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Sodium Nitrate Combustion Limit Tests

G. A. Beitel

April 1976

Prepared for the U.S. Energy Research
and Development Administration
Under Contract E(45-1)-2130



Atlantic Richfield Hanford Company
Richland, Washington 99352



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PREPARED FOR THE U.S. ENERGY RESEARCH AND DEVELOPMENT
ADMINISTRATION UNDER CONTRACT E (45-1) 2130

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SODIUM NITRATE COMBUSTION LIMIT TESTS

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April 1976

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SODIUM NITRATE COMBUSTION LIMIT TESTS

INTRODUCTION

A recent report discusses potential reactions between organic materials and nitrate-rich salt cake.^[1] To gain some practical experience and insight into sodium nitrate combustion processes, a series of tests was conducted with the objective of establishing combustion limits as a function of the concentration of nitrate, organic material, and moisture. The results presented in this report identify some combustion limits subject to specific conditions, and identify other conditions under which combustion processes should be investigated in greater detail.

SUMMARY

Sodium nitrate is a powerful solid oxidant. Energetically, it is capable of exothermically oxidizing almost any organic material. Rate-controlling variables such as temperature, concentration of oxidant, concentration of fuel, thermal conductivity, moisture content, size, and pressure severely limit the possibility of a self-supported exothermic reaction (combustion).

The tests reported in this document were conducted on one-gram samples at atmospheric pressure. Below 380° C, NaNO_3 was stable and did not support combustion. At

[1]*ARH-LD-119 (unclassified), March 1976, G. A. Beitel, "Chemical Stability of Salt Cake in the Presence of Organic Materials."

moisture concentrations above 22 weight percent, exothermic reactions did not propagate in even the most energetic and reactive compositions. Fresh resin and paraffin were too volatile to enable a NaNO_3 -supported combustion process to propagate. Concentrations of NaNO_3 above 95 weight percent or below 35 weight percent did not react with enough energy release to support combustion. The influence of sample size and confining pressure, both important factors, was not investigated in this study.

BACKGROUND

Most of the Hanford high-level liquid waste is a concentrated sodium nitrate solution. Under the present waste-management program, the liquid waste is being reduced to a solid through evaporation and crystallization. The resulting solid, known as "salt cake," contains as much as 95 weight percent sodium nitrate. During the course of waste generation and subsequent processing, some organic materials (paraffins, resins, sugars, a variety of solvents, and miscellaneous objects) have been added to the waste tanks in small but unknown quantities.

Sodium nitrate is a solid oxidant in a sufficiently high energy state to oxidize almost any organic material. The energy available per gram of any stoichiometric sodium nitrate - organic mixture is nearly equivalent to that of TNT. Therefore, a properly mixed sodium nitrate - organic composition can be expected to react with as much explosive force as TNT if kinetic factors will permit a sufficiently rapid reaction.

There are no known detonable mixtures of organic material and sodium nitrate, but many deflagrate explosively. The best known compositions are sodium-nitrate-based "black powders."

A deflagration is an explosive combustion process with the property that burning velocity (or propagation velocity) increases with increasing pressure.^[2] This condition leads to an explosion when a confined composition is ignited. Ignition is the process of initiating an exothermic process such that the heat-generation rate from the reaction is greater than the heat-loss rate. In many cases the time, location, and source of ignition are well defined and easily quantified. In other cases the fact of ignition or even the potential for ignition is indeterminate.

Comments similar to those applied to ignition can also be used for the term "combustion limit." Combustion limit refers to the concentration of either the oxidant or fuel at which self-supported combustion is just possible. Although it would be desirable to have a specific combustion limit for a given composition, this is impossible. Since the combustion process is controlled by pressure, heat transfer, and heat loss (as well as by both inert and active impurities), a combustion limit is applicable only to a single set of conditions (although the sensitivity to the other conditions is not always great). The dependence upon pressure places a dependence upon the initial environment and the ignition source. The importance of heat transfer and heat loss makes combustion limits dependent upon physical conditions such as grain size and the density, configuration, and size of the sample.

The size factor is perhaps the most difficult to determine. Testing of potential explosives in a laboratory is generally limited for reasons of safety to samples no larger than one gram. Outdoor testing is typically limited to one-kilogram samples.

[2] ARH-LD-119, page 5.

Our specific interest (stored salt cake) lies in the safety of kiloton quantities. Since actual testing on such quantities is totally impractical, we must draw conclusions by extrapolation.

In spite of the problems of extrapolation, considerable insight can be gained by conducting experiments on gram-sized samples to determine combustion limits. Any composition which supports combustion in a one-gram sample will certainly also support combustion in a larger sample. If, on the other hand, one is attempting to identify a combustion limit in a large mass, the fact that a small quantity will not support combustion does not prove that the large mass will not support combustion. It does, however, allow one to predict the relative size of an ignition source above which one must be concerned about combustion.

EXPERIMENTAL

METHOD

Various mixtures of organic materials and sodium nitrate were tested for self-supporting combustion. Similar samples in which the sodium nitrate was replaced by a simulated salt cake were also tested. Samples weighing approximately 1.0 gram were hand packed to a density of 1.1 - 1.4 g/cm³ in small aluminum foil capsules 8 mm in diameter and 25 mm long, constructed of a double wrap of household aluminum foil. The top 7 mm of the capsules were packed with a known combustible mixture of charcoal and sodium nitrate (the "starter," usually 80 percent NaNO₃ and 20 percent charcoal). The starter was ignited by touching the top surface with a small, heated nichrome wire (the nominal temperature was 1,000° C, even though the 80/20 starter mix would have ignited if the temperature of the igniter coil had only been slightly above

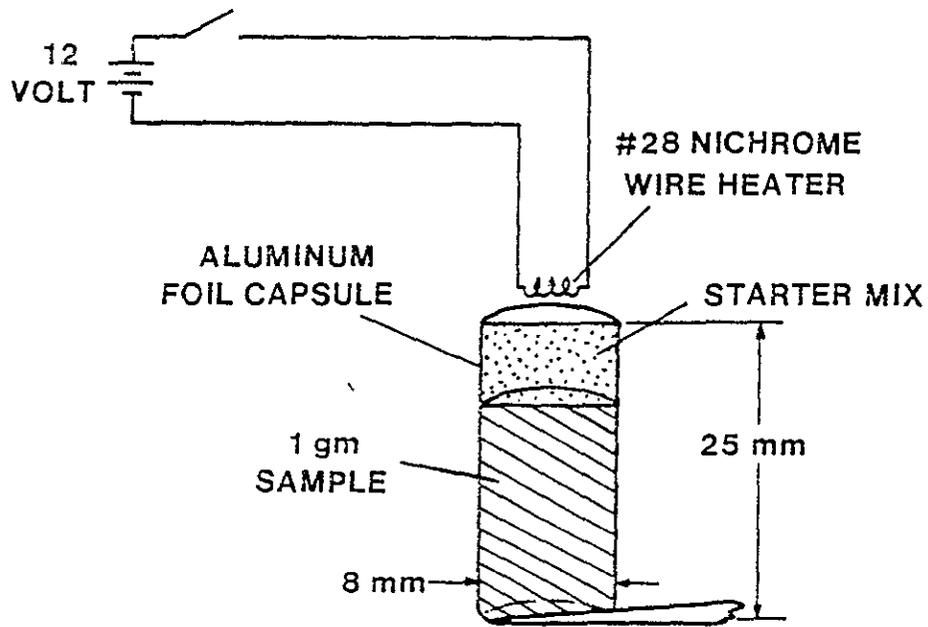
400° C). Within a few seconds, the starter burned violently and formed a molten drop approximately equal in size to its initial volume and at a fairly consistent temperature of about 900° C (bright red). This starter gave a reproducible high temperature, high-heat ignition source for each sample. Figure 1 shows the experimental setup.

Because the sample was essentially unconfined, the combustion process occurred at constant pressure, i.e. one atmosphere.

A stopwatch was used to record the time elapsed from initiation of the starter until the sample was completely burned. The combustion process was observed and recorded in qualitative terms: burned vigorously, violently, slowly, or uniformly; would not burn; splattered; etc. If the starter failed to ignite the test sample, the test sample was "prodded" with the heated nichrome igniter to give it additional encouragement to burn. Many of the samples would initially self-extinguish or fail to ignite but could be coaxed to burn by the igniter wire.

COMBUSTION DESCRIPTION

Ideally, it would have been preferable to measure either a flame temperature or propagation velocity or both for each sample. However, due to the size and configuration of the test capsule, "flame temperature" was either averaged out by rapid heat loss or impossible to see or measure. Theoretical flame temperatures as high as 3600° K were predicted; however, because of heat loss in the small samples, no evidence of temperatures greater than 1600-1800° K (white hot) was ever seen. In fact, the surface temperature rarely exceeded 930° K, as evidenced by failure to melt the aluminum foil capsule except in the hottest combustion events.



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FIGURE 1

EXPERIMENTAL SETUP
FOR COMBUSTION TESTS

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Because we tested a variety of compositions and were essentially unfamiliar with what would happen, we noted combustion characteristics at the time of the testing and constructed a scale of combustion descriptors after the experimentation was completed. The scale, in order of decreasing intensity, is shown and described in Table I.

MATERIALS

Reagent-grade sodium nitrate was used. It was crushed, weighed, and crushed and ground again while mixing. No attempt was made to dry the sodium nitrate.

Activated coconut charcoal was used for all charcoal tests. This has a higher carbon content and density, and lower ash content than the charcoal normally used in the manufacture of black powder. Analytical determinations were not made, but charcoal typically has 85 percent carbon, 3 - 5 percent absorbed gases, 1 - 5 percent moisture, and 1 - 3 percent ash, with the remainder consisting of heavy organic compounds. The charcoal was ground to a fine powder before mixing. Moisture content determinations which were made on the charcoal - sodium nitrate mixtures varied from 2.5 to 3.9 percent.

Dowex 21-K[®] (Dow Chemical Company) and Permutit SK[®] (Permutit Company) ion exchange resins were used. The inability to react fresh resin with sodium nitrate led us to bake the resin at 180 - 250° C in air, resulting in up to 65 percent weight loss before use.

Both paraffin oil and paraffin wax were tested. Each was used as received. The wax was melted so it could be mixed as a liquid, but ignition was attempted at room temperature.

Several tests were made with refined sugar in as-received condition. Some combustion tests were made with charcoal, resin, and sugar, and a simulated salt cake in place of sodium nitrate. The simulated salt cake was made according to a

TABLE II

COMBUSTION DESCRIPTOR SCALE

<u>Descriptor</u>	<u>Corresponding Observational Comments</u>
Violent	Total combustion time less than 20 sec; violent, very hot, explosive; sample spattered.
Rapid	Total combustion time of 20-40 sec; hot, fast.
Slow	Total combustion time greater than 40 sec, slow and controlled. Burned smoothly, quietly, continuously, uniformly.
Difficult	Time indefinite. Self-extinguished and reignited once or twice, or was hard to ignite (different from erratic in that at some point it would ignite and finally burn completely).
Erratic	Self-extinguished more than twice. Extremely difficult to ignite. Would only burn in presence of igniter. Entire sample would not burn.
No	Would not burn under any condition.

current popular recipe:

Dissolve 220 gm NaOH in 500 ml H₂O. Add 164 gm NaAlO₂, then add 138 gm NaNO₂ and 170 gm NaNO₃. Next add sufficient H₂O to bring the volume to one liter. Heat to 90 - 100° C and, while constantly stirring, add NaNO₃ until solution is saturated. Cool to 50° C and stir for 24 hours. Cool to 25° C and decant the supernatant liquid. The remaining solid is "salt cake."

The salt cake was dried at 150° C for several hours before weighing and mixing. No determination was made for the exact composition of the resultant salt cake. Other laboratory analyses have found that the salt cake from this recipe is 90 - 95 percent NaNO₃.

RESULTS

Sodium Nitrate - Charcoal

Combustion results for the NaNO₃ plus charcoal mix (with a nominal residual 3 percent moisture content) are summarized and displayed in Figure 2. The diameter of each data point shown is proportional to the number of tests at that point. The outer straight line boundary shows the maximum expected combustion behavior for a given nitrate concentration and the inner boundary identifies a minimum behavior. Rapid or violent combustion was observed for compositions from 65/35* to 85/15. This is consistent with black powder compositions which contain from 60 to 80 percent nitrate (generally KNO₃) but also contain

*We use the notation X/Y to represent the composition of any dry mix where X = the concentration, in weight percent, of oxidant (NaNO₃ or salt cake) and Y = the concentration of fuel (organic).

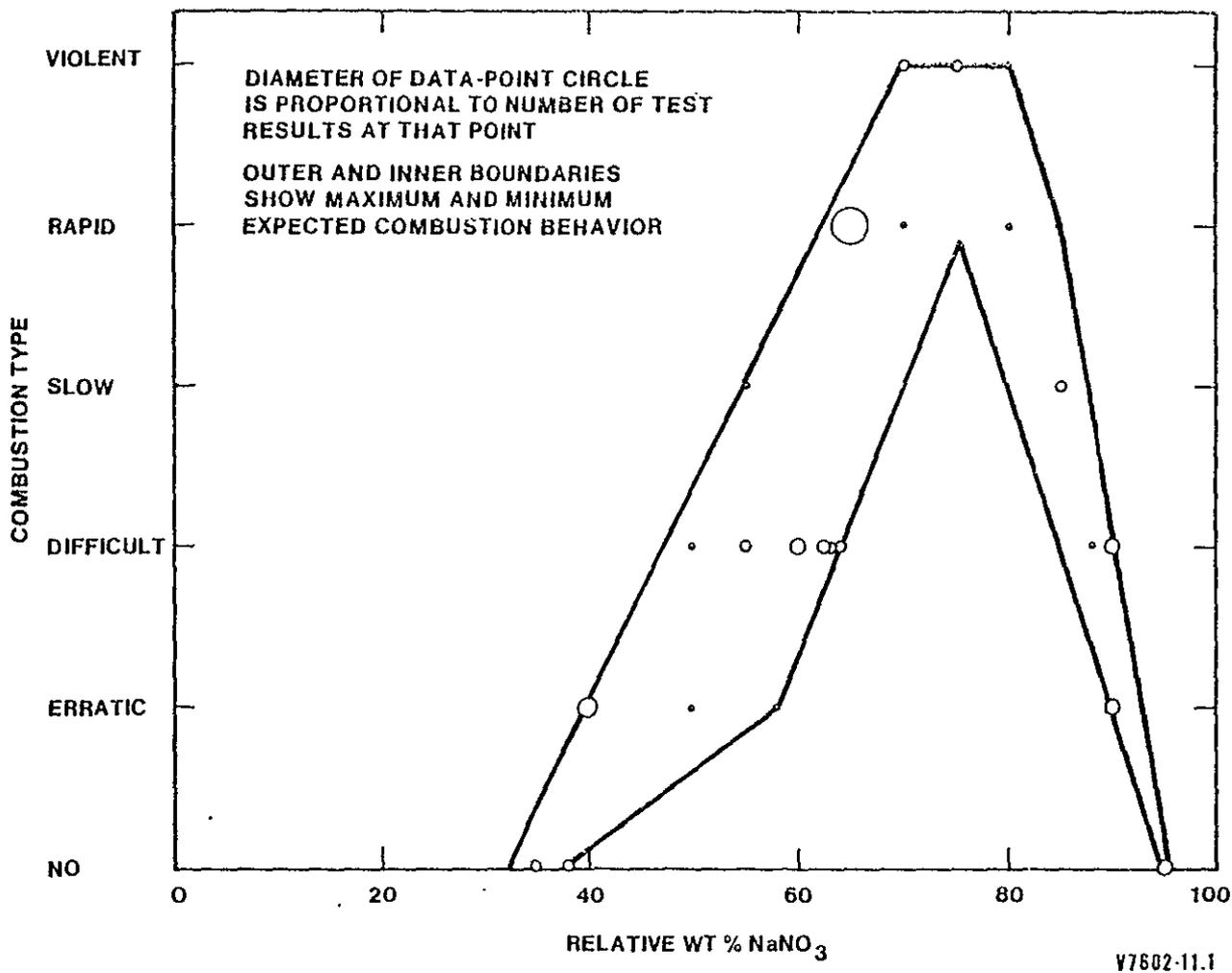


FIGURE 2

COMBUSTION RESULTS FOR
SODIUM NITRATE AND CHARCOAL

about 10 percent sulfur to promote burning. Optimum combustion occurred at about 75 percent nitrate instead of 85 percent nitrate, the stoichiometric composition. Combustion occurred anywhere between 40/60 to 90/10 mixtures.

At the fuel-rich limit (40/60) the carbon matrix can offer sufficient structural support and thermal insulation to allow ill-defined filamentary combustion in which a combustion process propagates through the matrix leaving the matrix unchanged in appearance but glued together with the product sodium carbonate. Since there is no supply of atmospheric oxygen, as soon as the sodium nitrate is depleted, the reaction extinguishes. The nonmelting nature of the carbon maintains a high temperature gradient to support localized combustion at nitrate crystals while maintaining the bulk temperature at 150 - 250° C. Thus, whether or not a specific 40/60 mix will burn depends on uniform mixing and packing density.

At the oxidant-rich limit, 90/10 to 95/5, excess reaction heat quickly melts any excess nitrate. The molten nitrate facilitates thermal equilibrium and quickly limits temperatures to the melting point of NaNO_3 (330°C), which is lower than the ignition temperature (400 - 440° C). In practice, the hot starter liquified the top several millimeters, heat was conducted rapidly throughout the capsule, and the combustion process was rapidly extinguished.

Salt Cake - Charcoal

Figure 3 shows the results of the combustion tests with salt cake and charcoal. Each point shown represents two tests. The behavior was much like the sodium nitrate - charcoal results except that the "rapid" or "violent" combustion region extended from 50/50 to 85/15 and the fuel-rich limit was lowered

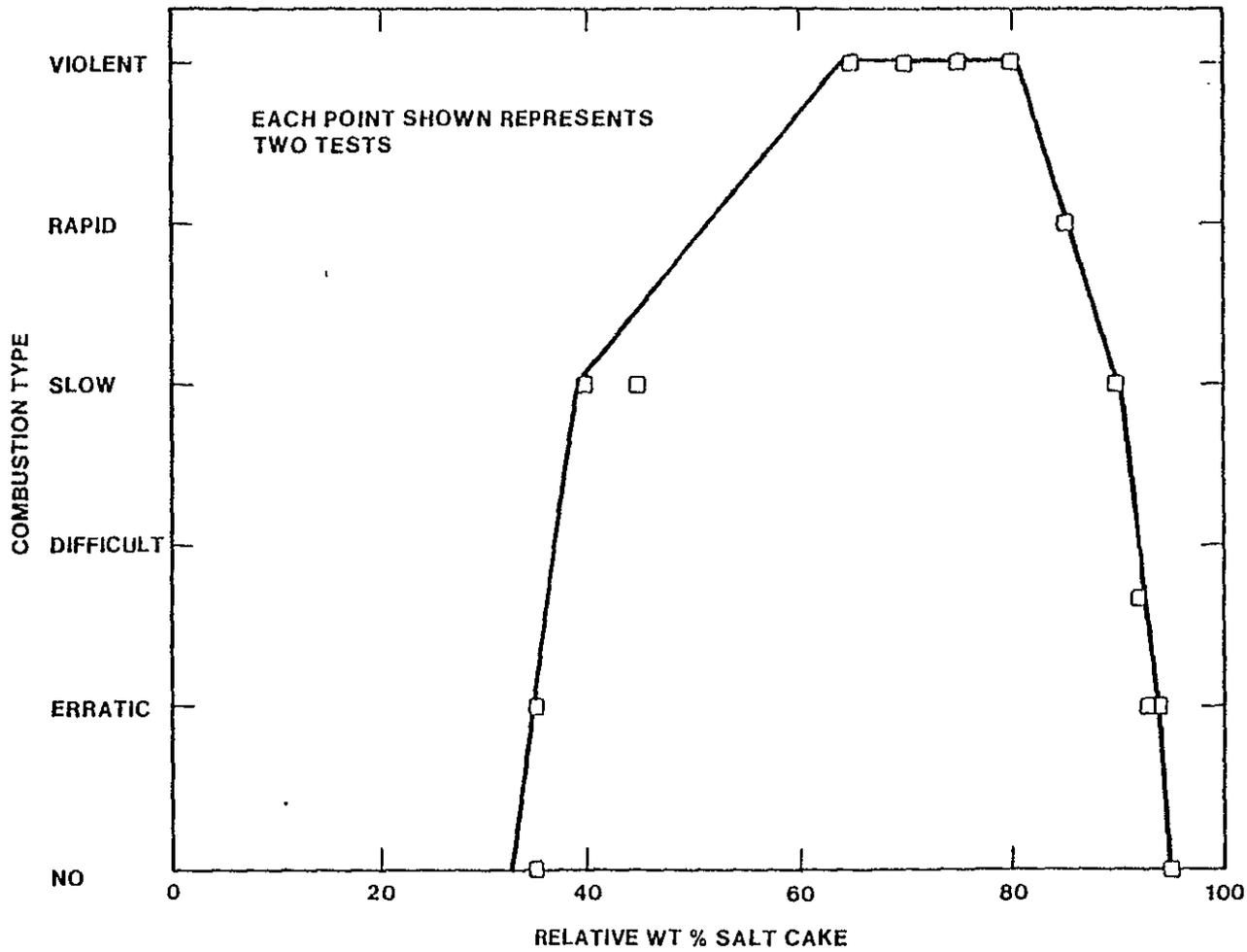


FIGURE 3
COMBUSTION RESULTS FOR
SIMULATED SALT CAKE AND CHARCOAL

from 40/60 to 35/65.

These effects were most likely due to the presence of NaNO_2 , which is more unstable (reactive) than NaNO_3 , even though it has a lower oxidizing power. The oxidant-rich limit appears to have increased just slightly so that 92/8, 93/7, and even 94/6 mixtures could be burned. Note, however, that 94 percent salt cake (which is 95 percent NaNO_3) is equivalent to 89.3 percent NaNO_3 (erratic). The 95/5 mix could not be burned under any conditions.

Sodium Nitrate - Charcoal - Moisture

Figure 4 shows the effect of increasing moisture content on an 80 percent sodium nitrate, 20 percent charcoal mixture. Up to 15 percent (by weight) moisture had no noticeable effect on the combustibility of the mixture. More precise quantitative measurements would show that the addition of water lowered the flame temperature and increased the gas pressure, but this was not detectable in our measurements.

As the mixture approached 20 percent moisture it became quite liquid, and the resultant increased heat transfer limited the temperature and made ignition more difficult. At 22 percent moisture the mixture could not be ignited or burned. The moisture content values shown in Figure 4 are based upon an accurately measured addition of liquid water plus an assumed 3 percent moisture content of the original mixture.

Dependence of Sodium Nitrate - Charcoal Combustion upon Initial Temperature

Very little effort was expended in this area because of the inherent problems in identifying initial temperature. For example, after the starter mix burned, but prior to ignition of the sample, the heat from the starter raised the temperature

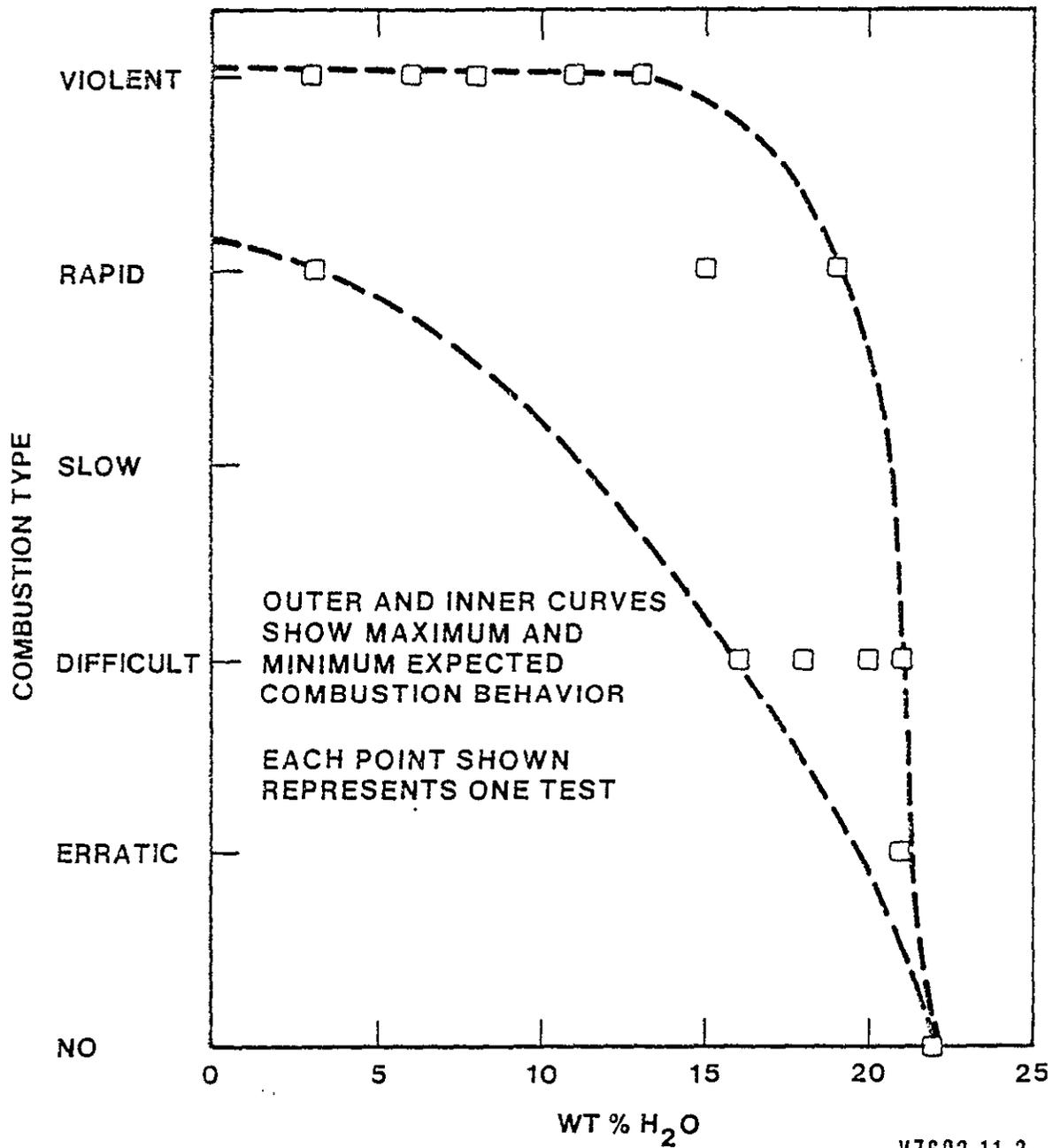


FIGURE 4

COMBUSTION RESULTS FOR
80% NaNO₃ - 20% CHARCOAL

TABLE II

INFLUENCE OF INITIAL TEMPERATURE ON COMBUSTION

Sample Composition (NaNO ₃ /Charcoal)	Initial Temperature	Combustion Type
88/12	25° C	Difficult
	150° C	Rapid
90/10	25° C	Erratic
	150° C	Erratic
	280° C	Slow - Rapid
92/8	25° C	No - Erratic
	280° C	Erratic - Slow
93/7	25° C	No
	280° C	Erratic
95/5	25° C	No
	120° C	No

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TABLE III

COMBUSTION TESTS WITH ION EXCHANGE
RESIN* - NaNO_3 MIXTURES

Pretreatment of Resin (% Wt Loss)	Composition Wt% Oxidant	Number of Tests	Type of Combustion	Comments
0	85	1	No	
0	85	1	No	Permutit [®] resin
0	65	1	Erratic	Sample size twice normal
0	75	3	No	Thermite starter
30	70	1	No	
40	85	2	No	Permutit [®] resin
50	70	1	No	
50	70	1	Difficult	Sample size twice normal
55	70	1	No	
55	70	2	Erratic	
55	70	1	Difficult	Sample size twice normal
65	35	2	No	Salt cake instead of NaNO_3
65	40	2	No	Salt Cake
65	70	1	Erratic	} Combustion dependent upon confinement of flames
65	70	3	Difficult	
65	70	4	Slow	
65	70	3	Violent	
65	90	2	No	Salt Cake
65	95	2	No	Salt Cake

*Dowex 21-k[®] unless otherwise noted.

90/10 mixtures with 65 percent weight loss resin could not be ignited.

Several tests, not shown in Table III, were made with a resin which had been boiled in a saturated NaNO_3 solution. The resulting dried resin, black in appearance, could not be ignited when mixed with nearly stoichiometric concentrations of NaNO_3 (70, 75, 80, and 85 percent).

Three of the resin mixtures which would not burn in the standard test capsule (8 mm diameter) were placed in a capsule with 4 times the volume (16 mm diameter). These samples exhibited "difficult" combustion.

Sodium Nitrate - Sugar

Six samples were tested using simulated salt cake and sugar with the following results: 75/25, violent combustion; 85/15, very slow (87 seconds, but did not extinguish until completely consumed); and 90/10, no combustion.

DISCUSSION

Although sodium nitrate is a powerful oxidizing agent (one gram of NaNO_3 has 40 percent of the energy of one gram of pure oxygen when oxidizing carbon, or, if viewed as an explosive, one ml of NaNO_3 has 450 times the energy of one ml of O_2) it is a stable molecule at temperatures below 300°C . Unlike most high explosives, sodium nitrate cannot decompose exothermically by itself at any temperature.

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Any exothermic reaction with sodium nitrate must be supported by a fuel.*

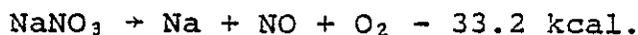
Even when mixed with a fuel (a compound or element in a more reduced state than NaNO_3), NaNO_3 is stable against both shock and decomposition at temperatures below 300°C . Sodium nitrate melts at 330°C ; even at this temperature, in the absence of some more unstable compound, ignition is impossible. Sodium nitrate boils incongruently ($\text{bp} = 380^\circ \text{C}$); that is, it decomposes to a vapor of a different composition than NaNO_3 . It is one or more of the decomposition fragments, NaO , O , O_2 , NO , NO_2 , or NO_3 , which participate in the oxidation process. (For the purposes of this discussion it is immaterial which decomposition product is primarily responsible.) This is the reason that ignition temperatures are so closely tied to the boiling point. All fuels which are sufficiently unstable to oxidize at 380°C will react with NaNO_3 at 380°C .

Pure carbon, which oxidizes at reasonable rates only above 550°C , will not ignite with NaNO_3 until then. Metallic powders (Al and Si in particular) tend to be even more stable and require ignition temperatures (again, with NaNO_3) of about $1100 - 1200^\circ \text{C}$. At about 700°C , NaNO_3 is so unstable that it reacts at "explosive" rates with almost any organic material which can be held in physical contact with it. Charcoal ignites with NaNO_3 at $380^\circ - 400^\circ \text{C}$ because small quantities of

*Many people confuse sodium nitrate, NaNO_3 , with ammonium nitrate, NH_4NO_3 , both common ingredients in explosives. Both are oxidants and both are mixed with fuels when used as explosives. However, NH_4NO_3 can detonate chiefly because it can decompose exothermically by itself (at 210°C) according to the reaction:



Sodium nitrate decomposes (at 380°C) endothermically according to:



more unstable hydrocarbons associated with the charcoal react first, raising the temperature high enough to support a carbon - NaNO_3 reaction.

This explains why charcoal is used as the fuel in black powder. The carbon fraction of charcoal does not vaporize in significant quantities below 2500°C . The small fraction of heavy organic compounds associated with charcoal do not volatilize in significant quantities below 700°C (typically the charcoal was charred at temperatures between $800 - 1100^\circ\text{C}$). The vaporizing, i.e. decomposing, NaNO_3 reacts with the nearby solid charcoal surfaces. Since gas - solid collisions are more likely than gas - gas collisions, the reaction rates are high and hence tend to be explosive.

The above combustion mechanism explains why combustion supported by sodium nitrate is totally different from an ordinary gas or candle flame. In a candle flame, a combustion zone is held above the fuel; the paraffin vaporizes (at still too low a temperature to react) and is convectively transported into the flame zone where it is heated to a temperature high enough to react. On the other hand, once NaNO_3 has been heated high enough to volatilize it is also hot enough to react. Consequently, the NaNO_3 -supported combustion occurs essentially within the solid or liquid phase. The combustion process actually occurs in the gas phase, but at a distance as small as 10^{-4} cm from the surface.

When combustion with paraffin or fresh resin was attempted, the paraffin and a significant fraction of the resin volatilized at temperatures well below 380°C . This evaporation rapidly cooled the test capsule. The vapor could not support combustion because the sodium nitrate was left behind, still a stable solid. Only when the resin was preheated to drive off most of the volatiles could a sodium nitrate combustion

process be supported. Sugar, on the other hand, is much less volatile than resin and, when it decomposes, leaves a large fraction of the original quantity as a solid carbonaceous residue, enabling it to react quite violently with the NaNO_3 . Sugar has a lower heat of reaction, gram for gram, than carbon, and therefore cannot support combustion at as lean a mixture; hence the 90/10 sugar mix would not burn.

These factors all contribute to confidence in the safety of the present high-level waste salt cake. The major organic additions to the waste tanks were resins and organic solvents. Even if they were present in concentrations theoretically adequate to support combustion, because of their volatility they are much more difficult to react than carbon (charcoal) and may, in fact, be impossible to ignite.

However, other factors confound the problem. The waste has spent many years at or near its boiling point. Therefore the lighter more volatile organic fractions have been removed, leaving behind the more stable components that may react more easily with sodium nitrate. After the waste has cooled below boiling, most of it is volume-reduced by an evaporation process which further removes volatile organic fractions.

The final point to be made concerns pressure. All tests reported here were conducted at atmospheric pressure. The reason that low-temperature volatilization of the fuel prevents combustion is that volatilization removes the fuel from the sphere of influence of the sodium nitrate. These volatile fractions could be held in contact with the sodium nitrate by means of increased pressure, a mechanism of major significance in the field of solid-propellant rocket engines, although not with respect to NaNO_3 . There probably exists a pressure above which NaNO_3 will react explosively with

paraffin oil (or any other organic). Such a pressure may or may not be beyond the physical realities of salt cake management.

RECOMMENDATIONS

A key factor yet to be established is the concentration of organic material within the salt cake. Special efforts are being made to ensure that total organic carbon determinations are part of the salt cake characterization program.

Additional tests with 5 - 10 percent resin (as received), charcoal, or paraffin in 50 - 100 kg lots with the ignition source (preferably a high temperature, low pressure source such as a one-pound mass of thermite) located in the center of the test material would be desirable. Because of the size and potential explosive nature of these tests, they would have to be conducted using all the precautions of high explosives testing.

Additional information should be obtained on the pressure dependence of paraffin or resin combustion with NaNO_3 . One-kg samples in sealed, pressure-limited containers should be tested with a variety of compositions at pressures of one to 100 atmospheres. Again, such tests have the potential of being destructively explosive and would require the respect accorded to similar quantities of TNT.

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