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NATIONAL BUREAU OF STANDARDS REPORT

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EXTINGUISHMENT EFFECTIVENESS
OF
SOME POWDERED MATERIALS ON HYDROCARBON FIRES

by

T. G. Lee and A. F. Robertson

NBS

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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NATIONAL BUREAU OF STANDARDS

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ABSTRACT

Laboratory methods are described for evaluating the effectiveness of powdered materials in extinguishing liquid hydrocarbon fires. These methods employ fire models 1 1/8-, 6-, and 22 3/4-in. diameter together with special equipment for controlled application of powder to the model fires. Extinguishment effectiveness was measured in terms of the minimum application rate required for extinction. The methods described were used to evaluate four different powdered materials of controlled particle sizes. Four additional materials were studied with the small model alone. The extinguishment effectiveness of the powders tested was found to depend upon (1) chemical nature (2) specific surface, and (3) particle size of the powder. On a surface-area basis, the most effective materials studied , potassium oxalate monohydrate and potassium iodide, were about four and two-and-one-half times, respectively as effective as sodium bicarbonate, the commonly used extinguishing agent.

The relative effectiveness of different powders did not change appreciably from one fire model to another. For a given powder and method of application, minimum application rate (surface-area basis), required for extinction of diffusion fires appears to be nearly proportional to the liquid burning rate of the fire model. Limited extinguishment data on large-scale fires of other workers seems to confirm the usefulness of the model method for evaluating powder effectiveness.

1. INTRODUCTION

In the search for a better understanding of the mechanism of action of powder-type extinguishing agents when used on burning liquids, a method has been needed to evaluate the effectiveness of various powders. This paper describes methods that have been developed for such evaluation, and presents results on several powdered agents, some of which were found superior to the powders now commonly used.

Following recent expiration of the patent rights covering dry chemical extinguishers, devices of this type have, because of their relatively high efficiency, found increasing use for control of flammable liquid fires. The extinguishing agent commonly used consists of about 96-98 per cent sodium bicarbonate in the form of a crystalline powder (0 to 50 micron

particle size), and a coating agent to improve free flow. It is discharged from containers varying in size from 2 to 1000-lb capacity by means of a compressed gas.

A review of literature in the field of combustion and flame extinction indicates the possibility of improving the effectiveness of powdered extinguishing agents. Although the mechanism by which powders extinguish flames is not well understood, it apparently involves breaking of chemical chain reactions by ions, and quenching by cold surfaces, as well as possible inerting by gases formed through decomposition. The ability of alkali halides to suppress gas explosions and to change the explosion-limit pressures of hydrogen-oxygen reaction has been studied by Hinshelwood and Willbourn. [1,2]*. Work by Jorissen, Snidders, and Vink[3], and more recently by Dolan and Dempster[4], on ignition and explosion suppression of the methane-air system showed that the specific chemical nature as well as the specific surface of the salt determines its effectiveness. Dufraisse, LeBras, and German[5], observed wide variations in salt effectiveness on small flames burning on premixed gases. Excellent reviews of the problem with literature compilations are given by Fryburg and by Friedman and Levy[6,7 & 8]. In general, the alkali salts with low decomposition temperatures were the most effective of those reported for inhibiting combustion reactions.

If these conclusions are applicable to full-scale (greater than 9 ft^2) gasoline fires, an improvement in the effectiveness of the present-day dry chemical ($NaHCO_3$) extinguisher by a factor of four or five is a distinct possibility. However, in extinguishing large-scale, free-burning liquid hydrocarbon fires, numerous factors are involved that do not appear in small flames burning premixed gases. For example, the problem of dispersing the powder and maintaining a sufficient concentration of powder in the flame zone is a critical one. No studies are available on the influence of such factors as chemical composition, particle size, discharge rate, and nozzle shape on extinction effectiveness. Nozzle shape of recent portable extinguisher models vary from a circular opening to a long, narrow slit, from diverging discharge to straight stream, depending, apparently, only on preference of the manufacturer.

McCamy, Shoub, and Lee[9]have reported the use of salts other than $NaHCO_3$ on field-scale fires. They indicate, qualitatively, that for a heptane fire burning on a 4 x 4-ft area, particle size and dispersibility of the powders influence their effectiveness. The powders used were mainly inert and poorly dispersible. Neill[10], recently reported that $KHCO_3$ is about twice as effective as Dry Chemical ($NaHCO_3$) on 3 ft square fires when applied at minimum critical rates. For fires up to 10 ft square with powder application at a given constant rate, the same order of effectiveness held for extinguishment on an area basis.

*Figures in brackets indicate literature reference at the end of this text.

Obviously, only a slight improvement in extinguisher effectiveness may provide the difference between success and failure in controlling a fire. Thus, a reliable laboratory method is needed to measure the extinguishment effectiveness of powders and to evaluate the factors upon which this effectiveness is based. A good method should be a small-scale one but should give reproducible results that can be correlated with those of much larger fires. The methods described in this paper appear to meet these requirements. These involve the extinguishing of liquid heptane fire models of 1 1/8-, 6-, and 22 3/4-in. diameter.

A measure of the effectiveness of any extinguishing agent can be obtained from (1) the minimum rate of application required to extinguish a standard fire, or (2) the time required for extinction under a given rate of application to a standard fire. The first method forms the basis of reporting efficiency here although both types of measurements were made.

2. MATERIALS INVESTIGATED

Table 1 lists the powdered materials that were tested and the preparatory treatment they received. All powder samples, with the exception of glass beads and dry chemical, were made from reagent-grade chemicals by grinding in a pebble mill. Two per cent of zinc stearate was added before grinding to most samples to improve their free-flow properties and to reduce moisture absorption. The powders were separated into fairly narrow fractions according to particle size by sieving and air elutriation. Beyond this, no definite tolerance was placed on particle size distribution. Measurement of particle size distribution of some samples by microscopic method showed that the cutoff was sharp on the large particle side of the distribution curve. Mean particle size was measured with a Fisher Subsieve Sizer based on the air permeability principle. The glass beads used were spherical and had a very narrow particle-size distribution.

3. EXPERIMENTAL PROCEDURES

3.1 One-inch Diameter Fire Model

The procedure used with the 1 1/8-in. diameter fire model was simple in principle and required only about 0.5g of powder for each run. Powder was dispersed in the air at a controlled rate while a small diffusion flame, supported by n-heptane liquid in a cup, was slowly introduced into the freely falling powder stream. Various rates of powder discharge were used to determine the minimum effective rate that would cause extinction.

Figure 1 shows the essential features of the apparatus. The dispenser consisted of a cylinder containing a stainless steel screen at mid-height to support the powder. The rate of discharge of powder was controlled by adjusting the amplitude of an electromechanical vibrator. A brass cup with an inside diameter of 1 1/8 in. was used as a burner. In use, the cup contained 1.0 ml of n-heptane floated on 3.0 ml of water, with fuel surface initially about 1/32 in. below the rim.

The burner was placed on a flat carriage which moved at a uniform velocity of 1.9 in./sec toward the powder stream. An identical but empty cup was placed on the carriage 2 in. ahead of the burner cup. The weight of powder collected in this cup served as a measure of discharge rate. A recording densitometer monitored the discharge stream for variation in the rate of discharge. The entire apparatus was enclosed in a draft-free cabinet. The fuel was ignited by a spark discharge and the flame was allowed to burn 35 sec before the carriage moved it into contact with the falling powder stream. At least 20 runs were made with each powder sample.

3.2 Six-Inch Diameter Fire Model

For the 6-in. diameter fire model, powder was propelled by air into a flame burning in a circular, stationary pan (Fig. 2). The powder dispenser involved use of a serrated rotor within a housing. Powder was fed to the moving rotor from a hopper at the top of the housing. A high-pressure stream of air directed through the bottom of the housing stripped the powder from the rotor serrations and carried it out through a nozzle. The powder discharge rate was governed by the speed of the rotor. In this way, uniform, reproducible discharge rates were obtained.

A solenoid-operated powder diverter, through which the powder was removed by means of a vacuum system, was used to limit the duration of powder application and was found necessary to reduce the effect of starting and stopping transients. Timing was controlled by a clock-driven stepping switch.

For the purpose of measuring the rate of powder discharge, the diverter was fitted with a cellulose filter thimble and used to collect powder for a measured period of 2 to 3 sec. The average discharge rate for a given dispenser speed was then calculated from the weight of powder collected in the filter and the time period of collection.

The flame was supported by 30 ml of n-heptane floating on water in a brass cup having an inside diameter of 6 in. A 12-in. diameter horizontal flange attached to the rim of this container was found desirable in stabilizing the flame. A distance of 1/8 in. was left between the fuel surface and the top of the flange. The flame was allowed to burn for 45 sec before the powder was applied.

Compressed air at a flow rate of about 1 ft^3/min was used to propel the powder. The powder was first discharged into the diverter for about 3 sec before it was directed into the fire. The distance from the nozzle to the center of the pan was 14.5 in.

The powder was aimed parallel to and slightly above the surface of the pan and flange. The nozzle provided a horizontally flat and diverging discharge pattern. The cross section of discharge was about 6 in. wide and 1 in. thick at the reagion near the fire. In reporting powder

application rates for the two large fire models no correction has been made for powder which did not enter the fire zone during application.

During each series of runs the discharge rate was varied stepwise from one run to the next and the time for extinction was determined in each case. As the discharge rate was decreased, the extinction time increased until a minimum discharge rate was obtained, below which extinction did not occur. Extinction time was taken as the period between arrival of powder at the flame and the time when the flame had decayed to 20 per cent of its initial value as recorded by a lead sulphide infrared detector located behind the fire.

3.3 Twenty-three Inch Diameter Fire Model

The apparatus and procedure for the 22 3/4-in. diameter fire model were similar to those used with the 6-in. model except:

(1) The distance from the nozzle to the center of the fire was 36 in.

(2) The air flow rate was about 7 ft^3/min providing a discharge cross section of about 26 x 4 in. at the center and above the fuel pan.

(3) A flange of 37 in. diameter was used around the top of the fuel pan.

(4) A freeboard of 1/2-in. was used above the fuel surface.

(5) Three hundred ml of practical heptanes (Eastman Kodak P-2215) was used as the fuel.

(6) A preburn period of 30 seconds was used.

Figure 3 shows the equipment being used for extinguishment of this large fire model. The photometers were not being used when this photograph was made.

4. RESULTS

4.1 One-Inch Diameter Model

Figure 4 presents data on minimum rate of powder application for extinguishment of the smallest fire model by various powders as related to mean particle size. Each point is plotted as a cross in which the vertical line represents the region of overlap where both extinction and non-extinction results occurred during the 20 runs. All runs at discharge rates above the top of the vertical line resulted in extinction; ONLY ~~all~~ non-extinctions occurred below the lower end of the line. The point at which the horizontal and vertical lines intersect indicates the best value for the minimum effective rate as determined by a statistical formula developed by C. S. McCamy [11].

This formula may be stated as follows:

$$W_m = a + \frac{x}{x+y}(b-a) \qquad \text{where:} \quad x = \sum_{n=1}^{j} (W_n - a),\ a < W_n < b$$

$$y = \sum_{e=1}^{k} (b - W_e),\ a < W_e < b$$

and, W_m = minimum effective rate

W_e = rate at which extinction occurred

W_n = rate at which non-extinction occurred

a = rate at or below which there were no extinctions

b = rate at or above which there were no non-extinctions

j = number of non-extinctions in interval between a and b

k = number of extinctions in interval between a and b

The values of a and b were experimentally determined and, in this case, were based upon 20 runs. All data were on a weight basis.

Hird and Gregsten/12/have shown that extinguishment behavior of $NaHCO_3$ powders of different sizes may be largely correlated when presented in terms of surface area of the powder particles. Accordingly, the curves of Figure 4 have been converted to a surface area basis on the assumption that the particles are spherical in shape and the Fisher Subsieve Sizer measurements are applicable. The resulting curves are presented in Figure 5.

It will be evident on inspection of this figure that properties other than surface area are important in determining effectiveness of the powders used. During the experiments it was observed that particles on the order of 5 micron or less were lifted by the hot gases above the fuel and in some instances this effect prevented extinguishment from taking place. The differences observed between the $NaHCO_3$ and "dry chemical" curves may be a result of dispersibility and particle shape differences perhaps resulting from different preparation techniques/13/.

Figure 6 shows the effectiveness of mixtures of 22-μ glass beads and 8-μ potassium oxalate monohydrate at various weight ratios. The experimental results are plotted as crosses in which the horizontal and vertical lines have the same significance as in Figure 4. These plotted results may be compared with the curve shown, which was calculated from the minimum effective extinguishment rate for 100 per cent 8-μ oxalate on the assumption that the glass beads have no effect in extinguishing the fire. (Thus, for example, the calculated minimum effective rate for the 50-50 mixture is twice that for the 100 per cent oxalate). These results seem indicative that an inert carrier with larger mass could be used to introduce a finely divided and chemically effective agent into the desired zone of the flame. Due to surface forces, the fine particles adhere firmly to the carrier, at least prior to its introduction into the flame. The higher momentum acquired by the much larger mass of the carrier would be able to overcome the buoyant forces created by upward draft above the combustion zone.

4.2 Six and Twenty-three Inch Diameter Fire Models

Figures 7 and 8 show the time to extinguishment as a function of application rate expressed on a surface-area basis. Various powders were used in these tests with 6-in. and 23-in. model fires. To avoid overcrowding, the plotted points in Figure 7 are shown only for the sodium bicarbonate curve. In general, these data seem similar to those of Hird and Gregsten [12] and show that for a given powder, extinction time increases as the discharge rate decreases. Eventually, the point of non-extinction is reached. At very high discharge rates it appears that the extinction time approaches a constant.

Table 2 presents summary results for the three fire model studies and each of the four powders tested. The results are presented in terms of minimum powder application rate on a surface-area basis, as well as in relative application rates with reference to $NaHCO_3$ as a reference material. Also shown is the fuel burning rate for each of the fires used. This latter shows fuel consumption rates roughly 60 per cent of those reported by Blinov and Khudiakov [14] for gasoline burning in deep fuel pans. When minimum $NaHCO_3$ application rates are divided by fuel consumption rates, the quotient is found to vary by a factor of 2.7 over the very wide range of fire sizes.

5. DISCUSSION

The results show that the fire extinguishing effectiveness of the powders tested depends on the physical and chemical nature of the powder. Thus, potassium oxalate monohydrate was more effective than sodium bicarbonate or potassium bicarbonate in all three models; and potassium iodide was about 2.5 times as effective as sodium bicarbonate in the two larger models on surface-area basis. However, the relative effectiveness appears to be somewhat influenced by method of powder application.

It was also found that, within a limited range of particle sizes, extinguishment effectiveness is a function of specific surface of the powder. For particles of smaller or greater size, buoyant or gravitational forces may limit their introduction and retention in the flame zone.

Figure 5 shows that, for a given powder, an optimum range of particle sizes exists for which the minimum powder application rate is effective for control of the 1 1/8 in. fire model. This range is between 15 and 20 microns for many of the powders tested. Although sufficient data were not obtained to confirm the existence of this behavior in the larger fires, a somewhat similar effect should be assumed. In this 1 1/8 in. model, powder particles approach the flame at or below the terminal velocity of free fall. Hence, the extinguishing behavior of the powders may be more sensitive to particle size, shape, and density than in the other models

where powder was propelled into the fire. The higher density of KI for example, appears to improve its effectiveness in this model. As particle size increases above the optimum, residence time in the flame zone shortens, the surfaces may not attain the decomposition temperature, and/or the total heat absorbed is less, thus lowering the efficiency. The generally flat shape of the potassium oxalate curve over a wide particle size range and for the smallest fire model may provide an indication of more rapid extinguishment action than achieved with bicarbonate salts.

One method of overcoming the poor physical behavior of fine powders is to employ a carrier to introduce a very finely divided yet chemically effective material into the flame zone. Reference to Figures 4 and 6 shows that a mixture consisting of glass beads and only 8 per cent 8-u potassium oxalate could be considered equivalent to the normally used 22-u dry chemical. Besides increasing effectiveness, proper use of a carrier may reduce toxicity hazards.

Figures 7 and 8 provide a good illustration of the need for use of minimum application rates in measuring relative effectiveness of different agents. Thus, it would be difficult to distinguish between effectiveness of different powders in the field if high discharge rates were used which resulted in extinguishment times being close to the minimum shown in these figures.

Individual points on Figures 7 and 8, and vertical lines on Figures 4 and 6 indicate the magnitude of the error. Since the methods developed are primarily for comparative evaluation, no analysis of error was made. However, the discharge reproducibility of the rotor dispenser, as measured at the nozzle, was within 3 per cent for all free-flowing powders used.

Table 2 presents results on the ratio of $NaHCO_3$ application rate to fuel combustion rate. Similar results for the other powders could have been presented. While this ratio does not appear to be a constant, the range within which it varies is on the order of 2.7 for all the powders studied. It thus becomes evident that in spite of the very wide range of fire sizes and powder application rates used there appears to be no drastic change in the character of the fire or the mechanism of extinguishment. It thus appears that the minimum powder application rate required for extinguishment is roughly proportional to the rate of fuel consumption.

This latter fact may perhaps be more clearly illustrated by the data shown in Figure 9. Here the extent to which the burning rate curve is parallel to the minimum application rate data serves to indicate the degree of proportionality.

In considering these results, it should be remembered that different techniques were used for powder application. For the 1 1/8-in. model, powder was applied to the flame under a condition of free fall. For the 6-in. and 23-in. models, powder particles were carried by a propelling air stream, discharged from a fixed nozzle. For the 3-ft square pan indoor, and the 10-ft square outdoor gasoline fire, Neill[10] used an especially adapted commercial extinguisher and attacked the fire manually by sweeping from side to side. Hird and Gregsten[12] used a fixed nozzle to cover the fire area together with a modified commercial extinguisher. Their fire was supplied by a 1 1/2-in. layer of gasoline in a 3-ft square pan involving a 1/2-in. freeboard.

Neill's results on extinguishment of gasoline fires shown in Figure 9 seem to present a reasonable extrapolation of our results. In plotting these data it was assumed that they could be shown as applicable to fires on circular pans having an equivalent surface area. Neill[10] did not mention specific surface in his paper, but found, later, that the $NaHCO_3$ powder had 1750 cm^2/gr [15]. Specific surface for the $KHCO_3$ was assumed to be the same on the basis of his results of sieve analysis. The extent of agreement between the two sets of results is encouraging especially when consideration is given to the differences in powder application techniques and the many other variables involved.

The higher application rates apparently required for Hird and Gregsten's experiments may have resulted from the need to cool the 2 1/2 in. rim of the fuel container below the flash temperature of the fuel air vapors.

The methods described have been shown to be effective in evaluation of different powder extinguishing agents when applied to flammable liquid fires. It is not known to what extent these measurement techniques will be applicable to very large fires although the work of Neill seems to indicate the possibility of such use. For study of second order effect, variables such as air velocity, powder concentration and particle size distribution must be more closely controlled than was possible in the work described.

6. SUMMARY

Laboratory methods have been developed to evaluate the fire extinguishment effectiveness of powdered materials. These methods employed 1 1/8-in., 6-in., and 22 3/4-in. fire models resembling the free burning of a liquid hydrocarbon. Dispensers which permitted controlled application of powder to the fire were designed and used for the evaluation.

The extinguishment effectiveness of various materials was determined by measuring the minimum rate of powder application required for extinction. The effectiveness of a powder was found to depend on (1) the chemical nature, (2) the specific surface and (3) particle size of the powder.

The most effective material studied was potassium oxalate monohydrate. It was about four times more effective than sodium bicarbonate of optimum particle size, the commonly used extinguishing agent. On a surface-area basis, when using the 1 1/8-in. fire model, particle sizes between 15- and 20-μ were generally most efficient.

There was found to be a correlation close to proportionality between fuel burning rate in a model and the minimum powder discharge rate required to extinguish it. Limited extinguishment data on large-scale fires of other workers seems to confirm the usefulness of the model method for evaluating powder effectiveness.

ACKNOWLEDGMENT

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TABLE 1. Materials used and Preparation Procedures of Powders

Sample	Source	Treatment
Dry chemical	Surface-treated $NaHCO_3$ from commercial extinguisher manufacturer 98% sodium bicarbonate	Size separation by sieving and air elutriation
$NaHCO_3$, $K_2C_2O_4H_2O$ KI, $KHCO_3$, $KTiC_2O_4$ Rbl, CsI	Reagent-grade crystalline chemicals	2% by wt. zinc stearate added, ground in ball mill, size separation by sieving and air elutriation
Mixture	All components were from above sources after treatment	Per cent based on wt., thorough mixing
Glass beads	Commercial glass microbeads	2% zinc stearate added, mixed and heated to 100°C. No treatment for some samples

TABLE 2. Minimum Effective Rates of Powder Application and Rates of Fuel Consumption for Various Fire Sizes and Powder Types in Average Size Range of 6-12 Microns

Diameter of Fire	Rate of Powder Application				Ratio of Application Rate			Fuel Consumption Rate	Rate Ratio $\frac{NaHCO_3 \text{ Application}}{\text{Fuel Consumption}}$
	$NaHCO_3$	$KHCO_3$	KI	$K_2C_2O_4 \cdot H_2O$	$\frac{NaHCO_3}{KHCO_3}$	$\frac{NaHCO_3}{KI}$	$\frac{NaHCO_3}{K_2C_2O_4H_2O}$		
in.	cm^2/sec	cm^2/sec	cm^2/sec	cm^2/sec				ml/sec	1/cm
1.12	68*	32*	15*	14*	2.1	4.5	4.9	0.0078	8.7 x 10^3
	90	42	20	15	2.1	4.5	5.5		11.5 x 10^3
6.0	2,100	800	900	500	2.6	2.3	4.2	0.23	9.1 x 10^3
22.8	25,500	9,500	9,500	5,500	2.7	2.7	4.6	6.0	4.3 x 10^3
40.6	200,000**							32.	6.3 x 10^3

* Data shown are for 20-μ particle size powders

** Data from reference /12/

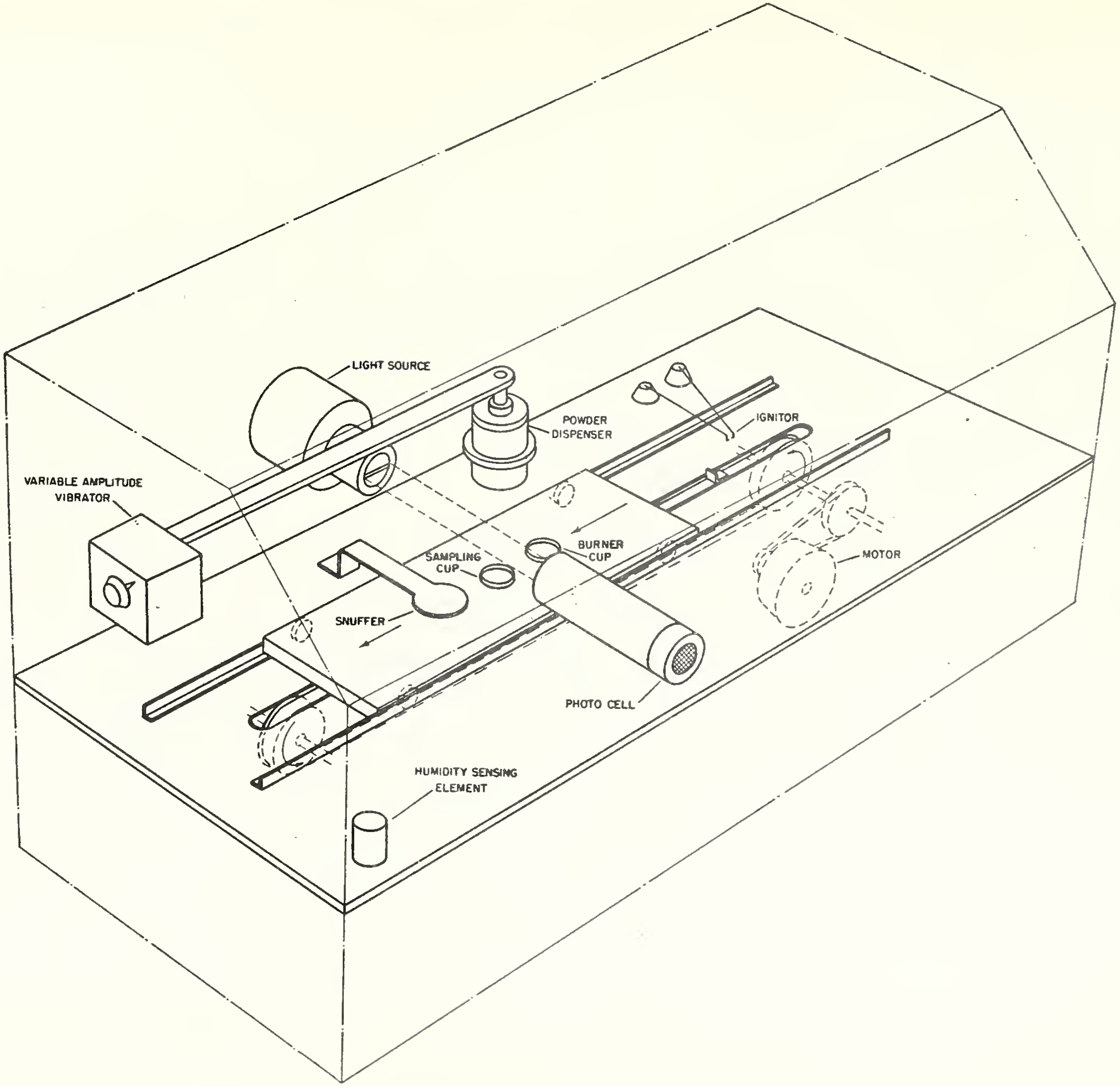

Fig. 1. Apparatus for evaluating the fire extinction efficiency of powders using the 1 1/8-in. fire model.

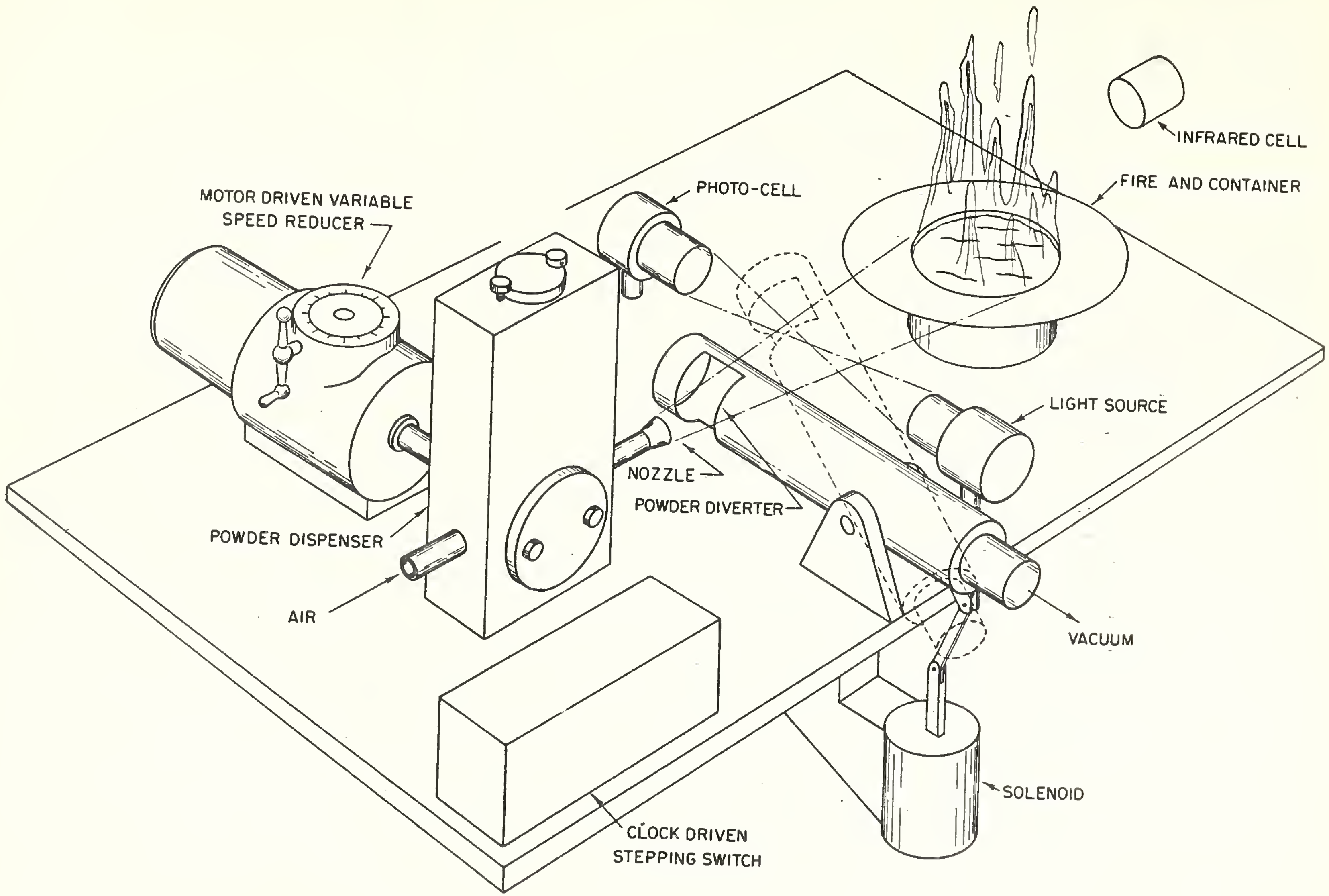

Fig. 2. Diagrammatic illustration of apparatus for evaluating extinction efficiency of powders using the 6- and 22 3/4-in. fire models.

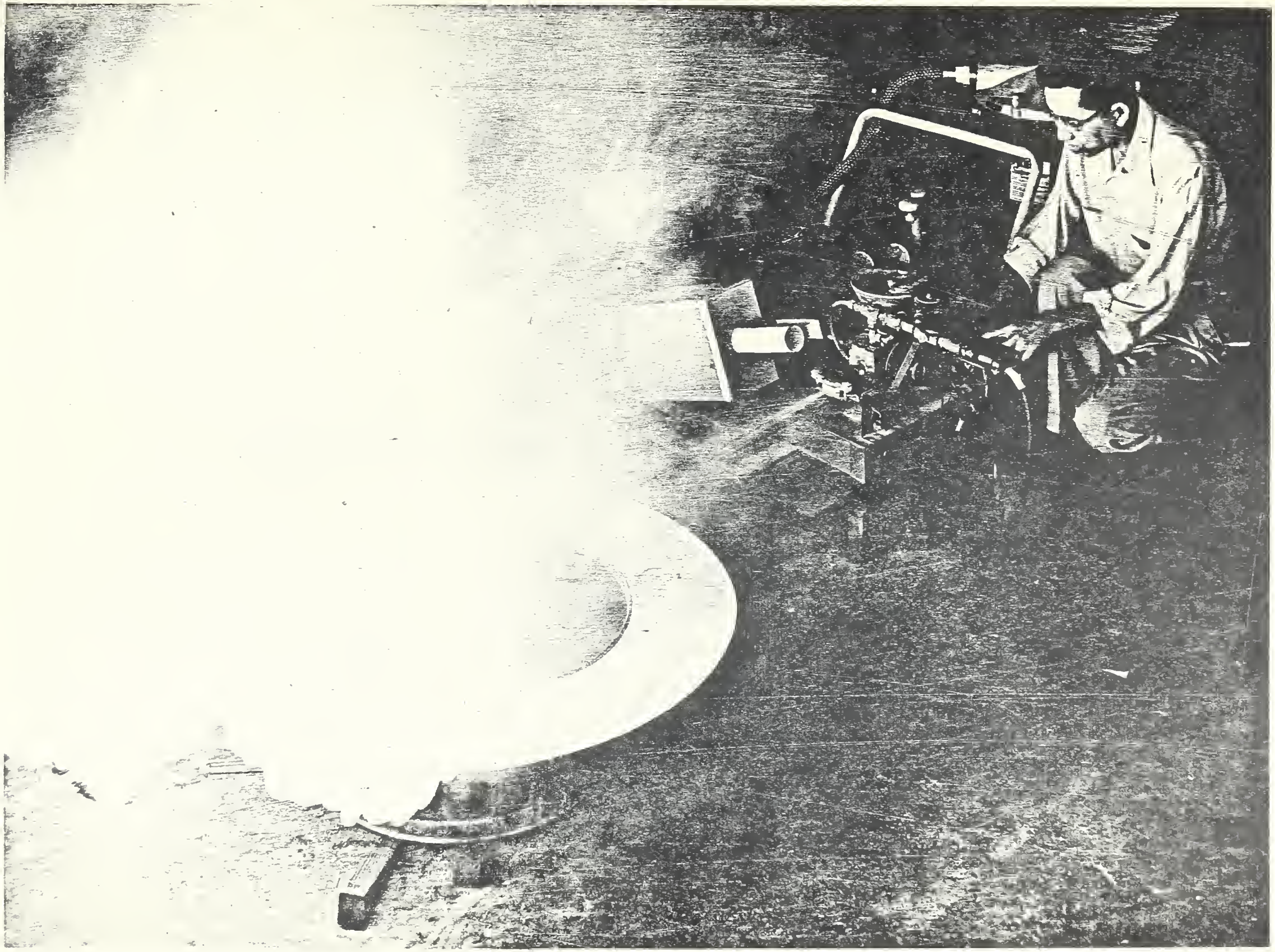

Fig. 3. Extinguishment of 22 3/4-in. fire model. Photometric equipment is not shown.

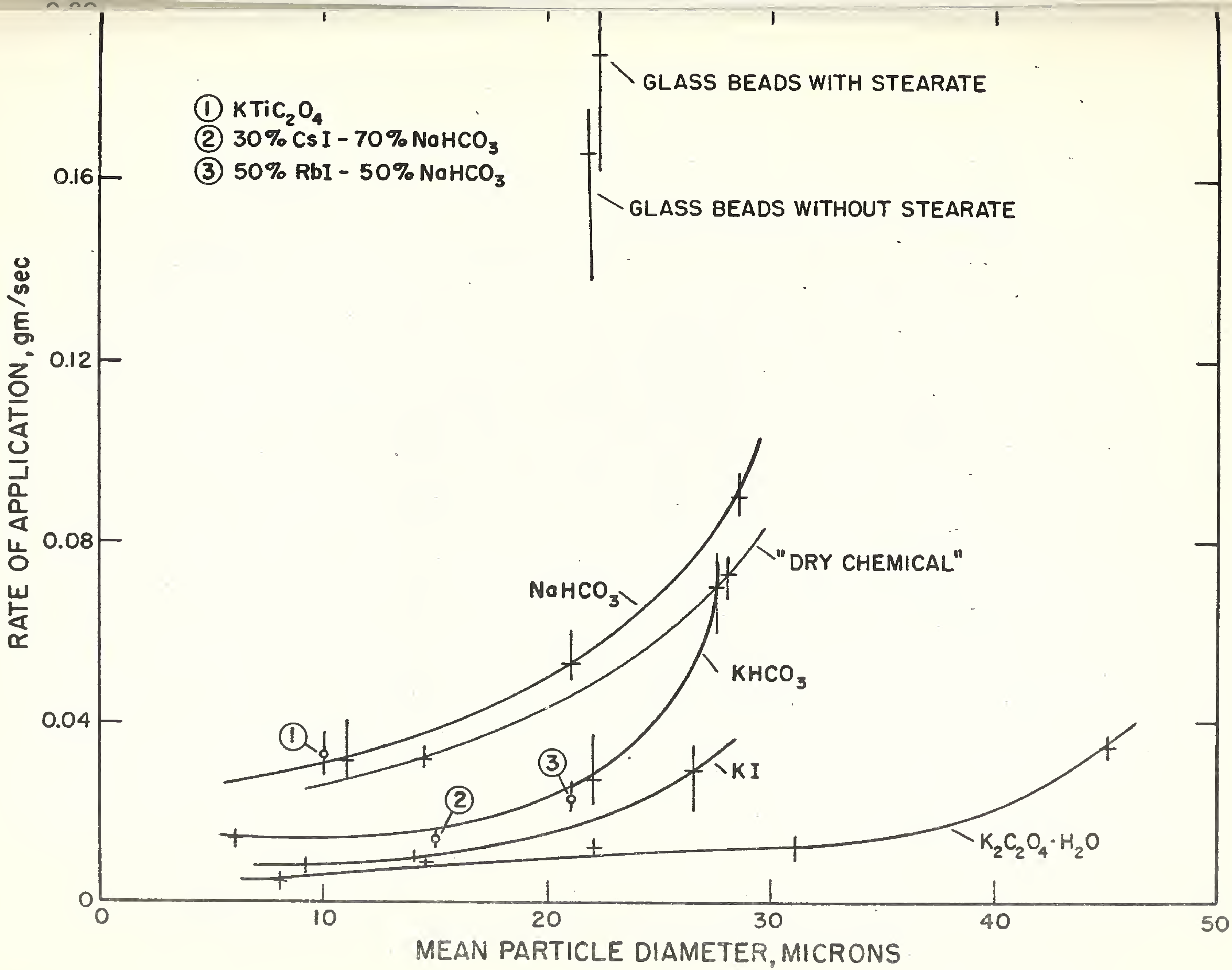

Fig. 4. Effective application rate for the 1 1/8-in. fire model.

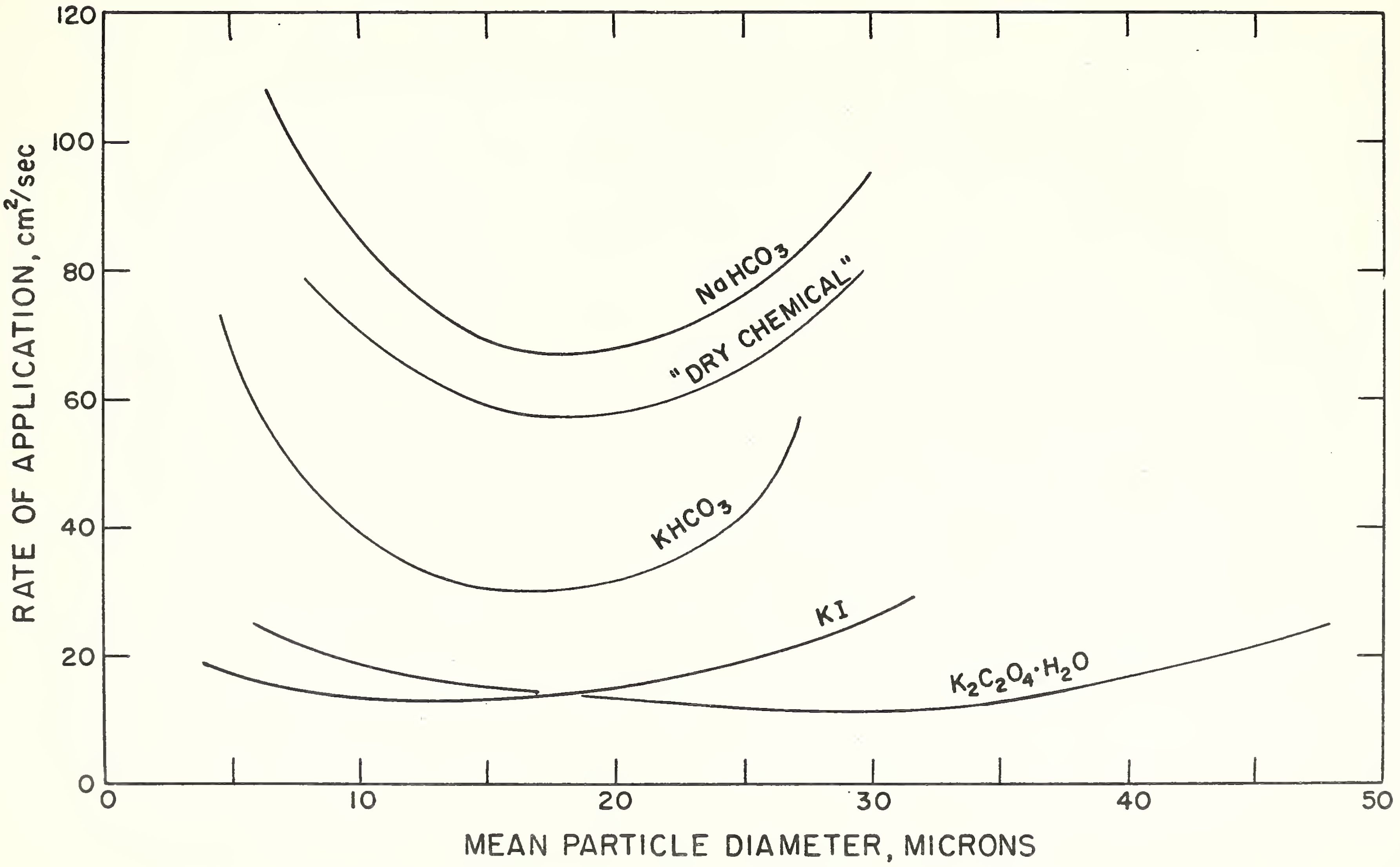

Fig. 5. Effective application rate in terms of powder surface area for the 1 1/8-in. fire model.

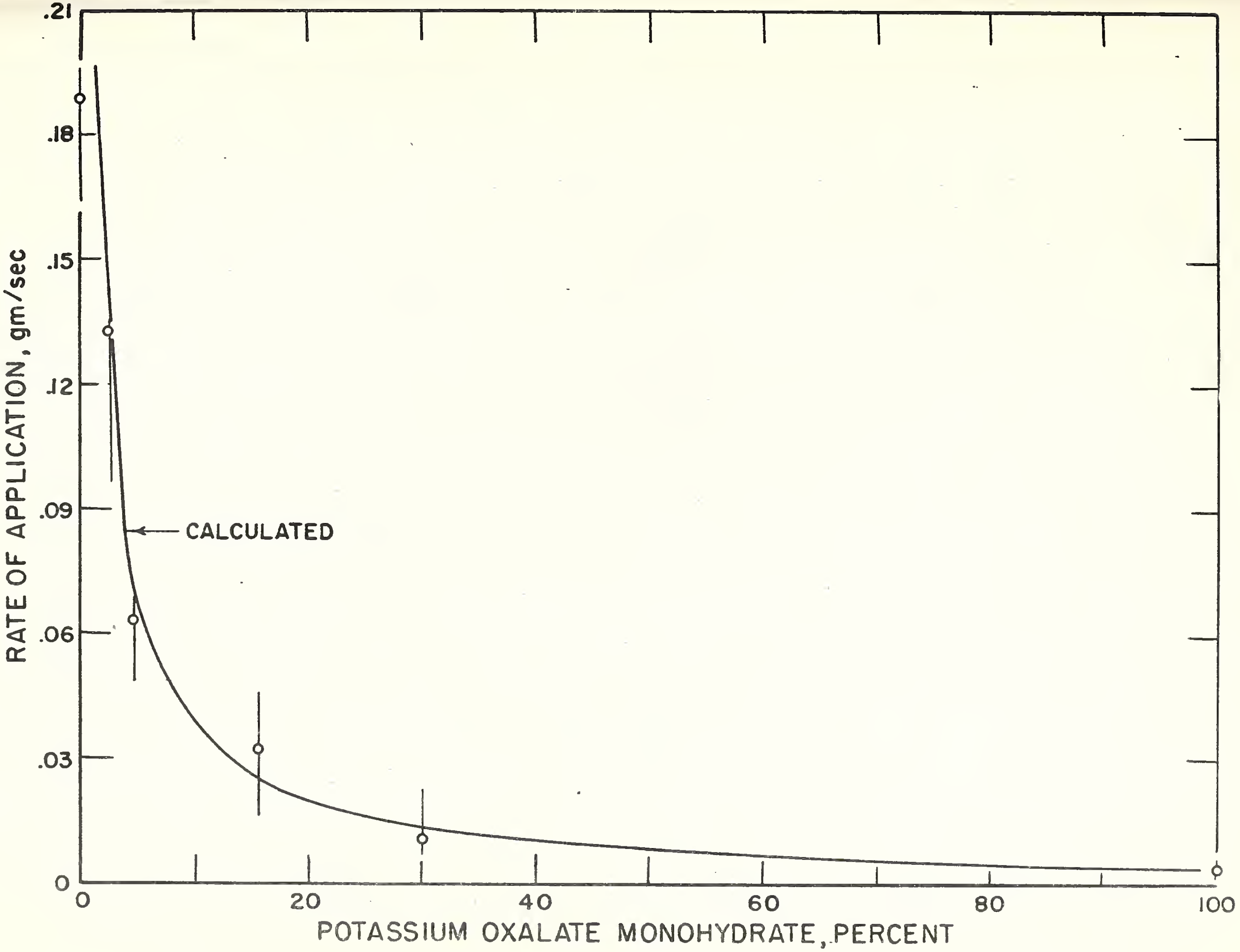

Fig. 6. Effective application rate of mixture of 22 micron glass beads and 8 micron $K_2C_2O_4 \cdot H_2O$, for the 1 1/8-in. fire model as influenced by the percentage of $K_2C_2O_4 \cdot H_2O$ present in mixture.

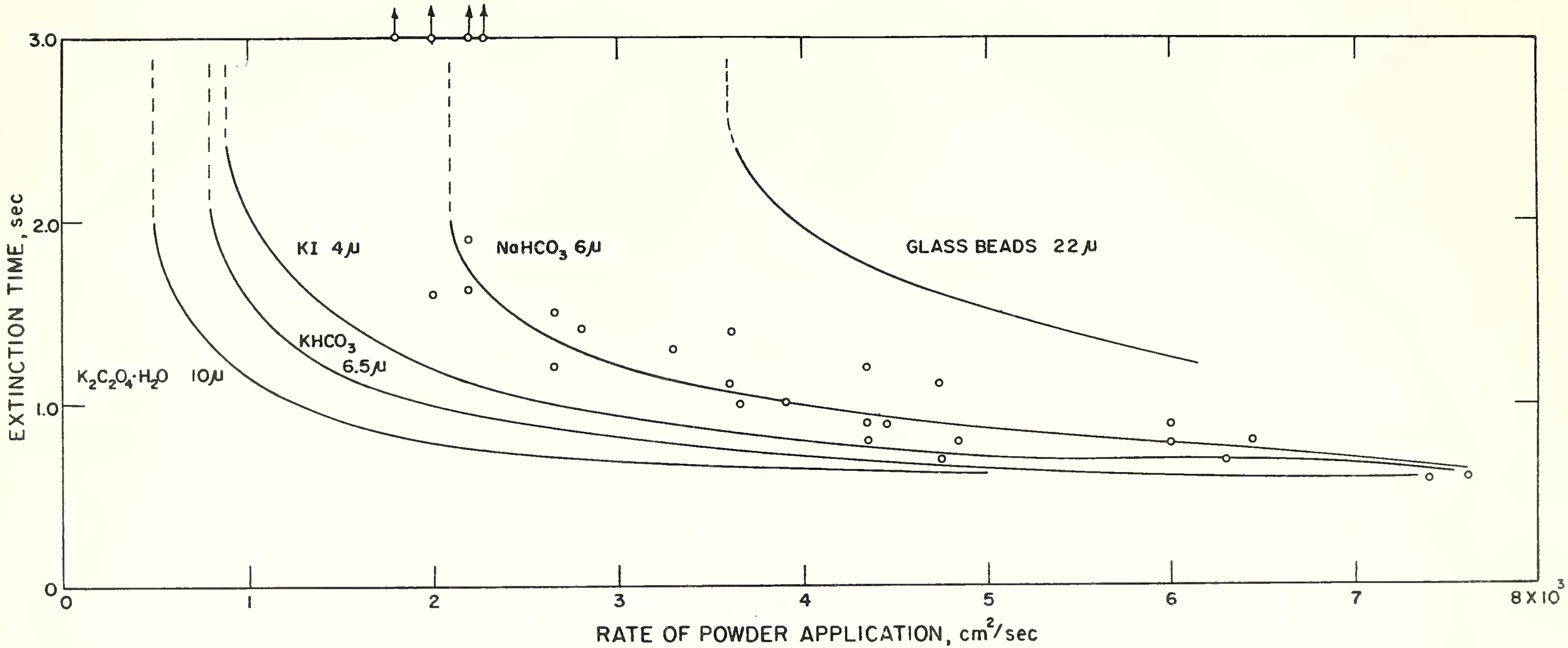

Fig. 7. Fire extinction time as influenced by rate of powder application for 6-in. fire model. Dashed lines represent minimum application rate for extinction. The data points shown were obtained with - $NaHCO_3$. The data with arrows represent trials in which no extinction was achieved.

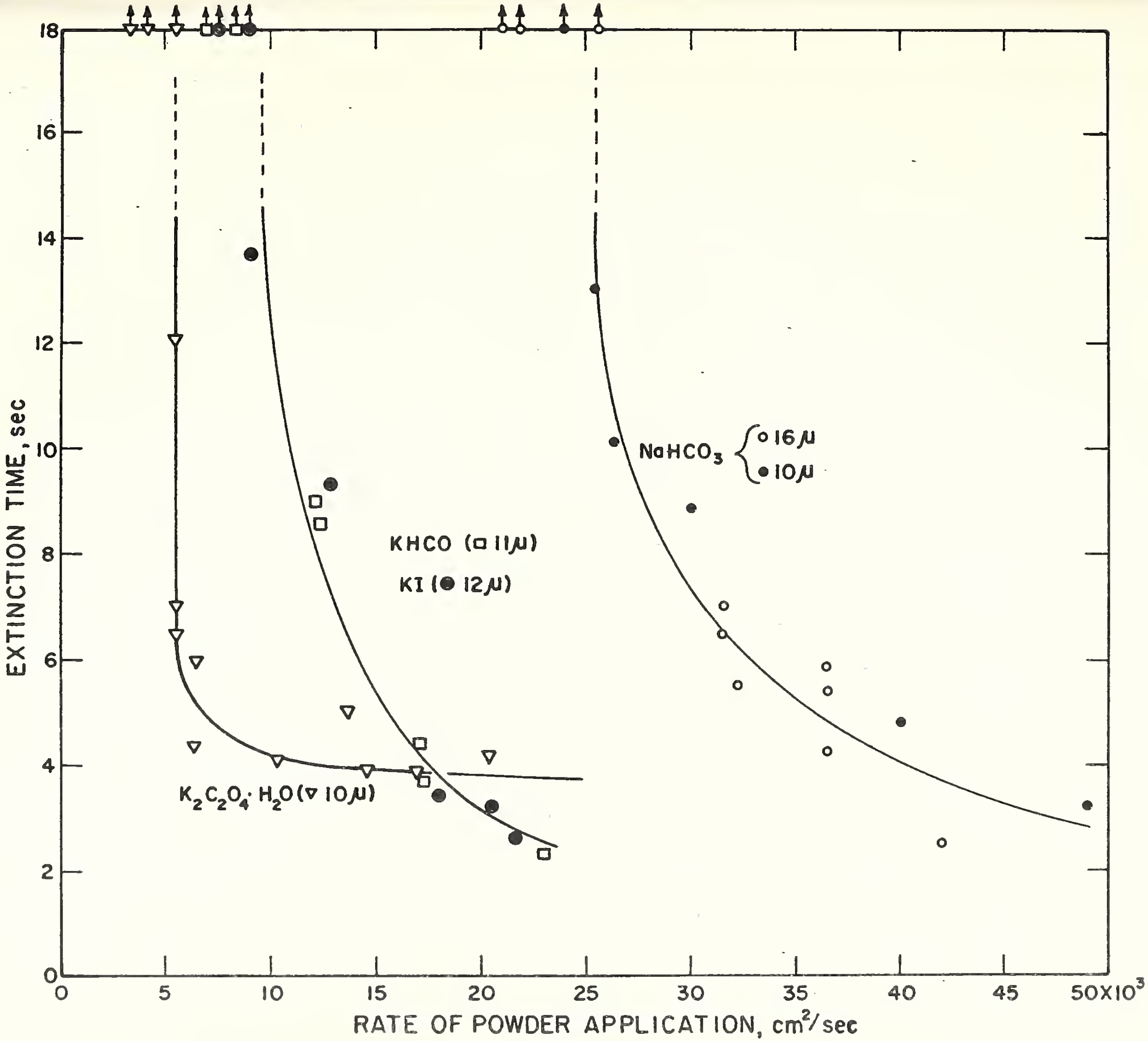

Fig. 8. Fire extinction time as influenced by rate of powder application for 22 3/4-in. fire model. Dashed lines represent minimum application rate for extinction. The data with arrows represent trials in which no extinction was achieved.

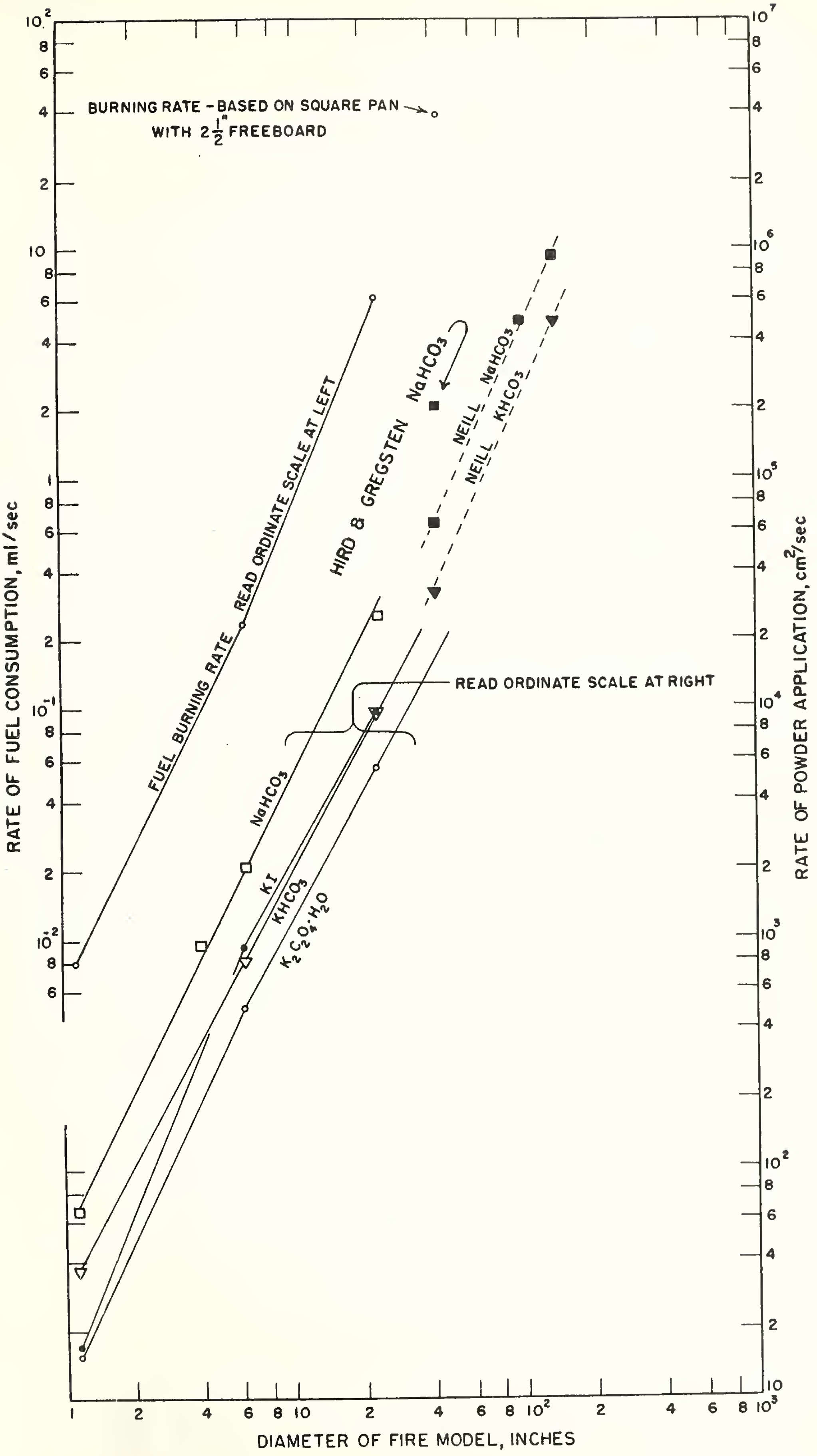

Fig. 9. Rate of fuel consumption and minimum rate of powder application for extinction of fires of different diameters.

U.S. DEPARTMENT OF COMMERCE
Frederick H. Mueller, Secretary
NATIONAL BUREAU OF STANDARDS
A. V. Astin, *Director*

THE NATIONAL BUREAU OF STANDARDS

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Radio Communication and Systems. Low Frequency and Very Low Frequency Research. High Frequency and Very High Frequency Research. Ultra High Frequency and Super High Frequency Research. Modulation Research. Antenna Research. Navigation Systems. Systems Analysis. Field Operations.


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