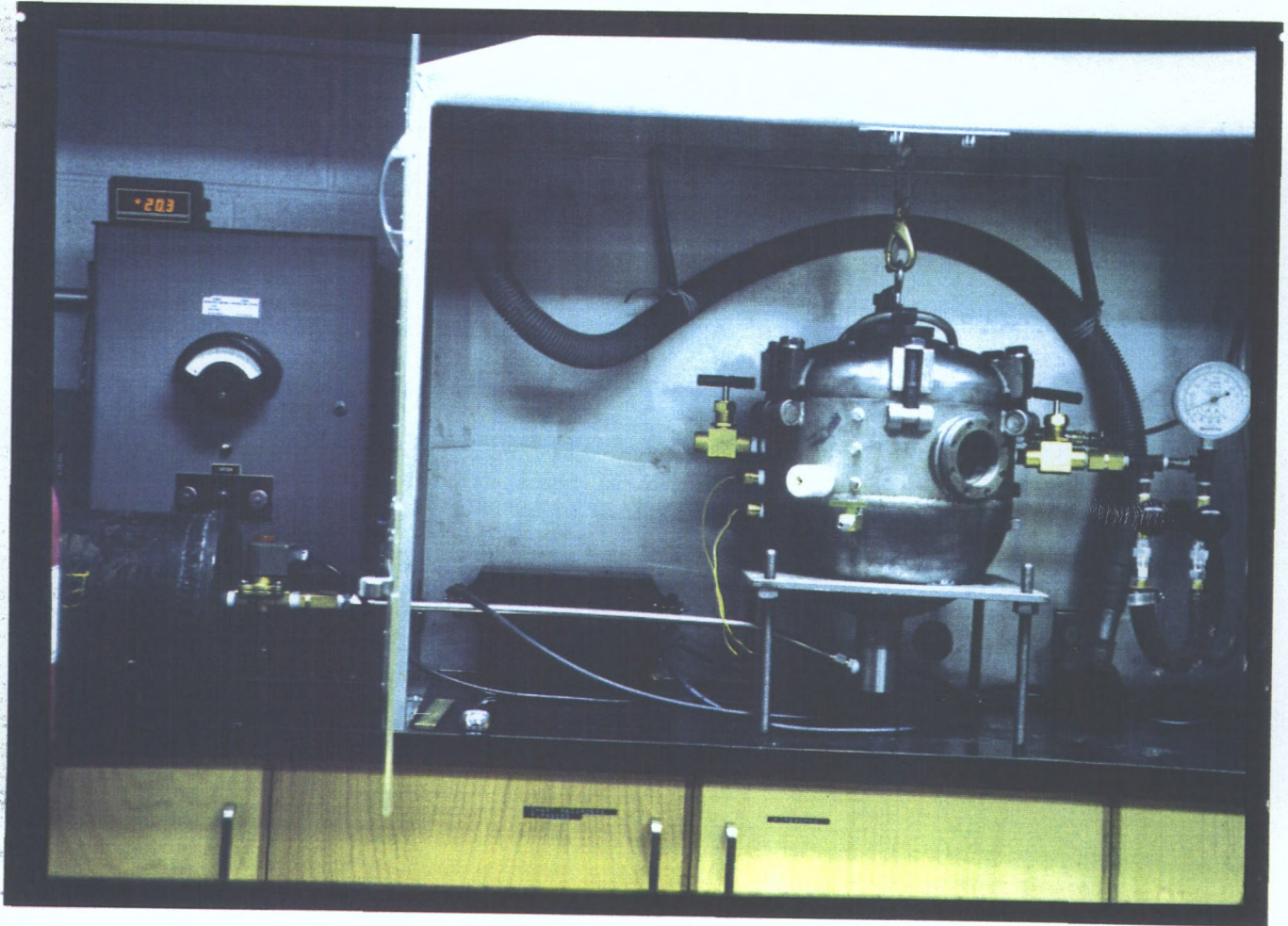


Fig. 5. Air chamber and oxygen analyzer on left side of 20-L vessel

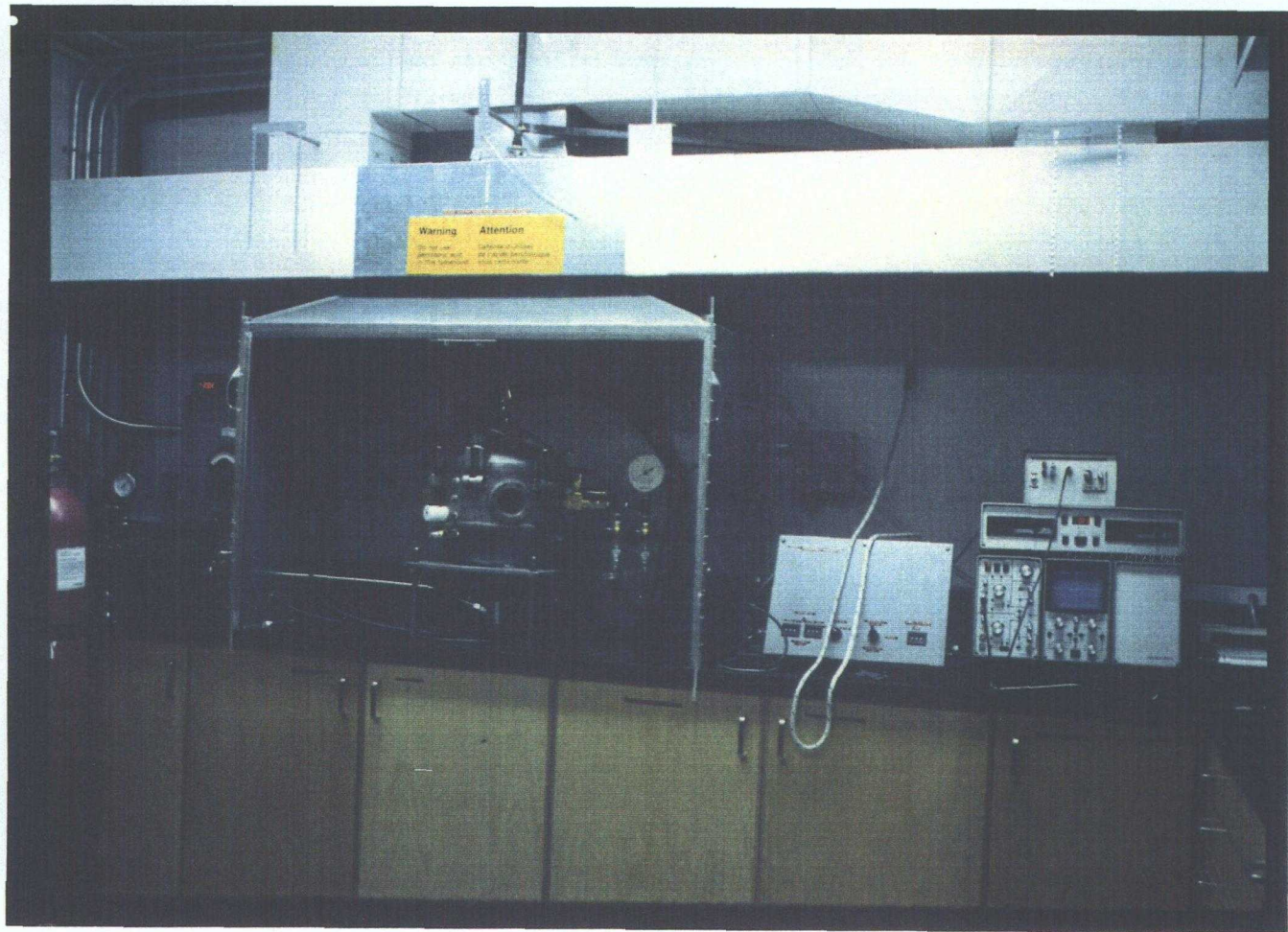


Fig. 6. Control box and digital storage oscilloscope on right side of 20-L vessel

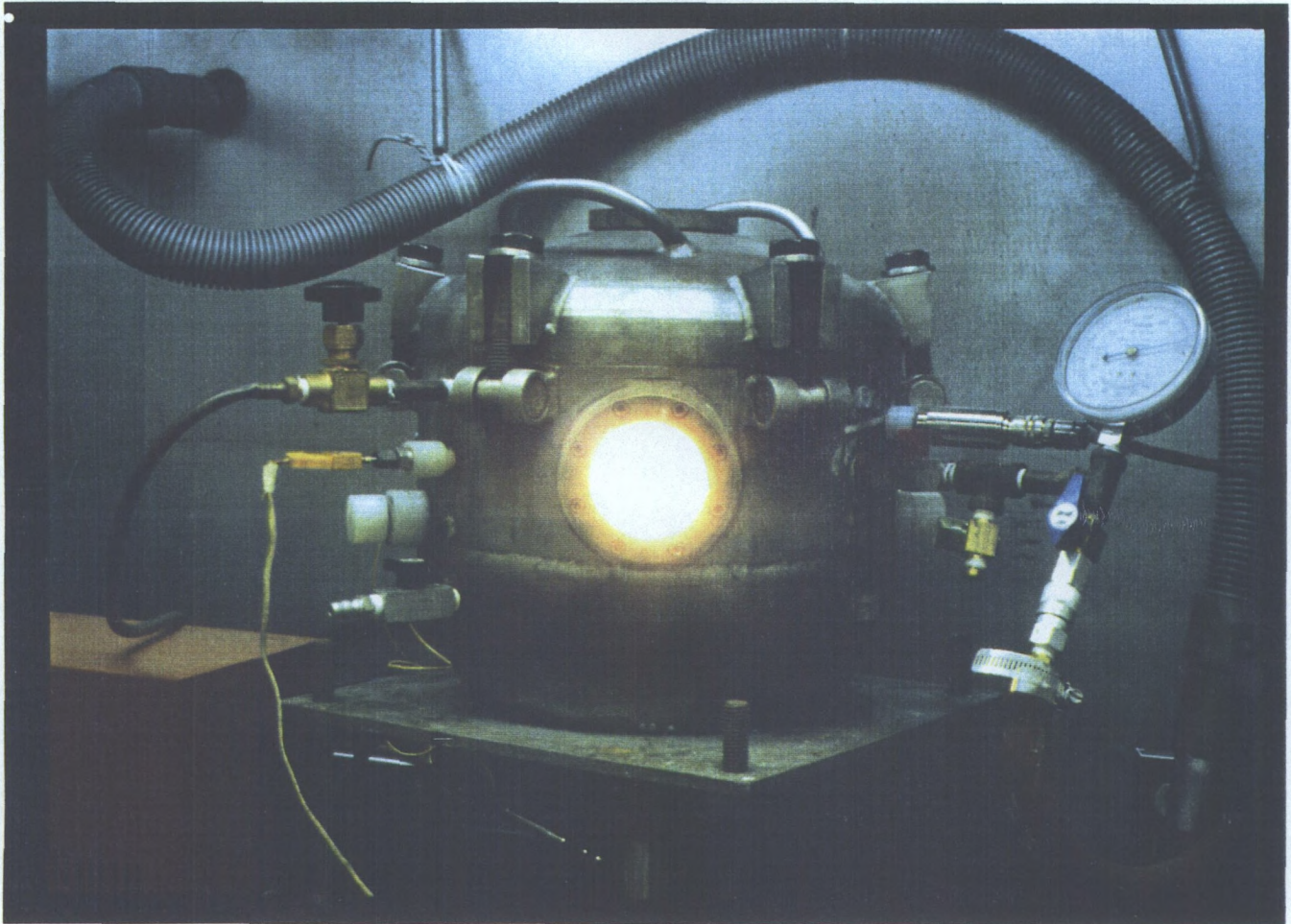


Fig. 7. An explosion test in the 20-L vessel

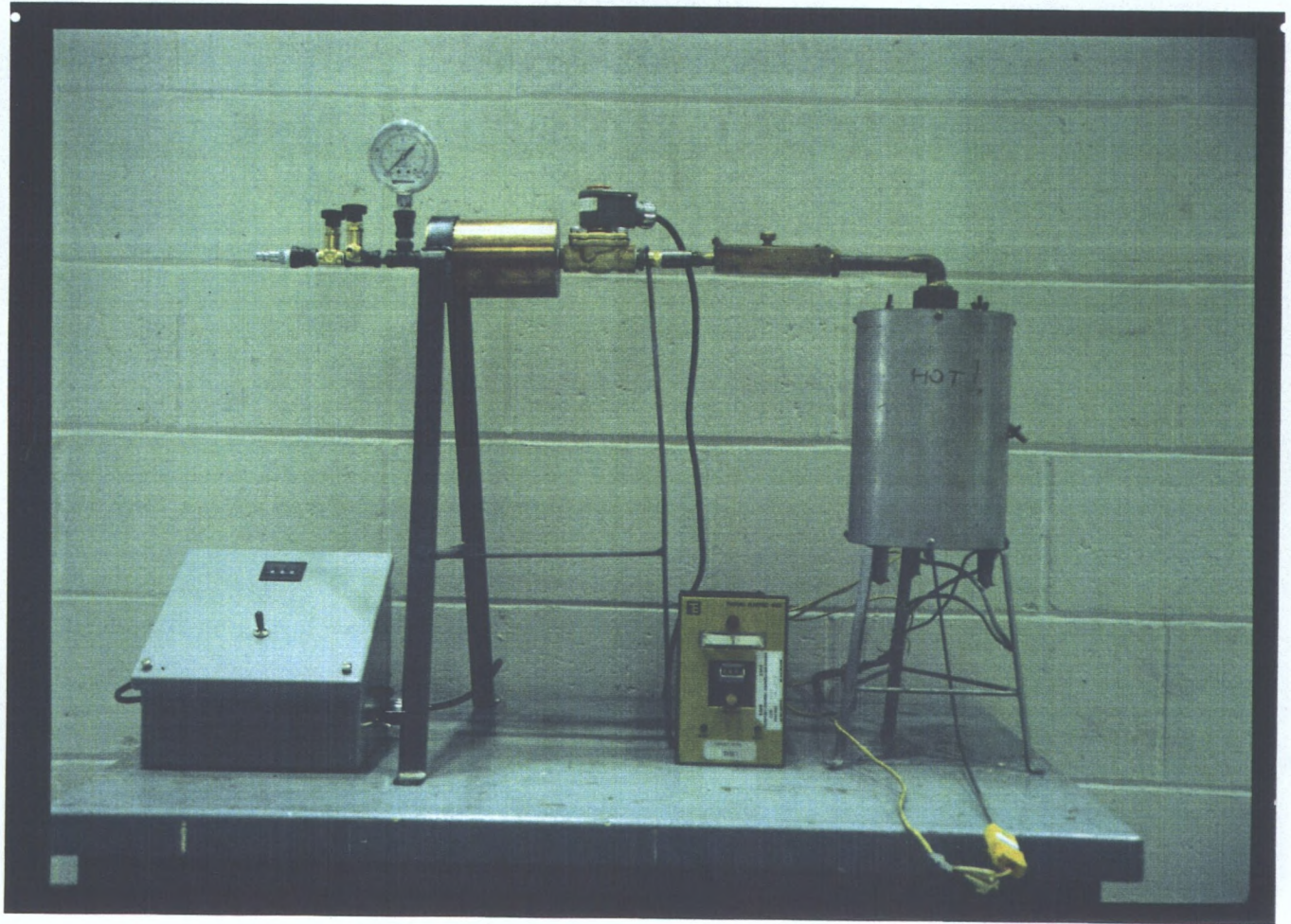


Fig. 8. Minimum ignition temperature apparatus for dust clouds
(the Godbert-Greenwald furnace)



Fig. 9. Image analysis equipment

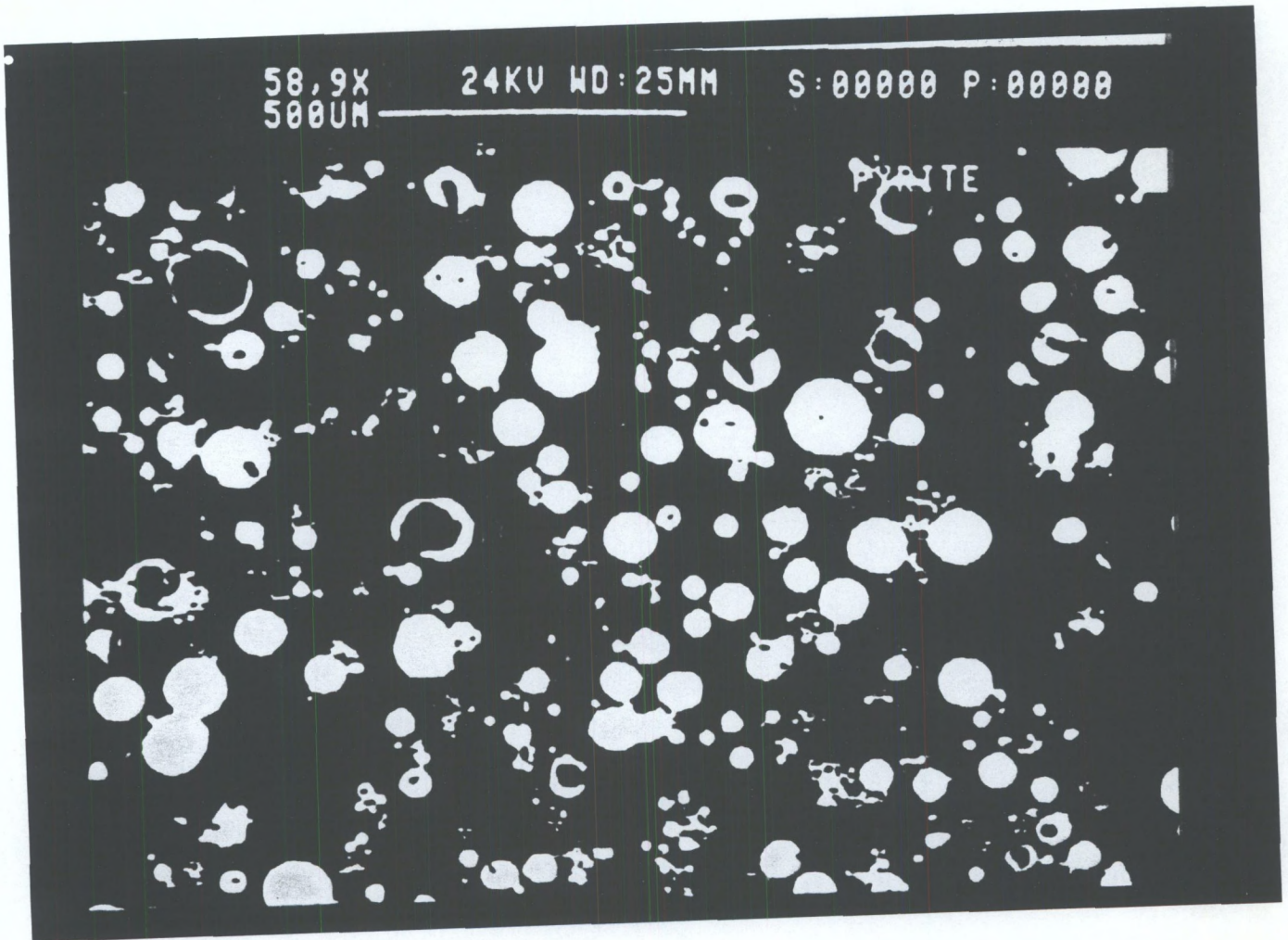


Fig. 10. Spherical particles resulting from an explosion test on pyrite dust

DUST EXPLOSIBILITY PARAMETERS

Minimum explosible concentration (lower flammability limit) g/m^3

Maximum explosion pressure kPa or bar

Maximum rate of pressure rise kPa/s

Minimum ignition temperature of dust clouds $^{\circ}\text{C}$

Minimum ignition temperature of dust layers $^{\circ}\text{C}$

Minimum oxygen concentration $\% \text{O}_2$

Minimum ignition energy J

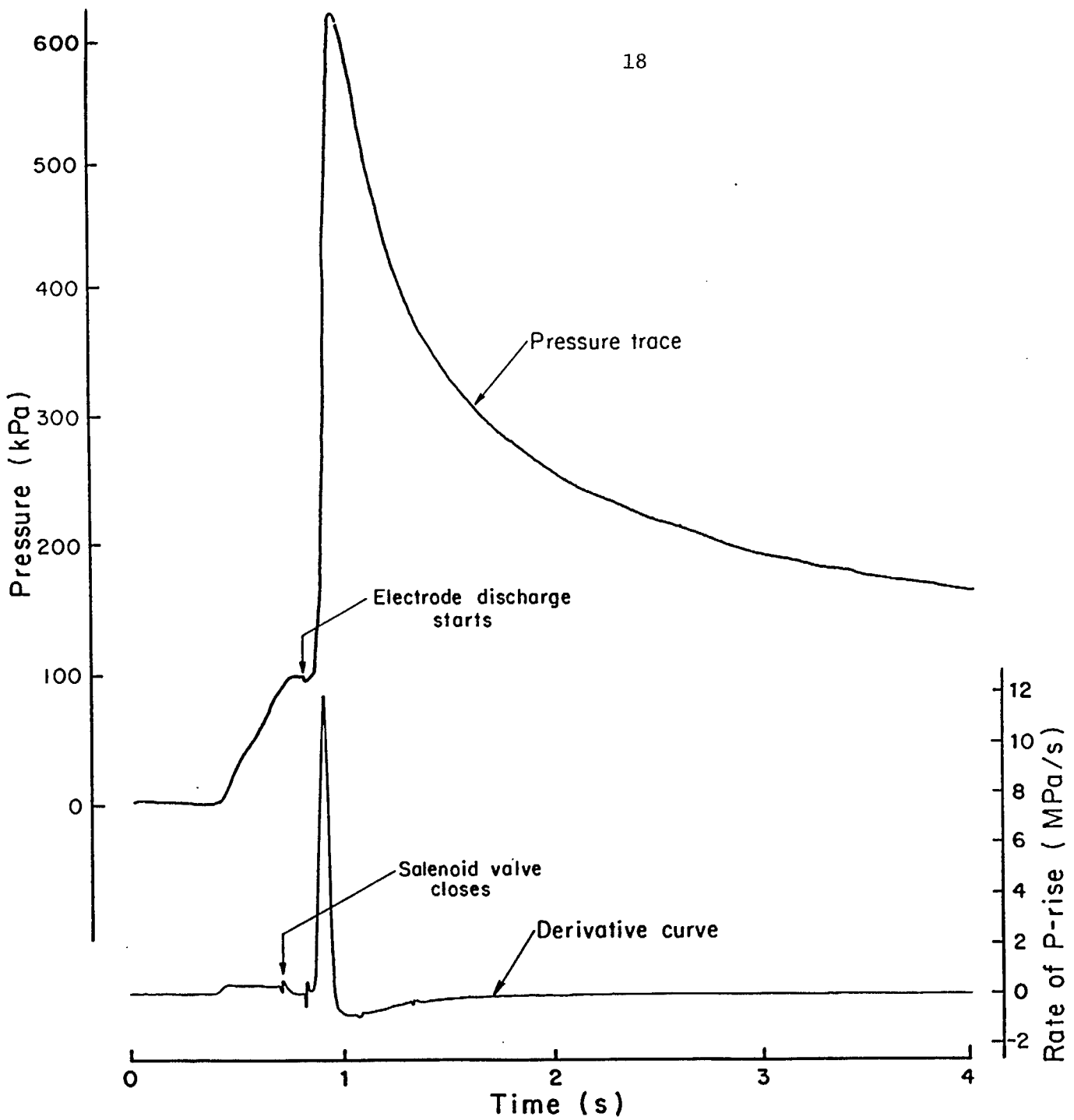


Fig. 11. Pressure and rate of pressure rise for a typical dust explosion test in the 20-L vessel, using an electric spark ignition

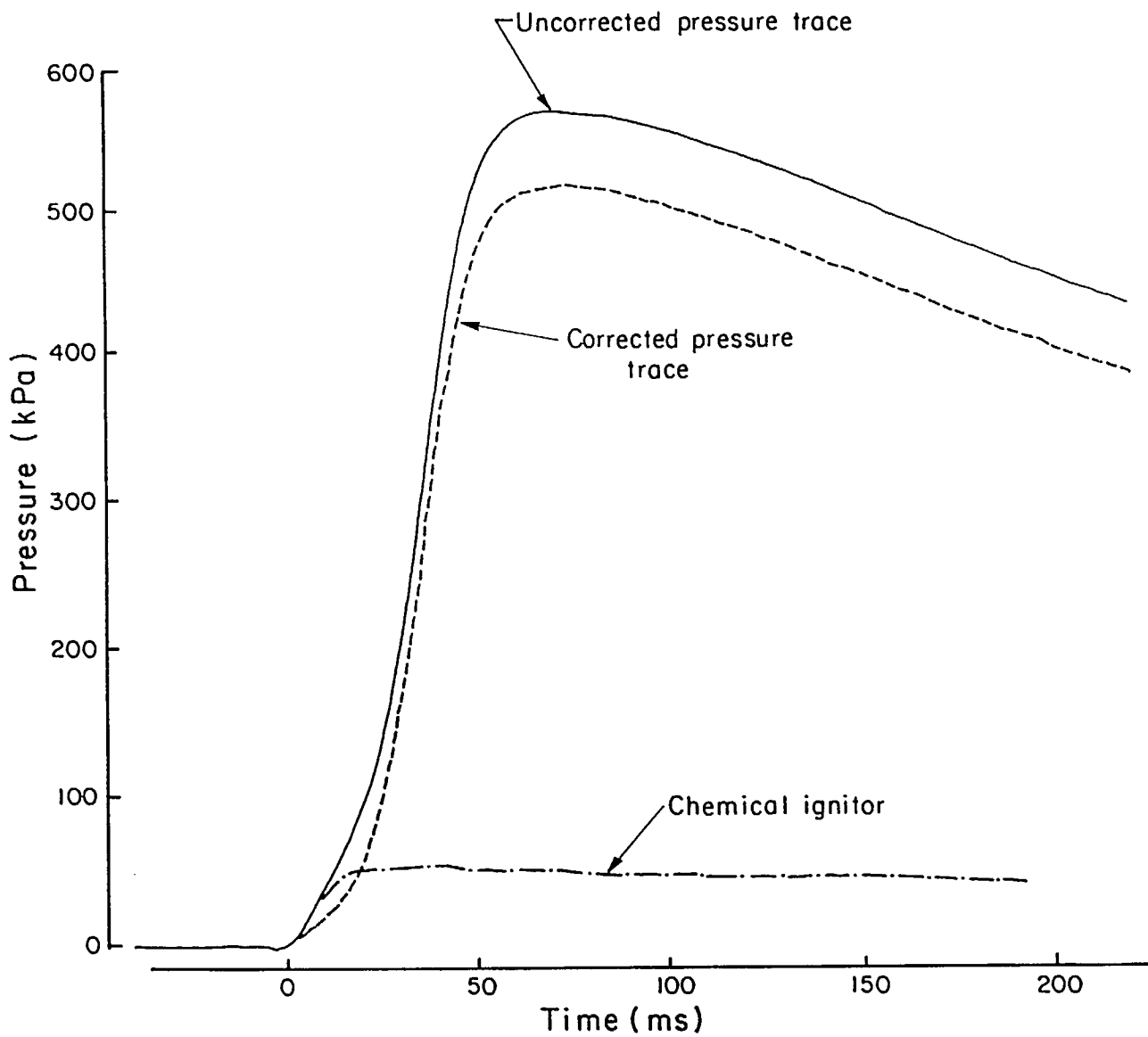


Fig. 12. A portion of the pressure trace for a typical dust explosion test, using a Sobbe (pyrotechnic) ignitor, plus the pressure trace for the Sobbe ignitor by itself

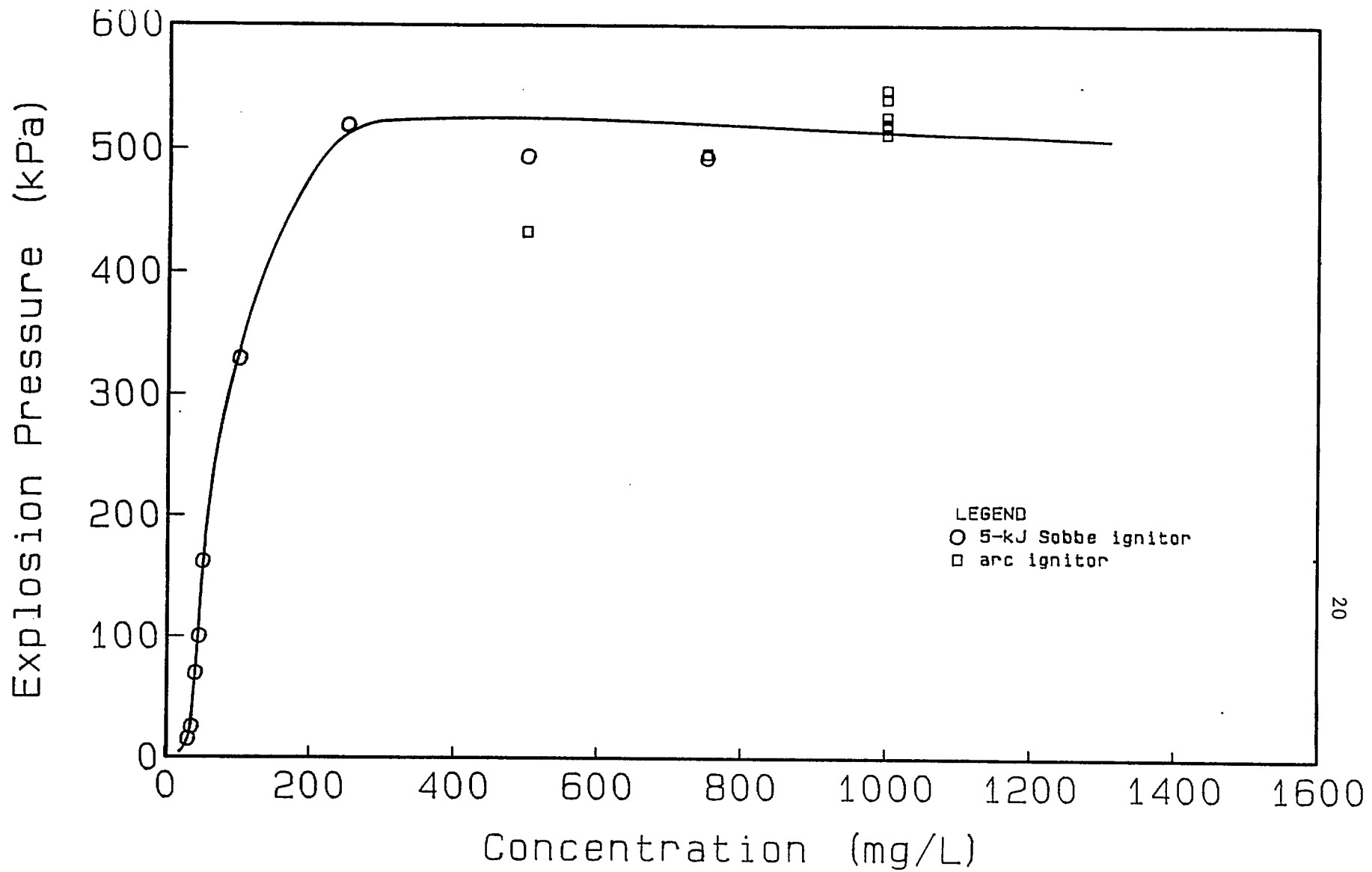


Fig. 13. Explosion pressure as a function of concentration, for Lingar. (Cape Breton Development Corporation) coal dust

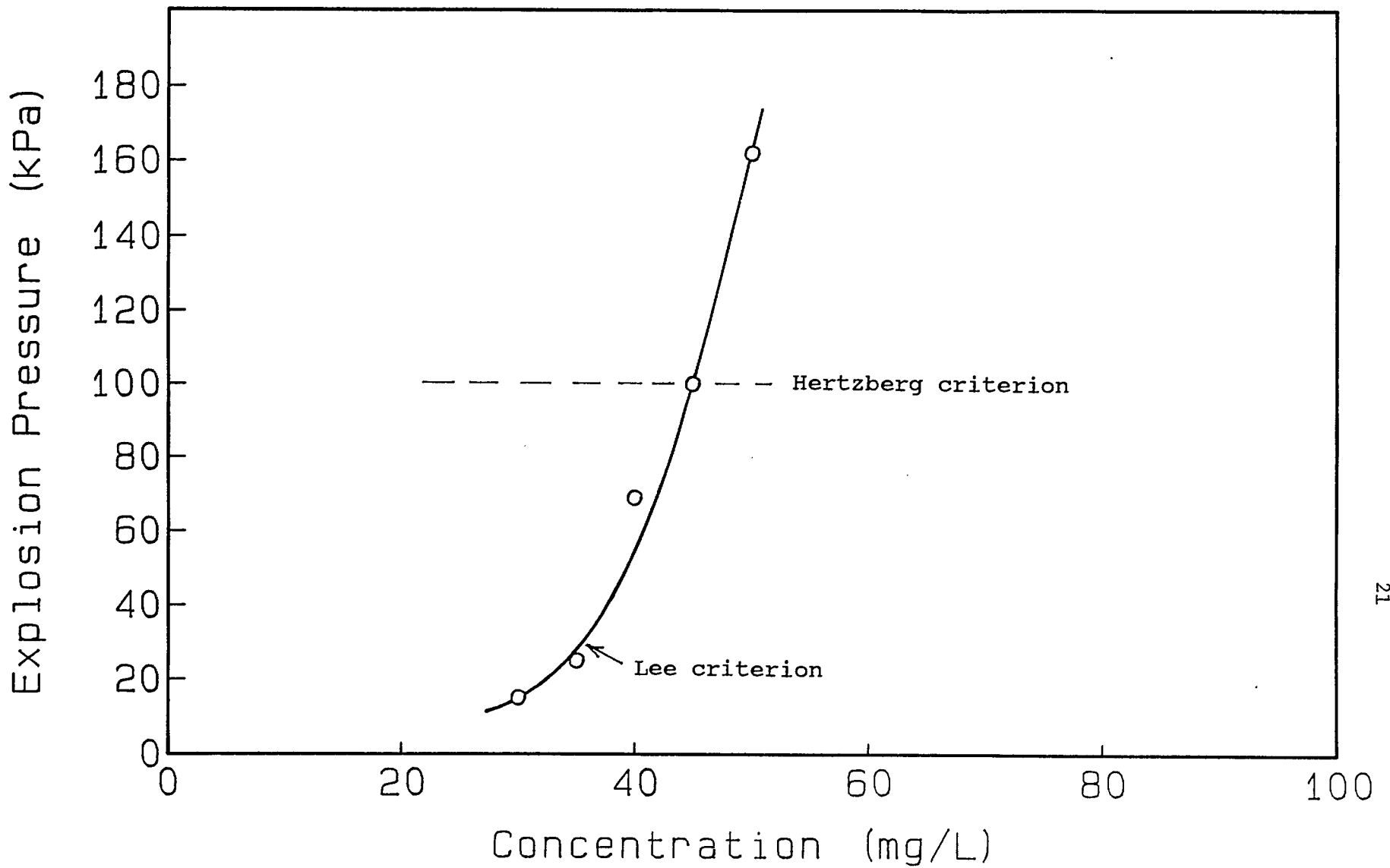


Fig. 14. An expanded portion of Fig. 13, showing the difference between two criteria for determining the minimum explosible concentration

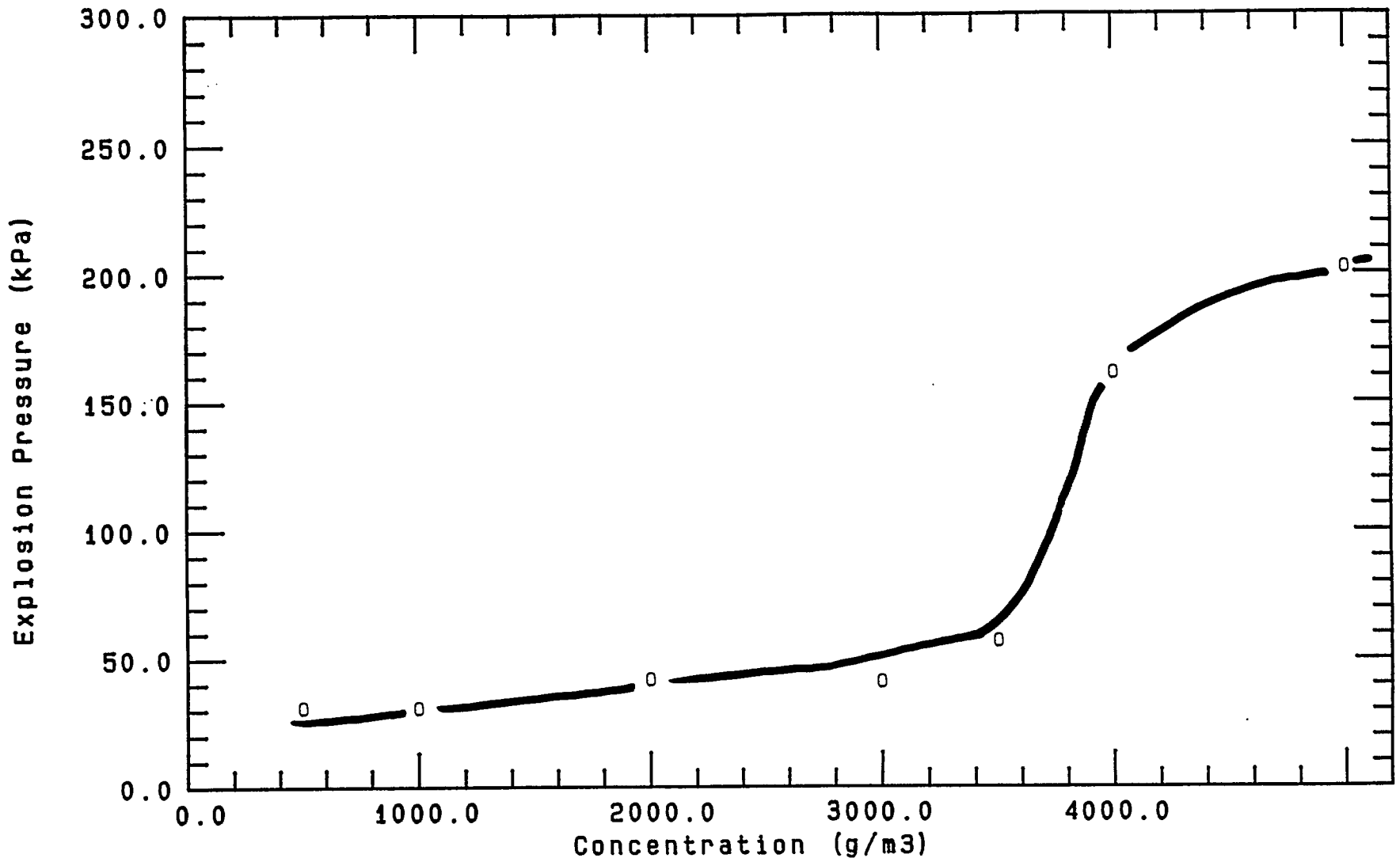


Fig. 15. Explosion pressure as a function of concentration for a weakly explosible sulphide ore dust

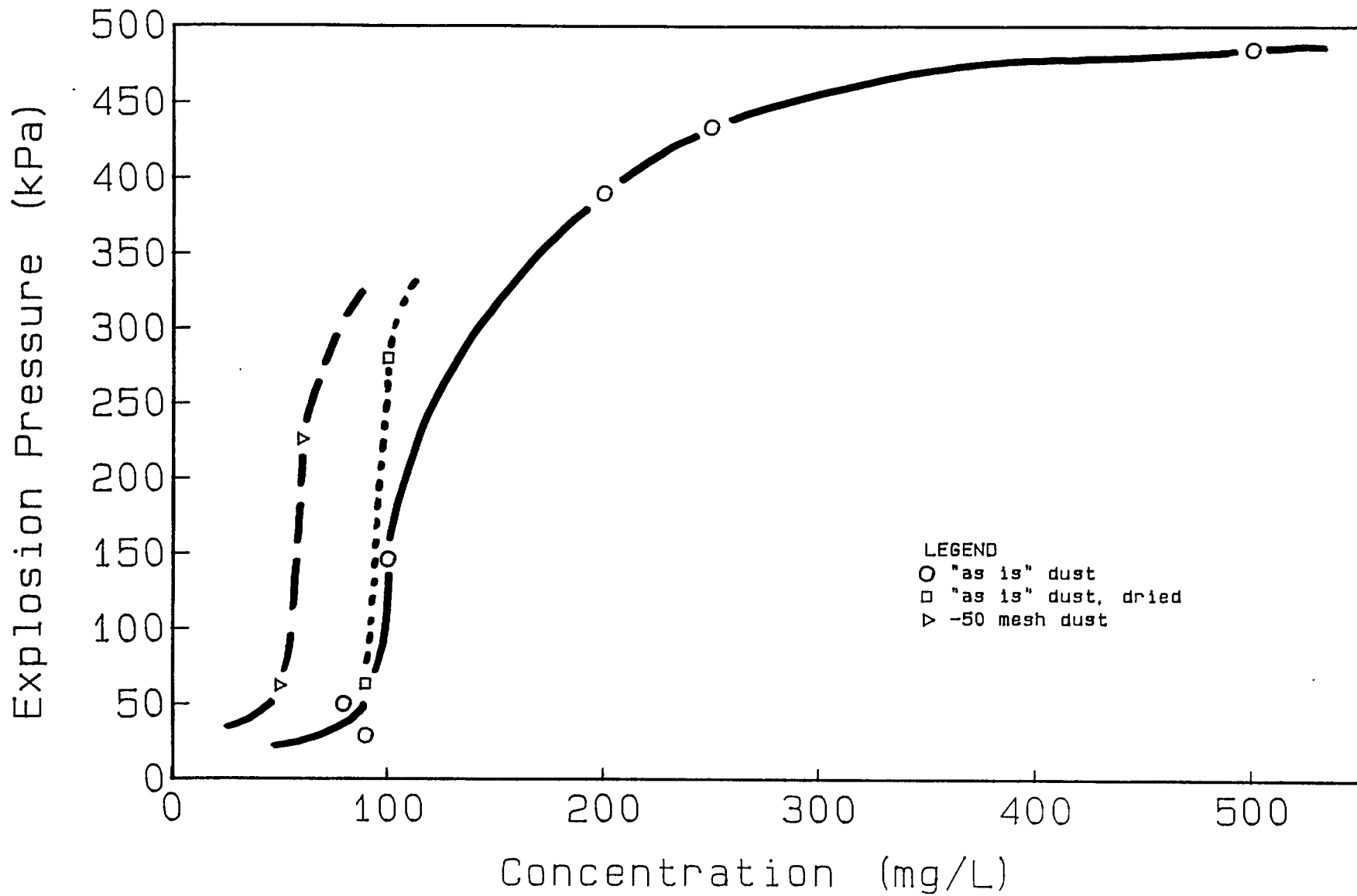


Fig. 16. Explosion pressure as a function of concentration for a petroleum coke

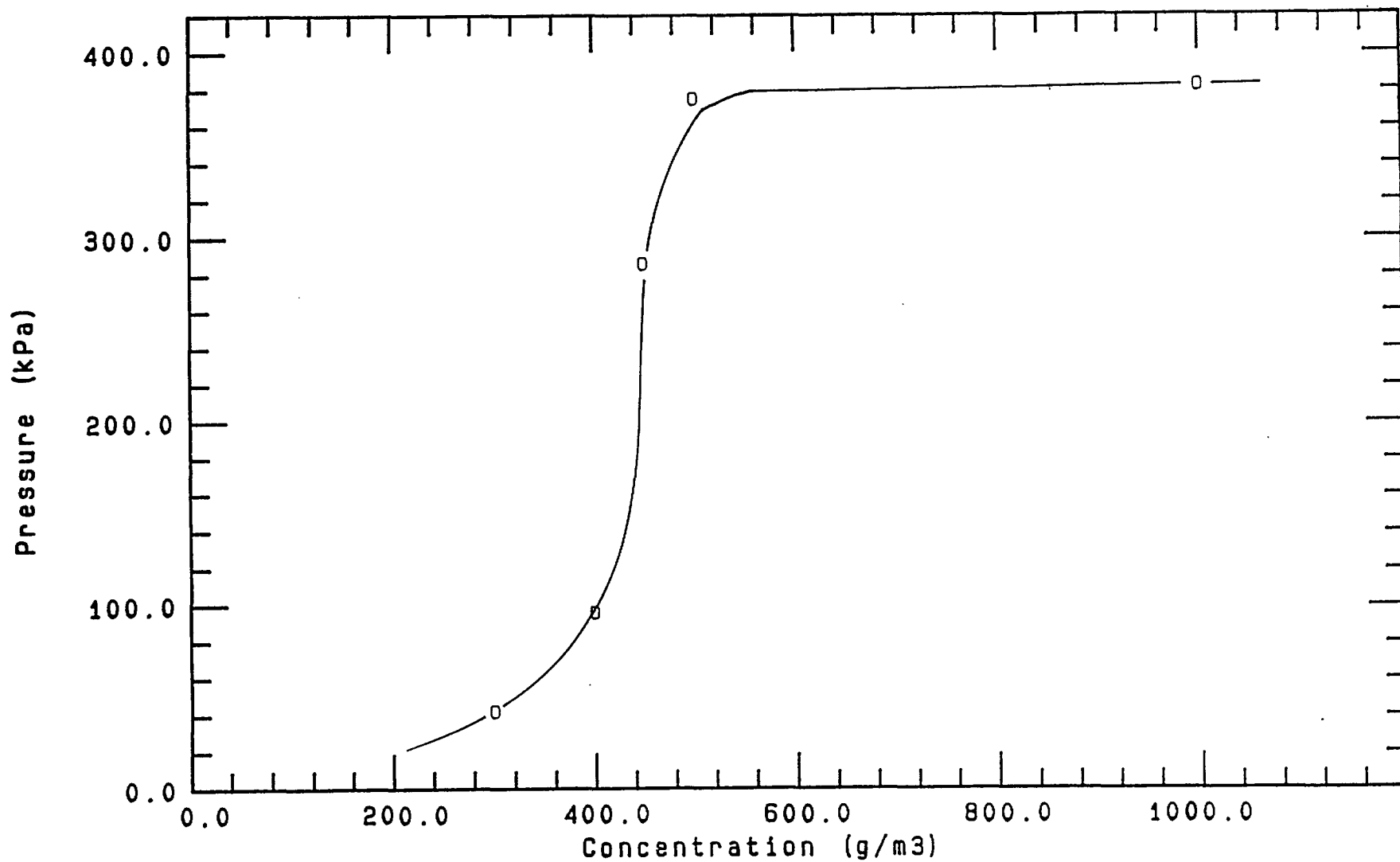


Fig. 17. Explosion pressure as a function of concentration for a coarse particle size of a petroleum coke

