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AN EXPLOSIVE HAZARDS ANALYSIS
OF THE
EUTECTIC SOLUTION NaK AND KO_2

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ABSTRACT

This report concerns planning, preparation, conductance, and evaluation of field tests to determine the explosive hazards associated with the combining of the sodium-potassium eutectic alloy (NaK) with the superoxide of potassium (KO_2) under various conditions of state, contamination, and detonation initiation. The planning and preparation was conducted by Aerojet Nuclear Company (ANC) at the Idaho National Engineering Laboratory, and the explosive hazards testing was done by Cook Associates, Inc., at IRECO Chemicals Pelican Point Research and Development facility in Utah. The test results showed that binary combinations of pure NaK and KO_2 could not be made to detonate, although the mixtures will spontaneously ignite and burn. However, tertiary combinations of NaK, KO_2 plus a water or hydrocarbon contaminate produced explosive hazards under a variety of conditions. The work was performed as part of the Decontamination and Decommissioning (D and D) of the first Experimental Breeder Reactor (EBR-I) and was funded by 189c I-215.

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AN EXPLOSIVE HAZARDS ANALYSIS OF THE EUTECTIC SOLUTION NaK AND KO₂

I. INTRODUCTION

The first Experimental Breeder Reactor (EBR-I) was designated a Registered National Historic Landmark in 1966; however, before the facility could be opened to the general public it was necessary to remove safety hazards contained within the facility. During assessment of the hazards, Aerojet Nuclear Company (ANC), prime contractor to the Environmental Research and Development Agency's Idaho Operations Office, determined that the primary hazard involved 5,000 gallons of bulk sodium-potassium eutectic alloy (NaK) stored in the reactor's primary and secondary coolant systems, of which 4,000 gallons in the primary coolant system were known to be radioactively contaminated.

Prior to removal of the hazardous material (NaK) from the reactor facility, a hazards analysis was performed which consisted of an evaluation of all known hazards involved with the NaK from both the chemical and radiological standpoint. Although the NaK systems had been maintained under an argon cover since deactivation of the facility, it was assumed that the ingress of oxygen and water vapor could not have been completely prevented. It was therefore postulated and subsequently confirmed that the most stable oxide of NaK, KO₂, would be present as films or oxide scum on the liquid metal surface.

A literature review and consultations with others having NaK handling experience revealed at best suspect evidence regarding the explosive potential of the reaction between NaK and KO₂ [1 through 7]. Experience reported in USAEC Health and Safety Information Bulletin Issue No. 251, Explosions Involving Metallic Potassium or NaK, suggested the reaction could be explosive; however, in all the experiences of NaK-KO₂ explosions, other contaminants such as water and/or hydrocarbon compounds were known or expected to have been present. People with extensive NaK handling experience from Allied Chemical Company (ACC) and Mine Safety Appliance Research (MSAR) have indicated no experience of incidents in NaK systems attributable to KO₂. They emphasized, however, that inert cover gas should always be maintained over the NaK systems and contamination of the NaK with hydrocarbon compounds should be avoided.

Following the review of all known available information regarding the NaK/KO₂ reaction, it remained unclear whether the reaction could be explosive and if so how violent in terms of TNT equivalency. It was therefore determined that an explosive hazards analysis and evaluation of the NaK/KO₂ reaction would be required prior to the initiation of any NaK removal from the EBR-I facility. This report, therefore, is concerned with the planning, preparation, conductance, and evaluation of field tests to determine the explosive hazards associated with the NaK/KO₂ reaction under various conditions of state, contamination, and detonation initiation.

II. OBJECTIVE

Historically, evidence exists that the superoxide of potassium (KO_2), NaK, and some form of presumed contaminants have produced unstable conditions leading to explosive reactions. In view of the potential of KO_2 formation in the EBR-I NaK systems, an explosive hazards analysis and evaluation of the NaK/ KO_2 reaction was initiated. Three major objectives of the study are listed below:

- (1) To determine the conditions and properties under which KO_2 becomes an explosive hazard
- (2) To evaluate the explosive parameters of specific compound/ KO_2 mixtures
- (3) To compare the magnitude of any explosion derived with that of other well-known explosives.

Given the successful completion of the study objectives, the means to evaluate the total hazard potential associated with the removal of NaK from the EBR-I facility would be available.

III. TECHNICAL APPROACH

1. REQUEST FOR TECHNICAL PROPOSAL

A request for technical proposals was prepared for conducting explosive tests using Government-furnished NaK and KO₂. The scope of work defined the test objectives and provided that the work be done in two phases.

Phase I. Combine pure NaK, KO₂, H₂O, and hydrocarbon in all possible groupings of two or more compounds and under various conditions of atmosphere, heat, shock, and friction; and determine the combinations of compounds and physical conditions which represent an explosive hazard.

Phase II. Evaluate the properties of any explosive combinations using tests similar to the Standard Military Explosives Tests found in TM9-1910 or NAV OD 44811, and prepare a written report covering both phases of work.

2. PROPOSAL EVALUATION

Proposals were received from two qualified companies, and were evaluated on the basis of technical approach, previous experience in comparable work, key personnel assigned to the work, and cost effectiveness of the proposal. Cook Associates, Inc., of Salt Lake City, Utah, was selected as the explosive testing subcontractor and Subcontract S-1306 was awarded on January 2, 1974.

3. PROGRAM PLANNING

Meetings were held at INEL on January 8 and 9, 1974 and attended by personnel from ANC, ACC, and Cook Associates, Inc. A program plan, which scheduled the work and final report to be completed by February 15, 1974, was approved. It was also agreed that the best approach to mixing NaK with other compounds was provided by using encapsulated NaK. NaK encapsulated in ampoules could be ruptured remotely into the various other components of the system under optimum conditions for producing ignition and/or an explosion. ACC provided laboratory grade NaK in sealed 50 cc ampoules and a stainless steel vessel containing about three pounds of NaK. ANC provided the KO₂ and an explosives expert to work with the subcontractor. Cook Associates, Inc., provided the test facilities, instrumentation, explosives, and related supplies. Work was initiated January 11, 1974, at the Intermountain Research and Engineering Company (IRECO) Explosive Test Site B at Pelican Point, 40 miles north of Salt Lake City.

4. PLANNED TESTS

The tests to be conducted included five series of tests as follows^[8]:

4.1 Series A: NaK/KO₂

- A-1 Blasting Cap Initiation of Encapsulated NaK in Coarse KO₂
- A-2 Impact Initiation of Encapsulated NaK in Fine KO₂
- A-3 Closed Bomb Containing Encapsulated NaK and Powdered KO₂
- A-4 Cap-Initiation of Encapsulated NaK/KO₂ in Closed Bomb
- A-5 KO₂ Mixed in Free NaK
- A-6 NaK Poured Over Powdered KO₂
- A-7 NaK Poured Over Coarse KO₂
- A-8 Encapsulated NaK/KO₂ (Coarse) in Closed Steel Bomb
- A-9 NaK/KO₂ Reaction in Dry, Inert Atmosphere
- A-10 Reaction of Uncontaminated NaK/KO₂ in Dry, Inert Atmosphere
- A-11 Impact Sensitivity of Frozen NaK-KO₂ Mixtures.

4.2 Series B: KO₂/Liquid Hydrocarbon

- B-1 KO₂/Encapsulated Kerosene
- B-2 Differential Thermal Analysis (DTA) of KO₂/Fuel Oil
- B-3 Detonability of KO₂ Penetrated by Fuel Oil
- B-4 Cap-Sensitivity and Strength of 91 to 9 KO₂/Fuel Oil
- B-5 Impact Sensitivity of 91 to 9 Powdered KO₂/Fuel Oil
- B-6 Detonation Velocity of 91 to 9 KO₂/Fuel Oil.

4.3 Series C: NaK/H₂O

- C-1 Explosion Potential of Spilled NaK/H₂O
- C-2 Pipe-Confined NaK/H₂O Reaction.

4.4 Series D: NaK/KO₂/Fuel Oil

D-1 NaK Initiation of 91 to 9 KO₂/Fuel Oil.

4.5 Series E: NaK Oxidation

E-1 NaK in Air

E-2 NaK in Oxygen.

IV. TEST PROGRAM

1. TEST FACILITIES AND EQUIPMENT

The explosive testing was conducted at the IRECO Test Site B, which was extensively equipped for explosives research. The particular facility used was an inside-out (investigators inside, test charges outside) bombproof shelter with remote viewing capabilities for test observation. The bombproof shelter was equipped with a multiple safety interlocked electric firing and siren warning system, periscopes, communications, streak and framing cameras, velocity of detonation timers, explosive monitoring oscilloscopes, and bombproof magazines for storage of standard explosive items such as PETN, IRAMITE, slurry explosives, pentolite boosters, detonation cord, and electric firing caps. Other equipment which was used in the test program included seismic geophones, airblast transducers, differential thermal analyzers, blast diaphragms, and explosive witness plates. Prior to initiation of the test program, the explosive monitoring equipment was calibrated against standard explosives; see Table I.

2. PURPOSE, DESCRIPTION, AND RESULTS OF TEST PROGRAM

The test program involved three binary (condensed phase) combinations of concern, namely: NaK/H₂O, NaK/KO₂, and KO₂/hydrocarbon (fuel oil); two tertiary combinations of concern, namely NaK/KO₂/fuel oil and NaK/KO₂/H₂O; and one quadri-combination consisting of NaK/KO₂/H₂O/fuel oil. The test program consisted of five series of tests, with each series consisting of one or more individual tests.

2.1 Series A: NaK/KO₂ Combination

2.1.1: A-1: Electric Blasting Cap (EBC) Initiation of NaK in Coarse KO₂. This test was designed to bring clean NaK into intimate contact with pure KO₂ at a maximum rate and to provide an initiation source to initiate any explosive potential of the NaK/KO₂ mixture.

The setup for Test A-1 is shown in Figure 1. One No. 8 EBC was attached to each side of a 50 cc NaK vial which was nested in a 400 ml. glass beaker filled with 2 to 10 mesh KO₂. The "witness" systems used for the test consisted of (a) a uniform paper diaphragm sandwiched between two sheets of plywood with two gauge holes in sizes of 1/8, 1/4, 1/2, and one and two inches in diameter; (b) an Altec Lansing microphone pickup for long-range (600-foot) airblast; (c) a seismometer monitored at a distance of 600 feet; and (d) a 1/8-inch-thick by 4-inch-square aluminum witness plate placed under the charge. All tests in this investigation were conducted in IRECO's No. 2 inside-out bombproof or in No. 1 inside-out go/no-go bombproof, which are shown in Figures 2, 3, and 4.

TABLE I

CALIBRATION DATA FOR ESTIMATING EXPLOSIVE ENERGY RELEASE OF TEST CHARGES

Charge	Weight ^[a] (Pentolite equivalent)	Long-Range Air Blast ^[b]		Short-Range Air Blast -- Minimum Diameter of Paper Gauge Broken at Five Feet	Seismic Wave at 600 feet ^[c]	
		Deflection (inches)	Overpressure (lbs/ft ²)		Deflection (inches)	Particle Velocity (in./sec)
IREPRIME	8	0.11	0.04	>2	0.41	0.0012
"6C" Booster	35	0.37	0.13	2 in.	1.26	0.0036
"2A" Booster	166	0.90	0.31	1 in.	2.20	0.0064

[a] Cast 50/50 pentolite (1,200 calories per gram).

[b] Altec Lansing microphone, d-c coupling preamplifier (x 1 attenuation).

[c] S-36 seismometer, low-level preamplifier (x 200 attenuation).

Fig. 1. Setup in Test A-1.

Initiation of the EBCs produced a sudden ignition and a thermite type reaction of the NaK/KO₂ mixture which resulted in a large coherent smoke cloud. The cloud raised about 100 feet into the air and floated coherently with the wind for about 200 yards prior to settling (still in a coherent cloud) to the ground. The noise and seismic record showed a response equivalent to the two No. 8 EBCs used to initiate the test. The aluminum witness plate was unaffected and none of the paper witness gauges were broken. The NaK/KO₂ reaction was not explosive and did not produce a gas (only solid particulate smoke); however, the reaction took place at a rate of speed associated with explosives.

2.1.2 A-2: Impact Initiation of Encapsulated NaK in Fine KO₂. This test was designed to bring NaK and KO₂ into intimate contact, using impact to initiate the reaction. The test was intended to provide a more realistic simulation of conditions which might exist at EBR-I, and to provide a means for evaluation of the potential hazard due to impact mixing of NaK/KO₂.

The setup for Test A-2 is shown in Figure 5. A flat steel plate weighing 60 pounds was suspended over a 400 ml glass beaker containing a 50 cc NaK ampoule nested in approximately one-half pound of finely ground KO₂. Photographic coverage was provided to monitor the smoke cloud; otherwise the monitoring equipment was identical to that used in Test A-1.

The EBC was initiated allowing the 60 pound steel weight to impact the encapsulated NaK and the powdered KO₂. The reaction was essentially the same as in Test A-1: The seismic and long-range airblast waves were of negligible intensity, paper gauges were

Figure 2a. Diagram of IRECO's No. 2 inside-out bombproof.

Fig. 2b Streak camera and other timing equipment in IRECO's No. 2 bombproof.

Fig. 3 Photograph of IRECO's No. 2 inside-out bombproof.

Fig. 4 Looking eastward to IRECO's No. 1 ("go/no-go") bombproof from top of No. 2 bombproof. Tall tower is IRECO's "artificial borehole".

Fig. 5 Arrangement in Test A-2.

unbroken, and the aluminum witness plate was undamaged (see Figure 6). The smoke cloud was again coherent and floated away much like that in Test A-1. The cloud was photographed from a distance of 600 feet by shooting as rapidly as possible with an ordinary camera (see Figure 7).

Fig. 6 Result in Test A-2.

Fig. 7 Smoke cloud from Test A-2 ($\text{NaK} - \text{K}_2\text{O}_2$).

2.1.3 A-3: Closed Bomb Containing Encapsulated NaK and Powdered KO_2 . This test was designed to bring NaK and KO_2 together in a confined space in order to determine whether confinement would produce overpressure or induce an explosion of the mixture.

The setup for Test A-3 consisted of a section of three-inch-diameter aluminum pipe, capped at both ends, containing a 50 cc ampoule of NaK and approximately one-half pound of powdered KO_2 . A small 35-gram 50/50 pentolite 6c charge was placed against the pipe section to attempt detonation of the NaK/ KO_2 within the pipe-bomb. Monitoring equipment was identical to that used in the previous tests.

The pentolite charge was detonated and did succeed in rupturing the NaK ampoule as well as producing a large hole in the aluminum pipe section. The NaK/ KO_2 produced no explosion, only the thermite type reaction previously noted in Tests A-1 and A-2 (see Figure 8).

Fig. 8 Bomb of Test A-3 after "6C" booster-rupture of the bomb.

2.1.4 A-4: EBC Initiation of Encapsulated NaK/ KO_2 in Closed Bomb. This test was designed to bring NaK and KO_2 together in a confined space and maintain the confinement during the reaction of the NaK/ KO_2 .

The setup for Test A-4 was identical to Test A-3 except that a No. 8 EBC was placed inside the bomb to rupture the NaK vial (see Figure 9).

Initiation of the No. 8 EBC ruptured the NaK vial and the reaction of the NaK/ KO_2 caused flames to shoot out of the bomb through the 1/8-inch hole provided for the EBC lead wires. There was no explosion or registration of the test on the seismograph record or the paper diaphragm gauges. The NaK/ KO_2 reaction continued to completion within the bomb which was undamaged by the reaction. However, the bomb became very hot and

Fig. 9 Setup in Test A-4.

remained too hot to handle for 10 to 15 minutes. The bomb was uncapped and exposed to water. There was no $\text{NaK}/\text{H}_2\text{O}$ reaction, which indicated that all of the NaK had reacted with the KO_2 . Figure 10 shows the inside of the bomb after Test A-4 and water boiling in the pipe section due to the heat of solution of the K_2O and Na_2O residue in water.

2.1.5 A-5: KO_2 Mixed into Liquid NaK . Test A-5 was designed to determine whether dry KO_2 and pure NaK could be mixed without producing a reaction similar to the thermite reactions which has resulted in Tests A-1 through A-4.

The setup for Test A-5 consisted of 10 ml of NaK , powdered, dry KO_2 , a 400 ml beaker and a mixing rod. The NaK was placed into a dry beaker and KO_2 particles were added slowly while the mixture was stirred remotely. No reaction occurred, so addition of KO_2 was continued until a stoichiometric mixture had been formulated. The mixture was allowed to stand undisturbed, and after approximately ten minutes the mixture spontaneously ignited into a rapid thermite type reaction. Heat from the reaction caused the beaker to break, however (as in prior tests), there was no evidence of detonation.

2.1.6 A-6 through A-11. The next six tests were all designed to bring NaK/KO_2 into intimate contact under varying conditions of containment, atmosphere, and ignition method.

These tests are described and illustrated in Cook Associates, Inc., report WIS-6-74, ANC Subcontract No. S-1306^[8].

None of the tests resulted in any evidence of NaK/KO_2 having explosive properties. All tests continued to indicate that the NaK/KO_2 reaction was merely a very fast solid-liquid thermite-type reaction, producing great heat and resulting in K_2O and Na_2O residue.

Inside of pipe after test A-4.

Boiling from adding water to residue after test A-4.

Fig. 10 Bomb after NaK - KO_2 excursion in Test A-4.

2.2 Series B: KO_2 /Liquid Hydrocarbon (Fuel Oil) Combination

2.2.1 B-1: EBC Initiation of KO_2 /Fuel Oil. This test was designed to bring pure KO_2 into intimate contact with fuel oil at a maximum rate and to initiate any explosive potential of the KO_2 /fuel oil mixture.

The setup for Test B-1 is shown in Figure 11. One No. 8 EBC was attached to each side of a 50 cc vial of fuel oil which was nested in a 400 ml glass beaker filled with 2 to 10 mesh KO_2 . The beaker was placed in a one gallon can filled with sand. Monitoring equipment was identical to that used in previous tests.

Fig. 11 Setup in Test B-1.

Initiation of the EBCs failed to produce a significant reaction. Only a small amount of smoke and two jets due to the EBCs could be seen issuing from the charge. The gallon can was not ruptured nor did any of the monitoring equipment register explosive forces other than that due to the EBCs.

2.2.2 B-2: Differential Thermal Analysis (DTA) of KO_2 /Fuel Oil. This test was designed to determine how heat influenced the explosive potential of KO_2 /fuel oil.

The setup for Test B-2 is shown in Figure 12. The equipment consisted of a solid aluminum cylinder three inches in diameter and six inches high wrapped with electric heat tape and drilled to accommodate a thermometer and two 10 x 75 mm sample tubes. A sample tube containing one-half gram of powdered KO_2 was placed in the sample well and 10 mg of fuel oil was added.

Fig. 12 Diagram of IRECO's DTA furnace (not drawn to scale).

Addition of the fuel oil caused no apparent reaction; therefore, the DTA heater tape was activated. When the sample temperature reached 115°C, a small endotherm was observed which continued until the sample temperature reached 175°C. The exotherm began to develop strongly at 200°C and the sample detonated at 207°C. The test was repeated with identical results. The DTA record for one of the two tests is shown in Figure 13.

2.2.3 B-3: Detonability of KO_2 Penetrated by Fuel Oil. This test was designed to determine the detonability of the KO_2 penetrated by fuel oil.

A 400 ml beaker was filled with powdered KO_2 and a stoichiometric measure of fuel oil was added. After the oil had penetrated the KO_2 , a No. 8 EBC inserted in an 8-gram charge of cast 50/50 pentolite was placed in the beaker as a detonator (see Figures 14a and 14b). Monitoring equipment was identical to that used in previous tests.

The detonator was activated and the charge appeared to detonate. There was no residue from the charge and the aluminum witness plate was bent (see Figure 14c). Both sets of two-inch and one-inch paper gauges and one of the two 1/2-inch paper gauges ruptured. The seismograph gave a peak-to-peak deflection of 2.34 inches compared with a deflection of 2.20 inches for a 2A booster (166 grams of pentolite). The particle velocity was 0.0067 inch per second compared with 0.0064 inch per second for the 2A booster. Based upon the seismometer report, the estimated energy release in the detonation was equivalent to 175 grams of 50/50 pentolite, or about 210 kilocalories, based on 1,200 calories per gram for 50/50 pentolite. The explosive strength of the 91/9 KO_2 fuel oil mixture was about 525 calories per gram.

2.2.4 B-4: Cap Sensitivity and Strength of Blended 91/9 KO_2 /Fuel Oil. This test was designed to determine the optimum strength, sensitivity, and velocity of a blended 91/9 mixture of KO_2 /fuel oil.

The test consisted of three shots. Each shot was prepared by mixing 387 grams of coarse KO_2 with 38 grams of fuel oil and placing the mixture into a 400 ml beaker (see Figure 15a). The first shot contained a No. 8 EBC, the second shot a No. 4 EBC, and the third shot a No. 5 EBC. The strength of a cap increases approximately in proportion to the numerical assignment; i.e., a No. 4 EBC is about half as strong as a No. 8 EBC.

The blended and balanced mixture of KO_2 /fuel oil when initiated with a No. 8 EBC detonated completely and strongly. Both sets of the two-inch and one-inch and the lower of the two 1/2-inch paper diaphragm gauges were broken; the seismometer readings at 600 feet were 2.35-inch peak-to-peak deflection and a 0.00674 inch per second particle velocity; the aluminum witness plate was deformed and the underlying steel plate slightly bent. See Figures 15b and 15c. Based upon the seismic results (Figure 16), the energy release was 530 calories per gram.

The second KO_2 /fuel oil charge when initiated with a No. 4 EBC failed to detonate; however, the charge produced a fireball and a large smoke cloud similar to that in Tests A-1

Fig. 13 DTA record for KO₂-FO.

a

b

c

Fig. 14 (a) KO_2 and FO before mixing, (b) after FO was poured into the KO_2 , and (c) the result of initiation with an IREPRIME (8-gram pentolite) booster.

a

b

Fig. 15 Test B-4 (a) KO_2 - FO before mixing, (b) witness plate and (c) the smoke cloud produced when detonated with a No. 8 EBC.

Fig. 16 Seismic record of short in Test B-4 compared with 2A Booster.

and A-2. The No. 4 EBC was not strong enough to initiate a detonation, only a part deflagration.

The third KO_2 /fuel oil charge, when initiated with a No. 5 EBC, also failed to detonate; however, the fireball was characteristic of an explosive deflagration. The two-inch paper diaphragm gauges were broken; however, the explosive energy release was less than one-fourth as great as when the No. 8 EBC was used. The detonation cap sensitivity of the 91/9 coarse KO_2 /fuel oil is possibly a No. 6 EBC and definitely a No. 8 EBC.

2.2.5 B-5: Impact Sensitivity of 91/9 Powdered KO_2 /Fuel Oil. This test was designed to compare the impact sensitivity of balanced KO_2 /fuel oil with PETN and TNT.

A small sample of 91/9 powdered KO_2 /fuel oil was prepared by mixing in the mortar of the drop-weight test apparatus. The drop-weight used was eight pounds.

The results of the impact sensitivity tests are shown in Table II. Dry samples (no adsorbed water) are shown to be quite sensitive, equivalent to fine PETN which is one of the most sensitive of the secondary high explosives. However, when the samples adsorbed water from the atmosphere (as could be seen by the reddish coloration that appeared on the sample with time) the sensitivity dropped off to less than a third as great as that for a fresh, dry sample.

2.2.6 B-6: Detonation Velocity of 91/9 KO_2 /Fuel Oil. This test was designed to measure the detonation velocity of the KO_2 /fuel oil mixture.

A charge of 400 grams of 91/9 coarse KO_2 /fuel oil was placed in a 1-1/2-inch diameter by 18-inch-long velocity tube containing two velocity probes placed 10 inches apart. The charge was mounted on a witness plate and had a No. 6 EBC and 35 gram 6C booster at an initiator. See Figure 17.

The velocity results for the balanced KO_2 /fuel oil are shown in Table III.

2.3 Series C: NaK- H_2O (Water) Explosion Potential

2.3.1 C-1: NaK/ KO_2 / H_2O Explosion Potential. This test was designed to bring NaK coated with KO_2 into contact with H_2O , and to determine the explosive potential of the combination.

A glass beaker containing 25 cc of NaK, which had been allowed to oxidize at the surface, was arranged over a one-gallon metal bucket containing about one quart of water. The beaker was arranged so that its contents could be spilled into the water remotely from within the bombproof (see Figure 18a and 18b). Monitoring equipment was identical with that used in previous tests.

The beaker was tripped to the bucket of water and a loud explosion occurred producing a large cloud of smoke. The bucket suffered a 1-inch-long by 1/16-inch rupture at

TABLE II

IMPACT SENSITIVITY OF BALANCES $\text{KO}_2\text{-FO}$

PETN		91/9 Fine $\text{KO}_2\text{-AN}$	
Height (inches)	Result [a]	Height (inches)	Result [a]
10	D	34	D
5	D	12	D
1.75	F	3	D
4.50	D	2	F
3.125	F	3	D
2.5	D	2.5	F
2	3F	3	D
2.5	D	2.5	3F
TNT (fine flaked)		3	2F
27	F	3.5	F
34	2F	4	F
		4.5	D
		3.5	F
		4	F
		4.6	D
		4.1	F
		4.6	D
		4.6	F
		5	F
		10	F

[a] D - explosion (8-pound weight)

F - failure

the waterline and was otherwise slightly expanded. However, none of the paper diaphragm gauges were broken. According to Table I, the explosion yielded less than 40 kilocalories heat of explosion and less than one mole of gas. Since the reaction of 25 grams of NaK in H_2O should generate 24 kilocalories and 0.4 mole of H_2 , which could react explosively with the oxygen in the atmosphere to produce an energy release greater than 40 kilocalories, it was concluded that NaK/ H_2O reaction was incomplete and/or that the H_2 did not react explosively with the oxygen in the air.

Fig. 17 Photograph and diagram setup for velocity measurements of $KO_2 - FO$.

TABLE III

VELOCITY RESULTS FOR BALANCES KO_2 -FO

(91/9) Coarse KO_2 -No. 2 FO ^[a]			(91/9) Ground KO_2 -No. 2 FO ^[b]		
Diameter (inches)	Density (g/cc)	Velocity (m/sec)	Diameter (inches)	Density (g/cc)	Velocity (m/sec)
2	0.76	540	1.5	0.85	1,160
3	0.77	733	1.5	0.89	1,050

[a] Owing to coarseness of KO_2 , FO did not mix uniformly with the KO_2 particles.

[b] All these charges were 18 inches in length.

2.3.2 C-2: NaK/ H_2O Reaction in Confined Bomb. This test was designed to bring NaK into contact with H_2O within a closed bomb in order to determine the explosive potential of the NaK/ H_2O mixture.

Five 10 cc ampoules of NaK were inserted into a 1-1/2-inch diameter by 12-inch-long steel pipe section. The pipe was capped at one end, filled with water, and then capped at the other end. The bomb was arranged at the top of a tripod so that it could be dropped onto a steel plate from a height of about 10 feet. A No. 8 EBC and a section of primacord provided the bomb release mechanism. See Figure 19a.

Initiation of the No. 8 EBC released the bomb which impacted the steel plate. The NaK vials were ruptured, releasing the NaK into the contained water. After impact, there was a short delay of less than one second, followed by a loud explosion and a cloud of smoke. The end pieces of both of the steel caps were blown off (see Figures 19b and 19c); however, none of the paper diaphragm gauges were ruptured, again showing that the explosion was of less magnitude than that of a 35 gram 50/50 pentolite charge (6C booster). Since the reaction took place in a closed system, there was no contributory energy release caused by the H_2/O_2 explosive reaction.

2.4 Series D: NaK/ KO_2 /Fuel Oil

2.4.1 D-1: NaK Initiation of 91/9 KO_2 /Fuel Oil. This test was designed to bring NaK into intimate contact with a stoichiometric blend of KO_2 /fuel oil in order to check for spontaneous initiation and to determine the explosive potential of the mixture.

One pound of blended KO_2 /fuel oil was placed in a 700 ml beaker, and a 25 ml ampoule of NaK was placed on top of the charge. A No. 6 EBC was attached to the ampoule (see Figure 20a).

a. Photograph before beaker was placed on support.

b. Arrangement for studying NaK - H₂O explosion potential.

Fig. 18 Test C-1

a

b

c

Fig. 19 (a) NaK - H_2O bomb ready for breaking of vials by impact, (b) and (c) bomb after impact and explosion.

a

b

Fig. 20 (a) Setup for Test D-1 coarse KO_2 (b) Result of rupture of NaK Vial with EBC.

The No. 6 EBC ruptured the 25 ml NaK ampoule and this action was accompanied by a large flame and much smoke. The KO_2 /fuel oil/NaK reacted completely; however, there was no explosion. The reaction was a fast deflagration (see Figure 20b).

2.5 Series E: NaK Oxidation

2.5.1 E-1: NaK in Air. This test was designed to allow NaK to oxidize in an air atmosphere to determine if the NaK would self-ignite with the KO_2 formed.

A glass beaker containing 25 cc of NaK was allowed to oxidize at the surface for a period of 40 minutes at 0°C .

An oxide film developed on top of the NaK; however, the sample did not self-ignite. The sample was later used in Test C-1.

2.5.2 E-2: NaK in Oxygen. This test was designed to expose NaK to pure dry oxygen over a prolonged period, and to determine the hazards associated with an oxide film rupture in the NaK.

A vial 1 inch in diameter and 14 inches long, having a stopcock at each end, was vacuum packed with glass wool and NaK. The glass wool was added to optimize the NaK surface area. Oxygen was provided via rubber tubing from an oxygen cylinder through a CaCl_2 drying tube to the vial stopcock. The stopcock was opened allowing dry oxygen to enter the vial. The oxygen pressure was set at 2 to 3 psig and the test setup was left to react from 1:30 p.m. January 22 until 10:45 a.m. January 23, 1975. See Figures 21a, b, and c.

A considerable amount of yellow superoxide (KO_2) had been formed during the 21-hour period; however, no spontaneous ignition had occurred. The vial was remotely manipulated in an attempt to mix the NaK and the KO_2 film. The attempt was unsuccessful, as the KO_2 film prevented the NaK from flowing, even when the pressure on the system was increased to 15 psig. The vial was finally broken, allowing some of the NaK to drop out on the snow covered ground where it reacted explosively. However, there was no ignition or explosion within the intact portion of the vial. See Figure 21d.

a

b

Fig. 21 Test E-2a - (a) Vial after connection of O_2 line (also showing drying tube), (b) after opening vial to introduce O_2 at 2-3 psig, (c) after opening stopcock on opposite end to force NaK out of tube, and (d) burning of NaK after rupture of vial.

V. DISCUSSION

Review of the test program shows that the greatest effort was expended on the Series A tests involving the binary system NaK/KO_2 and to a lesser extent the tertiary system $\text{NaK/KO}_2/\text{H}_2\text{O}$.

In the Series A tests, the NaK/KO_2 system was combined violently by electric blasting caps and by impact from a dropped weight; was confined in a bomb and triggered by an electric blasting cap; was hand-mixed at near stoichiometric measurements; was frozen and impacted; and was partially melted and impacted. The tests all resulted in ignition of the NaK and KO_2 and deflagration or rapid thermite-type burning with no measurable gas formation. The reaction time and rate were dependent upon the ignition means and the presence of water. When blasting caps and impact were used to initiate the reaction, ignition was immediate and the rate of deflagration very high. When the NaK and KO_2 were merely mixed, the ignition rate was slower, varying from 8 to 11 minutes in the open with moisture present, and taking 18 minutes in an inert atmosphere with no moisture present.

In the Series B tests, the KO_2 /liquid hydrocarbon system was combined violently by electric blasting caps, by mixing mechanically and heating, and by penetrating KO_2 with fuel oil and detonating. When it was determined that the KO_2 /liquid hydrocarbon system could be detonated, impact-sensitivity tests and detonation velocity tests were performed. It was determined that the dry system (no adsorbed water) was quite sensitive -- equivalent to PETN. It was also found that atmospheric water adsorbed by the KO_2 would lessen the sensitivity of the system by as much as a factor of three. Detonation velocity of the system varied from 540 to 1,160 meters per second depending upon the KO_2 particle size, with the finer particled system producing the highest detonation velocity.

In the Series C tests, the $\text{NaK/KO}_2/\text{H}_2\text{O}$ system and the $\text{NaK/H}_2\text{O}$ systems were briefly explored. Each of these systems reacted explosively during the mixing phase with no detonation device required. The NaK water reaction produced hydrogen gas (GH_2) and exothermic heat. The reaction was more violent when open to the atmospheric oxygen (O_2). No attempt was made to quantify the contribution made by the reaction of $\text{NaK/H}_2\text{O}$ or the GH_2/GO_2 reaction; however, the combined theoretical explosive potential could approach that of nitroglycerin. The Series C tests were held to a minimum since the EBR-I NaK system had been maintained under an inert gas atmosphere and would not be expected to contain free H_2O .

The Series D testing consisted of a single investigation of the NaK/KO_2 /fuel oil system. The test which consisted of spilling NaK onto a mixture of KO_2 and fuel oil did not produce an explosive detonation, only a fast deflagration. The testing was curtailed since the EBR-I NaK system was known to be free of hydrocarbons. Impact and shock testing of the system might well have produced an explosive detonation, however, similar to that produced in the KO_2 /fuel oil system.

The Series E testing provided for the natural formation of a KO_2 film over the NaK surface, considered more representative of potential conditions in the EBR-I NaK systems should KO_2 be present in the systems component parts. The KO_2 film which formed over the NaK was quite tenacious and attempts to force mix the KO_2 film with the NaK by mechanically shaking the containers failed. There was no spontaneous ignition of the system, due probably to the difficulty experienced in getting the KO_2 film to mix with the NaK.

VI. CONCLUSIONS

The conclusions drawn from the results of the test program conducted at the IRECO facilities are as follows:

- (1) Pure KO_2 can be mixed at ambient temperature with solid or liquid NaK with no immediate reaction either thermal or explosive; however, such mixtures will spontaneously ignite after an induction period of approximately 18 minutes. The ensuing reaction will not be explosive (high speed reaction producing gas, high pressure, and loud noise) but will be a nonexplosive, high speed, solid-phase, thermite-type burning reaction.
- (2) Pure KO_2 and liquid or solid NaK mixtures can not be made to explode in the "drop-weight" or "impact" tests. They will only ignite as previously indicated.
- (3) Pure KO_2 and liquid NaK mixtures can not be detonated using standard explosive detonators. The reaction is an extremely fast, condensed-phase nonexplosive reaction.
- (4) NaK and H_2O (water) react instantly, spontaneously, and explosively when brought into contact. However, the rate of pressure development in the NaK-water reaction is relatively slow, more like an explosive deflagration rather than a detonation. When NaK was poured into an excess of water, the observed explosion produced an energy release of somewhat less than 400 calories per gram of NaK, compared with about 1,000 calories per gram for TNT and about 1,300 calories per gram for nitroglycerin. However, if large quantities of NaK and water were allowed to react, the free hydrogen produced in the reaction could mix with the atmospheric oxygen, greatly enhancing the overall explosive potential to a theoretical 1,380 calories per gram, which is comparable to nitroglycerin.
- (5) Moisture in KO_2 reduces the induction period for spontaneous combustion of KO_2 and NaK; however, even when the moisture content is increased to cause instantaneous ignition, the mixture does not explode but results in an extremely rapid and hot condensed-phase combustion.
- (6) Mixtures of KO_2 and liquid hydrocarbon, e.g., No. 2 fuel oil (FO), can be mixed safely at ambient temperature. This is not true of all organic compounds however, since acetone will react spontaneously with KO_2 at ambient temperature. The KO_2 and

FO mixture is dangerous since it does explode spontaneously at about 207°C and is very sensitive to impact and friction. The impact sensitivity of dry KO_2 mixed with FO is comparable to that of PETN, one of the most sensitive solid secondary high explosives.

In summary, the study objectives were attained. It can be concluded that the binary combination of greatest concern, pure NaK and KO_2 , is not an explosive hazard even though the mixture will spontaneously ignite and burn. It can also be concluded that any of the other combinations involving NaK- KO_2 plus a contaminate (water or hydrocarbon) can produce an explosive hazard under a variety of conditions. It is concluded that explosions involving NaK and KO_2 referred to in the literature were caused by the introduction of a third or fourth component to the system in the form of organic or moisture contamination.

Comprehensive testing of contaminated NaK and KO_2 systems, although beyond the scope of this study, might clarify the exact mechanism which evolved the explosive situations referred to in the literature. Certainly the secondary reaction of hydrogen gas with atmospheric oxygen (ignited by the KO_2 -NaK thermite reaction) has not been completely investigated. However, since the system of concern (EBR-I) has been maintained in an inert atmosphere, the known absence of free oxygen within the system eliminates the potential hazard of a $\text{H}_2 + \text{O}_2$ reaction.

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