LABORATORY TESTING OF THE
INFLAMMABILITY OF COAL AND OTHER DUSTS
CONDUCTED BY THE BUREAU OF MINES

BY

H. P. GREENWALD
With foreword by GEORGE S. RICE
FOREWORD

One of the most important mine-safety researches conducted by the Bureau of Mines is on the inflammability or explosibility of coal and other dusts when raised into the air or into mixtures of inflammable gas and air. This research work is separated into two parts: One is concerned with large-scale testing, as in a gallery or mine passage, and the other with small-scale investigations.

From the earliest appreciation, in the middle of the last century, that combustible organic and mineral dusts might be inflamed when in a cloud, these two types of investigations have gone on more or less independently, but, for practical application, the results of laboratory work have depended upon the findings of large-scale tests. Although a certain amount of such dependence probably must continue, there has been a substantial advance in knowledge of laboratory testing. This progress makes it appear likely that a stage may be reached when laboratory investigations will, within limits, carry such conviction as to the relative explosibility of different dusts as will give the necessary confidence in applying the results.

As a step toward this stage H. P. Greenwald has critically reviewed the laboratory work done by the Bureau of Mines and, to a certain extent, by other experimental organizations both abroad and in this country. He has been concerned in the development and operation of instruments used in large-scale tests of the explosibility of coal dust at the Experimental Mine since 1914 and since 1927 has been in entire charge of these tests as supervising engineer of the Experimental Mine under the direction of the undersigned.

Determination of the relative explosibility or inflammability of different coal dusts in air was one of the principal objectives of the mine-safety researches begun by the Federal Government in 1908 after a series of mine disasters in the United States and abroad. These investigations were conducted first by the technological branch of the United States Geological Survey and were transferred to the Bureau of Mines when it was established in 1910. Laboratory studies of the subject were begun at the Pittsburgh Experiment Station simultaneously with large-scale testing in a steel gallery 6 feet 4 inches in diameter by 100 feet in length, located at the old arsenal grounds in that city. The results of this testing and reviews of what had been done in European countries were reported in a bulletin entitled “The Explosibility of Coal Dust.”

The laboratory work reported in that bulletin was carried on by Dr. J. C. W. Frazer, who employed a method (now generally abandoned) similar to that used abroad both in laboratories and in many galleries, namely, use of the pressure produced during explosions or inflammations of the dust as criteria of their relative explosibility.

Greenwald traces subsequent attempts to correlate results obtained in the bureau's laboratories with those found in the gallery already mentioned and, later, with those obtained in the Experimental Mine, in which testing was begun in 1911. The latter investigations were supplemented by field studies of mine-explosion disasters by the writer and his associate mining engineers of the bureau, with special reference to the part played in the disasters by coal dust.

To obtain data that might be useful investigations also were made of various grain-dust explosions. These led to the formation of a section for testing dusts of agricultural and forest products in the Bureau of Chemistry, Department of Agriculture, to which such work was transferred. The Bureau of Mines then confined its investigations to mineral and metallic dusts.

At various times serious explosions in grinding, handling, or transportation of dusts other than coal dust, like dusts of pyriticiferous ores and native sulphur, dusts of metals, such as aluminum, magnesium, and zinc, and pitch and tar dusts like those produced from the processing of coal, have caused the bureau to make investigations so as to be able to give advice to the respective industries as to means of lessening the hazard of ignition and giving protection to workers if ignition accidentally occurs. Such means are the use of tightly closed machinery and connecting conduits in grinding and moving the material, the simultaneous use of inert atmospheres in the enclosed spaces, the providing of explosion vents, and the removal of or protection of all sources of ignition.

In coal mining some suitable form of rock dusting has been found thoroughly effective in preventing widespread propagation of local ignitions of coal dust, and rock dusting has already prevented the loss of thousands of lives by extinguishing incipient explosions. While this problem has by no means reached a final solution and while improved methods are sought constantly, it is only occasionally that it is feasible, even in the case of coal dust, to obtain from mines long distances away large enough samples to carry out large-scale tests of dusts in the Experimental Mine; whereas in laboratory work tests can be made quickly with small amounts of material, and certain fundamental problems undoubtedly can be studied best under controlled laboratory conditions. It is feasible to use rock dust to neutralize coal dust only in mines, and other remedies must be sought when the danger of explosions of coal or other dusts is to be lessened in other places.

Research on laboratory testing has been done independently in the past by many research laboratories in different countries; but in April, 1931, under existing cooperation between the Bureau of Mines and the Safety in Mines Research Board of Great Britain, Dr. A. L. Godbert, of the latter organization, was detailed to the Pittsburgh Experiment Station of the bureau to collaborate with H. P. Greenwald to standardize and, if possible, to develop further methods of laboratory testing which will not depend upon direct correlation with large-scale testing.

George S. Rice,
Chief Mining Engineer.
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LABORATORY TESTING OF THE INFLAMMABILITY OF COAL AND OTHER DUSTS CONDUCTED BY THE BUREAU OF MINES

By H. P. Greenwald

INTRODUCTION

The Bureau of Mines has conducted laboratory tests of the inflammability of coal dusts more or less continuously since its establishment as a separate organization on July 1, 1910. A number of investigators have worked on the problem, each with his own method of attack. Some of the results have been published in various places; others remain unpublished. This paper endeavors to collect and describe all of the work in proper order, as briefly as is consistent with a good understanding thereof, and attempts to evaluate it in the light of present-day knowledge derived from large and small scale experiments in the United States and foreign countries.

ORIGIN OF LABORATORY INVESTIGATIONS

Laboratory studies of the ignition of coal dust by various means were begun in England and France more than 50 years ago; more detailed reference to these appears later. The question at that time was whether or not coal dust, which lay everywhere in the mines, played a part in the explosion disasters which periodically swept different mines in European countries; of the danger of firedamp (methane) explosions there was no doubt. Could coal dust be responsible for a disaster when no firedamp was present? Years of experiment supplemented by observations in mines were required to produce widespread belief in affirmative answers to this question.

Until 1908 studies of the explosibility of coal dust in the United States lagged behind those in European countries, as operators were content to await receipt of the results from abroad. Furthermore, those engaged in coal-mine operations were concerned chiefly with the tremendous development going on in various bituminous-coal fields. The first great coal-dust explosion in the United States, that of the Pocahontas Mine in 1884, was studied carefully, but the problem attracted little added attention until the practice of shooting coal from the solid was introduced in the early nineties. After that time explosions occurred fairly frequently because of the large increase in quantity of explosive, chiefly black blasting powder, used per shot under conditions favorable to ignition of dust. The climax was reached in 1907 when a series of terrible explosions claimed nearly

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2 Work on manuscript completed October, 1931.
3 Supervising engineer, Experimental-Mine section, Bureau of Mines.
900 lives; 700 men died from this cause in December alone. The organization established by act of Congress in the Geological Survey on July 1, 1908, to investigate the causes of these and other mine accidents became the nucleus of the Bureau of Mines at the time of its formation some two years later.

**PURPOSE OF LABORATORY INVESTIGATIONS**

The engineers in charge of the investigations were convinced that coal-dust explosions could best be studied on a large scale—that is, in a mine—but that concurrent laboratory investigations would be a valuable aid. The purpose of such investigations would be: (1) To obtain as much information as possible in advance of large-scale testing, which would of necessity be somewhat slow; (2) to study factors not readily amenable to treatment on a large scale; and (3) to develop a method of testing which would duplicate results obtained in large-scale tests of different coal dusts and which could be substituted for those more expensive and time-consuming tests, especially when large samples had to be sent long distances from mines in different parts of the country.

Need of the first purpose has passed; the large-scale experiments, now numbering over 1,200, conducted in the bureau's Experimental Mine, have given a clear insight into the nature of coal-dust explosions and means of preventing and limiting them. The second and third purposes are still in process of achievement. Many important details can best be studied on a laboratory scale, with subsequent confirmation of results by large-scale tests. The need of an inexpensive method for routine tests is made evident by the mounting costs of testing explosions in the Experimental Mine.

It should be made clear that those in charge of the large-scale tests have always been directly interested in results obtained in the laboratory and have been anxious that the objectives mentioned above be achieved, particularly in the earlier stages when need of information far exceeded the rate at which data could be obtained in the Experimental Mine. George S. Rice, chief mining engineer of the bureau, was in direct charge of tests in the Experimental Mine until 1916, when his headquarters were moved from Pittsburgh to Washington. Until then he took an active interest in the laboratory testing and participated regularly in the various developments in an advisory capacity. The laboratory galleries, described below, were developed in the Experimental-Mine section, a part of the division of the bureau of which he is head.

**TERMINOLOGY**

Before proceeding it is necessary to state what is meant by the terms "inflammability" and "explosibility" as used in this paper and applied to suspensions of coal dust in air. Different investigators have used these terms with different meanings, and exact definitions have been made only in recent years. The following definition of the term "explosibility" was published in Technical Paper 464: "A coal-dust explosion is defined * * * as any combus-

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tion of a cloud of coal dust in air irrespective of its velocity, violence, or extent, provided the combustion, inflammation, or explosion extends beyond the influence of the heat or flame of the igniting source." Stated in other words, any self-sustained combustion of coal dust in air in a mine or in testing work is called an explosion, whether weak or strong, extended or limited. On the other hand, the term "inflammability" may be reserved for combustion which does not extend outside the sphere of influence of the igniting source. No hard and fast line can be drawn between the two, because in many instances they grade into each other insensibly. Thus, pure fine dust of a bituminous coal—from the Pittsburgh bed, for example—will give an extremely violent self-sustained explosion. On addition of inert dust in successive quantities the violence is first reduced, together with the flame velocity; ultimately, a point is reached where the explosion will not be self-sustained over some arbitrary distance, say 300 feet, although the sphere of influence of the source may be only 50 feet. Between explosibility and inflammability there is then a twilight zone of transition not readily removed by attempts at exact definition.

The laboratory tests have not been on a sufficiently large scale to warrant use of the term "explosibility"; that is, one cannot say that the combustion would always have been self-sustained if the dust cloud had been of indefinite extent. For this reason the term "inflammability" is used in describing these tests to emphasize the difference between them and the large-scale tests; however, the fact that no definite line of demarcation exists between explosibility and inflammability as here defined must not be forgotten. They are in reality only different phases of a continuously varying phenomenon governed in its entirety by the laws of physics and chemistry.

**STANDARD OF REFERENCE IN LABORATORY WORK**

Practical application of the results of tests of the explosibility of coal dusts leads to the conclusion that the ultimate standard of reference must be conditions in the mines where the dusts are found. Such conditions vary widely, and in testing one chooses those most favorable to the initiation and propagation of an explosion. This result is achieved by the arrangements in the Experimental Mine to a far greater degree than in any method of laboratory or gallery testing so far devised, and for this reason the bureau has always taken Experimental-Mine test results as the standard with which laboratory investigations must be compared. A statement of the factors influencing explosibility in the Experimental Mine is then necessary. The relative explosibility of different dusts is measured by the proportion of inert material (including moisture and ash of the coal dust) required in a mixture to prevent continued propagation of an explosion under a standard set of conditions.

**FACTORS INFLUENCING EXPLOSIBILITY IN EXPERIMENTAL MINE**

Experimental-Mine tests have shown that the proportion of inert dust required to prevent propagation of an explosion is altered by variation in seven factors. These are as follows: (1) The size of coal and inert dust used, the former of primary and the latter of
secondary importance; (2) the composition of the coal dust, the volatile content being of primary and the moisture and ash content of secondary importance within the limits met in coal beds worked at present in the United States; (3) the presence of inflammable gas in the atmosphere; (4) the quantity of dust raised to form the cloud; (5) the distribution of the dust, which determines the ease with which a cloud is formed; (6) the strength of the source of ignition, that is, its dust raising and igniting power; and (7) surrounding conditions which alter the rate at which energy is taken from the burning dust either by direct absorption of heat or release of pressure. The first four of these have been investigated more thoroughly than the last three. Some are interdependent in several ways. The large-scale tests have established the behavior of factor 3 sufficiently well for all practical purposes, but further study of factors 1, 2, and 4 in suitable laboratory apparatus should yield important information when confirmed by large-scale tests. The last three factors are not readily amenable to laboratory treatment.

INVESTIGATION OF FACTORS IN THE LABORATORY

All of the first four factors have been dealt with directly in the laboratory work described in the following pages. Unfortunately none of them has been the subject of sufficient experimentation to yield results duplicating those in the Experimental Mine at all points, because suitable apparatus has not yet been developed. In contrast with large-scale testing one of the difficulties encountered in laboratory work has been the handling of dusts of high moisture content. This moisture may be external to the coal, as in damp or wet mines; or it may be inherent; as in low-grade bituminous and subbituminous coal mines. Studies of factors 5 and 6 have been made indirectly, as it was necessary to produce and reproduce dust clouds of proper density for the work and as the source of ignition had to be adequate for all the different dusts tested.

As a result of tests in the Experimental Mine the coarse limit of dust has been set at 20 mesh; that is, all material passing a standard 20-mesh sieve is considered as dust. The openings of a 20-mesh sieve are 0.033 inch (0.83 mm) square, and a laboratory apparatus must handle particles ranging from this size to microscopic if it is to be considered successful. It must grade dusts of widely varying composition in the same order of explosibility, both qualitatively and quantitatively, that are found in the Experimental Mine, and in general it should show the same variation with dust-cloud concentration found therein. The value of the methods tried may then be judged according to whether they did or did not fulfill these conditions.

CLASSES OF EXPERIMENTATION

The laboratory methods pursued by the various investigators fall readily into two classes: (1) Tests in closed systems with measurement of the pressure produced by the inflammation and (2) tests

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5 For a detailed discussion of these see Rice, G. S., and Greenwald, H. P., work cited, p. 9 and following.
in open systems with measurement of extension of the flame of the igniting source caused by the dust or determination of the proportion of added inert dust required to prevent flame extension. Class 2 may be subdivided into two types: (a) Tests in laboratory galleries designed to simulate conditions in a mine passageway to a greater or less degree; and (b) tests in furnaces with inflammation of the dust by hot surfaces. The first apparatus used by bureau investigators belonged to class 1; class 2 was developed later.

CLASS 1.—EXPERIMENTS IN CLOSED SYSTEMS

WORK OF FRAZER

The first laboratory work on the inflammability of coal dust in the bureau was conducted by J. C. W. Frazer, chemist, who was with the staff when the bureau became a separate organization. He reviewed the work of previous investigators abroad and wrote a summary thereof which was incorporated in Bulletin 20. L. A. Scholl, junior chemist, was employed to assist Frazer in January, 1911, and E. J. Hoffman, chemist, also started work on the problem in October of the same year. Frazer resigned at the end of 1911, and Hoffman was then in charge for a short period.

REVIEW OF LITERATURE

In Bulletin 20 Frazer reviewed the work of Galloway, Vital, Hall and Clark, Abel, Mallard and Le Chatelier, Thorpe, Engler, Bedson and Widdas, and Holtzwarth and Meyer, as well as large-scale tests conducted at Liéven by Taffanel. He based his apparatus on the second used by Bedson and Widdas in which the dust was inflamed by a heated platinum coil. The pressure produced was noted by visual observation of a water manometer.

The Safety in Mines Research Board of Great Britain recently has published a historical review of laboratory investigations of the inflammability of coal dust, including some of the work covered by Frazer, some which he did not include in his review, and a number of more recent researches.

FIRST APPARATUS

Frazer’s first apparatus consisted of an explosion flask constructed from a 2-liter aspirator bottle with a platinum igniting coil suspended in it. The dust was blown against the coil from below. An opening in the top of the flask was connected to a horizontal glass tube in which there was a snugly fitting steel ball. Pressure in the flask caused projection of the ball, and the distance it was thrown was taken as a measure thereof.

Instantaneous ignition of all dusts tried was obtained, with the exception of anthracite, natural coke, and charcoal, but flame was confined to the upper half of the flask and did not appear to fill that

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9 For a complete description see Rice, G. S., and others, work cited, pp. 140–142.
half completely. Results of tests of 7 bituminous coals, 1 cannel coal, and 3 lignites were reported. The steel ball was projected farthest by 2 of the lignites; but the distances recorded for the other samples were only slightly less, with one exception.

This first apparatus, like preliminary devices in many other investigations, gave its originator an insight into the problem being investigated and showed what modifications were desirable for more accurate and consistent results. Concerning the work Frazer says:

The coals are not greatly differentiated here, probably because the heat supplied by the wire was too great in comparison with that supplied by the dust in burning, so that the differences of the dusts were masked.

A great deal more work would have to be done before any positive statement could be made concerning the relative behavior of different coals. The same is true with respect to the effects of variations in the current used and in the pressure with which the dust is brought into the bottle. A definite strength of current does, however, seem to be required for complete ignition, and beyond that strength variation of current has no effect. The initial pressure of the ignition mixture is probably of considerable importance.

Subsequent work has shown nothing that leads one to question the accuracy of this summary.

SECOND APPARATUS

Frazer's second apparatus was developed from the first. The general principle of the two was the same, and the alterations made were designed to give better control of the experiments and to provide a more accurate means of measuring the pressure developed. It has been called the Frazer apparatus in recognition of its designer. It was in use before Bulletin 20 went to press, and the results of some early experiments are published therein. The balance of Frazer's work until his resignation was published in Bulletin 50.

DESCRIPTION

The apparatus is shown in Figure 1, and a detailed description appears in Bulletin 50, page 5. A spherical glass explosion vessel, a, of 1,600-cm³ capacity, was used with a heater coil, b, at its center. The dust-injecting system was altered only slightly. The pressure developed acted on a steel ball, d, ground to a tight fit, with a tube, c, projecting from the top of the explosion vessel. A flask, e, weighted with mercury, was placed on the ball; and by repeated experiments 2 weights differing by 5 g were found, the lower of which permitted the ball to rise whereas the higher did not.

Tests were made with current through the coil ranging from 5 to 7 amperes in half-ampere increments. The coil was heated exactly 3 minutes before the dust was injected, and the resultant pressure was released 1, 2, and 2¾ minutes after the start.

FIRST EXPERIMENTS WITH COAL DUST

The results obtained in this apparatus with 19 bituminous and subbituminous coals from six States were published in detail in
Bulletin 20. The dusts were ground so that all would pass a 100-
mesh sieve, but the fineness below that sieve was variable. Pressures
produced in the apparatus ranged from 3.7 to 11.5 pounds per square
inch. In general, the pressure increased with increasing volatile
content of the coals, but there were considerable variations. Certain
coals had a high moisture as well as a high volatile content, and the
moisture apparently caused a reduction in pressure when the dust
was inflamed.

The question of size of dust to use was important in view of tests
of five of the samples reground so that all passed 200 mesh; pressures
3 to 60 per cent higher were obtained. The effect of inherent mois-
ture was not determined quantitatively, and class 1 apparatus can not
handle dusts containing sufficient extraneous moisture to cause them
to cohere.

In a general summary Frazer
cites the following factors as in-
fluencing the order of relative in-
flammability: Amount and char-
acter of the volatile matter, ease
of its expulsion from the coal,
character of the source of igni-
tion, amount of moisture and ash,
and pressure prevailing in the
neighborhood of the flame. On
the last he was particularly in-
sistent, but it is not clear just
what influence the
pressure was sup-
posed to have; possi-
bly, its influence on the rate of chem-
ical reactions in-
volved was intended.
No other investigators seem to
have considered the pressure as
a factor in the combustion;
rather, they took it as a meas-
ure of relative inflammability determined by other conditions and
without reflex action on the inflammation itself. The other conclu-
sions cited have all been recognized in later work.

LATER EXPERIMENTS WITH COAL DUST

The balance of the work by Frazer was published in Bulletin 50. The
test method was standardized further, and all dusts were tested
at five different temperatures determined by currents of 5 to 7
amperes through the coil. Each sample was air-dried and ground
to pass a 200-mesh sieve. Curves showing the relation of pressure
developed to current in the coil were plotted for each of the 68

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samples tested. The inflammability of the coals was graded according to the pressures obtained at the highest temperature used, but a different order existed for each of the other temperatures. From the general shape of the curves one may expect that a still higher temperature would be needed to give a similar grading of the coals at two temperatures 100° apart.

It was thought that additional information might be obtained by plotting the pressures produced by the coals at the highest temperature against their volatile content when moisture and ash free in a manner similar to that used for plotting explosibility of coals tested in the Experimental Mine. The plot shows that, of two coals having equal moisture and ash content, the one having the higher volatile content gave the higher pressure; also, of two coals having the same volatile content, the one having the lower moisture and ash content gave the higher pressure, the differences being considerable in many cases. It was evident that no simple expression could be derived covering all the differences and that pressure produced varied with volatile content in a manner different from variation of explosibility of similar dusts as determined in the Experimental Mine.15

EXPERIMENTS WITH ROAD DUST

Frazer tested 21 road dusts gathered from 10 mines in five States. Their moisture and ash content varied considerably, but all except two were from mines working high-volatile bituminous or subbituminous coals. The higher the proportion of moisture and ash in the sample the lower the pressure obtained on inflammation. The data do not permit quantitative correlation of pressures with composition of the samples, but increments in moisture and ash content caused rapid reduction in pressure produced on inflammation.

EXPERIMENTS WITH MIXED DUST

The inflammability of dust of five coals was determined when pure and when mixed with 10 to 50 per cent of inert dust. The inert dust was shale in four cases and calcium carbonate in the fifth. Tests were made at all five of the standard temperatures, but the effect of the added inert dust was shown most clearly at the highest temperature; that is, with seven amperes passing through the coil. Each addition of inert dust caused a decrease in pressure with all of the coals, but the absolute effect varied from coal to coal. Reductions in pressure caused by successive increases of 10 per cent inert dust were irregular in three of the five cases, and the order of inflammability of the pure coal dusts was different from that obtained when there was 50 per cent inert dust in the mixtures. It is impossible to say whether the irregularities are to be charged to the test method or are to be taken as indicative of different behavior of the coals, and the matter is of minor importance in view of the changes made in the apparatus by Frazer’s successor, Clement.

EXPERIMENTS WITH DUST OTHER THAN COAL

Frazer tested 11 samples of miscellaneous dusts, comprising asphalt, wood, flour, starch, sugar, and lycopodium. Pressures obtained

15 Rice, G. S., and Greenwald, H. P., work cited, p. 18, fig. 2.
ranged from 7 to 11.4 pounds, except with the lycopodium, which gave 20 pounds. None of the cereal dusts gave as great a pressure as did Pittsburgh coal dust (11.4 pounds); and it is believed that this is an indication of poor dispersion of the dust in the explosion flask, as subsequent work has shown that sugar and starch are more inflammable than any coal dust. This idea is supported by the fact that the readily dispersible lycopodium gave a pressure much in excess of the others.

It may be noted that the bureau, from its inception, was interested in grain and cereal dust explosions. At that time (1910) the study of the explosibility of such dusts in the United States had been limited. Frazer worked on this problem as well as on the inflammability of coal dusts and designed an apparatus to study the oxidation of grain dusts under controlled conditions. There is record of tests of gluten dust sent from a mill at Granite City, Ill., in which an explosion on August 7, 1910, caused the death of two men and injury of seven others. 16

INITIATION OF ILLINOIS COOPERATIVE INVESTIGATION

In July, 1911, the bureau entered into a cooperative agreement with the Illinois State Geological Survey and the mining department of the University of Illinois under which a detailed study of the coal beds and mines of the State was to be made. 17 A study of the inflammability of all of the coals was included, and Frazer arranged this part of the program. After some preliminary work at Pittsburgh during September and October, Scholl established a laboratory at Urbana in November, 1911, and continued the work there. As the work was done mainly under Frazer's successor, Clement, details are given later.

SUMMARY OF FRAZER'S WORK

Frazer was the first investigator to study the inflammability of coal dusts under the program inaugurated by the Bureau of Mines. With the aid of one and, for a short time, two assistants he devised an apparatus for measuring inflammability, improved it, and collected a mass of information through its use. He also studied the inflammability of grain dust, a subject continued by his successor.

WORK DIRECTED BY CLEMENT

After Frazer's resignation the inflammability testing was directed by Hoffman for a period of two months and was then placed under the supervision of J. K. Clement, physicist, Hoffman taking up studies of the constitution of coal. Clement was in charge of all of the work in physics being conducted at the Pittsburgh station of the bureau and the apparatus used in recording the physical phenomena of coal-dust explosions in the Experimental Mine. Consequently, his connection with the inflammability studies was prin-

17 For a detailed statement of the aims and organization of this work, see Illinois Coal Mining Methods Cooperative Agreement, Preliminary Report on Organization and Methods: University of Illinois, Bull. 1, Urbana, 1913, pp. 43 and 67.
cipally to plan and supervise the work of his assistants. Scholl continued the cooperative work at Urbana until September, 1914, when he resigned—just before the investigation was completed. L. V. Walker, assistant physical chemist, worked under Clement at Pittsburgh during 1912 as did F. F. Rupert, junior physical chemist, from June, 1912, to October, 1913. Walker was succeeded by J. N. Lawrence, assistant physical chemist, who joined the staff in May, 1913, and resigned in October, 1915. The cereal-dust investigation was augmented in August, 1913, when D. J. Price, assistant mining engineer, was transferred from another section of the bureau to conduct field investigations. Beginning in February, 1914, he was assisted by H. H. Brown, organic and physical chemist. Part of this work was financed by the Millers’ Association of Buffalo until the fall of 1914, when it was transferred to the Bureau of Chemistry and Soils, Department of Agriculture, where Price and Brown developed a separate organization. Clement continued in direct charge of the bureau’s laboratory inflammability studies until June, 1916, when he was called to military duties. This ended his active work as a member of the bureau, and he resigned in April, 1917. The work done under his direction falls naturally into four divisions.

ILLINOIS COOPERATIVE AGREEMENT

The work done under the Illinois cooperative agreement was reported in Bulletin 102. More than 500 samples from 100 representative mines in the State were tested. Face samples of the coal and road and rib dust samples were collected in each mine. Full details of the methods of collecting and analyzing the samples are given in Bulletin 102.

APPARATUS

The apparatus used was the second developed by Frazer, described on page 6 and illustrated in Figure 1, with some modifications. The most important change was the use of a method of determining the temperature of the coil, so that tests were made at 100° intervals from 800° to 1,200° C. The currents required through the coil to produce the desired temperatures were determined and used during the experiments. It was found that ash deposited on the wire of the coil during inflammations of dust changed the calibration, and special means of checking it were employed daily.

RESULTS

Detailed data of all tests made were given in tabular form in Bulletin 102; these tables cover 13 pages. The results were also summarized by districts of the State. The primary intention was to divide the dusts into three classes: (1) Those which, in suspension, might be ignited by a flame from a blow-out shot or an oil lamp and initiate an explosion, (2) those which could not be so ignited but were capable of propagating an explosion started in a gas

mixture or in more inflammable dust, and (3) those which could not
propagate an explosion. The dividing lines were set by comparison
with similar laboratory tests of dusts of coals whose limits of ex-
plosibility had been determined in the Experimental Mine. The
experimental work on Illinois dusts was completed before the re-
quired data were obtained from the Experimental Mine; when these
data were obtained it appeared that any dust developing a pressure
of 0.5 pound or more in the laboratory apparatus might be ignited
by the flame of a blow-out shot, but certain dusts that developed no
measurable pressure in the laboratory apparatus were capable of
propagating an explosion. Evidently, the laboratory method was
not sufficiently sensitive to distinguish between dusts of Groups 2
and 3, and a modification was necessary, the details of which are
described later.

Face samples.—Dusts prepared from 95 face samples and ground
so that all passed a 200-mesh sieve were tested in this investigation;
they gave pressures ranging from 10.1 to 4.9 pounds per square inch
at a coil temperature of 1,200° C. An attempt was made to corre-
late the pressure developed with the volatile content as determined,
ash content, and B. t. u. value of the coals by the methods of least
squares, a straight-line relation being assumed. The agreement be-
tween computed and observed values was not satisfactory. Present
knowledge indicates that volatile, moisture, and ash contents are the
factors to be used, and volatile matter should be on a moisture and
ash free basis instead of as determined. Recalculation on this basis
gives fair agreement between computed and observed values of pres-
sure, but it is evident that the relation is a curve and not a straight
line and good agreement could be obtained only by use of a more
complicated mathematical expression.

Rib-dust samples.—Rib-dust samples produced little pressure when
all material passing a 20-mesh sieve was used; about 40 per cent of
those tested produced no measurable pressure at all, and 80 per cent
gave less than 0.5 pound. Different results were obtained when they
were ground to pass a 200-mesh sieve or when the portion of the or-
iginal sample through 200 mesh was sieved out. The inflammabilities
were much the same by either method. It was evident that the rib
dusts were much less inflammable than the pure coal dusts, as one
would expect from their higher ash contents.

Road-dust samples.—The road-dust samples were higher in ash
content and less inflammable than the rib-dust samples. Most sam-
ples that did not develop sufficient pressure to raise a 25-g weight
(0.9 pound per square inch) were not tested with smaller weights,
as it was not expected that any dusts developing so small a pressure
in the laboratory apparatus would propagate an explosion. The
fact that such dusts would do so was shown later by tests in the
Experimental Mine. Road dusts from one district were then tested
in the Clement-Frazer apparatus described below, and better gra-
dation of the samples was obtained.

SUMMARY

The tests made under the Illinois cooperative agreement showed
that the coals of that State produced dust that was highly inflam-
mable; and, as might be anticipated, the dusts in many Illinois mines were found to be inflammable. Such a result was foreshadowed by the explosions in mines of that State. Tests of large samples sent to the Experimental Mine later gave similar evidence.

The principal difficulty with the laboratory apparatus was in interpreting the results obtained, as the apparatus was not sufficiently sensitive to differentiate dusts of low inflammability, and modifications were required to overcome this. The lack of large-scale tests prevented recognition of this difficulty sooner.

DEVELOPMENT OF CLEMENT-FRAZER APPARATUS

The need of increased sensitivity led to modifications of the Frazer apparatus, and the new form has been commonly known as the Clement-Frazer apparatus. Details of the latter are shown in Figure 2.

DESCRIPTION

The principal alterations from the Frazer apparatus concerned the construction of the igniting coil, the pressure measuring apparatus, and the use of oxygen instead of air to inject the dust. The igniting coil consisted of a refractory tube, on which the platinum wire was wound, with a subsequent coating of alundum cement and application of a sheath of sheet platinum. A thermocouple was fused to this sheath. A Crosby gas-engine indicator was used to record the pressure.

The apparatus was arranged so that atmospheres containing combustible gas could be used if desired. The gas was injected through ports in a special fitting placed in the base of the explosion flask. The fitting appears in detail in Figure 2.

In operation a weighed quantity of dust was placed in the dust container and the apparatus was assembled. The coil was heated in a standard manner to the desired temperature and the dust injected by oxygen under a pressure of 15 to 20 cm of mercury, according to the quantity of dust used. The pressure shown by the Crosby indicator

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was corrected for the increase due to the injected oxygen, and that reported was the average of at least three determinations. More than three determinations were made when required for satisfactory duplication. The standard method of preparing the dust was to air-dry and grind it to pass a 200-mesh sieve.

When tests were made with combustible gas the heating element was brought to the temperature outside the explosion flask while the gas-air mixture was prepared in it. When all was ready the element was lowered and clamped in place rapidly and the dust injected. As this operation consumed not over 5 seconds, the composition of the gas-air mixture was not changed greatly prior to inflammation of the dust.

**STANDARDIZATION OF LABORATORY METHOD**

Five coals were tested at temperatures ranging from 750° to 1,200° C. to determine the best working temperature of the apparatus. The results are plotted in Figure 2 of Technical Paper 141 and show that the highest temperature was best.

The pressures developed by a wide range of mixtures of fine Pittsburgh coal dust and shale dust were determined next. The results obtained were used as a basis of comparison for stating the inflammability of other dusts when tested, as any dust could be said to have the same inflammability as the mixture of Pittsburgh coal dust producing an equal pressure on test. However, it was difficult to state the behavior of such a dust in a working mine. The method used is best described by the following quotation:

Dusts may conveniently be classified as follows:
1. Dusts that, when suspended in air, may be ignited by a flame and give rise to an explosion.
2. Dusts that, when suspended in air, can not be ignited by a blow-out shot, or burning gas, or by an electric arc, but may propagate an explosion set up in a cloud of more inflammable dust or in an explosive gas mixture.
3. Dusts that will not propagate an explosion.

The dividing line between these classes was set arbitrarily by the standard ignition and propagation tests of the Experimental Mine. It was possible, then, to classify a dust of unknown inflammability by determining the pressure it gave in the laboratory apparatus, selecting the mixture of Pittsburgh coal dust and shale dust that gave an equal pressure, and assigning it to the class in which this mixture fell.

**TESTS OF MIXED DUST**

The pressure-composition curves of four additional coals were determined by the method outlined above for Pittsburgh coal dust, and the propagation and ignition limits deduced from the results were compared with determinations made in the Experimental Mine. The propagation limits agreed within the error of interpretation of Experimental-Mine tests then allowed, but similar agreement was not obtained with the ignition test limits.

Tests were also made with up to 4 per cent of gas in the laboratory apparatus. These tests showed the effect of gas on the inflammability

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limit, a check on Experimental-Mine work. It may be noted that at that time investigators conducting large-scale tests in the United States and abroad did not agree that an increase in explosibility of dust was caused by small percentages of gas in the air current.\textsuperscript{22}

Since this work was done the number of coals tested in the Experimental Mine has increased to 46. It is doubtful if laboratory tests of all of these would have agreed with the relation established by the first five, but this question is less important than the fact that the fine or pulverized dust required by this apparatus is not the average dust found in mines and that the apparatus was not developed to handle the coarser dusts that were used in increasing proportion as the Experimental-Mine work progressed. No relation has been found by which the explosibility of one size of a given dust can be predicted from tests of another size in the Experimental Mine, although work along this line is now in progress, and recommendations for safety must be based on the results of tests of the size found in the particular mine in question. This difficulty was recognized early in the laboratory work.

The effect of gas in the air on the explosibility of coal dust has been studied so thoroughly by tests in the Experimental Mine that it can no longer be considered a suitable subject for laboratory experiment. Also, the classification of dusts quoted previously cannot be applied at all rigidly. The propagation and ignition limits found in the Experimental Mine have fixed meaning only under conditions obtaining there. Considerable alteration of the size or configuration of the passageways in which the tests are conducted will give different results with the same dust and source of ignition. Moreover, results of tests in which dust explosions have been initiated by strong gas explosions have shown that a still higher propagation limit may be obtained with Pittsburgh and some other coal dusts than is obtained in standard propagation tests.

TESTS OF COAL AND MINE DUST

As noted in Technical Paper 141, page 23, over 600 coals taken from mines in 21 different States and 2 Provinces of Canada were tested. The results obtained with 93 of them are given in Table 1 of that publication. It is stated that "no definite relation is to be found between inflammability and chemical composition. Usually, the higher the content of volatile matter the greater the inflammability."

Reexamination of the data of Table 1 in the light of present knowledge confirms the above statement in so far as a "definite" relation is concerned. Plotting pressure against volatile ratio gives a rough curve which is concave down and can be approximated by a formula involving the square root of the volatile ratio. Coals having a volatile ratio less than 0.10 were noninflammable. It should be noted that the curve of explosibility versus volatile ratio derived from Experimental-Mine tests and given in Technical Paper 464\textsuperscript{23} is of a different shape from that mentioned above. It rises

\textsuperscript{22} This disagreement was brought out in discussion of a paper by Rice, G. S., American Coal-Dust Investigations: Trans. Inst. Min. Eng., vol. 49, 1914–15, pp. 769–788.

more sharply for coals of low volatile content and becomes more flat for those of high volatile content. This shows that the definite or quantitative relationship of explosibility of pulverized coal dusts as determined in the Experimental Mine is different from their inflammability as determined by the Clement-Frazer apparatus, despite the fact that both methods may place them in the same order qualitatively.

Table 2 of Technical Paper 141 lists over 60 road and rib dusts tested in the Clement-Frazer apparatus. The pressures are lower than those developed by pure coal dusts of the same composition because of higher ash contents; and, with constant volatile ratio, the higher the ash the lower the pressure. This accords qualitatively with Experimental-Mine results. It is noted on page 25 of that publication that the original inflammability of these samples was increased an unknown amount by grinding them through 200 mesh when received in the laboratory. This point is, in fact, the principal objection to the Clement-Frazer apparatus, and it was not retained as a standard laboratory apparatus because coarse dusts could not be tested in it.

**LARGE-SCALE APPARATUS**

Mention should be made of an apparatus designed for studies of the effect of inflammable gas on the explosibility of coal dust on a much larger scale than the Clement-Frazer apparatus. The results were incomplete and have not been published previously.

The apparatus, constructed of standard 4-inch cast-iron pipe, is outlined in Figure 3. It was in the form of a closed circuit made up of two T's at the top and two L's at the bottom connected by short pieces of pipe with flanged joints. The inclosed volume was about 19.3 liters. A fan, A, supported by a shaft which passed through the
cover of the left T, gave a strong blast of air through the circuit designed to mix gas and air and put the coal dust in suspension prior to a test. There was a stopcock, B, near the bottom through which gas was admitted and an opening, C, for draining out water when the apparatus was cleaned by washing. Cleaning could be effected also by an air blast blown through cock B. On the right side were three openings. A heating coil or arc igniter was placed in the lowest opening D; the second one E was an observation window; and to the upper one F was fitted the dust injector. This injector consisted merely of a plunger passing along a tube in which the desired amount of dust was placed. Arrangements were made for sampling and analyzing the mixtures of natural gas and air used and the gaseous products of combustion. A connection on top of the right leg led to a Crosby gas-engine indicator used to measure the pressure developed by the explosions in the system. The apparatus was gastight, except at the packing surrounding the fan shaft, where some difficulty was experienced in producing a tight joint.

The first tests were attempts to obtain a dust explosion in the absence of gas, using an electric arc as the source of ignition, but trouble was experienced in maintaining the 6 to 7 ampere arc between brass electrodes against the strong current of air produced by the fan. A coil similar to the one used in the Clement-Frazer apparatus also was unsuccessful. An arc carrying 20 to 39 amperes between carbon electrodes was finally successful. The carbons were inserted through the upper flange and a hole in the side near the dust injector.

With this arrangement and a charge of 650 g of Pittsburgh coal dust per cubic meter small inflammations were obtained which gave pressures up to 3.5 pounds per square inch; but guncotton tufts 18 inches from the arc were not burned, showing that the flame was localized around the arc.

The next step was to determine the pressures developed in the apparatus by natural gas-air mixtures with no dust present. A mixture containing 4.43 per cent of gas failed to give an explosion, whereas with 4.98 per cent the pressure was 31 pounds. Pressure increased as the gas content was raised, reaching 82 pounds with an 8.35 per cent mixture, but it is noticeable that the pressure varied considerably for different tests that were duplicates in so far as gas content of the air was concerned. The pressure declined from this point to 15 pounds at 14.59 per cent of gas, and no explosion was obtained with 15.47 per cent of gas. The limits of inflammability thus established are practically the same as those recognized to-day for propagation in turbulent mixtures of this natural gas.

The next series of tests used mixtures containing 4.7 to 14.6 per cent of natural gas with sufficient coal dust in the apparatus to produce a dust-cloud density of 650 to 1,400 g per cubic meter. It does not appear likely that all of this dust was put in suspension by the fan. So far as can be determined from the limited data, the dust caused no greater alteration in the pressure produced by the gas explosion than was recorded in essentially duplicate tests with no dust present. The most definite finding was that a gas-air mixture containing 14.59 per cent of gas would not propagate an explosion in two trials with dust present whereas it had done so with dust absent, evidence of the absorption of heat by the dust that one would anticipate.
Additional experiments with small percentages of gas and 7 to 8 amperes in the arc gave better results. Coal dust in the absence of gas gave 20 to 30 pounds pressure; with 1 to 2 per cent of gas it gave 70 to 80 pounds pressure. What this apparatus might have done if further developed is, of course, pure speculation. It might have been useful in determining the concentrations of dust and gas required simultaneously to propagate flame when neither is in sufficient quantity to do so alone. On this matter there is still no reliable information.

TESTS OF GRAIN DUSTS

Studies of the inflammability of grain and cereal dusts begun by Frazer were continued and extended in Clement’s laboratory. The investigation developed principally after the explosion in a Buffalo, N. Y., feed-grinding plant on June 24, 1913, in which 33 men were killed and 70 more injured. Engineers of the bureau inquired into this disaster, after which the bureau entered into a cooperative agreement with the millers’ committee of Buffalo to study the subject of grain and cereal dust explosions. Dr. George A. Hulett, chief chemist, directed the cooperative work for the bureau, and the field investigation was made by Price and Brown, as previously noted; the laboratory work was reported by Clement. The results of this investigation were published in 1914.24 This report deals with the origin of the study, extent of the grain industry, investigation of recent explosions, history of explosions, origin and distribution of dusts, dust-collecting systems, and laboratory investigations. It closes with a discussion of causes and prevention of grain-dust explosions. The laboratory studies occupy about half of the publication. The work of Professors Peck and Peckham25 after the flour-mill disaster at Minneapolis in 1878 and that of Wheeler26 in England are reviewed, the latter in considerable detail.

The first work done in the bureau’s investigation was the analysis of a set of samples collected from various mills; this was done in the United States food and drug laboratory, Chicago, by H. H. Brown under the direction of Dr. A. L. Winton. The next step was the determination at Pittsburgh of the ignition temperatures and relative inflammabilities of these dusts. A survey and study of conditions in the industry were conducted at the same time.

Ignition temperatures were determined by a modification of Wheeler’s method and were probably accurate to 10° C. The results indicated that oat and yellow corn dust were the most ignitible, approached only by a sample of fine dust taken from the side wall of a wheat elevator. Flour dust had a considerably higher ignition temperature.

Tests to determine the relative inflammability of the dusts were made in the Clement-Frazer apparatus, using air to inject the dust.

The temperature of the coil was 1,200° C. The samples of dust were not of uniform size and this made the results less consistent. The oat and corn dusts gave the highest pressures, with wheat and flour dusts appreciably lower.

The investigation brought out forcibly the dangers, partly unrecognized until then, which accompanied the handling of grains and cereals. The following quotation is an excellent summary:

Although sufficient work has not been done to allow of any absolute statements, the results thus far indicate that all dusts that are made in the handling and working up of grain into food products can be ignited under proper conditions, and also will propagate a flame, most of them with explosive violence.

As the work progressed it became evident that a thorough investigation of all phases of grain-dust explosions required more time and a greater expenditure of funds than was possible under the agreement in force, and arrangements were made to transfer the work to the Bureau of Chemistry and Soils, Department of Agriculture, where it has been conducted since November, 1913. A large number of publications dealing with various phases of the work have been issued by Price and his associates. Since 1913 the Bureau of Mines has studied only the inflammability and explosibility of mineral dusts, but information has been exchanged and apparatus loaned from time to time; also, joint work with the dust explosion hazard committee of the National Fire Protection Association has kept the two bureaus in contact. Through the dust explosion hazard committee the bureau has participated in the formulation of recommended codes, which have been approved by the American Standards Committee and are designed to bring about safety in the handling of various combustible dusts in plants or factories. Coal dust other than that in mines is included.

**IMPORTANT POINTS OF WORK DIRECTED BY CLEMENT**

In attempting to point out the advances in laboratory studies made under Clement's direction one must remember that the work developed gradually point by point and received frequent setbacks when new results obtained in the Experimental Mine did not agree with the laboratory work. Frazer's apparatus was the basis from which a start was made. Improvements resulted in greater ease and rapidity of manipulation, and lack of sensitivity was overcome by the use of oxygen instead of air. It was then possible to grade fine or pulverized dusts in the same order of inflammability as that shown by Experimental-Mine tests; but quantitative agreement was obtained only partly, a fact that is not surprising in the light of present knowledge. Of greater importance was the inability of this apparatus to handle coarse dusts—up to 20 mesh in size.

An unfortunate result of progressive development of any research apparatus or method is that each new improvement casts doubt on the value of the results obtained before. Thus, the results obtained under the Illinois cooperative agreement are not as valuable as they would have been if made in the Clement-Frazer apparatus instead of the Frazer apparatus; however, this does not alter the fundamental conclusion drawn, namely, that the dusts in Illinois mines
were explosive, and the need of precautions against explosions was pointed out.

In November, 1915, it was decided that an entirely new line of attack was needed to obtain the results desired, and work was begun on the first laboratory gallery, a device that belongs in the class of experiments in open systems treated in the second part of this paper. Before this work is described some further use and modification of the Clement-Frazer apparatus will be noted.

**USE OF CLEMENT-FRAZER APPARATUS, 1916–1922**

From 1916 to the middle of 1922, a period of intensive work in attempting to develop an efficient laboratory gallery, the Clement-Frazer apparatus was used intermittently for a number of purposes. No changes were made in the method of handling it, and oxygen was used to inject the dust. The apparatus was used principally for auxiliary tests of large samples of coal received at the Experimental Mine from various parts of the United States for determining explosibility; tests of small samples of road and rib dusts and coal received from different parties who desired to know the explosibility thereof; and tests of special dusts concerning which information was desired, such as dusts from plants using pulverized fuel, lampblack, aluminum, gilsonite, zinc, and oil shale. These tests were usually paralleled by trials of the same material in the laboratory gallery in use at the particular time in question. If the tests in the Clement-Frazer apparatus were reported to the parties submitting the samples, the inflammability of the dust was given as equal to that of a mixture of pulverized Pittsburgh coal and shale that would give an equal pressure in the apparatus.

Use of the apparatus for dusts other than coal frequently showed its limitations. This was true to some extent in the tests of aluminum.\(^{27}\) It was necessary to alter the heating coil so that no platinum was exposed to prevent the hot aluminum from alloying with it. The thermocouple also had to be removed; the temperature of the coil was first determined for different currents flowing through it. The heat capacity of the coil was also too small to give satisfactory results when the ignition temperature of aluminum was wanted. Thus, a temperature above 900° C. was required in the apparatus, whereas a 6-inch diameter incinerating dish heated to 800° C. would ignite a cloud of aluminum dust blown across it.

**Mill dusts.**—Tests of dusts collected in mills using pulverized coal were made in connection with an investigation of explosion hazards in such plants and have been reported in Bulletin 242.\(^{28}\) They showed that dust—including partly consumed coal dust—commonly found in such plants was highly inflammable. The paper cited also gives numerous examples of fires and explosions caused by such dusts.

**Lampblack.**—Tests of lampblack or gas black were made at the request of a company using that material in its manufacturing processes and desiring to know what hazards accompanied that use. This material contained 6.1 per cent of volatile matter, 1.7 per cent of

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\(^{28}\) Tracy, L. D., Explosion Hazards from the Use of Pulverized Coal at Industrial Plants: Bull. 242, Bureau of Mines, 1925, pp. 18–22.

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moisture, and 0.2 per cent of ash. It failed to ignite in the Clement-Frazer apparatus, but inflammations were obtained in the laboratory gallery then in use. Some difficulty was experienced in producing a good cloud of the dust. A report of the work was published in the India Rubber World. 29

Gilsonite.—Tests of gilsonite were made at the request of a company mining this mineral. The sample received analyzed 87.4 per cent of volatile matter and 0.7 per cent of moisture plus ash. This material is highly inflammable and easily ignited, and explosions and fires have occurred in mines producing it. Laboratory experiments showed that it could be ignited by small flames, induction-coil sparks, sparks from the igniter of a safety lamp, and sparks produced by a loose brush on a small 220-volt direct-current motor. Gallery tests were made, as will be described later. An attempt was made to determine the ignition temperature of gilsonite in the Clement-Frazer apparatus; the coil had to be heated to 950° C. before ignition took place. On the other hand, a 6-inch incinerating dish heated to 600° C. caused ignition when a cloud of the dust was blown across it, another illustration of the unsuitability of the Clement-Frazer apparatus for this type of work. The conclusions were that the gilsonite was much more inflammable and explosive than any coal dust. The report of this work was not published.

Oil shale.—Tests of the inflammability of oil-shale dust were made at the request of the State Commission of Mines of Colorado. The shale received contained 37.3 per cent of volatile matter and about 60 per cent of ash plus moisture. Tests in the Clement-Frazer apparatus gave a pressure of 5 pounds, the same as that given by a mixture of 35 per cent of pulverized Pittsburgh coal dust and 65 per cent of inert dust. Laboratory-gallery tests and a more extensive investigation were made of the material at a later date. 30 It was concluded that the dust was not highly inflammable but would propagate an explosion under favorable conditions.

Zinc dust.—Tests of fine zinc dust were made at the request of a company manufacturing it and interested in hazards incident to transportation. It was ignited readily by the coil of the Clement-Frazer apparatus; other tests, including some in a laboratory gallery, fully confirmed its inflammability. Zinc dust, because of its weight, forms a cloud in the air much less readily than coal or other light inflammable dusts; but it also reacts with moisture with the production of hydrogen gas and heat, and fires may be started in this way.

WORK OF BOUTON

By July, 1922, laboratory galleries had been developed sufficiently to give some idea of what they might do and what their limitations were; details of this work appear later. It was evident that they were not doing all that was expected of them; whether they could do so or not was a matter on which interested parties disagreed. In any case, the investigation as a whole again needed a new line of attack. C. M. Bouton, associate research chemist, was transferred

from the Berkeley (Calif.) station of the bureau and took up a new branch of the work at that time, continuing it until his resignation from the bureau in May, 1927. He was assisted at different times by V. C. Allison, assistant chemist, and by J. Malcolm Pratt, J. H. Hayner, Garnet Phillips, and C. H. Gilmour, research fellows working on coal-mining investigations under the auspices of the Carnegie Institute of Technology, Bureau of Mines, and advisory board of coal-mine operators and engineers.

Bouton made a study of the problem and concluded that it could be attacked best by studying the time relation of pressure, temperature, and rate of chemical reaction.

DEVELOPMENT OF BOUTON APPARATUS

Bouton started with the Clement-Frazer apparatus but made so many changes that his device has been termed the “Bouton apparatus.” A time record of pressure development was obtained by use of a French-type manometer after experiments with a piezoelectric manometer were unsuccessful. Later a Bureau of Mines manometer was used.\(^{31}\) Experiments were made with a number of igniting devices with final decision in favor of an induction-coil spark. Several different methods of injecting the dust were tried. Because of the large number of changes made it is necessary to consider two forms of the apparatus.

**FIRST FORM, 1925**

Results obtained with the first form were published in 1925.\(^{32}\) This form \(^{33}\) differed from the Clement-Frazer apparatus in that air was used to inject the dust, the French manometer gave a time record of pressure, and the method of dispersing the dust was improved. At first the dust was dispersed by the method developed by the experimenters of the Bureau of Chemistry and Soils,\(^{34}\) but this was abandoned in favor of a device which gave a whirling motion to the air that picked up the dust.\(^{35}\) The number of operations during a test was so large that automatic control by electromagnets was required.

The weight of dust was that required to consume all the oxygen of the air present, namely, 100 mg, but combustion was less than one-fifth complete. The dust was injected by air at a pressure of 24.5 cm of mercury. The igniter of the Clement-Frazer apparatus was used at a temperature of 1,200° C.

*Preparation of dust.*—The investigation was confined to dusts of minus 200-mesh size which could be separated only by elutriation. Much time was spent on design of an air elutriator, and the earlier

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\(^{33}\) For a detailed description, see Bouton, C. M., and Hayner, J. H., work cited, pp. 10–15.


\(^{35}\) For details of this disperser, see Bouton, C. M., and Hayner, J. H., work cited, p. 13, fig. 5.
work was described in a cooperative bulletin. The final form was described in Bulletin 22 of the same series. Dust 0 to 10, 10 to 15, 15 to 25, and 25 to 74 microns in size was prepared. It was found that the elutriated fractions differed somewhat in composition from the original, probably because of separation of materials of different gravities.

Results of tests.—Tests of Pittsburgh dust with oxygen as the injecting medium gave the surprising result that the highest pressures were obtained with the coarser sizes. Repetition of the work with air gave different results, and air was used exclusively thereafter. It then appeared that the 10 to 15 micron size gave the highest pressure with both Pittsburgh and Pocahontas dusts, but there was a disturbing lack of agreement between duplicate tests except with the coarsest size (25 to 74 microns). In a third series the 15 to 25 micron size of Pittsburgh dust appeared the most inflammable.

Careful investigation led to the belief that the discrepancies could be due only to nonuniform injection and dispersion of the dust cloud after certain irregularities in assembling and operating the apparatus were eliminated. The difficulty of producing and maintaining dust clouds of uniform density was fully recognized, gravity and agglomeration being the disturbing factors, as shown by the following quotation:

The difficulty is an inherent one, but it is essential and, in the opinion of the authors, practicable to disperse a dust with such a degree of uniformity that small-scale apparatus will give consistent and convincing evidence as to the properties of the material used.

Bouton and Hayner also prepared a paper read before the meeting of the American Chemical Society in Baltimore in April, 1925. The results given above were discussed from a theoretical standpoint. The results of 10 experiments were selected, and the increase in pressure with increasing size was noted. Time from initiation of the experiment to maximum pressure was given; this time increased with increasing particle size. The average temperature rise required to produce the pressure recorded was computed and found to have a maximum value of 199° C. The quantity of heat required to cause the computed rises in temperature was calculated as ranging from 21 to 60 calories. Quoting work of Thornton it was shown that the actual energy liberated may have been double this amount, but then it was only 6 to 16 per cent of that obtainable by complete combustion of the quantity of dust used. Possible explanations of the results were then discussed. Apparently, this paper was not printed subsequently.

One may note in passing that, at the request of different parties, this apparatus was used to determine the inflammability of a number of dusts, among them dusts of high sulphur content and alloys of nickel and aluminum and copper and zinc.

37 See Bouton, C. M., and Hayner, J. H., work cited, pp. 4–6.
38 For details see Bouton, C. M., and Hayner, J. H., work cited, pp. 17–21.
During the 18 months ending in May, 1927, Bouton and his assistants made and tested a number of changes in the apparatus just described. The changes concerned the method of introducing and igniting the dust. It will suffice here to deal with the apparatus in its final form, as described in a report of investigations.\textsuperscript{40}

\textit{Description of apparatus.}—The apparatus is shown in Figure 4, and a detailed description of it appeared in Report of Investigations 2927. The dust-injection and ignition systems were greatly altered, also the explosion flask was evacuated before a test was made. The dust was placed in a special dispersion chamber $a$, from which it was blown by a blast of air confined in the reservoir $b$. Part of the

air was by-passed through a tube and rejoined the dust-laden air at \( d \). The stream was then divided, part entering at the top of the flask and part at the bottom. The tubes extended nearly to the center of the flask, and the opposing jets caused the dust to be thrown out sideways through a surrounding wire gauze. Ignition was caused by an induction-coil spark between the gauze and a spark point outside it. The various operations had to be nicely timed, and an automatic control \( e \) was provided. Pressure was measured by a Bureau of Mines manometer.

The early paragraphs of the report discuss the effects on dust dispersion of turbulence, agglomeration, and separation of different-size particles in eddies and show in what ways the apparatus tended to overcome these difficulties.

**Typical records.**—Figure 5 gives three typical curves obtained in this apparatus; they are reproduced from Report of Investigations 2927. Concerning them the report says:

The films measure 41 by 9 cm and travel in the direction indicated by the arrows at a rate of 1 cm in 0.021 to 0.023 second. Instead of films, paper coated with a special rapid "oscillograph" emulsion was used for most of the work and was preferred to films because of the sharpness of the black lines upon the white ground. The lowest line at the left of each record corresponds to the pressure in the evacuated flask. The instant of the entrance of the dust cloud into the flask is shown by a clearly marked bend in this pressure line, and the pressure rises at first almost linearly. The next line above is traced before the flask is evacuated and gives a base line at atmospheric pressure. The beginning of the inflammation is shown by another upward turn of the pressure line. Here there is no discontinuity but a rounding of the curve upward. The slope of the curve now becomes rapidly steeper, reaches a maximum steepness, and then decreases to zero as maximum pressure is attained. The maximum pressure is maintained for an instant and is followed by a gradual decline corresponding to the cooling of the gases.

The instant of ignition is shown on the records by a heavy dot near the upper edge; a series of lighter dots, controlled by the 50-cycle tuning fork, marks intervals of 0.02 second. The instant of ignition has been shown in the figure by the vertical line across the three curves, and every fifth timing dot only is reproduced.
Results of tests.—Better reproducibility of results was obtained with the second form. Six duplicate tests of Pittsburgh coal dust gave maximum pressures ranging from 41.4 to 42.8 pounds. There was always a lag on ignition ranging from 0.024 to 0.076 second. It recalled the similar lag on ignition found in certain gas mixtures, notably methane and air.\(^2\) It was hoped that the rate of pressure rise would give significant information on the ability of different coals to propagate explosions, but there was too much variation in the results of duplicate tests.

The fact that the pressure curves did not rise to a sharp maximum but had a horizontal portion at the peak was considered significant. Analogy was drawn to a similar phenomenon in gas explosions which has been explained\(^4\) as a readjustment of thermal balance between exterior and interior portions of the gas; but the analogy does not appear to be good at some points, and confirmation is needed before the theory is accepted for coal-dust inflammations.

The tests of segregated sizes of fine Pittsburgh coal dust were repeated in the second form of apparatus, and more consistent results were obtained. The finest size was the most inflammable and gradation with size was regular. Evidently the dust was not properly dispersed in the first apparatus. The authors concluded Report of Investigations 2927 with the following paragraph:

The experiments reported in the foregoing table were to have been preliminary to an investigation (1) of the effect of concentration of dust on its inflammability as measured by pressure produced, (2) of the intensity of spark necessary for inflammation, (3) of the amount of combustion as indicated by gas analyses of residual gases, and (4) of the effects of varying the size and kind of dust; but the separation of the writers from the Bureau of Mines has brought the present work to a close. The apparatus, as thus far developed, is here described as a promising tool for research, but its complicated nature and the unknown effect of various suggested mechanical improvements cause the writers to hesitate to suggest its use as an implement for testing the inflammabilities of various dusts.

This apparatus undoubtedly was complicated, and unpublished data show also that Bouton found it unsatisfactory for tests of dusts of low inflammability; this difficulty led the next investigator first to alter and then abandon it. Nevertheless, the apparatus was a considerable advance over the Clement-Frazer type, particularly in the thoroughness of dispersion of the dust, as is witnessed by the fact that Bouton using a given dust dispersed by air obtained pressures two to three times greater than those recorded in the Clement-Frazer apparatus with dust of the same coal dispersed by oxygen.

**WORK OF GRIFFIN**

After Bouton, H. K. Griffin, physicist, took up the study of the inflammability of dusts. He was assisted at different times by F. A. Hartgen, junior metallurgist; P. L. Golden, assistant scientific aid; and Donald L. Reed, research fellow, Carnegie Institute of Technology. The problem from May, 1927, to May, 1928, was to study the relative ignitibility and relative ease of flame propagation of suspensions of powdered coal and semicoke in air.


ALTERATION OF BOUTON APPARATUS

In using the Bouton apparatus for the above-mentioned problem the investigators encountered some difficulties that were overcome as they appeared. The system of operating the valves was revised so that it was more reliable. The apparatus as finally used by Griffin is shown in Figure 6. Details of the changes appear in a cooperative bulletin.43

The first tests of semicoke were to determine its relative ignitibility. It was found that it could not be ignited in either the Clement-Frazer or Bouton apparatus under the usual test conditions.

Preliminary work had to be done before tests of relative inflammability could be made. It was found that better reproducibility of time-pressure curves was obtained when secondary air was not used. A satisfactory source of ignition was found in a pellet made by compressing a mixture of magnesium powder and barium peroxide in combining proportions. The pellet was fired by passing an electric current through it. It was also found that better results were obtained with semicoke when air was used in the dispersion chamber at a pressure of 2 atmospheres gage. Inflammability of the semicoke decreased with increase of particle size. The differences between 0 to 5, 5 to 15, and 15 to 30 micron sizes were not large, but there was a wide difference between 15 to 30 and 30 to 74 micron sizes.

RESULTS OF TESTS OF COAL DUST

Effect of ignition ratio.—The ignition ratio was defined as the ratio of time interval between start of injection of dust and ignition thereof to the total time of injection of dust. The time of injection was taken as the time during which the valve in the inlet line was open. If ignition took place before the valve was closed the ratio was less than unity; if after, it was greater than unity. Tests of Pittsburgh coal dust were made with different ignition ratios; and it was found that maximum pressures were obtained when the ratio was approximately unity, but pressure rose most rapidly when the ratio had a lower value. Some difficulty was found in duplicating results when the ratio was low. A ratio of 1 appeared to give the best combination of desired characteristics in the time-pressure curves.

Concentration.—Pittsburgh coal dust was tested with concentrations ranging from 0.53 to 2 g per liter. The effect of change of concentration was not as great as that of change of ignition ratio; peak pressures did not vary greatly for the various concentrations used. The minimum and maximum concentrations that would give an inflammation were outside the range used.

Effect of secondary air.—Definitely better reproducibility of curve characteristics was obtained when no secondary air was used. The cause of this was not clear. There was no way of measuring the concentration of the dust cloud in the flask, and explanations of the effect found are conjectures.

Spark versus pellet ignition.—Blank experiments showed the heating effect of the pellet to be negligible as compared with the inflammation proper. The lag on ignition disappeared when the pellet was used, with some few exceptions. Pellets weighing 0.075, 0.15, and 0.30 g were tested, and no significant effect of size was found. The largest was adopted as a matter of convenience in preparation.

Comparisons of spark and pellet ignition were made with a Kanawha gas coal. When the pellet was used the rate of pressure rise was considerably greater, but the maximum pressure was somewhat less. It was suggested that the latter might be caused by uncontrolled change in the effective ignition ratio.

44 See footnote 43.
The results are discussed in much detail in the cooperative bulletin (see footnote 43), but the purpose of the present paper requires consideration primarily from the standpoint of the relative inflammability of coal dusts. The important point is the wide variation in results obtained by altering details of the method of operating the apparatus.

**OTHER WORK IN CLEMENT-FRAZER AND BOUTON APPARATUS**

There has been a small amount of additional work coincident with and subsequent to the work of Griffin. In 1927, Hartgen and Golden made tests in the Clement-Frazer and Bouton apparatus of a mill dust containing zinc and anthracite. Analysis of the dust showed 3.5 per cent of zinc and a residue after ignition of 42.6 per cent.

Tests in the Clement-Frazer apparatus with charges of 0.1 to 0.5 g and igniting-coil temperatures of 800 to 1,200° C. resulted in no inflammations. Tests were also made in the Bouton apparatus. Pittsburgh coal dust and dusts of aluminum, zinc, and a 50-50 nickel-aluminum alloy were tested at the same time. The coal and aluminum dusts gave about the same pressure—40 to 42 pounds per square inch. The alloy gave 1.0 to 5.5 pounds per square inch, depending on the concentration used; the highest pressure was obtained with a 3.8-g charge. Five grams of zinc dust gave 4.5 pounds pressure. The mill dust gave no pressure rise with charges up to 1 g. Evidently, it was noninflammable or of very low inflammability. This work was not published.

In 1928 Smith and Hartgen made tests in the Clement-Frazer apparatus on the inflammability of suspensions of pure soap dust in air. Examination under the microscope showed that the soap-dust particles were uniform in size and about 0.05 mm² in area. Violent explosions were obtained with them, and the minimum concentration giving inflammation was lower than that of Pittsburgh coal dust. Evidently, precautions against disaster are required in the manufacture of this product.

The Bouton apparatus has not been used since the work of Griffin, Hartgen, and Golden. The Clement-Frazer apparatus was used a few additional times to determine the probable relative inflammability of dusts, principally those other than coal.

**SUMMARY AND DISCUSSION OF EXPERIMENTS IN CLOSED SYSTEMS**

The various steps in the study of the inflammation of coal and other mineral dusts in closed systems taken over a period of 18 years may be summarized as follows:

1. Frazer was the first worker for the bureau; he based his apparatus on that of Bedson and Widdas. He tested a large number of coal and mine dusts and made some experiments with grain dusts. A study of the inflammability of Illinois coals was made in his apparatus under the direction of his successor, Clement.

2. Clement altered Frazer's apparatus to promote speed and ease of handling. As results of Experimental-Mine tests became available

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it was evident that those obtained in the laboratory did not parallel those in the mine, and the laboratory apparatus was made more sensitive by using oxygen instead of air in it. The results then agreed better, and a method of stating the inflammability of various dusts in terms of the inflammability of mixtures of Pittsburgh coal and inert dusts was worked out. From present knowledge it appears that the agreement with Experimental-Mine tests was qualitative. The apparatus could not handle dust much coarser than 200 mesh; this reduced the relative scope of the agreement, and the apparatus became of less importance as work in the Experimental Mine progressed.

3. Bouton endeavor to develop the Clement-Frazer apparatus into a research tool for studying the phenomena of dust explosions from a more fundamental standpoint. His work was stopped by his separation from the bureau.

4. Griffin, following Bouton, altered the Bouton apparatus to overcome mechanical difficulties and to obtain better agreement on duplicate experiments. He investigated the effect of a number of variations in method of operation on the time-pressure curves obtained. His opinion—expressed verbally to the author—is that the apparatus requires additional study and possibly modification before it can be considered entirely satisfactory.

One should recall here the original and still active purposes of the investigation; that is, to develop an apparatus by which the explosibility of a mine dust can be determined without tests in the Experimental Mine and which can be used for studies of factors not readily amenable to treatment in the Experimental Mine. It is evident that neither of these objectives was attained by the devices described. As to the first—duplication of Experimental-Mine work—the author believes that it never will be attained in an apparatus which uses pure coal dust and grades inflammability according to the pressure produced by the inflammation. There is no a priori reason why the pressure produced in inflammations of different coal dusts should bear a definite relation to the amount of added inert material required to prevent inflammation or explosion in those dusts, and the latter method is used in the Experimental Mine. Moreover, it is the basis of modern dust-explosion prevention in mines, where it is termed rock dusting. The experimental results show beyond question that if any relation exists between pressure produced on inflammation and amount of inert required to prevent it the relation is too complicated to have more than theoretical significance.

The Bouton apparatus was intended primarily for work along different lines. Duplication of Experimental-Mine results was not a factor in the matter, except as it might be derived from more fundamental studies. One hesitates to pass judgment on this apparatus at present. Certainly additional work and probably further modifications are needed. The results obtained by Griffin show how sensitive it is to variations in experimental conditions. The superiority of this apparatus over the Clement-Frazer as regards dispersion of dust has been cited, yet all evidence points to the conclusion that the variation in the time-pressure curves was governed primarily by the formation of the dust cloud which varied with different tests and different conditions. It is on this point that work is most needed.
Nothing is known of the actual density of the dust cloud at any point in the apparatus, and until this is found all of the work is based on suppositions involving the original weight of dust used and the volume of air in which it is dispersed. Information might be obtained by utilizing the obscuring power of the dust cloud on a powerful beam of monochromatic light; but the development of such a method will involve considerable calibration and reference to a standard based on weighing, which will also have to be worked out, so that it is a major problem in itself.

CLASS 2.—EXPERIMENTS IN OPEN SYSTEMS

The open system differs from the closed in that some part of it is continuously in free communication with the surrounding atmosphere, and any pressure developed therein causes ejection of gas and dust. There are two subclasses—galleries and furnaces. Pressure is not measured, and inflammability is graded according to the length of flame produced or the amount of added inert dust required to suppress flame or prevent extension of the flame given by the source of ignition. The term “furnace” is used here to designate any device in which inflammation is obtained by blowing the dust through a heated tube whose hot walls ignite the dust.

LABORATORY GALLERIES

The laboratory galleries used by different investigators were essentially attempts to reproduce on a small scale conditions in the test zone of the Experimental Mine. It is evident that the agreement between laboratory and mine results will depend largely on the degree to which test conditions in the latter were reproduced in the former. Unfortunately, such reproduction can be only a first and in some ways poor approximation. The work of the various investigators was a series of attempts to reproduce results, usually through alteration of such factors as were capable of close control.

WORK OF WHITE

The first work with a laboratory gallery was done by E. C. White, junior physical chemist, in 1916. At that time it was agreed by those interested that the Clement-Frazer apparatus was not giving a sufficiently close parallel to Experimental-Mine results, and the idea of a laboratory gallery was advanced as a new line of attack. White’s work was halted by his transfer to other duties, and his results have not been published hitherto.

TESTS IN IRON TUBES

The first experiments were made in a 5-foot length of 3-inch iron pipe, to one end of which was screwed, by means of a coupling, a cannon made from a cylindrical steel block. A hole 1 inch in diameter by 5 inches in depth was drilled along the axis of the block, and another one-half inch in diameter by 4½ inches in depth was drilled midway between the first hole and the periphery. The explosive used in the cannon was black blasting powder stemmed with clay.
The dust was placed either on the bottom of the tube or, more often, on a framework designed to represent the side and cross shelves of the Experimental Mine. Charges up to 10 g of powder stemmed with 10 g of shale were used in the central bore of the cannon, apparently without causing ignition of the dust. Ten g seemed to be the minimum charge that would sweep all of the dust from the shelves, but even 5 g of powder with no dust gave too long a flame to permit distinction between it and ignition of some of the coal dust.

Tests were then made with charges of powder placed in the small bore, which was close to the bottom of the gallery. Tests with a charge in each hole, the first acting as a dust raiser and the second as an igniter, were made with negative results. Finally, a 3-inch glass tube was used, so that the results could be observed; but the third test shattered the tube, and a different course was then pursued.

Tests in Wooden Gallery

A wooden gallery 8 feet long by 6 inches square inside and having walls 2 inches thick was constructed next, and a frame with side and cross shelves was made to fit in it. One end of the gallery was closed by a block pierced with a hole for the cannon. Charges of 5 and 10 g of black blasting powder were fired into loadings of 10 to 100 g of coal dust placed on the frame or on the floor of the gallery; two shots fired in quick succession were also used, but all results were negative. Moving the cannon inside the gallery was also without effect.

The gallery was then mounted on rollers in a frame and rotated at various speeds up to 30 revolutions per minute by a motor. The purpose was to promote formation of a dust cloud, but the highest speed attainable was not sufficient; the cloud of dust in the gallery was thin enough to be transparent throughout its length.

The next procedure was to place a paper diaphragm across the gallery about 2 feet from the closed end. Dust in the confined space was suspended by an air blast prior to the cannon shot; a very thick cloud resulted from this process. Dust was also placed on the framework representing shelves throughout the remainder of the gallery. Shots fired under these conditions tore out the diaphragm but did not ignite the dust beyond it. An electric arc between carbon electrodes was also tried in place of the cannon shot without success.

Finally, attempts were made to start an explosion by means of a pocket of natural gas at the face of the gallery. Mixtures supposedly containing as much as 30 per cent of gas were used. For some reason the gas would not explode when mixtures thoroughly stirred and presumably in the explosive range were used. Black-powder igniters and electric arcs were used in attempts to ignite the gas. Either of these is capable of igniting any gas-air mixture that is within the limits of inflammability. It follows that in no case did White have an inflammable mixture; otherwise he could not have failed to get a gas explosion. The gas probably leaked away in some manner not apparent either then or now.

Failure to get ignition of the dust was probably due to lack of a proper dust cloud. Laboratory galleries gave few signs of success until a mechanism for raising the dust was provided that was independent of the igniting source.
WORK OF LEIGHTON

Alan Leighton, assistant chemist, took up work on the laboratory-galley problem in May, 1917, and continued it with interruptions until September, 1920, when he joined the staff of the Department of Agriculture. He was assisted, beginning in February, 1919, by H. L. Lentz, assistant chemist, who left the service about three weeks after Leighton. Their work was interrupted frequently by intrusion of more pressing problems, particularly those having to do with war work. Hence, the actual time spent on the development of a laboratory gallery was considerably less than the above dates indicate. The work was not completed by Leighton and Lentz, and their results are published in this paper for the first time.

During the summer of 1917 preliminary work was done to determine the form a gallery should take. The first experiment was made in White's wooden gallery erected in a vertical position. Two and one-half ounces of pulverized Pittsburgh coal dust were placed in it, and a charge of 10 grams of black blasting powder was fired at the bottom. A violent explosion resulted which split the gallery into pieces. Flame was projected several feet from the open end. This result proved conclusively that a laboratory gallery would work and reduced the problem to a study of conditions required.

PRELIMINARY EXPERIMENTS

After this experiment a section of 3½-inch pipe, 10 feet long, was tried in both horizontal and vertical positions with little success. Experiments were also made in a glass tube 2½ inches in diameter by 6 feet in length to observe dust-cloud formation. There are records of tests in a new gallery 6 inches square by 10 feet long and in a gallery 18 feet long, the cross-sectional size of which is not evident. A study was made of the flame produced by different quantities of black blasting powder used in a number of ways, as well as of different methods of placing the dust and the effect of the powder shots thereon. It was decided that a gallery 14 feet long placed horizontally would give best results, and one was constructed.

HORIZONTAL GALLERY

Figure 7 is a longitudinal cross section of the 14-foot horizontal wooden gallery; its interior cross section was 6 by 6 inches. The sides and bottom were integral, and the top was a cover recessed into the sides and held in place by bolts passing through crosspieces
2 feet apart. There were similar crosspieces on the bottom; and the bolts, provided with wing nuts at the top, were fastened in these. The cover was removed between tests to clean the gallery and introduce fresh dust. Later, refrigerator fasteners were substituted for the bolts to speed operation. As first used the dust was placed in the gallery, but as this was not satisfactory a separate dust-dispersing system was added, consisting of a 1¼-inch pipe placed longitudinally under the gallery with ¾-inch risers into it at 1-foot intervals. On the top of each pipe and about one-half inch below the gallery floor was placed a steel disk which carried the dust. This appears at b in the enlarged section of Figure 7. Over this, on the gallery floor, was a slip of 10-mesh wire gauze (a in fig. 7) held in place with small strips secured in turn by bolts and wing nuts. A small cannon at the end of the longitudinal pipe was used to give a concussion, which threw the disks up against the wire gauzes, thus projecting the dust into the gallery. A shot from a second small cannon fastened to the end of the gallery or placed entirely inside it served as a source of ignition. The gallery was provided with glass-covered peep holes at 2-foot intervals along the side. Length of flame was determined by burning wisps of guncotton suspended in the gallery at 6-inch intervals.

Reproduction of Experimental-Mine results.—Having the gallery in this form, the next step was to reproduce results obtained in the Experimental Mine with Pittsburgh coal dust and rock dust of the sizes used therein; that is, so to arrange conditions that mixtures which propagated flame in the mine would also propagate flame in the gallery and that those which failed in the mine would likewise fail in the gallery. Factors that could be varied were the quantity of powder in and arrangement of the igniting shot, time from raising to ignition of dust, quantity of dust, tightness of gallery, presence of a valve in back to prevent formation of a vacuum behind the flame, and nature and amount of obstructions along the gallery.

The first trouble encountered was that explosibility limits obtained in the gallery were low; mixtures that propagated explosions throughout the test zone in the Experimental Mine would not propagate flame through the gallery. The first system tried to overcome this was to use larger charges of powder and place obstructions near the open end of the gallery. A valve opening inward was also placed at the rear. The net result of these changes was to materially raise the limits obtained. Later the obstructions were removed, and a charge of 5 g of powder laid loosely in the rear of the gallery was substituted for the igniting cannon. An interval of 1 second then elapsed from firing of the dust-raising cannon to ignition of the loose powder. The inward-opening valve at the rear was retained. At the close of the investigation it was stated that any mixture that was explosive under the standard ignition test in the Experimental Mine would give flame throughout the gallery. A mixture that would barely give propagation in the standard propagation test in the Experimental Mine gave 9 to 10 feet of flame in the gallery. The powder alone gave 6 to 7 feet of flame. An experienced investigator could judge the propagation limit of a dust within 5 per cent of that obtained in Experimental-Mine tests. That better results were not obtained was due largely to interruptions caused by other work.
It should be noted that difficulty was experienced from time to
time in duplicating results. The behavior of explosions in the gal-

lery sometimes changed, and it became evident that the alteration in
behavior was caused by change in tightness of the gallery. Leaks
sometimes occurred along the cover. Slight changes in retention or
release of pressure in a laboratory gallery affect markedly the results
obtained. This result is paralleled by results obtained on a larger
scale.46

Tests of various dusts.—Tests of various inflammable dusts were
made from time to time as explosions occurred with them in industry
or as they were regarded with suspicion by those who handled them. Among these were gas black, dusts
from mills burning powdered coal, petroleum coke, mixtures of petroleum coke and pitch, grain dusts,
sulphur-bearing dusts, and coals from a number of
mines. The work on grain dusts was done at the
request of D. J. Price, of the Department of Agri-
culture, who was in charge of that branch of the
work, as previously noted. Tests of coal dusts were
made in parallel with tests of large samples in the
Experimental Mine. The arrangement of the gallery
was not the same in all of these tests consequently
direct comparisons are not justified. The gallery did
show qualitatively whether or not explosions might
be expected if clouds of the dusts were exposed to a
sufficiently strong source of ignition; in fact, inflam-
inations were obtained in the gallery with all of the
dusts mentioned.

VERTICAL GALLERY

The success attending the first shot in a vertical
gallery was an incentive to further work along that
line, but it was the summer of 1919 before such a
gallery was used regularly. The one constructed was
much the same as the horizontal gallery, except that
its length was 10 feet. Figure 8 is a cross section
showing how the dust-injection system was altered;
the pipes holding the dust projected through the side
of the gallery at an angle of 30° from the vertical.
Figure 9 is a picture of this gallery in the laboratory.
Different sources of ignition were used, such as a
charge of powder in a pipe at the bottom of the gallery, a candle
flame, and an alcohol flame from a can of so-called solidified alcohol.

This gallery had some merit in testing dusts other than coal, par-
cularly when data on their sensitivity to ignition by flames were
wanted, but it did not show promise when used with coal dust.
Thus, the addition of 10 per cent of rock dust to pulverized Pitts-
burgh coal dust was sufficient to prevent flame traveling through the
cloud. The gallery gave information of value when used with

of Pressure on their Development: Safety in Mines Research Board (Great Britain),
Paper 14, 1926. See also Rice, G. S., and Greenwald, H. F., Coal-Dust Explosibility
Factors Indicated by Experimental-Mine Investigations, 1911 to 1929: Tech. Paper 464,
grain dusts and with such dusts as gilsonite, naphthalene, and zinc. A mixture of gilsonite and shale containing 70 per cent of the latter propagated flame throughout the gallery. Comparison of this result with that obtained with Pittsburgh coal dust gives some idea of its high inflammability. Pure zinc dust and naphthalene dust were ignited readily by candles in this gallery.

Objections to vertical galleries have been made on the ground that the convection currents caused by the inflammation will alter results obtained, and with dusts of non-uniform size segregation will be caused by gravity. The latter is true, undoubtedly, if a single charge of dust is dispersed in the gallery. One way to overcome this might be to inject dust continuously; the continued fall of the heavier particles through the lighter would tend to produce a mixture at any point approximating that of the original dust. Ignition would have to be delayed until this condition was obtained. A vertical gallery is, in some ways, similar to an elutriator, and size separation is avoided only with difficulty.

Figure 9.—Vertical gallery installed in laboratory
V. C. Allison, assistant fuel chemist, took up the work on laboratory galleries in October, 1920, after the resignation of Leighton and Lentz. He continued, with numerous interruptions caused by other work, until his own resignation at the close of 1920. A description of his apparatus, with a summary of the results obtained, was published in July, 1926.47

A few tests were made in Leighton's horizontal gallery; these led to the conclusion that an entirely new apparatus was needed, and one was constructed of steel pipe. The vertical gallery was used for a few tests but was not particularly satisfactory, and it was abandoned some months later.

The laboratory steel gallery was developed quite rapidly, and only the final form need be described, with mention of further study that occurred near the close of the investigation. Considerable data were available by the summer of 1922 and with additional work—totaling over 1,100 tests—was the basis of a general report finished in the summer of 1924 but not published. The paper referred to in footnote 47 was an abridgment of this report. The final work was an investigation looking to substitution of a photographic method of measuring flame length for the use of guncotton tufts and a study of the effect of different sources of ignition.

DESCRIPTION AND OPERATION OF LABORATORY STEEL DUST GALLERY

As seen in Figure 10, the laboratory steel dust gallery consisted of a steel pipe a 17 feet long and 8 inches in diameter. The dust was placed upon steel disks, seven-eighths inch in diameter, inside short vertical or riser pipes b below the gallery, which in turn were joined to a horizontal manifold pipe c extending lengthwise of the gallery. When in place the upper ends of the risers made tight joints against holes bored in the bottom of the gallery. The dust-injecting mechanism could be lowered from the gallery by a single operation when dust was to be put in place. There were 11 points of dust injection at 1-foot intervals beginning 1 foot from the closed end. Attached to the end of the manifold pipe was a steel bottle d 0.07 cubic foot in capacity, in which air was pumped to a pressure of 100 pounds. The air was released by a trigger valve e and threw the disk valves upward, discharging the dust into the gallery. This method of raising the dust by means of compressed air superseded the use of a small cannon attached to the manifold pipe.

The ignition mechanism was in the center of the breech of the gallery; it consisted of a chamber f into which gunpowder could be introduced and a standard, bolt-action 22-caliber rifle mechanism g by which a blank cartridge with the wadding removed could be fired into the gunpowder. Three automobile valves h, also in the breech, opened inward under slight tension to prevent formation of a vacuum behind the flame by cooling of the gases.

Length of flame in the gallery was measured by burning wisps of guncotton, suspended centrally at 6-inch intervals from a frame that

was supported at the top of the gallery and could be pulled out for examination and replacement of burned guncottens.

The open end of the gallery could be closed by a gas-tight parchment-paper seal when tests were made with inflammable gas in the atmosphere. In such tests metered amounts of natural gas were admitted; and the contents of the gallery then circulated through an external system which contained a motor-driven pump, a sampling tube, and the necessary valves. This system was connected to the gallery near the two ends. A sample of the mixture used in each test was taken for analysis.

When a test was to be made the requisite quantities of dust were placed on the disk valves, the manifold was raised and locked in position, and the igniting mechanism was loaded with 9.32 g of FFGG black powder and the blank cartridge. The air receiver was pumped up to pressure; and at the proper moment the air was released, the igniting shot being fired one second later. The gallery was cleaned with a circular wire brush and a swab after each shot.

Early in the work, an extensive series of tests was made to determine the proper time interval between raising and ignition of the dust. An accurate means of timing this interval was required, and the dust was raised by a shot from a small cannon placed at the end of the manifold with discharge of the cannon and ignition of the dust cloud controlled automatically.

RESULTS OF TESTS

Scope of investigation.—Allison investigated singly and between the limits shown the following factors influencing the length of flame in the gallery: (1) Quantity of dust injected, 0.034 to 0.873 ounce per cubic foot; (2) size of dust, four sizes, all through 20, 48, 100, and 200 mesh, respectively; (3) ash in mixture, 3.4 to 39.3 per cent; (4) water in mixture, 1.3 to 30 per cent; (5) natural gas in air, 0.83 to 7.70 per cent; (6) carbon dioxide in air, 0.95 to 33.2 per cent; (7) time interval between raising and ignition of dust, 1 to 60 seconds; and (8) temperature of room and gallery, humidity, and barometric pressure, over such ranges as occurred naturally during the test work. Some of these factors received more attention than others; also, factors 7 and 8 were found to have too little effect to justify their inclusion in the final correlation.

Method of correlating results.—When one of the factors mentioned above was varied between the limits shown a series of flame lengths was obtained. These were plotted against the corresponding values of the variable, and a curve was drawn through them which could be represented by some simple mathematical formula. When all of the individual formulas were available, they were combined into a single formula designed to give the flame length in terms of the values of all of the variables when active simultaneously.48

Various substitutions were then made, and a second formula was evolved which purported to give the percentage of added incombustible in a limiting mixture of a dust under standard propagation test conditions in the Experimental Mine; this value was in terms of the size, volatile ratio, moisture, and ash of the coal and the amount

48 For formula see Allison, V. C., work cited, p. 396.
of carbon dioxide and methane in the air. In general, one might say that the work consisted of developing a test method designed to duplicate results in the Experimental Mine and of constructing a formula to replace the test.

Duplication of results.—All of Allison’s work rested on two assumptions: (1) Extension of the flame of the igniting source used in the gallery was a measure of explosibility, and (2) the change in length of flame caused by any variation in test conditions could be correlated directly with that variation. The second assumption is not reasonable unless there is a satisfactory agreement of results in duplicate tests. It follows that a constant length of flame from the igniting shot was a prime requisite. The author has spent much time in studying the original data of the tests and is forced to conclude that the results of duplicate tests did not agree satisfactorily. The two following illustrations are cited to show this:

The wisps of guncotton used to measure flame length were placed at 6-inch intervals along the gallery. In 32 consecutive shots with no dust present the flame length from the igniting source ranged from 9.5 to 12 feet, or 20 per cent of the highest value. At least five tests would be required to give an average that would not differ markedly from the average of a second similar group, and any flame length reported with dust present would also have to be the average of five tests. Such averages were available in some but not all of the work.

The second illustration has to do with variation in length of flame with changes in the quantity of dust injected into the gallery. There were 138 shots in the series considered here, and the results obtained are shown in Figure 11,⁴⁹ which also gives the curve proposed as a correlation of them. It is seen that the variation in length of flame is very wide at all concentrations with which a large number of tests were made, and in several instances the results do not tend to group themselves about any one point. As all results are given equal weight it is permissible to take averages for each concentration used. This has been done for the concentration at which there were five or more duplicate tests, and the averages expressed to the nearest 0.5 foot appear in Figure 12. On the basis of this figure one would say that concentrations above 115 ounces per 1,000 cubic feet were without effect on the flame length.

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⁴⁹ For original figure see Allison, V. C., work cited, p. 397.
The rock-dust formula.—The formula developed to predict Experimental-Mine test results consisted of terms governed by variation in size and composition of the dust and composition of the air in which the dust was dispersed. All terms were of the first degree and directly additive. No such simple relations have been found in Experimental-Mine testing. As a further trial the formula was used by the author to compute the limiting mixture in 52 cases in which that limit had been determined in the Experimental Mine with an error not greater than 5 per cent. The agreement of computed and observed values was poor; in only eight cases was the discrepancy less than 8 per cent, and in 18 cases it was over 20 per cent.

One is forced to conclude that the formula has no value in predicting results of tests in the Experimental Mine.

USE OF PHOTOGRAPHIC FLAME RECORDER

Experiments in the laboratory steel gallery were resumed in the last half of 1926 to develop a method of photographing the flame to replace the use of guncotton tufts. A thin brass frame was hung in the gallery in place of the frame supporting the guncotton tufts. In this frame was clamped a strip of motion-picture film to which light had access through small holes at 1-inch intervals. It was necessary to exclude extraneous light from the gallery; this was done by placing a flap valve over the open end, so arranged that it would open outward when there was a slight pressure in the gallery. It was found that this valve reduced the length of flame given by the igniting source about 1 foot, as measured by the burning of guncotton tufts, that is, from 10.5 to 9.5 feet in succeeding experiments. The photographic method gave an average flame length of 5.3 feet, showing that guncotton tufts beyond that point had been burned by hot material whose temperature was below that required to produce a radiation that would affect the film. The result given by the film is more reliable; but seven tests gave lengths ranging from 4.0 to 6.5 feet, showing that the difference between duplicate tests had not been reduced and probably was due to variation in the burning of the powder.

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50 Allison, V. C., work cited, p. 402.
LABORATORY FURNACES

The first extensive study of the inflammability of coal dust in a laboratory furnace was conducted by Taffanel in France in 1910–11. The core of the furnace was 4.4 inches in length and 1.6 inches in diameter, and it was heated electrically. A cloud of dust was blown through the heated core by means of compressed air, and the volume of flame produced at the mouth of the tube was photographed. The photographs were divided into eight classes lettered A to H in ascending order of flame size. There was a fair degree of correlation between size of flame and volatile content of the coal. Studies of the effect of fineness and added incombustible were also made, as were tests of inflammable dusts other than coal.

This work was reviewed in some detail by Godbert of the Safety in Mines Research Board of Great Britain in a publication dealing with laboratory methods of studying the inflammability of coal dusts; this publication was a survey of the problem prior to his experiments at Sheffield, England.

Godbert’s work in developing a furnace for laboratory studies was published as Safety in Mines Research Board Paper 56. The furnace was similar in most details to the one described in the following section. Oxygen was used to inject the dust, and the amount of added inert dust required to suppress all flame was determined for each coal dust used.

This method appeared to be of value; and arrangements were proposed to have Godbert detailed to the Pittsburgh Experiment Station of the bureau as an exchange investigator under the cooperative agreement in force between the bureau and the Safety in Mines Research Board, in order that the method might be developed further and made applicable to American conditions. In the meantime, preliminary work was started at Pittsburgh by the author.

It may be noted in passing that bureau investigators had been using furnaces to study the rate of combustion of coal-dust particles, an investigation more in line with fuel studies than with safety in mines.

WORK OF GREENWALD

A furnace similar to the one used by Godbert was set up in the Pittsburgh laboratory early in 1930; Figure 13 is a photograph of it. The furnace a stands on a tripod and has a central tube 1 inch in internal diameter by 6 inches in length, with a nichrome winding to supply heat. The tube is centered in the transite ends of the outer shell, which is 5 inches in diameter and packed with diatomite. A regulating resistance b and ammeter are provided for control of the current. Temperature is measured by a thermocouple c inserted

from the bottom, whose junction rests close to the wall at the center longitudinally. The dust-injection system consists of a compressor and reservoir \( d \), a bulb \( e \) of 142-cm\(^3\) capacity with a mercury manometer attached, and a tube \( f \) leading to the top of the furnace. This tube is in two pieces, with a ground-glass joint at \( g \) to permit insertion of a small open-ended nickel boat holding the charge of dust. One boat \( h \) rests against a piece of transite which leans against the front leg of the tripod. The transite is used as a cover over the furnace between tests.

![Figure 13. Laboratory furnace used by Greenwald](image)

Preliminary experiments indicated that a furnace temperature of 775° and an air pressure of 85 cm of mercury in the glass bulb were satisfactory. In conducting an experiment the boat containing the dust was put in place, the air pressure adjusted, and the air released suddenly by turning tap \( i \). The blast of air blew the dust downward through the furnace. If the dust was inflammable, flame was projected from the bottom of the furnace. The size of flame was reduced, as addition of inert dust reduced inflammability, until finally only dust and possibly a few sparks were projected.

*Temperature gradient in furnace.*—It was evident that the furnace was hottest at the center, and exploration with the thermocouple gave the temperature curves shown in Figure 14. The hottest spot
was a little above the center, and the bottom was considerably cooler than the top. Sufficient time had to be allowed in heating the furnace to establish constant conditions throughout, otherwise results were apt to vary. This was particularly true if the furnace had been overheated. Sudden cooling produced a temperature of 775° over a much greater area, and limits of inflammability were increased.

**Size and kind of inert dust.—**A study was made to determine the best kind and size of inert dust to mix with the inflammable dust under test. The inert dust should have little tendency to agglomerate and should preferably be of the same specific gravity as the inflammable dust. While the latter property is desirable, it can not be obtained, as inert dusts are all heavier than coal or other organic dusts. Calcined fuller's earth was found to be best, and a segregated size—150 to 200 mesh—was used. The results obtained in the furnace are affected by changes in size of both the inflammable and inert dust, and these must be kept constant.

**Inflammability of Pittsburgh coal dust.—**Tests were made of Pittsburgh coal dust which passed a 150-mesh sieve and was retained on 200 mesh. Five trials each were made of mixtures containing 10, 12.5, 15, and 17.5 per cent of coal dust. The first two mixtures gave no flame; the third gave flame twice; and the last gave flame in every trial. The 15:85 mixture was close to the borderline of inflammability, and it appeared that there would be no difficulty in distinguishing between the inflammabilities of dust mixtures differing by 2.5 per cent of added inert material. Direct comparison with Experimental-Mine tests is not possible, because a segregated size of dust was used. From the above figures one would set the limit for this size of dust in the furnace at 86 per cent of added inert dust, as compared with 75 per cent for pulverized dust in the Experimental Mine.
The experiments showed that tests could be made in such a furnace easily and rapidly, that the agreement between duplicate experiments was as good or possibly better than that obtained in the Experimental Mine, and that limits of inflammability could be obtained with all desired accuracy. The furnace described was considered satisfactory for the routine testing of small samples of dust received by the bureau from time to time. A much more extensive investigation was required to determine whether tests in a furnace would duplicate mine results at all points, and one of the first things to be investigated was the ability of the furnace to handle dust as coarse as 20 mesh.

WORK OF GODBERT

Dr. A. L. Godbert, of the Safety in Mines Research Board, was assigned to the problem of developing a furnace at Pittsburgh under the cooperative agreement in effect between that organization and the Bureau of Mines. This work is in progress at the time of writing and will be the subject of a separate publication. A new and somewhat larger furnace has been constructed and has been found capable of handling dust as coarse as 20 mesh, and tests with coal dusts of different compositions are giving encouraging results. It is further proposed to determine the inflammability of closely graded sizes of Pittsburgh coal dust and mixtures thereof to determine what relation exists between the values obtained.

GENERAL SUMMARY

1. Early laboratory work conducted by the bureau was designed to obtain information on the explosibility of coal dusts in advance of the findings of Experimental-Mine tests and subsequently to devise a means of duplicating the results of such tests. The method used was to inflame small quantities of dust in a closed vessel and record the resulting pressure.

2. The results in the Experimental Mine did not agree with those in the laboratory, and a number of modifications of the laboratory apparatus were made to eliminate the differences. These were only partly successful, as the apparatus evidently could not deal with the coarse dust that was required to duplicate tests in the Experimental Mine.

3. Attempts to devise laboratory galleries simulating a mine passageway on a much reduced scale were not carried to a conclusion. The tests showed that there were a number of difficulties to be overcome, and the development in England of a furnace method opened a new line which was then pursued.

4. Tests in a laboratory furnace showed that it would prove satisfactory for the comparison of inflammabilities of small samples of dust. Further development of this method is in progress, and results obtained justify the prediction that this method will come nearer duplicating Experimental-Mine results than any methods which have preceded it. It can also be used to study the effect or inflammability of factors not readily subject to large-scale experiments.