EXPLOSION HAZARDS OF OIL SHALE DUSTS: LIMITS, PRESSURES, AND IGNITABILITY

Kenneth L. Cashdollar, Martin Hertzberg, and Ronald S. Conti

U.S. Department of the Interior, Bureau of Mines
Pittsburgh Research Center
Cochrans Mill Road
Post Office Box 18070
Pittsburgh, PA 15236

ABSTRACT

The Bureau of Mines, under a cooperative agreement with the Colorado Mining Association, has investigated the explosion hazards of fine oil shale dust. Six grades of dust of varying oil assay (from 20 to 55 gal/ton) but with similar size distributions were studied in a 20-L explosibility chamber and in a 1.2-L ignitability furnace. Lean limits of flammability, maximum explosion pressures, and maximum rates of pressure rise were measured in the 20-L chamber and thermal autoignition temperatures were measured in the 1.2-L furnace. Electrical spark ignition measurements were made in both systems. For comparison with the oil shales, Pittsburgh seam bituminous coal, gilsonite, sulfide ore, and anthracite coal were also tested.

The lean limits of flammability vary inversely with oil assay and all grades of shale dust with oil assays of 20 to 24 gal/ton or greater are capable of generating dust explosions at concentrations above their respective lean limits. Even the 50 gal/ton shale dust is less flammable than Pittsburgh bituminous coal. In terms of electrical ignitability, the shales are at least an order of magnitude less ignitable than the coal. The shale dust clouds are, however, somewhat more easily ignited thermally than the coal.

Microscopic observations of the various grades of dust were made in order to determine whether the particle size variable could be isolated and studied independently of kerogen content of the shale dust. The implications of preliminary data, which suggest that those two variables are not readily separable, are also considered.

INTRODUCTION

For a number of years the Bureau of Mines has conducted laboratory and full-scale mine experiments on the explosion hazards involved in the mining and processing of oil shale dusts. The earliest work was by Allison and Bauer1 who showed that oil shale dust could indeed propagate an explosion. More recent and more comprehensive mine testing of the explosibility of oil shale dusts was conducted by Richmond, Sapko, and others.20-23 Additional Bureau research involved the monitoring of methane emissions in oil shale mines and the study of large oil shale rubble fires.21-23,25

Supplementing this work, a Bureau contractor7 evaluated the fire and explosion hazards of oil shale mining. Various accident scenarios were postulated, mine dust loadings were measured, and laboratory tests were conducted.3,7,8,28 The laboratory dust flammability testing under the contract used a Hartmann apparatus10 and found that oil shale dust could be ignited only after altering the standard procedures.7,28 Recent Bureau research16 has shown that the 1.2-L Hartmann apparatus has several severe deficiencies, such as nonuniform dust dispersion and inadequate ignition energy, which limit its usefulness, particularly for hard-to-ignite dusts such as oil shale.

The Bureau of Mines recent laboratory dust flammability testing16 has been conducted in an 8-L chamber in which optical probes were used to monitor the uniformity of the dust dispersion and strong chemical igniters were used to initiate the explosion tests. The data reported in the current paper are from a 20-L chamber that is similar in design to the 8-L chamber.

There are two aspects to the explosion hazard of dusts. One is related to the probability of having a flammable volume of dust dispersed in air. To evaluate this hazard, it is necessary to measure the lean concentration limit of flammability for the dust and to compare that number to the actual dust loading in the mine volume. The second aspect is related to the probability of igniting the flammable dust cloud.
To evaluate this, the minimum thermal autoignition temperature and minimum ignition energy can be measured. If both a flammable dust cloud and a sufficiently strong ignition source are present, an explosion will occur, and the explosion pressure and rate of pressure rise will provide a measure of the severity of the explosion.

Because of the complexity and large scale of the experimental mine tests, personnel and time demands are considerable for each test. The laboratory tests in the 20-L chamber can be conducted much more easily and quickly. Various comparison experiments\(^5,14\) have shown good agreement between laboratory and mine tests. Therefore, the laboratory chambers are now used for preliminary screening before full-scale mine tests are conducted. The full-scale mine tests are, however, still essential for the final evaluation of the true explosion hazard.

**APPARATUS AND EXPERIMENTAL METHOD**

The properties and characteristics of the oil shale dusts tested are shown in table 1. The first column in the table is an identification number used in previous Bureau publications\(^20-21\) for the same fine sized oil shale dusts. The Fischer assay\(^26\) are shown in English and metric units in columns 2 and 3. The percent volatiles in column 4 are the sum of the oil and gas amounts from the Fischer assays.\(^21\) The heating values in columns 5 and 6 were measured in an adiabatic bomb calorimeter.\(^2\) The size analyses were from a combination of sonic sieving data and Coulter counter data (reference to specific equipment does not imply endorsement by the Bureau of Mines). \(D_S\) is the surface mean diameter and \(D_W\) is the volume or mass mean diameter.

**TABLE 1. - Analyses of oil shale dusts**

<table>
<thead>
<tr>
<th>Number</th>
<th>Fischer assay</th>
<th>Vol.</th>
<th>Heating value,(\text{cal/g})</th>
<th>Size (\mu\text{m})</th>
<th>(D_S)</th>
<th>(D_W)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5082...</td>
<td>20</td>
<td>83</td>
<td>9</td>
<td>1810</td>
<td>1010</td>
<td>29</td>
</tr>
<tr>
<td>6114...</td>
<td>24</td>
<td>100</td>
<td>11</td>
<td>2140</td>
<td>1190</td>
<td>22</td>
</tr>
<tr>
<td>6238...</td>
<td>35</td>
<td>146</td>
<td>16</td>
<td>3230</td>
<td>1790</td>
<td>22</td>
</tr>
<tr>
<td>0000...</td>
<td>44</td>
<td>184</td>
<td>20</td>
<td>4100</td>
<td>2280</td>
<td>24</td>
</tr>
<tr>
<td>5084...</td>
<td>50</td>
<td>209</td>
<td>22</td>
<td>4700</td>
<td>2610</td>
<td>18</td>
</tr>
<tr>
<td>5777-5.</td>
<td>55</td>
<td>235</td>
<td>25</td>
<td>5260</td>
<td>2920</td>
<td>28</td>
</tr>
</tbody>
</table>

Four additional dusts were studied for comparison with the oil shales; they are listed in table 2. The volatility and heating values for the coals and gilsonite were measured by the standard ASTM methods.\(^2\) The size analyses are in the same form as those in table 1. Pittsburgh seam bituminous coal was used for comparison because of the large amount of practical data on its explosion hazards in the coal mining and electric power industries. Gilsonite (or uintahite) is a mined asphaltic material that is even more hazardous than coal. Anthracite coal was chosen as a material which has a long mining history with no record of any pure dust explosion.\(^13\) The sulfide ore was chosen as a material that is difficult to ignite but that has caused secondary explosions in mines.\(^12,18\)

**TABLE 2. - Analyses of comparison dusts**

<table>
<thead>
<tr>
<th>Number</th>
<th>Dust</th>
<th>Vol.</th>
<th>Heating value,(\text{cal/g})</th>
<th>Size (\mu\text{m})</th>
<th>(D_S)</th>
<th>(D_W)</th>
</tr>
</thead>
<tbody>
<tr>
<td>11/74...</td>
<td>Gilsonite</td>
<td>85</td>
<td>17,770</td>
<td>9,870</td>
<td>15</td>
<td>38</td>
</tr>
<tr>
<td>78/83...</td>
<td>Pittsburgh coal</td>
<td>37</td>
<td>13,810</td>
<td>7,670</td>
<td>29</td>
<td>47</td>
</tr>
<tr>
<td>12/83...</td>
<td>Sulfide ore</td>
<td>--</td>
<td>3,460</td>
<td>1,920</td>
<td>27</td>
<td>44</td>
</tr>
<tr>
<td>3/83...</td>
<td>Anthracite coal</td>
<td>5</td>
<td>12,860</td>
<td>7,140</td>
<td>24</td>
<td>41</td>
</tr>
</tbody>
</table>

The 20-L laboratory chamber used for the flammability and ignitability testing of the dusts is shown in figure 1. The optical dust probes\(^16\) are used to measure the dust dispersion uniformity. The dust is placed in the reservoir at the bottom of the chamber and is dispersed through the holes in the nozzle by a blast of air from a dispersion reservoir (not shown). The standard procedure is to partially evacuate the chamber to 0.1 atm absolute so that the blast of air (which disperses the dust) raises the chamber pressure to 1 atm absolute at ignition. Various ignition sources, such as electric sparks, chemical matches, and strong chemical ignitors, can be used. A more detailed description of the experimental procedures can be found in reference 16. The 1.2-L furnace\(^6\) used to measure the thermal and electrical ignitability of the dusts is shown in figure 2. For the thermal ignition tests, the furnace is set at a predetermined temperature and the dust is placed in the dispersion receptacle. Then the receptacle is quickly inserted into the bottom of
the furnace, and an air blast from the reservoir disperses the dust into the furnace. A fiberglass filter diaphragm on the top of the furnace confines the dust so that the maximum time of exposure of the dust cloud to the furnace temperature is at least several seconds, after which the dust begins to settle out. The criteria for ignition are that the diaphragm ruptures and flame is observed emitting from the top of the furnace. Because of its larger volume, more uniform dispersion, and longer residence time, the 1.2-L furnace generally gives somewhat lower minimum autoignition temperatures than does the Godbert-Greenwald furnace used in earlier Bureau studies.

The electrical circuit shown in figure 2 was not a part of the system during the thermal ignitability testing, but it was used for spark ignitability testing in the furnace at ambient temperature and at elevated temperatures below the autoignition temperature of the dust cloud. A similar spark ignition circuit was also used in the 20-L chamber for ignitability testing at ambient temperature.

Figure 1. - 20-L dust explosibility test chamber.

Figure 2. - 1.2-L ignitability furnace.

EXPERIMENTAL DATA

Some of the thermal autoignition data for the oil shales and for Pittsburgh coal are shown in figure 3. The areas above the curves are the temperatures and dust cloud concentrations that will thermally autoignite in the 1.2-L furnace. The areas below the curves will not ignite thermally, although much of this region would propagate an explosion if a point ignition source such as a spark or match flame were present. Both the low grade (19-24 gal/ton) and high grade (50-55 gal/ton) oil shales have slightly lower minimum autoignition temperatures (AIT) than the Pittsburgh coal dust. The rich oil shale reaches its minimum AIT at about the same concentration as the
Pittsburgh coal, but the 19-24 gal/ton shale reaches its minimum AIT at a significantly higher concentration. The minimum AIT’s, 475°-500°C, measured for oil shale in the 1.2-L furnace are significantly lower than the 560°-620°C minimum AIT’s measured for the shales in the Godbert-Greenwald furnace.28

The complete data for the minimum AIT’s for the oil shales and the comparison dusts are shown in table 3. The oil shales have minimum AIT’s similar to that of gilsonite, somewhat lower than those of Pittsburgh coal and the sulfide ore.

The minimum spark ignition energies for the oil shales were measured at room temperature in the 1.2-L furnace and 20-L chamber. The minimum ignition energy is generally apparatus dependent because it is a function of the turbulence level generated by the dust dispersion and the circuit efficiency for transferring stored electrical energy into the gas in the spark gap. Therefore, the values reported here should be considered only as relative values for comparing different dusts and not as absolute values. The data reported in table 3 are mainly from the 20-L chamber; the minimum values from the 1.2-L furnace were about the same for the shale but larger for the Pittsburgh coal and gilsonite. The data are reported both as the stored energy on the capacitor, 1/2 CE², where C is the capacitance and E is the voltage and also as an effective energy deposited into the gas in the spark gap. To compare different types of ignition sources, the effective energy of each is obtained by measuring the pressure rise, Δp, due to the ignition source by itself in a fixed volume V.

For the sparks, the effective energy is defined as 2.5 W·p.

For either the stored or effective minimum ignition energy, even the richest oil shale tested is at least an order of magnitude more difficult to ignite than the Pittsburgh coal, which is itself somewhat more difficult to ignite than gilsonite. The 44 gal/ton shale could not be ignited at ambient temperature by the strongest sparks available. At above ambient temperatures of 100°-200°C, this shale could easily be ignited by sparks. This lowering of the minimum ignition energy at elevated temperatures has been

![Figure 3. - Thermal ignitability data for oil shales compared to Pittsburgh coal.](image)

**TABLE 3. - Thermal and electrical ignitability data for the oil shales and comparison dusts**

<table>
<thead>
<tr>
<th>Dust</th>
<th>Fischer assay, gal/ton</th>
<th>Minimum autoignition, °C</th>
<th>Stored, 1/2 CE², Effective</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oil shale</td>
<td>50</td>
<td>475</td>
<td>80-100</td>
</tr>
<tr>
<td></td>
<td>44</td>
<td>500</td>
<td>--</td>
</tr>
<tr>
<td></td>
<td>35</td>
<td>500</td>
<td>--</td>
</tr>
<tr>
<td></td>
<td>24</td>
<td>500</td>
<td>--</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>500</td>
<td>--</td>
</tr>
<tr>
<td>Gilsonite</td>
<td></td>
<td>490</td>
<td>0.140</td>
</tr>
<tr>
<td>Pittsburgh coal</td>
<td></td>
<td>540</td>
<td>0.310</td>
</tr>
<tr>
<td>Sulfide ore</td>
<td></td>
<td>550</td>
<td>--</td>
</tr>
<tr>
<td>Anthracite coal</td>
<td></td>
<td>675</td>
<td>--</td>
</tr>
</tbody>
</table>
discussed previously. The 24 gal/ton oil shale could only be ignited with a very strong chemical ignitor. Even a chemical ignitor with a calorimetric energy of 2,500 J and a VAp energy of 400 J was not able to ignite a uniformly dispersed cloud; however, one with a calorimetric energy of 5,000 J and a VAp energy of 770 J did ignite the 24 gal/ton shale. Such difficulty in igniting pre-dispersed oil shale dust clouds of low assay was also observed by Richmond and others in mine tests.

The dust explosion data from the 20-L chamber for the oil shales and the comparison dusts are shown in figure 4 as a function of dust concentration. Data points are shown only for the oil shales; smoothed curves are shown for the comparison dusts. In figure 4B, the explosion pressure ratio is the maximum

Figure 4. - Explosibility data for oil shales compared to Pittsburgh coal, sulfide ore, and anthracite coal.
explosion pressure (corrected for the small pressure rise due to the ignitor itself) divided by the pressure at ignition, which is about 1 atm. The criterion used to define the lean limit is a pressure ratio of two or alternatively a pressure rise of approximately 1 atm above the pressure at ignition.\textsuperscript{14,16} This is a reasonable definition for comparison among different dusts.

The data shown in figure \textsuperscript{4B} are for the strongest chemical ignitor, with 5,000 J calorimetric energy. For the dusts shown, Pittsburgh bituminous coal dust has the lowest lean limit and highest maximum explosion pressure. Gilsonite has a lower lean limit and about the same maximum pressure as the Pittsburgh coal, as shown in table 4. The 50 gal/ton oil shale has a higher lean limit concentration and significantly lower maximum pressure than the Pittsburgh coal. The lower assay oil shales have progressively higher lean limits and lower maximum pressures. In coal mines, there is usually enough dust to propagate an explosion if the dust is dispersed; therefore, rock dusting is required. Preliminary dust sampling\textsuperscript{7,8} in oil shale mines has shown that the accumulated dust, if dispersed, would normally be below the lean explosibility limit.

The variation in the measured lean limits with ignition energy, shown in table 4, indicates the ease or difficulty in igniting the various dusts. Pittsburgh coal can be ignited at almost as low a concentration with the 2,500 J ignitor as with the 5,000 J ignitor. The more difficult to ignite 50 gal/ton shale can be ignited at much lower concentrations with the stronger ignitor. The same is true for the 35 gal/ton shale. The 24 gal/ton shale and the sulfide ore could not be ignited with the 2,500 J ignitors. As discussed in previous publications\textsuperscript{14,16} the true lean limit concentration for dust explosibility is the value measured with a high enough ignition energy so that the limit is independent of ignition energy. The data in table 4 show that this is probably true for the coal, but that even with the 5,000 J ignitors, the measured limits for the oil shales may not yet be ignition energy independent.

The 20 gal/ton oil shale produced two explosions out of six tests at very high concentrations (0.8 -1.2 kg/m\textsuperscript{3}). Therefore, even the 5,000 J ignitor is probably not strong enough to reliably ignite this dust. The explosions that did occur, however, produced pressures of approximately 2 atm above ambient. These results are comparable to the full-scale mine tests\textsuperscript{21,23} which found that 22 gal/ton shale could produce explosions at high concentrations, but that 19 gal/ton shale did not

<table>
<thead>
<tr>
<th>TABLE 4. - Explosibility data for oil shale and comparison dusts</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dust</td>
</tr>
<tr>
<td>gal/ton</td>
</tr>
<tr>
<td>Oil shale</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>Gilsonite</td>
</tr>
<tr>
<td>Pittsburgh coal</td>
</tr>
<tr>
<td>Sulfide ore</td>
</tr>
<tr>
<td>Anthracite coal</td>
</tr>
</tbody>
</table>

Note: NF means nonignitable and the dashes mean no data were obtained.

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propagate explosions.

The sulfide ore, which has been involved in secondary mine explosions during blasting, 12,18 is intermediate in explosion hazard between the 24 and 35 gal/ton oil shales. For the anthracite coal, the slight pressure rise observed was only due to a small amount of burning around the ignition source and does not signify flame propagation. Therefore, this 5 pct volatile anthracite is considered nonexplosive in air.

The data in figure 4A for maximum rates of pressure rise are similar to the pressure data except that the differences among the various dusts are more pronounced. These data are also listed in the last column of table 4. The rate of pressure rise is very dependent on the turbulence in the chamber, and therefore the data should only be used for a relative comparison among the various dusts. A higher level of turbulence would increase the rates of pressure rise for all the dusts.

THE PARTICLE SIZE VARIABLE AND MICROSCOPIC OBSERVATIONS

In a previous study 15 of coal dust and polyethylene powder, it was possible to isolate the particle size variable and to measure the effect of particle diameter on the lean limit of flammability and the minimum autoignition temperature. Using narrow size distributions for coal and polyethylene with average diameters ranging from 2 µm to over 400 µm, it was determined that the lean limits were insensitive to particle size below some characteristic diameter. Above these characteristic diameters of 50 µm for Pittsburgh seam coal dust and 100 µm for polyethylene, the lean limit concentrations increased markedly with increasing particle diameter. The minimum autoignition temperatures for coal dust and polyethylene powder displayed a similar particle size dependence except that at the elevated temperatures involved, both the characteristic diameters were larger.

It was, therefore, quite logical to attempt to initiate a similar study of the effect of particle size on the flammability limit and thermal ignitability for the oil shale dusts studied here. In the case of polyethylene, which is a totally volatilizable, homogeneous solid, the particle size and volatility variables are readily separable. Even though the coal structure is heterogeneous and contains separate macerals, mineral phases, and pyritic inclusions, the data in figure 5A, 8 show that there is no tendency for these separate phases to concentrate in either the fine or the coarse fractions of the sieved dust. The volatile content and heating value are essentially independent of particle size, despite the heterogeneous structure of the coal.

Figure 5C shows a different behavior for a broad size distribution of 35 gal/ton oil shale which was sieved to obtain the various sizes. There is an increase in the heating value with increasing particle size until the heating value levels off at the 300 to 1,000 µm particle size range. The heating values can be related to Fischer assays as shown in a previous publication. 21 This observation of lower heating values or lower assays associated with the finer sizes of oil shale has been reported previously. 3,20 This variation in assay with particle size is a reflection of the basic heterogeneous structure of the oil shale and the way in which that heterogeneity affects its

Figure 5. - Heating value for oil shale as a function of particle size compared to heating value and volatile content of coal.
pulverization. This variation is consistent with the structure studies of Tisot and Murphy who showed that the average particle size of the mineral matter in the Green River shale deposit was less than 44 μm. The parent oil shale rock is a heterogeneous agglomerate consisting of fine mineral particles bound together by a soft kerogen glue or matrix in some regions and by an inorganic cementing agent in other regions. When the shale is pulverized, the fine mineral particles are more easily separated from the matrix leaving a higher kerogen concentration in the larger particles. This structural heterogeneity and grinding behavior of the oil shale also serves as a basis for the physical concentration method for oil shale enrichment described by Fahinstrom. Independent studies of comparable methods were later reported by others at the 16th Oil Shale Symposium in 1983.

This structural heterogeneity is clearly illustrated by the optical and scanning electron

Figure 6. - Optical microscope photograph of oil shale particles (A) and the corresponding SEM X-ray maps for the elements calcium (B), silicon (C), and aluminum (D).
microscope (SEM) data shown in figures 6 and 7. A second sample of the same 35 gal/ton shale as that used for figure 5C was sieved and the various sieved fractions were sampled and analyzed with an optical microscope and with the X-ray imaging feature of an SEM. Figure 6A shows a collection of particles from several of the various sieved fractions. In the upper left portion of figure 6A is an approximately 600 μm diameter particle from the 40 x 30 mesh sieved fraction. This particle was selected because of its high optical transparency. The three accompanying SEM X-ray maps show the distribution of the elements calcium, silicon, and aluminum for the same field of view as for figure 6A. The maps are made by collecting only the X-rays from a particular element to form an image showing the distribution of that element over the field of view. A comparison of the three X-ray images corresponding to the 600 μm transparent particle shows that the particle contains a large amount of calcium and only trace amounts of

Figure 7. - Optical microscope photographs of the 270 x 200 mesh and the 200 x 140 mesh oil shale size fractions together with their corresponding silicon X-ray maps.
silicon and aluminum. Therefore, that particle is most likely calcitic in composition.

In the upper right quadrant of figure 6A is a particle from the 40 x 30 mesh sieved fraction that was selected because it was one of the more opaque particles observed with the optical microscope. This particle is clearly much more heterogeneous in structure than the calcitic particle. Some areas of the particle appear quite dark and are probably high in kerogen content; lighter areas are probably mineral matter. The corresponding X-ray maps in figures 6B, C, D show a strong Si X-ray signal and weaker signals from both the Ca and Al for this particle. Thus, its heterogeneous structure seems to contain the organic kerogen matrix together with mineral matter such as quartz, feldspar, spurrite, calcite, dolomite, etc.4

The dark particle in the lower left quadrant of figure 6A was selected from the 100 x 70 mesh fraction. This particle is even darker than the previous particle, and its corresponding X-ray maps show some Si and smaller amounts of Al and Ca. This particle appears to be another highly heterogeneous agglomerate but with a higher kerogen content than the particle in the upper right quadrant.

The lower right quadrant contains a large number of very small particles from the minus 400 mesh sieved fraction. Probably each of these small, individual mineral particles is fairly homogeneous in structure,27 and the data in figure 5C show that these smaller particles are lower in organic content.

Figure 7 shows numerous particles from the intermediate mesh size fractions. The 270 x 200 mesh sample is shown in figure 7A together with its corresponding Si X-ray map in figure 7B. The 200 x 140 mesh sample is shown in figure 7C together with its corresponding Si X-ray map in 7D. The optical microscope photographs show a range of opacities from dark to light for the particles in both size fractions. The Si X-ray maps generally correlate negatively with opacity. The darker, more opaque particles are generally richer in kerogen and, therefore, have weaker Si X-ray signals which are associated with the mineral components.

These structure studies show that the particle size and oil content variables are not independent. The fact that the heating value (kerogen content) decreases with decreasing particle size would have a moderating effect on the potential explosion hazard of dust generated in a practical mining situation. The fundamental heterogeneous structure of the oil shale deposits also has implications for the effective design of retorting or pyrolysis systems for the efficient recovery of the shale's oil content.11

CONCLUSION

The fundamental conclusion of this paper is the same as in previous Bureau studies: fine oil shale dust is a potential explosion hazard. The flammability and ignitability data reported here has, however, broadened the comparison of various grade oil shales to include other dusts with more extensive mining histories. Fine 50 gal/ton oil shale is only slightly less hazardous than Pittsburg coal in terms of its lean explosive limit concentration and explosion pressures generated, but it is at least an order of magnitude more difficult to ignite with an electric spark. The lowest assay shales that could propagate an explosion were 20 to 24 gal/ton. Sulfide ore, which has a history of dust explosions in mines, is comparable in explosion hazard to oil shale of about 30 gal/ton. All of the oil shales tested were somewhat more easily ignited thermally than was the Pittsburg coal.

Future laboratory testing of oil shale should include a study of the effect of particle size on flammability and a study of the effect of an added 1 or 2 pct methane on the ignitability and flammability data.

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