

Reduction of Incendivity of Hot Gases to Methane and Coal Dust

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ABSTRACT

Hot gases produced by explosives are known to constitute a possible ignition hazard in coal mines. Experiments in the large test gallery of the Bureau of Mines have established that sodium chloride reduces the incendivity of explosives. In the present investigation the effect of sodium nitrate was explored. This study was conducted in two phases. Gallery experiments showed that sodium nitrate reduced the incendivity of certain explosives to 8 percent natural gas in air, but increased their incendivity to coal dust predispersed in air. Laboratory experiments using hot jets from explosions of stoichiometric mixtures of methane-oxygen-nitrogen showed that both sodium chloride and sodium nitrate reduced the incendivity to methane, to mixtures of coal dust and methane, and to coal dust. The difference between the gallery and laboratory results with respect to coal dust is attributed to temperature-time effects. In the gallery experiments, the sodium nitrate probably forms sodium oxide which affects the incendivity of the hot gases. In the hot jet case, it is possible that the nitrate is not as completely decomposed and has a different effect on incendivity.

INTRODUCTION

The hot gases produced by explosives constitute a possible ignition hazard in coal mines. Components of explosives known to affect incendivity of explosives are sodium nitrate and sodium chloride. Large scale gallery experiments with sodium chloride had confirmed that it reduces the incendivity of explosives,^{5/} but the effect of sodium nitrate had not been systematically explored. As sodium nitrate is an oxidant, it was conceivable that it might increase the incendivity of explosives to methane or coal dust dispersed in air. On the other hand it might have an inhibiting action similar to that of sodium chloride. The present study sought to determine whether sodium nitrate inhibits or promotes the ignition of mixtures of methane, coal dust, or both with air.

In one phase of this study, an investigation was conducted in a gallery 6-1/3 feet in diameter, with a 20 foot long section filled with 8 percent natural gas or coal dust predispersed in air. Concentrations of coal dust in air were about 300 mg/liter. This and the 8 percent natural gas concentration in air are both much higher than the lean flammability limit of the respective fuel. Explosives containing natural and synthetic sodium nitrate were fired in the gallery and their incendivity

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 - ^{5/} Hanna, N. E., G. H. Damon, and R. W. Van Dolah. Probability Studies on the Incendivity of Permissible Explosives Effect of Small Percentages of Sodium Chloride, BuMines Rept. of Inv. 5463, 1959, 22 pp.

was determined by the Bureau's standard up-and-down method.^{6/} Sodium nitrate reduced the incendiivity to methane but increased the incendiivity to coal dust. Another phase of the study sought to model gallery conditions through small scale laboratory experiments in which the ignition sources were small pulsed hot gas jets produced by explosions of stoichiometric mixtures of methane-oxygen or such mixtures diluted with nitrogen. These small jets were "salted" or not, so that the relative promoting or inhibiting effect could be determined. The fuel-air mixtures exposed to these jets when not salted were fuel-lean mixtures of methane plus coal dust (hybrid mixtures) or coal dust. Concentrations of coal dust and methane in the hybrid air mixtures were generally below concentrations at their lean flammability limits. The oxygen index [oxygen/(oxygen + nitrogen)] of the ignition source was the index of incendiivity. It was used to vary the temperature of the hot jet which increased as the oxygen index was increased. Temperatures of these jets are noted in table 1 along with other characteristics. The ability of the technique to detect changes in incendiivity was confirmed by experiments with sodium chloride. Sodium nitrate was found to reduce the incendiivity of the hot gases with respect to all three of the fuel systems. This experimental technique should make it possible to survey a large number of ignition inhibitors to select the materials which are most effective in reducing the ignition hazard of hot gases from detonating charges in coal mines.

ACKNOWLEDGEMENT

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EXPERIMENTAL EQUIPMENT AND PROCEDURE

Gallery Experiments

Equipment used in these experiments were the same as those used to evaluate the permissibility of explosives according to schedule 1-H.^{7/} The first experiment had a 2 x 3 factorial design and was conducted by firing from a stemmed cannon into 8 percent natural gas-air mixtures (gallery test 7).^{7/} Its purpose was to compare the six explosive formulations in table 2, which are based on the composition of two different permissible explosives (A and B). The samples based on composition A contained about 4 percent sodium nitrate; those based on composition B contained 10 percent sodium nitrate. Both synthetic (99.5% pure) and natural (98.5% pure) sodium nitrate was used, with the natural in two ranges of particle size. The average particle diameter of the coarse natural, ground natural, and synthetic sodium nitrate was 1,170, 317, and 437 microns, respectively. Each of the six explosives was carried through the up-and-down sequence, varying the weight of explosive to obtain W₅₀ values.^{8/} Ten pairs of ignition-nonignition results were obtained in each up-and-down sequence.

The second experiment was also given a 2 x 3 factorial design to compare the incendiivity of the same explosives to coal dust (300 mg/liter) predispersed in air. The coal dust was of Pittsburgh Seam coal; its average particle diameter was 80 microns and the proximate analysis was 2.0 percent moisture, 34.9 percent volatile, 55.4 percent fixed carbon, and 7.6 percent ash. In this experiment, 5.5 kilos of

^{6/} Hanna, N. E., P. A. Richardson, and R. W. Van Dolah. An Improved Method for Evaluating the Incendiivity of Explosives to Coal Dust: A Preliminary Report. Restricted International Conference of Directors of Safety in Mines Research, Sheffield, England, 1965, Paper No. 12.

^{7/} Federal Register, March 1, 1961, v. 26, No. 39. Title 30 - Mineral Resources, Schedule 1-H, p. 1761.

^{8/} W₅₀ is weight of explosive producing 50 percent probability of igniting a natural gas-air atmosphere; W₅₀ values increase as incendiivity decreases.

Table 1. - Characteristics of hot turbulent gas jets. (ignition sources)

Oxygen index <u>1</u> /	Adiabatic flame temperature, ° K	Observed ² / maximum temperature, ° K	Jet velocity, ³ / m/sec	Length ³ / of jet, cm
1.0	3050	2740	137	12.8
.75	2975	2650	98	9.0
.50	2813	2480	59	6.4
.35	2620	2270	36	5.4
.30	2510	2510	28	5.0
.26	2400	2020	22	4.7
.21	2200	1820	16	4.3

1/ O₂/(O₂ + N₂) of stoichiometric mixture containing methane.

2/ Experimental values determined by the sodium D line reversal technique.

3/ Velocity of advance of head of jet from orifice. Velocity was constant between time zero and growth of jet to maximum length (point of sharp decrease in luminosity and start of break-up of jet).

Table 2. - Incendivity of products from explosives containing sodium nitrate and sodium chloride

	A formulations				B formulations			
Chemical analysis:								
Moisture	0.3	0.2	0.2	0.4	0.3	0.4	0.3	0.4
Nitroglycerine	8.8	8.7	8.8	10.6	10.7	10.5	10.7	10.5
Nitrocellulose	.3	.3	.3	.3	.3	.2	.3	.2
Ammonium nitrate	68.3	67.6	68.1	57.6	57.1	56.9	57.1	56.9
Sodium nitrate	1/ 3.8	2/3.9	3/3.8	1/10.3	2/9.3	3/9.9	2/9.3	3/9.9
Sodium chloride	9.2	9.5	9.1	10.4	10.1	9.8	10.1	9.8
Antacid	.4	.3	.5	.3	.5	.5	.5	.5
C.C.M. ^{4/}	8.9	9.8	9.2	10.1	11.6	11.8	11.6	11.8
Physical properties:								
Apparent specific gravity	1.06	1.05	1.05	0.84	0.82	0.81	0.82	0.81
Grams wrapper per 100 g explosive	7.3	7.7	7.3	8.2	8.5	8.5	8.5	8.5
Ballistic mortar, percent strength of TNT	96	97	96	89	89	90	89	90
Rates of detonation, meters per second	2470	2360	2480	2630	2570	2580	2570	2580
Oxygen Balance, grams oxygen per 100g explosive	-11.6	-13.3	-12.5	-15.5	-18.1	-19.1	-18.1	-19.1
Incendivity, Gallery Results:								
W50, grams ^{5/}	615	600	590	670	650	670	650	670
WCD, grams ^{6/}	300	290	490	80	90	90	90	90

- 1/ Coarse natural nitrate, average particle size 1171 microns.
- 2/ Fine synthetic nitrate, average particle size 437 microns.
- 3/ Ground natural nitrate, average particle size 317 microns.
- 4/ Carbonaceous combustible material.
- 5/ Weight for 50 percent probability of ignition in 8 percent natural gas-air; stemmed cannon shots.
- 6/ Weight for 50 percent probability of ignition in predispersed coal dust (300 mg/liter) - air; suspended shots.

coal dust was dispersed in the first section of the gallery 1/2 second before firing an explosive charge suspended in the center of this section which was isolated from the rest of the gallery by a paper diaphragm. The coal dust was spread evenly over a length of 30-grain per foot detonating cord laid in a 20-foot long steel Vee trough made from 6-inch by 6-inch angle and mounted 7 inches above the gallery floor; the detonation of the cord disperses the dust. With this test arrangement, $W_{CD}^{9/}$ were determined, again using up-and-down technique.

LABORATORY EXPERIMENTS

The effect of sodium nitrate on incendivity of hot gas jets was investigated using mixtures in air of methane, coal dust, or hybrid mixtures of the two as the acceptor charge. The coal dust was an ultrafine grind (83.5 percent less than 17 microns) of Pittsburgh Seam, Mathies mine coal. The elemental composition in percent by weight was: $H_2 = 5.3$, $C = 78.9$, $O_2 = 8.0$, $N_2 = 1.6$, $S = 1.3$, and ash = 4.9. The proximate analysis in percent by weight was: Moisture, 0.7; volatile matter, 37.0; fixed carbon, 57.5; and ash, 4.8. All gases were obtained in cylinders and were chemically pure grade except for air. Sodium chloride and sodium nitrate of chemically pure grade were ground to minus 20 micron particle size.

The hot gas ignition apparatus (figure 1) and coal dust disperser (figure 2) have been described earlier.^{10/} The explosion vessel consists of two chambers, one

Figure 1. - Hot Gas Ignition Apparatus.

Figure 2. - Coal Dust Disperser.

partially within the other (figure 1). The small chamber A (76 cc) was capped and communicated with the large chamber B (2.1 liters) through a straight channel, 0.5 cm in diameter and 1.0 cm long. After purging and filling chamber A with a stoichiometric methane-oxygen-nitrogen mixture, the cap was removed and the contents were spark-ignited near the channel opening. The explosion products vented through the channel into the flammable mixture in chamber B. These jets differed in temperatures as shown in table 1, both by their calculated theoretical temperatures assuming adiabatic combustion, and by measured temperatures using the sodium D line reversal technique. Natural gas used in these experiments contained about 91 percent methane and 6 percent ethane. Chamber B was equipped with top plates having an array of small venting holes, a blowout pressure release diaphragm at the sidewall, and viewing windows. The outer face of the diaphragm was inerted with nitrogen to eliminate spurious luminosity due to secondary combustion in surrounding air of the hot, partially burned explosion products. The premixed coal dust-methane-air mixture to be ignited flowed through chamber B at approximately 140 cc/sec, giving a constant average linear upward speed of 2.2 cm/sec.

In determining an ignition limit, the coal dust concentration was held constant and the methane concentration was increased until ignition occurred. The salt being investigated for its inhibiting or promoting action was added to chamber A prior to spark ignition by mechanically vibrating the duster (figure 1, view C) that had previously been filled with the requisite amount of additive. Amounts placed in

9/ W_{CD} is the weight of explosive producing 50 percent probability of igniting a coal dust atmosphere. W_{CD} values increase as incendivity decreases.

10/ Singer, J. M. Ignition of Mixtures of Coal Dust, Methane and Air by Hot Laminar Nitrogen Jets. Ninth International Symposium on Combustion, 1963, Academic Press, Inc., New York, N. Y., pp. 407-414.

Singer, J. M. Ignition of Coal Dust-Methane-Air Mixtures by Hot Turbulent Gas. BuMines Rept. of Inv. 6369, 1964, 24 pp.

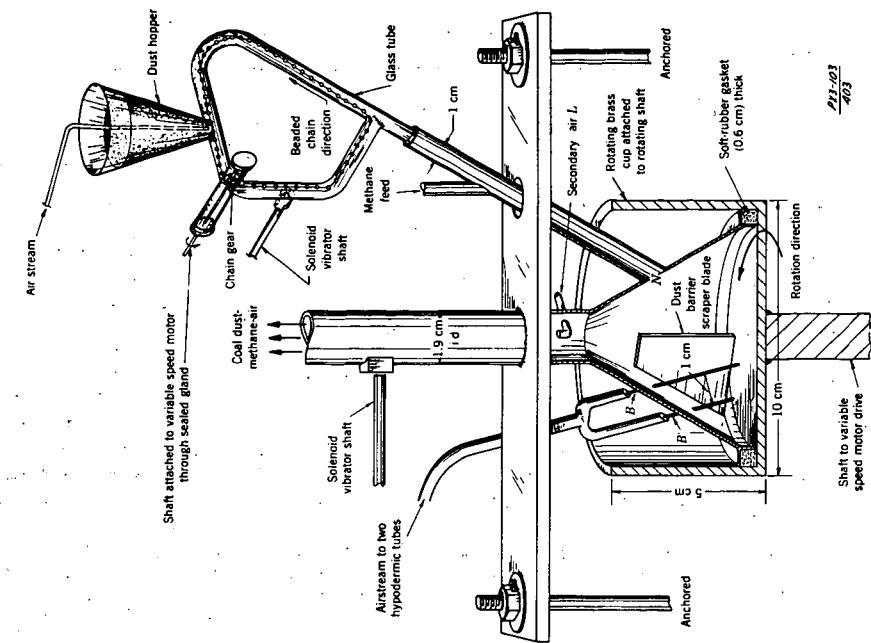


Figure 2. - Coal Dust Disperser.

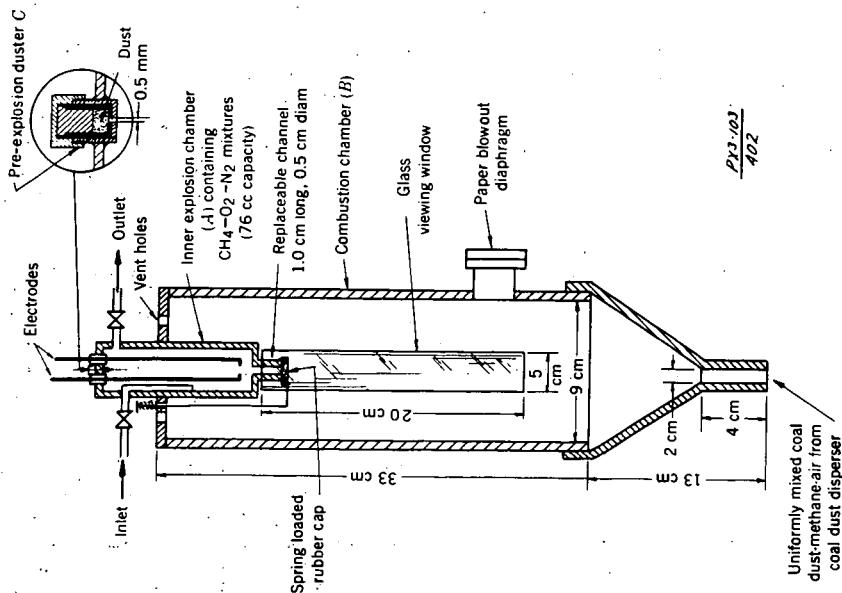


Figure 1. - Hot Gas Ignition Apparatus.

chamber A are stated in figure 3. The actual amount of additive entrained by the

Figure 3. - Effects of sodium salts on incendivity of hot jets to methane-coal dust-air mixtures.

hot jet is not known. The criteria of ignition after the hot jet entered chamber B was the luminosity observed throughout B, and the simultaneous luminous flame shooting out of the ruptured pressure release diaphragm. Duplicate runs made at each ignition point showed that methane-in-air concentrations were reproducible to 0.15 percentage units, oxygen indices to within 0.02 units and coal dust concentrations to within 20 percent. Coal dust concentrations were determined on separate runs by filtering the entire mixture at different levels of chamber B through a 4 cm diameter glass-wool cartridge and weighing after a selected time interval. Local concentrations of dust were determined by filtering through a paper extraction thimble on a 1.0 cm diameter iso-kinetic sampling probe. Local concentrations of dust at various heights and radii of chamber B were constant to within 20 percent. Concentrations of fuel in chamber B at the ignition limit are termed "lower ignition limits", and correspond to lower flammability limits, except that ignition limits are dependent on apparatus factors.

The coal dust disperser in figure 2 was continuously fed by an endless beaded-chain carrier that removed dust from the hopper at a rate determined by its rotation speed. Methane was added through an inlet to the dust disperser. Not all of the air could be added through the air jets; the balance was added through duct L. Hypodermic needles, dust carrier tubes, and chamber B were continuously vibrated to facilitate dust movement and to prevent dust deposition.

RESULTS AND DISCUSSION

Gallery Experiments

The W_{50} values obtained for the six samples are given in table 2. The values for the three explosives containing the low percentage of sodium nitrate varied from 590 to 615 grams; and the values of the three high sodium nitrate explosives varied from 650 to 670 grams. A statistical analysis of these data showed that the type and fineness of the sodium nitrate had no significant effect on the incendivity of the explosives to natural gas air mixtures. However, the samples with the high sodium nitrate content were consistently less incendive than those with low sodium nitrate concentrations.

The W_{CD} values for the low sodium nitrate explosives (a) formulation varied from 290 to 490 grams; the W_{CD} values for the high sodium nitrate explosives (b) formulations varied from 80 to 90 grams. As in the previous experiment the type and fineness of the sodium nitrate does not appear to have any significant effect on the incendivity to coal dust. The 10 percent sodium nitrate explosives were more incendive in coal dust-air atmospheres than the 4 percent sodium nitrate explosives. The reverse was the case for natural gas-air, but differences were much smaller.

The concentration of other constituents of the explosives were changed; nitro-glycerin increased about 17 percent between A and B formulations, ammonium nitrate decreased by 18 percent, sodium chloride increased by 10 percent and the combustible carbonaceous matter increased by 19 percent. The sodium nitrate increase was much greater, being about 155 percent. The net result of these changes in chemical composition is that the oxygen balance is more negative for the B formulations than for the A group of explosives. (The oxygen balance is the deficiency or excess of oxygen required for stoichiometric explosion, in units of grams of oxygen per 100 grams of explosive.) Problems of interpretation due to changes in composition, other than changes in sodium nitrate concentration will be discussed later.

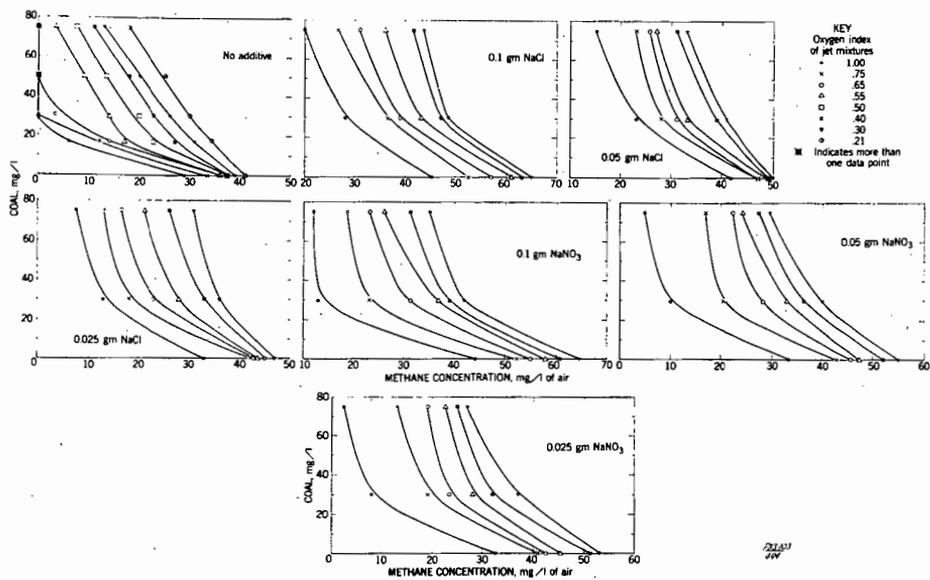


Figure 3. - Effects of Sodium Salts on Incendivity of Hot Jets to Methane-Coal Dust-Air Mixtures.

Laboratory Experiments

Lower ignition limits with and without sodium nitrate in the hot gas jet which served as an ignition source are shown in figure 3; no ignition is obtainable to the left of any curve with the jet used. Each jet is characterized by its oxygen index. The fuel-air mixtures subjected to these jets range from methane-air (lean limit of flammability for methane, 35 mg/liter) to hybrid mixtures of methane-coal dust-air to coal dust-air mixtures (stoichiometric concentration of coal dust used, 109 mg/liter). As the incendiivity of the hot jet is decreased by decreasing the oxygen index or adding sodium salts, the lower ignition limits are displaced upward for constant methane concentration and to the right for constant coal dust concentration. Table 3 gives the reduction in incendiivity as measured by an increase in oxygen index of the jet required to ignite a given fuel mixture; the composition of the fuel mixture is given in terms of concentrations of methane and coal dust. Table 4 summarizes in another way, the change in incendiivity due to the sodium salts. As the incendiivity of the jet decreases due to the salts (comparisons for constant oxygen index and coal concentrations) more methane must be added to the fuel mixture to keep it ignitable. The increase in methane is indicated in table 4 by the increase in total fuel concentration.

The data for sodium chloride shows that the hot jet technique is capable of demonstrating the known effectiveness of sodium chloride in reducing incendiivity.^{5/} The data for sodium nitrate parallel the trend of the sodium chloride data, but at a lesser level of effectiveness. Both salts reduce incendiivity of the hot jet toward methane, a hybrid mixture of methane and coal dust or coal dust only. This result was not the case in the gallery experiments in which sodium chloride reduced incendiivity to gas and to coal dust, but sodium nitrate only reduced incendiivity to gas while increasing incendiivity to coal dust.

It is difficult, without further investigation, to pinpoint the reason for the difference in the two sets of results. The gallery determinations were done with coal dust concentrations of 300 mg/liter in air, and the average particle diameter of the coal dust was 80 microns. The coal dust used in the hot jet determinations was of a much finer grind; concentrations used were below 80 mg/liter. It is not known whether these differences would lead to any specific chemical effect between coal dust and sodium nitrate. There are also differences between the two sets of formulations of the explosives (table 2). The overall changes in stoichiometry between formulations A and B resulted in more combustible materials being present in the products of explosion of the B formulation than in those of the other. Perhaps secondary burning in air may have contributed to the greater incendiivity of the B formulations, rather than the greater concentration of sodium nitrate. However, afterburning of the richer fuel concentration in the explosion products should increase incendiivity to natural gas as well as to coal dust unless the ignitibility of coal dust is far more dependent than gas on the temperature of the ignition source.^{11/}

The striking difference in the two experiments may be due to the condition of the sodium nitrate when the hot gases are injected into the fuel-air mixture. In the case of the gallery experiments, the nitrate may be completely reacted with fuel materials in the detonation front or shortly thereafter. A usually assumed product of its reaction is sodium oxide. In the hot jet experiment it is possible that less sodium oxide is formed in the time available. Thus the possibility exists that sodium compounds derived either from sodium chloride or sodium nitrate in the detonation gases may be a specific inhibitor for the ignition of methane, and that sodium oxide may have a specific ignition promoting mechanism for coal dust. In the hot jet case, sodium chloride and sodium nitrate may play essentially equal roles.

^{11/} Singer, J. M. and J. Grumer. Equivalences of Coal Dust and Methane at the Lower Ignition Limits of Their Mixtures. Restricted International Conference of Directors of Safety in Mines Research, Sheffield, England, 1965, Paper 13, 22 pp.

Table 3. - Incendivity of hot gas jets: minimum oxygen indices of jets required to ignite coal dust-methane-air mixtures.

Fuel Compositions		Additives in ignition jet ^{1/}						
Coal dust Concentration, mg/liter	Methane Concentration, mg/liter	No additive	0.1 gm NaCl	0.1 gm NaNO ₃	0.05 gm NaCl	0.05 gm NaNO ₃	0.025 gm NaCl	0.025 gm NaNO ₃
0	35	0.54	n ^{2/}	n	n	0.95	0.94	0.94
	40	.23	n	n	n	.80	.80	.77
	45	.21	n	1.0	0.88	.65	.44	.55
	50	.21	0.86	.81	.30	.50	.21	.40
30	0	.75	n	n	n	n	n	n
	10	.60	n	n	n	1.0	n	0.9
	20	.50	n	0.80	1.0	.75	0.70	.70
	30	.21	0.90	.60	.70	.65	.50	.50
50	40	.21	.65	.38	.35	.30	.26	.30
	0	.65	n	n	n	n	n	n
	10	.55	n	n	n	0.9	1.0	0.85
	20	.30	n	0.80	1.0	.73	.65	.65
75	30	.21	0.80	.60	.55	.53	.40	.35
	40	.21	.55	.26	.26	.30	.21	.21
	0	.60	n	n	n	n	n	n
	10	.45	n	n	n	0.85	0.85	0.80
75	20	.21	1.0	0.73	0.88	.70	.70	.65
	30	.21	.70	.45	.45	.30	.35	.30
	40	.21	.45	.21	.21	.21	.21	.21

1/ Weight of additive in primary chamber before ignition.

2/ n = Not ignitable with oxygen index of unity.

Table 4. - Total concentrations^{1/} of coal dust and methane at the lower ignition limits of their mixtures with and without the additives sodium chloride and sodium nitrate.

Additives in ignition jet ^{2/}	None		0.1g NaCl		0.1g NaNO ₃		0.05g NaCl		0.05g NaNO ₃		0.025g NaCl		0.025g NaNO ₃								
	1.0	0.55	0.3	1.0	0.55	0.3	1.0	0.55	0.3	1.0	0.55	0.3	1.0	0.55	0.3						
Oxygen index of jet	1.0	0.55	0.3	1.0	0.55	0.3	1.0	0.55	0.3	1.0	0.55	0.3	1.0	0.55	0.3						
Coal dust concentration, mg/l:																					
0	0.45	0.55	0.59	0.68	0.92	0.95	0.68	0.88	0.95	0.62	0.71	0.72	0.52	0.71	0.80	0.50	0.65	0.70	0.50	0.69	0.79
30	.30	.46	.65	.68	.90	.96	.48	.82	.89	.60	.75	.86	.42	.75	.82	.48	.68	.81	.40	.68	.83
50	.45	.58	.75	.80	1.03	1.12	.62	.93	1.02	.73	.92	1.0	.53	.91	.99	.53	.83	.95	.50	.83	.93
75	.65	.70	.85	.94	1.13	1.27	.80	.98	1.15	.90	.98	1.1	.72	.95	1.01	.70	.94	1.02	.65	.95	.98

1/ Fraction of stoichiometry of coal dust-methane-air for complete combustion.

2/ Weight of additive in primary chamber before ignition.

The effect of additives on ignition has been examined recently by several investigators. The experiments of Singer using hot laminar nitrogen jets^{12/} and hot pulsed jets^{13/} as ignition sources indicated that (1) gaseous inhibitors were more effective when added to hot laminar nitrogen jets than to the fuel mixtures to be ignited, and (2) inhibitors suppressed ignition by hot pulsed turbulent jets less efficiently than ignition by hot continuous laminar jets. In ignition by hot pulsed turbulent jets, higher temperatures and high rates of heat and mass transfer to the dust mixtures nullified the effect of the volatile inhibitors. In hot continuous laminar jets, the same compounds were powerful ignition suppressors, apparently because they inhibited slow chemical reactions coupled to the slow diffusion of oxygen and fuel into the slower moving hot gases.

Most of the work reported by other investigators^{14/} concerns suppression of flames of gas mixtures by well-known inhibitors such as halogenated hydrocarbons and alkali metal compounds. Flame inhibition mechanisms suggested for gas mixtures usually relate the condition that chain branching of the active species will equal chain breaking in flames of mixtures with a minimum (or zero) flame velocity or in flames at the upper and lower flammability limits. Dust inhibitors are presumed to act either as coolants or as chemical inhibitors attacking free radicals responsible for chain reaction. One explanation is that the efficiency of a chemical inhibitor is related to the ease of removal of a free valence electron by a colliding radical. Sodium nitrate and sodium chloride are classed as both thermal and chemical inhibitors.

^{12/} First work cited in footnote 10.

^{13/} Second work cited in footnote 10.

^{14/} Abrams, M. C. Chemical Flame Quenching Theory. Pyrodynamics, v. 1, 1964, pp. 131-141.

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CONCLUSIONS

1. The incendiivity to methane or natural gas-air mixtures was reduced by the presence of sodium nitrate in explosives or in hot jets from methane-oxygen-nitrogen explosions.
2. The incendiivity to methane-coal dust-air mixtures was reduced by sodium nitrate in the hot jets.
3. The incendiivity to coal dust was increased by sodium nitrate in explosives and decreased by sodium nitrate in the hot jets.