

tractions were made with two 10 cc. portions of chloroform from 50 cc. of aqueous solution, made alkaline with 0.5 cc. of NH_4OH :

TABLE XVIII				
Wt. strychnine used	Total wt. found	Corr. for residue in chlor.	Corrected wt.	Percentage found
0.1594	0.1623	0.0015	0.1608	100.8
0.1594	0.1622	0.0015	0.1607	100.8
				Calculated, 100.0

Using 50 cc. of water, 0.5 cc. of NH_4OH , one 10 cc. portion of the 3 : 1 ether-chloroform mixture and 4 cc. of ether (allowed for saturating the water) for the first extraction and 10 cc. of the mixture for the second extraction, the following results were reached:

TABLE XIX				
Wt. strychnine used	Total wt. found	Corr. for residue in ether-chlor.	Corrected wt.	Percentage found
0.0638	0.0582	0.0010	0.0572	89.7
0.0626	0.0570	0.0010	0.0560	89.5
				Calculated, 90.8

We must conclude, then, that chloroform alone is to be preferred in this extraction. It is possible that some authors may have other reasons for using the mixture.

CONCLUSIONS

I—The distribution coefficients of a number of substances have been studied with a view to finding the best set of conditions under which to make extractions.

II—It has been shown that chloroform serves to extract aconitine and codeine from aqueous solution better than ether. Chloroform extracts strychnine better than mixtures of chloroform and ether, as suggested by many authors, while ether serves very satisfactorily for the extraction of cocaine alkaloid.

III—The distribution of citral between lemon oil and 45 per cent and 50 per cent alcohol has been discussed from this point of view.

IV—A study of the extraction methods for morphine has been made and none has been found to be short and accurate for the analytical chemist.

V—Under morphine, a use of the distribution ratio has been suggested to avoid laborious and continued extractions.

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VERMILION, SOUTH DAKOTA

LABORATORY AND PLANT

THE EXPLOSIBILITY OF GRAIN DUSTS¹

By HAROLD H. BROWN
Received October 8, 1914

As a result of a number of explosions in grain mills and industrial plants in this country and in Europe, and more especially as a result of an explosion in a feed-grinding plant at Buffalo, New York, in June, 1913, by which thirty-three men lost their lives and upwards of seventy were injured, a coöperative movement between milling interests generally and the Bureau of Mines was arranged for the purpose of making a scientific study of the explosibility of grain dusts, and of methods pertaining to the prevention of such explosions. The milling interests were represented in the conduct of the work by Messrs. Lawrence E. Harmon, President of Buffalo Cereal Company; Frank F. Henry, Manager Washburn-Crosby Company; and George P. Urban, Secretary George Urban Milling Company, all of Buffalo, New York.

This work was started August 1, 1913, being placed under the direction of Prof. George A. Hulett, Chief Chemist of the Bureau of Mines. David J. Price was assigned to the field-engineering work, and on February 1, 1914, Dr. H. H. Brown began a laboratory study of the problem.

During the preliminary study thirteen explosions were investigated which have occurred since 1905. Three of these took place in Iowa, three in New York, two in Illinois, and one in Vermont, Indiana, Kansas, Ohio and Texas. These explosions were classified among the various lines of milling as follows: Cereal mills, 4; elevators, 3; feed mills, 2; starch factories, 2; glucose factory, 1; flour mill, 1. It is reported that, as a result of these explosions, at least 78 men were

killed and 119 injured. The total damage to property exceeded \$2,000,000.

Since 1911 four explosions have occurred in Europe, two in dextrine works, one in a provender mill, grinding peas, beans, and wheat, and one in a linseed mill. As a result 47 men were killed and 119 injured.

In order to make a laboratory study of the problem, samples of the following dusts were collected, and the conditions under which they were produced were studied:

- 1—Dusts produced during the process of elevating and handling grain, and known as elevator dusts
- 2—Wheat-flour dusts from rolls, bolters, purifiers, conveyors, packing machines, etc.
- 3—Wheat-flour dusts from beams, rafters, elevator heads, etc.
- 4—Dusts produced during the cleaning of oats
- 5—Dust from grinding white corn
- 6—Dust from grinding yellow corn
- 7—Dust from grinding oat hulls
- 8—Oatmeal dust from packing machines
- 9—Floor dusts from elevator sweeping
- 10—Oat-groat dusts after aspirator

These dusts were first analyzed in the United States Food and Drug Laboratory, Chicago. Determination of moisture, ether extract, proteins, crude fiber, ash, and nitrogen-free extract or carbohydrates, were made, to ascertain, not only the chemical nature of the materials, but also wherein they might differ from the grains from which they originated.

Experiments were then started in the Bureau of Mines, Pittsburgh, to determine the ignition-temperature of these dusts, using the method of Wheeler.¹ This consisted in forcing the dust in a cloud through a

¹ Abstract of a Preliminary Report by David J. Price and Harold H. Brown, published by the Millers' Committee of Buffalo, N. Y.

¹ "Report on the Inflammability and Capacity for Transmitting Explosions of Carbonaceous Dust, Liable To Be Generated on Premises, under the Factory and Workshop Acts," 1913. R. V. Wheeler.

glass tube, 3 inches in diameter and 55 inches long, against a heated platinum coil, which was 15.75 inches from one end of the tube. The temperature of the coil was obtained by a Pt-PtRh thermocouple, having its hot junction within the quartz tube upon which the coil was wound. Using this method, Wheeler

While this work gave the relative ignition-temperatures it did not give the lowest temperature of ignition or the relative inflammability. This latter was determined by means of an apparatus developed in the Bureau of Mines. It consists of an explosion flask of about 1400 cc. (85.36 cubic inches) capacity having

two tubulures, a platinum coil, a device for driving a dust cloud against the coil, and a Crosby pressure-gauge for measuring the pressure developed. In each determination 50 mg. (0.00176 oz.) of the dust are forced in a cloud against the coil, which has been previously heated to a known temperature determined by a thermocouple. The temperature is that inside the coil and, therefore, higher than the actual temperature on the outside of the coil. The dust is ignited by the heated

coil and a pressure developed within the flask, which is registered by the gauge. The relative inflammability at any temperature is measured by the difference in the pressures developed within the flask.

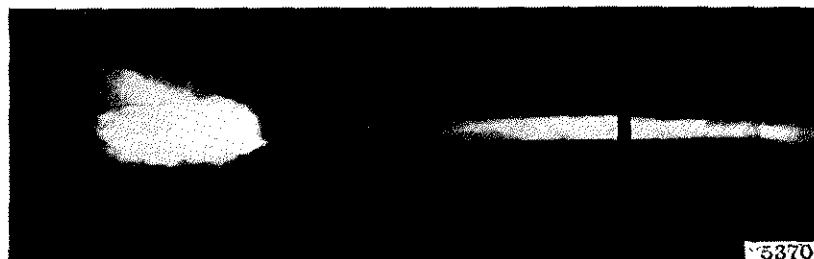
Determinations were made of pressures developed by the different dusts, as received and dried at 105° C., when they were forced against the coil heated to 1200°, 1100°, 1000° and 900° C. As no standard has been taken for carbonaceous dusts, other than coal dust, Pittsburgh standard coal dust, which is very constant in its properties, and which is used as a standard in the Bureau of Mines, was taken as a standard and all determinations run against it and checked against the average value obtained for it at each temperature.

Tables and curves are given which indicate that most, if not all, the grain dusts are more inflammable than Pittsburgh standard coal dust, higher pressures being developed in most cases, and especially so at the lower temperatures. The results also seem to indicate that the dusts from oats and yellow corn are more inflammable than those from wheat or other grain. However, the results are only very preliminary and it is possible that later work will change this supposition, and probably will change the curves, extending them to still lower temperatures.

It is interesting to note the difference in the inflammability of the dried and undried dusts. In nearly every case the pressure developed was appreciably increased after drying. Three are especially noticeable. These gave 0.5 pound pressure or less at 1200° when undried and over 8.0 pounds when dried, Pittsburgh standard coal dust giving 9.0 pounds at the same temperature. This is an indication of what may be expected when the humidity of the air is decreased.

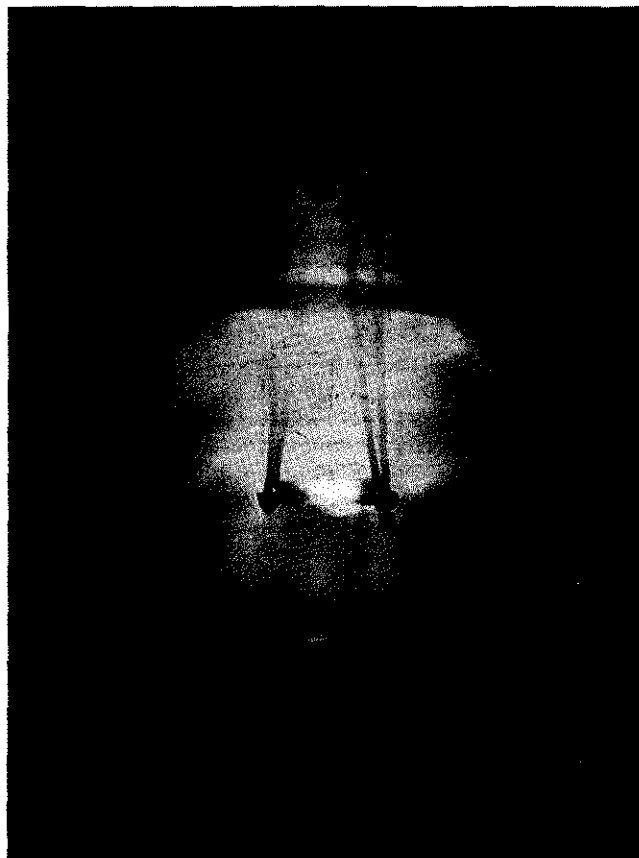
Experiments carried out by the Bureau of Mines¹ have shown that an explosion could be produced when there was only 0.032 ounce of coal dust suspended in each cubic foot of air, or one pound in 500 cubic feet of air. In the experiments of M. J. Taffanel, at the Lievin Experiment Station in France, in one instance as low a weight as 0.023 ounce of coal dust per cubic foot of space was sufficient to produce an

¹ "The Explosibility of Coal Dust," *Bull.* 20, Bureau of Mines, p. 102.



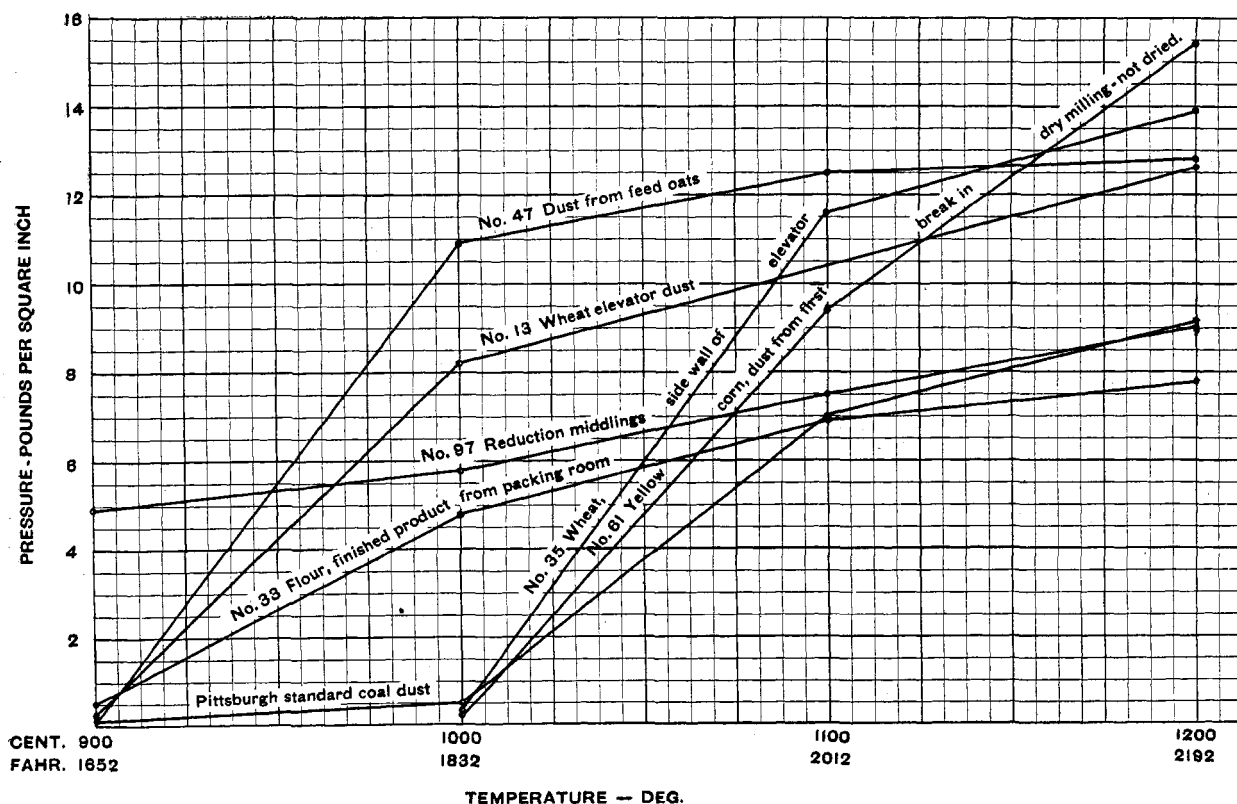
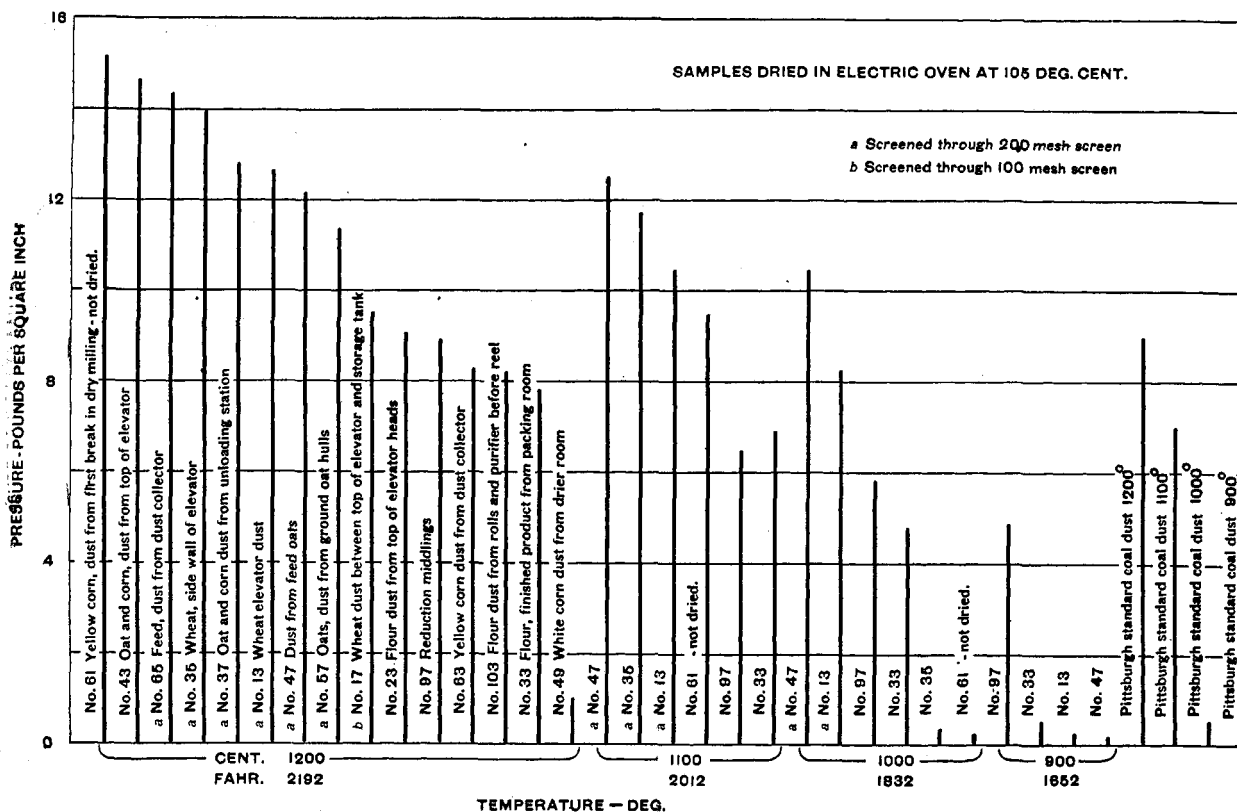
EXPLOSION IN WHEELER'S APPARATUS—No. 61, YELLOW CORN—1150° C. (2102° F.)
DUST FROM FIRST BREAK IN DRY MILLING, AS RECEIVED

determined the ignition-temperature—temperature of propagation—of many dusts, obtaining results varying from 805° C. for sugar, 960–1035° for starch, 990° for oat husks, 995° for grain (flour-mill), to 1060° for flour and 1100° C. for castor-oil meal. The results obtained upon grain dusts by the author varied



EXPLOSION IN BUREAU OF MINES INFLAMMABILITY APPARATUS
No. 35, WHEAT—PRESSURE, 11.6 POUNDS
DUST FROM SIDEWALLS OF ELEVATOR, DRIED

from 995° C. for oat and corn elevator dusts, 1015° for feed dust from dust collector, 1020° for ground oat hulls, 1025° for yellow-corn dust, to 1115° for wheat elevator dust and 1235°–1270° for flour dusts. Wheeler worked with samples which had been dried at 107° C.; the author used the samples as received from the mill.



ignition. Since preliminary experiments already conducted indicate that many of the grain dusts have relatively a lower ignition-temperature than many kinds of coal dust and are relatively more inflammable, it may be possible that an ignition of dust of this nature

might be produced with a smaller proportion per cubic foot than is necessary for coal dust.

During the investigations it has developed that the following causes have been assigned to many of the explosions in milling plants in this country and abroad:

1—Use of open lights, or naked flames, such as lamps, torches, gas jets, lanterns, candles, matches, etc.

2—Property fires.

3—Introduction of foreign material in grinding machines.

4—Electric sparks from motors, fuses, switches, lighting systems.

5—Static electricity produced by friction of pulleys and belts, grinding machines, etc.

The investigation has indicated that a large number of the recent explosions and fires have been caused by the introduction of foreign material into grinding

by those pulp mills using the soda or the "sulfate" process and by the alkali works has long been a serious problem for a number of the mills. In most cases it is not lawful to dump this material into the streams, hence the nearest available low ground is used as a dumping pond. These ponds have in many instances grown to alarming proportions, often using space that is needed for plant extensions.

Another and a more important phase of the problem is that this waste lime itself carries so much potential and actual value that it should not be thrown away after all. It is doubtful if in the average soda pulp mill practice the amount of alkali that goes to the dump will fall below $1\frac{1}{2}$ per cent calculated as sodium hydrate. This means a value of about 30 cents per ton. The average price paid for lime is probably \$4.00 per ton delivered into the plant. The actual ultimate cost of disposing of lime mud varies at different plants with conditions, but will not fall below 25 cents per ton in any case, all factors being taken into consideration. This waste lime, therefore, represents a possible value of not less than \$4.50 per ton to the average pulp mill or alkali works if burned back to quicklime. In other words, the mill can afford to spend \$4.50 per ton upon a recovery process and still break even.

Some two years ago the writer made a study of the problem simultaneously for a large alkali works and a pulp mill. Burning tests of their sludge were made in a 20-foot test kiln, and all available data were submitted to them. After due consideration, both of these companies have installed recovery plants which are now in successful operation.

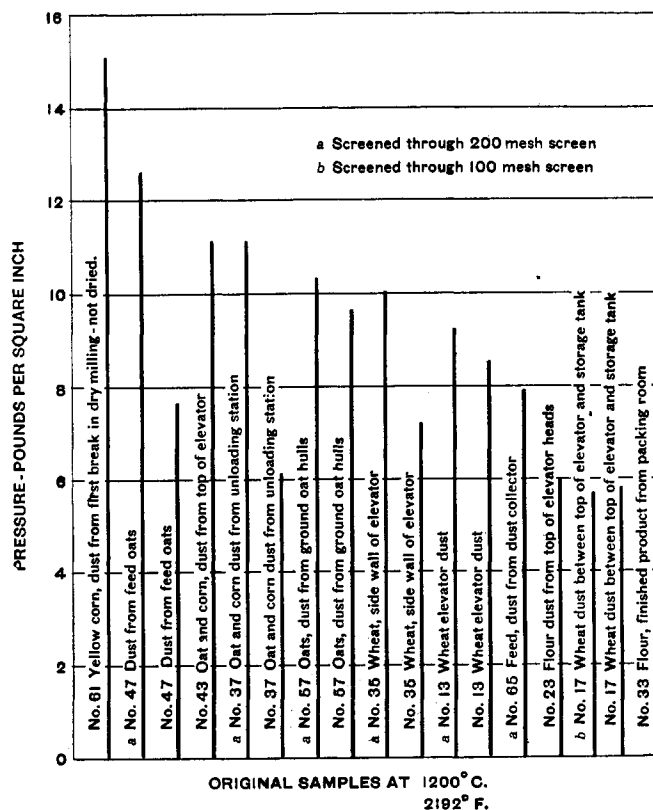
A brief summary of the facts in the case may be of interest to the readers of THIS JOURNAL.

STATE OF THE ART

About 1900, one of the large pulp mills installed a rotary kiln 6 ft. in diameter and 60 ft. long preceded by a rotary driver 4 ft. in diameter and 40 ft. long which used the hot gases from the kiln. A 10-ft. producer was used to furnish gas. The results were so unsatisfactory as regards output and fuel economy that the simple 4-ft. driver was later replaced by a Ruggles-Coles drier, with a fan at the outlet of the system to produce sufficient draft. This introduced a dust trouble so serious that the plant has never been able to operate successfully.

About the same time the Western beet sugar plants began to use the rotary kiln for re-burning their spent lime. The results in their case were entirely satisfactory and today a number of kilns are in operation. The size, at first 6 ft. \times 60 ft., has later been increased to 7 ft. \times 60 ft. and even larger. Beet sludge differs from causticizing mud in that it contains less alkali and is of coarser grain, so that filter cakes with as low as 35 per cent water can be produced. These plants use oil as fuel exclusively.

About 1905 one of the alkali works installed a rotary kiln 6 ft. \times 100 ft. for re-burning causticizing mud. The rotary was preceded by a tunnel system of drying the cakes which used waste heat from the kiln. After drying, the cakes were crushed to about 1 in. size and fed to the kiln. The tunnel system seriously inter-



machines. It would appear that a possible means of prevention would be to devise some system by which the foreign material might be removed before it reached the mill. Other preventions suggested are: a complete electric-lighting system, the use of portable electric lamps instead of lanterns or naked lights, the inclosing of the electric-light bulbs in strong wire guards or protectors, and the possible use of vapor-proof globes, and the locating of all fuses, switches, starting boxes, motors, etc., at points where no dust is present. It is also advised to have the receiving bins from the grinding machines as small as practical with the operations, as increased size gives increased space for dust clouds and, therefore, opportunity for a more violent and destructive explosion.

THE RECOVERY OF THE SPENT LIME FROM CAUSTICIZING OPERATIONS

By JAMES H. PAYNE

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The disposal of the large quantities of lime sludge that are daily produced in the causticizing operation