Flammability limit measurements for dusts in 20-L and 1-m^3 vessels

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Abstract

Two types of flammability limits have been measured for various dusts in the Fike 1-m^3 (1000-L) chamber and in the Pittsburgh Research Laboratory (PRL) 20-L chamber. The first limit is the minimum explosible concentration (MEC), which was measured at several ignition energies. In addition to the three dusts studied previously (bituminous coal, anthracite coal, and gilsonite), this work continues the effort by adding three additional dusts: RoRo93, lycopodium, and iron powder. These materials were chosen to extend the testing to non-coal materials as well as to a metallic dust. The new MEC data corroborate the previous observations that very strong ignitors can overdrive the ignition in the smaller 20-L chamber. Recommendations are given in regard to appropriate ignition energies to be used in the two chambers. The study also considered the other limiting component, oxygen. Limiting oxygen concentration (LOC) testing was performed in the same 20-L and 1-m^3 vessels for gilsonite, bituminous coal, RoRo93, and aluminum dusts. The objective was to establish the protocol for testing at different volumes. A limited investigation was made into overdriving in the 20-L vessel. The LOC results tended to show slightly lower results for the smaller test volume. The results indicated that overdriving could occur and that ignition energies of 2.5 kJ in the 20-L vessel would yield comparable results to those in the 1-m^3 vessel using 10.0 kJ. The studies also illustrate the importance of dust concentration on LOC determinations. © 2000 Elsevier Science Ltd. All rights reserved.

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1. Introduction

Many explosibility measurements are needed for safety or hazard analyses. These include the basic explosibility parameters, maximum pressure and rate of pressure rise, as well as explosibility limit parameters such as fuel concentration, oxidant concentration, and ignition energy. The fuel concentration limit, often referred to as Minimum Explosible Concentration, MEC (or lean flammable limit, LFL) is the lowest concentration of dust dispersed in air that can propagate an explosion. Today, most MEC measurements are made in either a 20-L vessel or a 1-m^3 (1000-L) vessel. The 20-L vessel is considerably more convenient to use; the 1-m^3 vessel is expected to produce data that are more representative of industrial scale explosions. Another limit measurement of use in hazard analysis is the Limiting Oxygen Concentration (LOC) or Minimum Oxygen Concentration (MOC). The LOC, which is the term used in this paper, is the oxygen concentration at the boundary between propagation and nonpropagation of the dispersed dust cloud. LOC data are used, along with an appropriate safety factor, to establish safe inerting levels in industrial processes.

Since the MEC and LOC values are experimentally determined in the laboratory, one of the on-going concerns is that of overdriving the system by a large ignition source. This is of particular concern with the smaller 20-L vessel. Dust clouds are inherently more difficult to ignite than gases and therefore stronger ignition sources are used in testing. A “true” limit measurement should be independent of ignition energy. When the ignition source is too weak, both the measured MEC and LOC will be higher than the true value. The system is underdriven and the results are based more on ignitability than flammability. In theory, the ignition energy is increased until the limit measurements are independent of energy. At some point, however, the energy level is excessive for the size of the vessel and the system is "overdriven". In this situation, the energy contributed by the ignitor is sufficient to combust enough dust so that the result appears to be an explosion although there is no real propagation beyond the ignitor flame. Similarly, an overly strong ignitor can markedly change the initial test conditions by raising the overall temperature of the system, which in turn would lower the apparent limits and a nonexplosible system would appear to be explosible. A plot of ignition energy versus the measured limit would ideally have a vertical asymptote where the limit is independent of energy. For most dusts, however, this is not the case, particularly in the 20-L vessel.

Comparison measurement between the 20-L and 1-m^3 vessels can be used to evaluate the overdriving effect. Overdriving is generally unlikely to occur in the 1-m^3 vessel and in principle the 1-m^3 vessel can be used to establish the energy independent limit value. Such comparisons of vessel size have been made by Hertzberg, Cashdollar and Zlochower (1988) comparing 20-L vs. 120-L limit data for gases, by Cashdollar and Chatrathi (1992), comparing 20-L vs. 1-m^3 MEC data for dusts, and by Bartknecht (1989) and Siwek (1988), comparing 20-L vs. 1-m^3 data for dusts. Bartknecht (1989) did not report the effect of different energy sources in the 20-L vessel, but did note that the LOC results from a 10-kJ ignitor in a 20-L vessel were ~1.6 times lower than those found in a 1-m^3 vessel with the same ignition energy. This difference was attributed to the energy of the ignition source affecting the entire vessel volume and not acting like a point source. Siwek (1988) extended this work by considering the effect of various lower energy ignitors in the 20-L vessel. He noted that the 10 kJ ignitor gave much higher LOCs in the 1-m^3 vessel in comparison with those found in the 20-L vessel. Using an ignition energy of 2.5 to 1 kJ in the 20-L vessel brought its LOC into better agreement with the 1-m^3 values. Siwek also compared the MEC values for 16 dusts measured with 10 kJ ignition in both 20-L and 1-m^3 vessels. The issue of overdriving in a 20-L vessel and its relation to ignition source energy for MEC testing has also been discussed by Chawla, Amyotte and Pegg (1996).

The current testing was performed in a 20-L chamber at the NIOSH Pittsburgh Research Laboratory (PRL),[1] located near Pittsburgh, PA and in the 1-m^3 vessel located at Fike Corporation in Blue Springs, MO. Both vessels have been used extensively for dust and gas testing. This paper reports on the new comparative MEC tests for RoRo93 (a tetramethylpiperidine derivative used for a round robin test in 1993), lycopodium, and iron powder and comparative LOC tests for gilsonite, Pittsburgh coal, RoRo93, and aluminum powder. Limits were determined versus energy levels with the goal of establishing the appropriate 20-L ignition energy that yields data equivalent to 1-m^3 data.

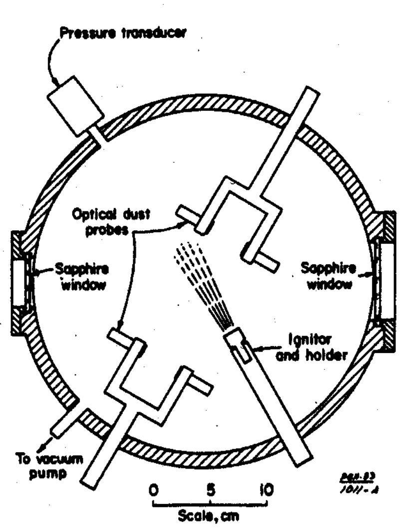

Fig. 1. Vertical cross-section of Pittsburgh Research Laboratory 20-L chamber.

2. Experimental

The 20-L dust explosibility data were obtained in the PRL 20-L laboratory chamber (Cashdollar & Hertzberg, 1985) shown in Figs. 1 and 2. The near-spherical chamber is made of stainless steel and has a pressure rating of 21 barg. The hinged top is attached with six 19-mm diameter bolts which are not shown. Strain gauge pressure transducers measured the explosion pressure. The data were collected by a high speed personal com-

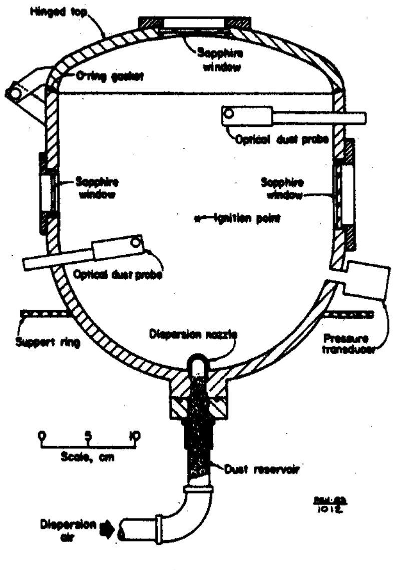

Fig. 2. Horizontal cross-section of Pittsburgh Research Laboratory 20-L chamber.

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[1] The Pittsburgh Research Laboratory was part of the U.S. Bureau of Mines before its transfer to NIOSH in October, 1996.

puter (PC) based data acquisition system. The dust to be tested can be placed either in the dust reservoir or on top of the dispersion nozzle at the bottom of the chamber (Fig. 1). After the dust and igniter have been placed in the chamber, the top is bolted on and the chamber is partially evacuated to an absolute pressure of 0.14 bara. Then a short blast of dry air (0.3 s duration from a 16-L reservoir at 9 barg) disperses the dust and raises the chamber pressure to about 1 bara. The ignitor is activated after an additional delay of 0.1 s. This results in a total ignition delay of 0.4 s from the start of dispersion until ignition. The experimental dust concentration reported in this paper is the mass of dust divided by the chamber volume, i.e. the nominal dust loading.

The Fike Corporation 1-m^3 chamber (Figs. 3 and 4) was also used to measure dust explosibilities. The 1-m^3 chamber is spherical with an internal diameter of 1.22 m and a wall thickness of 9.5 mm. It has a pressure rating of 21 barg. The two halves of the sphere are connected by 12 bolts of 51 mm diameter. Two variable reluctance pressure transducers were used to measure the explosion pressure. Data from the instruments were collected by a high speed PC based data acquisition system.

The dust injection system for the 1-m^3 chamber consists of a 5-L dispersion reservoir, a 19-mm pneumatically activated ball valve, and a rebound nozzle (Fig. 3). In previous work (Cashdollar & Chatrathi, 1992), a ring nozzle was used. To create a dust cloud, a weighed sample of dust is placed in the dispersion reservoir. The reservoir is pressurized with dry air to 20 bara and the chamber is partially evacuated to 0.88 bara. Activation of the ball valve disperses the dust and air into the 1-m^3 chamber through the rebound nozzle and raises the chamber pressure to about 1 bara. The ignitor is fired 0.6 s after activation of the ball valve. The reported experimental dust concentration for the 1-m^3 chamber is the mass of dust divided by the vessel volume.

The dispersion time and measured K_{st} values (and presumably the turbulence level) in the Fike 1-m^3 chamber are comparable to those in European 1-m^3 chambers (Bartknecht, 1989). This is the turbulence level in VDI Standard 3673, ISO Standard 6184/1, and ASTM Standard E1226 used to determine the maximum rate of pressure rise of a dust explosion. The K_{st} and turbulence levels in the PRL 20-L chamber are lower, but this should not significantly affect measurements of the MEC or LOC (Cashdollar & Chatrathi, 1992). The main effect of increased turbulence at low dust concentrations is to make the dust cloud more difficult to ignite (Amyotte, Chippett & Pegg, 1989). However, with the strong ignitors used for the tests, the somewhat higher turbulence level in the 1-m^3 chamber should have little effect on the measurements.

The ignition sources used for the tests were electrically activated chemical ignitors manufactured by Fr. Sobbe of Germany. The ignitors are composed of 40% zirconium, 30% barium nitrate, and 30% barium peroxide. They are activated electrically with an internal fuse wire and deliver their energy in about 10 ms. The Sobbe ignitors are available in energies of 0.25, 0.50, 1.0, 2.5, 5.0 and 10.0 kJ. These are nominal calorimetric energies based on the mass of pyrotechnic powder in

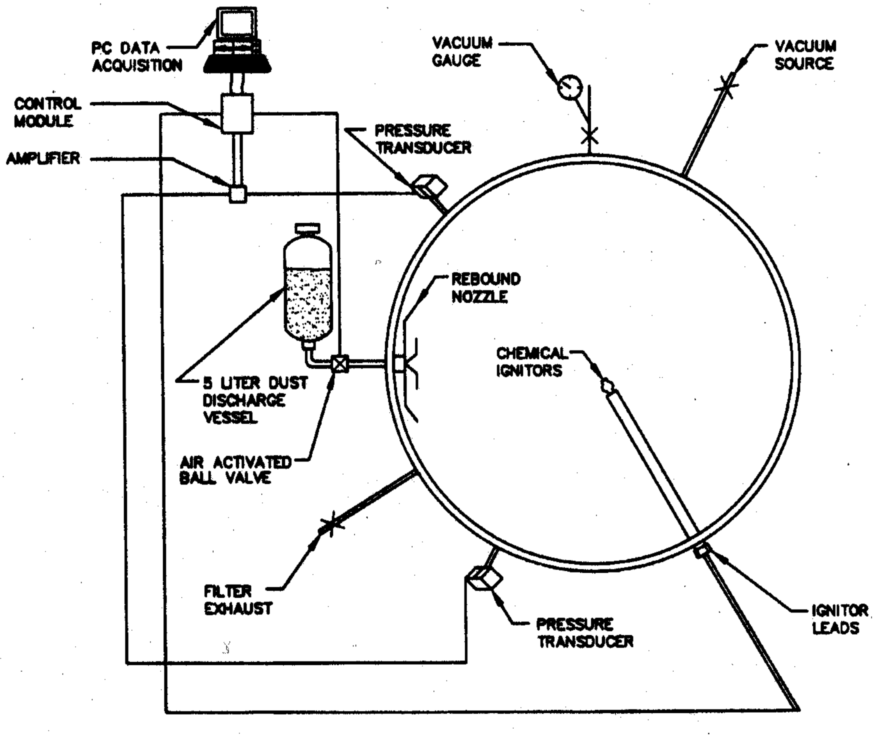

Fig. 3. Vertical cross-section of Fike 1-m^3 vessel.

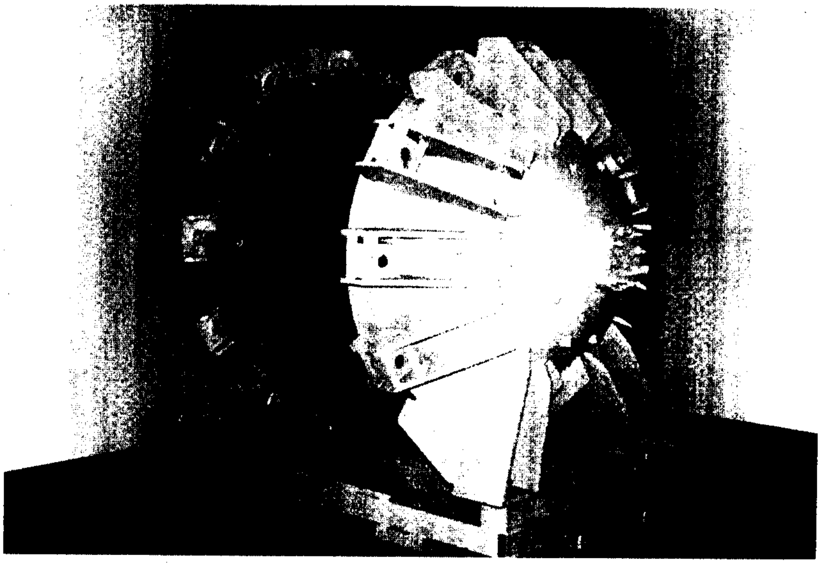

Fig. 4. Photograph of Fike Corporation 1-m^3 vessel.

each ignitor. The 5000-J ignitor by itself produces a pressure rise of about 0.5 bar in the 20-L chamber but only about 0.01 bar in the 1-m^3 chamber. Physical and chemical properties of the dusts are shown in Table 1.

The gilsonite is an asphaltic material mined in Utah. The bituminous coal is from the Pittsburgh seam; this dust has been used for decades as a standard test dust (Rice & Greenwald, 1929; Cashdollar, Sapko, Weiss & Hertzberg, 1987). RoRo93 was distributed worldwide by A. Kuhner AG, of Switzerland, in 1993 as a round robin test material for $P_{\max}$ and K_{St} testing. RoRo93 is a 2,2,6,6-tetramethylpiperidine derivative (light stabilizer). Lycopodium clavatum (reticulate form) is a plant spore obtained from the Meer Corporation. The iron is a minus-325-mesh powder. The aluminum was Alcoa atomized aluminum powder, grade 123. The size distributions were determined from a combination of sonic sieving data, Coulter counter data, and laser diffraction particle size data.

3. MEC data and discussion

In a previous report (Cashdollar & Chatrathi, 1992) in this series, the MECs of three dusts were evaluated in the PRL 20-L chamber and in the Fike 1-m^3 chamber following the test procedures in ASTM E1515. The MEC values for gilsonite dust and bituminous coal dust were measured in each chamber at several ignition energies. Chemical ignitors with energies from 0.5 to 10 kJ were used in the tests. These results, given as the top two dusts in Table 2, indicated that the 20-L chamber may be overdriven with high energy ignitors. The MEC values measured in the 20-L chamber with 2.5-kJ ignitors were comparable to those measured in the 1-m^3 chamber with 10-kJ ignitors. At higher ignition energies in the 20-L chamber, there was evidence of overdriving. The explosibility of anthracite coal was also studied in the two chambers, but the data are not listed in the table. The anthracite did not ignite at 2.5 kJ, but appeared to

Table 1
Physical and chemical properties of fuels

Parameter	Gilsonite	Pittsburgh coal	RoRo93	Lycopodium	Iron	Aluminum
Surface mean diameter, D_s (µm)	19	30	~12	26	~16	~18
Mass mean diameter, D_w (µm)	37	50	~38	28	~22	~24
Mass median diameter, D_{med} (µm)	28	44	~29	28	~23	~20
<75 µm (%)	91	81	~89	100	97	100
<20 µm (%)	36	16	~37	1	-	~42
Moisture (%)	1	1	0	3	0	0
Volatiles (%)	84	37	100	92	NA	NA

Table 2
Summary of MEC testing results (g/m^3)

Dust	20-L vessel				1 m^3 vessel		
	1 kJ	2.5 kJ	5 kJ	10 kJ	2.5 kJ	5 kJ	10 kJ
Gilsonite	50±5	35±5	30±5	30±4	39±3	41±3	36±3
Pittsburgh coal	90±5	80±10	60±10	50±10	90±5	85±5	80±5
RoRo93	–	36±3	28±4	25±5	34±4	35±4	35±4
Lycopodium	–	45±4	30±5	–	41±2	42±2	42±2
Iron	–	250±30	~200±40	–	210±10	195±5	195±5

ignite at 5 kJ in the 20-L chamber. It did not ignite even at 30 kJ in the larger 1-m^3 chamber.

The previous study (Cashdollar & Chatrathi, 1992) has now been extended to two additional carbon-based dusts as well as to a metallic dust with the goal of evaluating the extent of the overdriving phenomenon more thoroughly. The organic dusts were selected to be non-coal in order to widen the study. RoRo93 was selected as it has been widely studied in recent years; lycopodium was chosen due to its universal acceptance as an explosibility standard (because of its uniform size). Explosibility tests using RoRo93 were done with 2.5, 5, and 10-kJ ignitors in both chambers. The results are shown in Figs. 5 and 6.

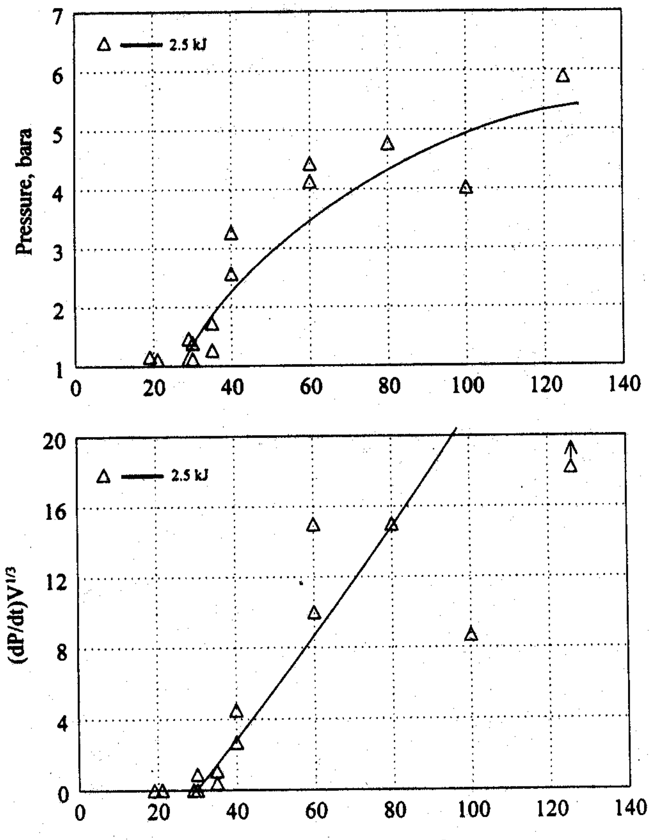

Fig. 5. MEC data for RoRo93 from the 20-L chamber.

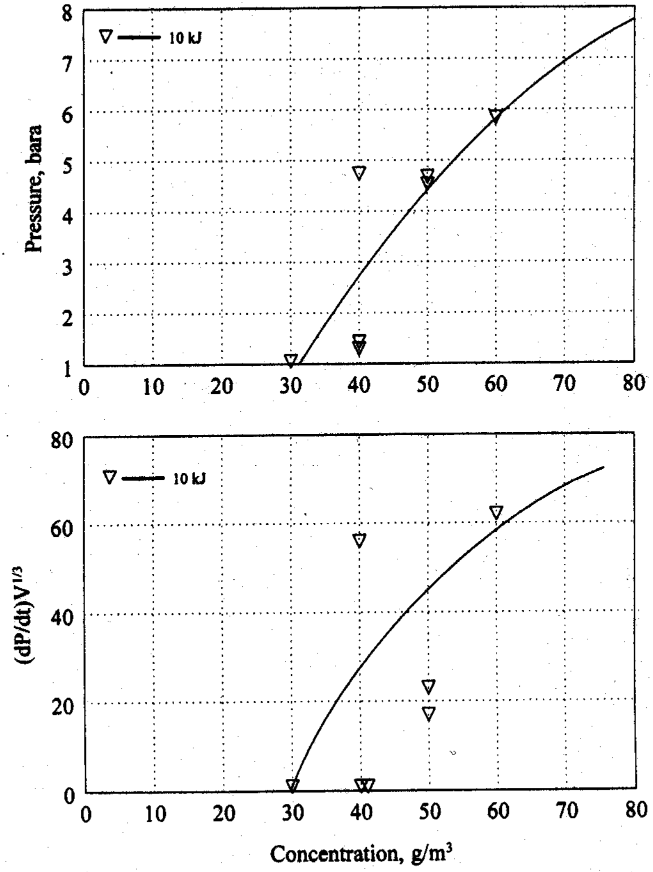

Fig. 6. MEC data for RoRo93 from the 1-m^3 chamber.

In these and subsequent figures only the data points and curves for one or two ignitors will be shown for purposes of clarity. The top portion of each graph shows the maximum absolute explosion pressure plotted against dust concentration. The effect of the ignitor is partially corrected by subtracting the pressure rise due to the ignitor itself from the maximum explosion pressure. The lower portion shows the maximum rate of pressure rise, normalized by the cube root of the chamber volume, $(dP/dt)V^{1/3}$. When tested at the standard turbulence level of ASTM E1226, this is known as the K_{St} value. The value $(dP/dt)V^{1/3}$ is proportional to the maximum flame speed (Amyotte et al., 1989; Hertzberg & Cashdollar, 1987; Hertzberg, Cashdollar & Zlochower, 1988). The primary criterion for flame propagation in the 20-L tests was a 1 bar pressure rise, corrected for the pressure rise of the ignitor itself. However, an additional criterion of a pressure rate of rise of 1.5 bar·m/s was also used in evaluating the 20-L results (Cashdollar & Chatrathi, 1992). Using these criteria, the MEC for RoRo93 with a 2.5-kJ ignitor in the 20-L chamber was found to be 36 g/m^3. The results shown in Table 2 show the change in MECs at higher ignition energies. The criterion for flame propagation in the 1-m^3 chamber is 1 bar pressure rise or an absolute pressure of 2 bara. Based on this criterion, the MEC for RoRo93 is 35 g/m^3 with a 10-kJ ignition source in the 1-m^3 vessel. There was no significant pressure rise at lower concentrations while the pressure continued to rise at higher concentrations. The experimental MEC values, given in Table 2, show little dependency on ignition energy in the 1-m^3 chamber.

Lycopodium testing was performed at 2.5 and 5 kJ in the 20-L vessel and at 2.5, 5, and 10 kJ in the 1-m^3 vessel. The results are shown in Figs. 7 and 8 for one ignition energy and the summary data are given in Table 2. The ~41 g/m^3 MEC for the 1-m^3 vessel was clearly independent of ignition energy. The 20-L vessel showed an energy dependency and gave an MEC of 45 g/m^3 at 2.5 kJ and 30 g/m^3 at 5 kJ.

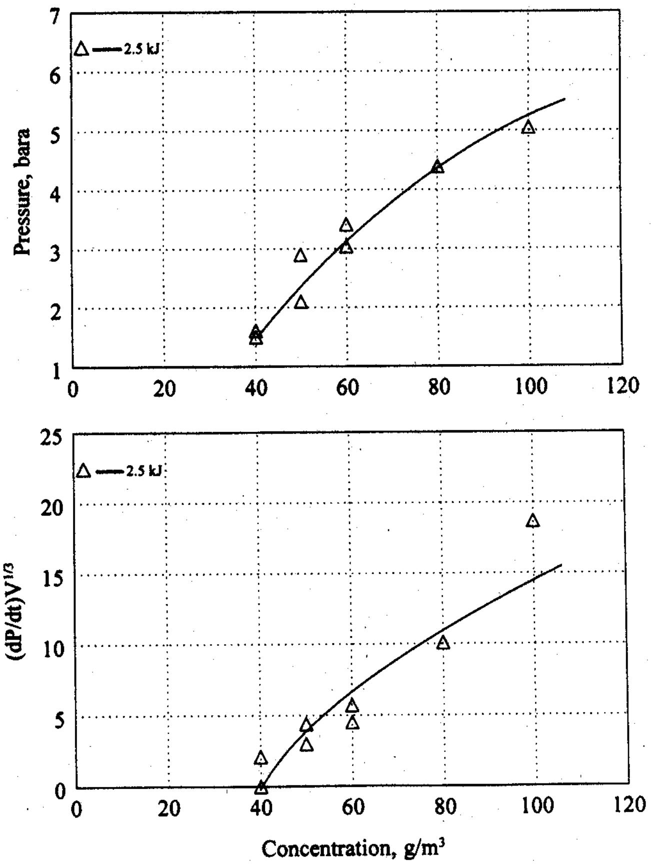

Fig. 7. MEC data for lycopodium from the 20-L chamber.

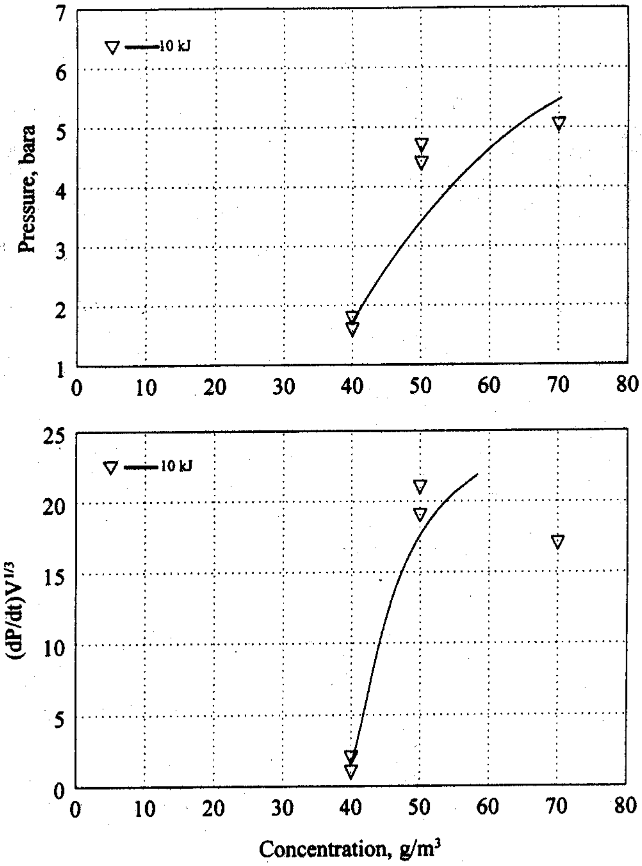

Fig. 8. MEC data for lycopodium from the 1-m^3 chamber.

For the RoRo93 and lycopodium, as well as for gilsonite and bituminous coal, the 2.5-kJ MEC data in the 20-L chamber agreed better with the 10-kJ data in the 1-m^3 chamber. At higher ignition energies, there was no evidence of overdriving in the 20-L chamber.

The iron results are shown in Figs. 9 and 10 and are listed in Table 2. The MEC was ~195 g/m^3 at 10 kJ in the 1-m^3 vessel. In the 20-L chamber, the MEC was ~200 g/m^3 at 5 kJ and 250 g/m^3 at 2.5 kJ. In this case, the 5-kJ ignitor data in the 20-L chamber agreed better with the 1-m^3 data using the 10-kJ ignitor.

The effect of ignition energy on MEC measurement in the 20-L and 1-m^3 chambers was studied in a previous report (Cashdollar & Chatrathi, 1992). The results of those tests along with the current results for RoRo93, lycopodium, and iron powder are summarized in Table 2 and Fig. 11, where the measured or apparent MEC is plotted versus ignition energy. The pattern observed previously for gilsonite and Pittsburgh coal in the 1-m^3 was again seen with the three new dusts. That is, the asymptotes are nearly vertical and the measured MECs from the 1-m^3 vessel are essentially independent of ignition energy over the range studied. The 20-L tests did not, however, show the same independence of ignition energy. As the energy increased from 2.5 kJ, the apparent MEC decreased and was definitely less than the