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Modified Ammonium Perchlorate Incorporating Potassium, Permanganate, and Dichromate Dopants [U]

by

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ABSTRACT

(C) The present study of ammonium perchlorate which is grown as whisker crystals from porous glass by a recently discovered process is extended to doped ammonium perchlorate. The selection of dopants in whisker ammonium perchlorate is limited initially to those materials capable of isomorphic substitution in the ammonium perchlorate lattice structure. These dopants include potassium perchlorate, ammonium permanganate, and ammonium dichromate. The characterization of the doped whisker ammonium perchlorate is reported together with burning rate data of propellant incorporating the doped oxidizer.

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W. J. Moran, RADM, USN . . . Commander

H. G. Wilson . . . Technical Director

FOREWORD

(C) The program "Propellant Applications of Modified Ammonium Perchlorate" is based on the discovery of a process for growing whisker ammonium perchlorate. The technical objective of the work is to study various aspects of the new whisker ammonium perchlorate synthesis and to evaluate the use of this new oxidizer in propellant and explosive combustion and behavior. The present report focuses on the incorporation of several dopants in the whisker ammonium perchlorate for possible catalytic effects in propellant burning rates.

(C) This project was supported under the Independent Exploratory Development Program ZFXX 212 001 at the Naval Weapons Center and by the Director of Laboratory Programs, Task Assignment No. ZR011-01-01. The financial support is enabling the program to explore the many faceted implications of a whisker oxidizer in propulsion, explosive, and pyrotechnic technology.

Released by
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1 November 1971

Under authority of
HUGH W. HUNTER, Head
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CONTENTS

ACKNOWLEDGMENT

(U) The authors wish to acknowledge with appreciation the contribution of Dr. C. Howard Shomate for assistance in the mathematical analysis of the burning rate data.

INTRODUCTION

(C) Modified ammonium perchlorate is a descriptive term to denote whisker ammonium perchlorate (AP) grown by a new method of preparing crystal AP of rather fine particle size. The whisker crystals of AP are grown by allowing an aqueous solution to pass through porous glass tubing and to evaporate on the surface of glass under near-ambient conditions. A full description of the method of growth is given in an earlier report (Ref. 1). That report summarized the initial characterization of the whisker AP, the laboratory scale-up of facilities for preparation of the modified oxidizer (Fig. 1), and comparative burning rate data in a model composite propellant. Recommendations for future work identified a number of important research and development areas requiring further investigation. Two research areas of interest centered on the whisker AP itself: the incorporation of selective dopants in the whisker AP for possible catalytic effect in influencing propellant burning rate and secondly, control of whisker diameter in the submicron range.

(C) The present report covers the characterization of whisker AP doped with potassium, permanganate, or dichromate ions and the burning rates of a model propellant formulation incorporating the doped oxidizer whiskers.

GROWTH OF DOPED WHISKER AMMONIUM PERCHLORATE (C)

(C) The procedure adopted for the growth of doped whisker AP was essentially that employed in the whisker AP facility (Ref. 1). The tubes, nominally 25 mm OD, were arranged in a bay protected from laboratory drafts and heated to about 35°C with banks of infrared lamps. The 3-ft tubes which were thinly coated with the Silastic RTV 732 silicone rubber were fed the doped AP solution from polyethylene containers in a self-leveling arrangement. The usual concentration was 165 grams of AP per liter of water. In several instances the use of doped AP solutions required a dilution of the standard solution in order to prevent crystallization of the doped AP inside the porous glass tubes.

(C) The selection of dopants for AP whiskers was limited to those materials capable of isomorphic substitution within the orthorhombic lattice structure for AP. The dopants include potassium perchlorate, ammonium permanganate, and ammonium dichromate. These dopants in single crystals of AP have been studied previously (Ref. 2) and thus would provide a rather definitive basis for observation of unusual or otherwise anomalous effects. The potassium perchlorate and ammonium dichromate were Reagent Grade chemicals; the ammonium permanganate was freshly prepared as described by Bircumshaw and Tayler (Ref. 3) immediately prior to use. The ammonium permanganate was used directly without analysis. Since neither the solid nor solution of ammonium permanganate is very stable under ambient conditions, lower oxides of manganese were present in undetermined amounts.

(C) The doped whisker AP was grown from aqueous solutions containing the ammonium perchlorate and the respective dopant. The solutions had compositions within the ranges shown in Table 1. The composition of solutions is specified on a weight percent of dopant relative to the total weight of solids (essentially AP) in solution. For example, a 1% potassium perchlorate solution (or a 0.28% potassium ion solution) would contain 1 g of potassium perchlorate, 99 g AP, and water. The nominal solution compositions were 1.00% MnO_4^-; 2.08% $Cr_2O_7^=$; 0.28, 1.00, 1.41, and 2.02% K^+. The analysis of the whisker AP for dopants is shown in the last column of Table 1. The analyses were done by flame emission or atomic absorption spectroscopy. The source or sources of the variation in the potassium analyses is not fully understood, although a plausible explanation is discussed later in this report.

(U) In a separate laboratory experiment the feed solution was sampled within the porous tube at the same time the whiskers were removed for analysis. The sampling was done every other day. The results are shown in Table 2. The above results indicate a distribution coefficient of about 1.5 which favors retention of the potassium within the solution rather than selective incorporation in the whisker crystal. Independent data from the growth of large, single AP crystals under equilibrium conditions indicate a distribution coefficient greater than 10 for preferential incorporation of the potassium ion within the AP crystal.

(C) TABLE 2. Distribution of Potassium Between Solution and Whiskers

K^+ in feed solution, %	0.74	0.74	0.64
K^+ in whiskers, %	0.49	0.49	0.45
Distribution ratio	1.51	1.51	1.42

(C) With other dopants, the single crystal studies indicated that permanganate was present in the crystal at values directly related to the solution concentration, while dichromate was present at much lower concentrations. Qualitatively, the analyses in Table 1 would suggest no preferential incorporation of potassium or permanganate and partial rejection of the dichromate in the whisker AP.

(C) TABLE 1. Composition of Whisker Ammonium Perchlorate and Feed Solutions

Whisker designation	Dopant	Solution Composition[a]			Whisker analysis for dopant ion, %
		Dopant, %	Dopant ion, %	g Solid/1 H_2O	
KW-1	$KClO_4$	1.00	0.28 K^+	165	0.34 K^+
KW-1A	$KClO_4$	3.53	1.00 K^+	99	1.20, 1.34 K^+
KW-5	$KClO_4$	5.00	1.41 K^+	165	1.09[b] K^+
KW-2	$KClO_4$	7.14	2.02 K^+	82.5	2.08, 1.49, 1.20, 1.30, 1.44[c], 2.36[c], 0.33[d,e], 1.11[d], 0.40, 5.63, 4.32, 0.18, 2.77, 2.61, 2.50, 1.97, 2.50 K^+
MnW-1	NH_4MnO_4	1.15	1.00 MnO_4^-	165	0.89 MnO_4^-
CrW-1	$(NH_4)_2Cr_2O_7$	2.42	2.08 $Cr_2O_7^{=}$	165	0.25, 0.27 $Cr_2O_7^{=}$
Undoped				165	0.008 K^+

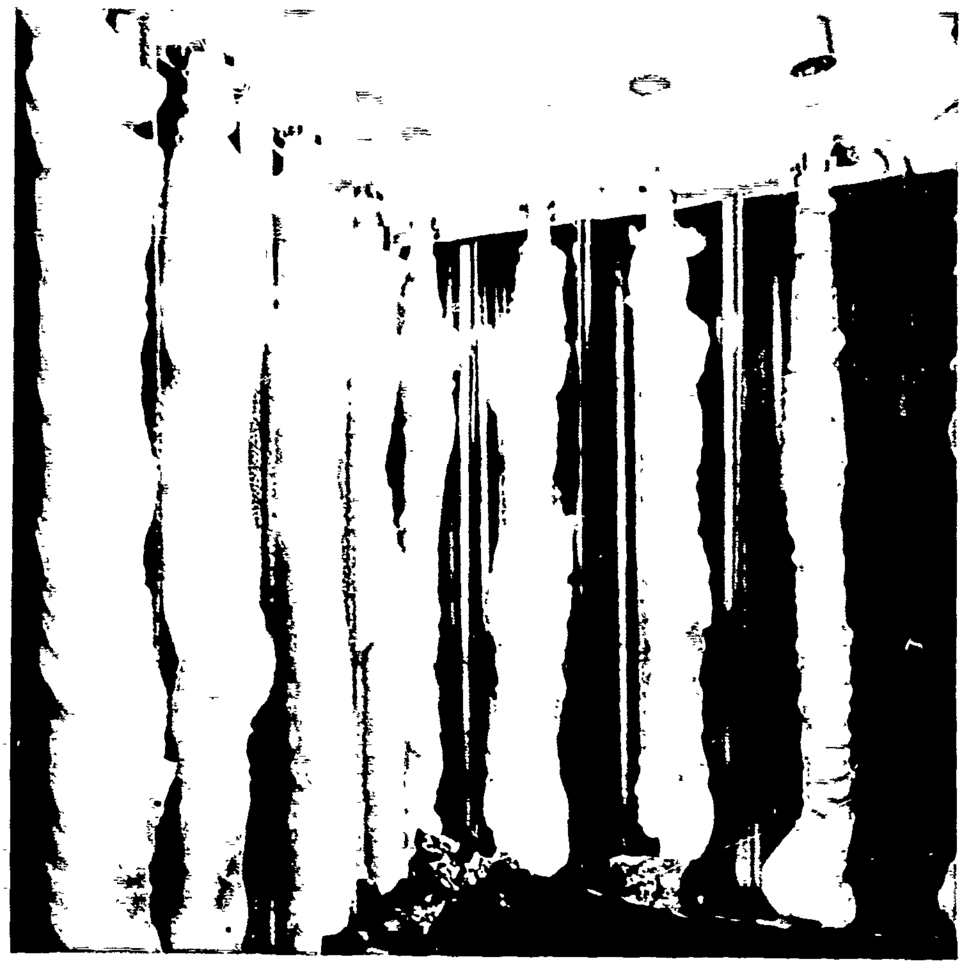
[a] Dopant or dopant ion is given as weight percent of solid used to make solution.

[b] Some solid came out of the feed solution in the solution reservoir.

[c] Analyses marked "c" are from the same whisker sample.

[d] Analyses marked "d" are from the same whisker sample.

[e] A crystal growing in the feed solution in the bottom of the tube was 1.83% K^+.



(C) FIG. 1. A View of the Laboratory Whisker Facility Employed for the Growth of Pure and Doped Ammonium Perchlorate Whiskers from Porous Glass Tubes.

CHARACTERIZATION OF DOPED WHISKER AMMONIUM PERCHLORATE (C)

(C) The doped whisker AP was characterized by three techniques: average whisker diameter, impact sensitivity, and differential thermal analysis. The microscopic determination of whisker diameters was that method employed previously (Ref. 1). The whiskers were photographed at 160 or 400X and their size calculated from distances measured on the photographs with a travelling microscope. The average values for the doped whisker AP are listed in Table 3.

(C) TABLE 3. Diameters of Doped Whisker Ammonium Perchlorate

Dopant ion	Average diameter, μ	Whisker designation
MnO_4^-	3.84	MnW-1
$Cr_2O_7^=$	3.58	CrW-1
K^+	3.47	KW-1
K^+	2.49	KW-5
K^+	1.77	KW-1A
K^+	3.55	KW-2

(C) The impact sensitivity of both doped and undoped AP whiskers as well as 15-μ ground AP was determined using 20 mg samples of material on garnet paper and a 2 kg weight. The results are tabulated in Table 4.

(C) TABLE 4. Impact Sensitivity of Whisker Ammonium Perchlorate

Whisker designation	Dopant ion	50% Point, cm	Nominal diameter, μ
W-14	None	90	40
W-22	None	92	3
15-μ ground	None	39	--
W-production	None	115	5
CrW-1	$Cr_2O_7^=$	117	4
MnW-1	MnO_4^-	113	4
KW-2	K^+	112	4

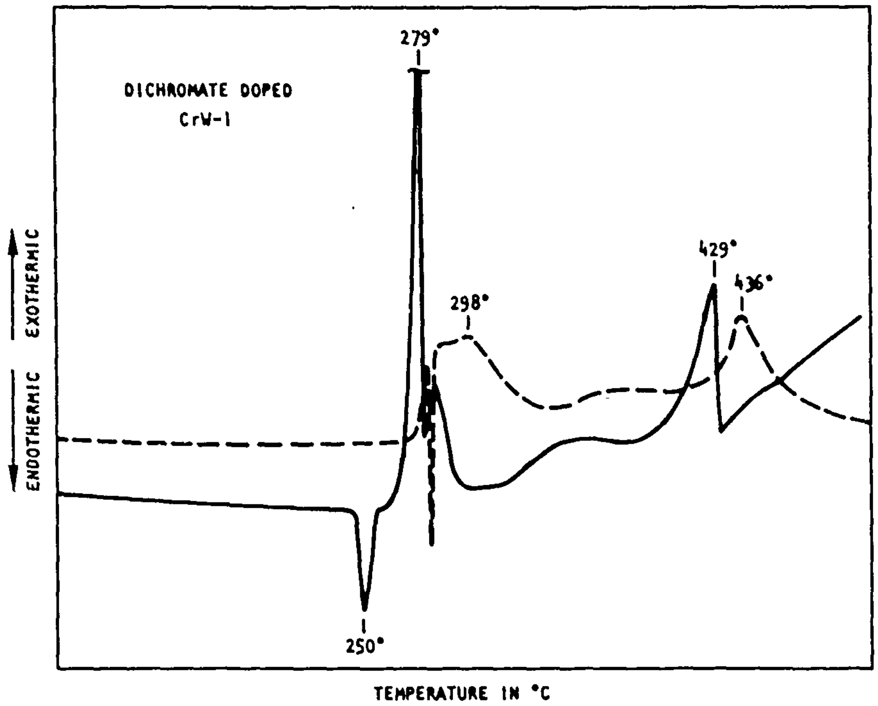
(C) The differential thermal analysis (DTA) and gas profiles for potassium doped, permanganate doped, dichromate doped, and undoped AP whiskers are shown in Figs. 2-5. The thermograms were run on a differential scanning calorimeter (Perkin-Elmer DSC-1B). The temperature was raised at 10°C/min and nitrogen was used as the purge gas.

PROPELLANT BURNING RATE DATA

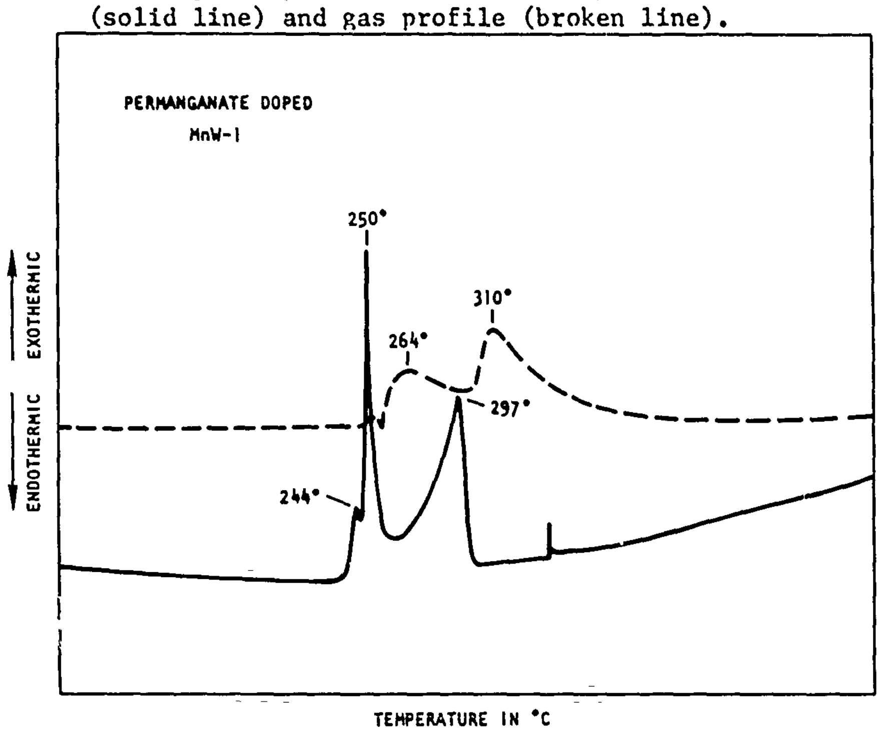
(C) Propellant strands were formulated using 76% doped whisker AP and 24% binder according to a previously described procedure (Ref. 1). The binder was Butarez II, which is a low molecular weight carboxyl terminated butadiene prepolymer. No aluminum was incorporated into any of the propellant formulations utilizing doped whisker AP. Burning rate data are reported for propellants with dichromate, permanganate, and potassium doped whisker AP. The strand burning rate curves were determined through the use of a least squares program. The slopes of the resulting lines give the pressure exponents according to the equation $r = aP_c^n$, where r is the burning rate, a is a constant, P_c is the pressure, and n is the pressure exponent. Fig. 6 shows the burn data and least squares lines for strands made from permanganate doped AP whiskers and dichromate doped AP whiskers. For purposes of comparison, the curve for nominal 3-μ whisker AP (undoped) is also given.

(C) The burn data for propellant strands made from potassium doped AP whiskers at various dopant levels are shown in Fig. 7. The particular interest in potassium doped whisker AP coupled with the large variation in the potassium analysis of the doped whisker AP led to the preparation of a quantity of whisker AP grown from feed solutions doped with 2.02% potassium ion (KW-2). The resultant whiskers were thoroughly mixed mechanically to give a uniform distribution of the potassium throughout the material (analyzed as 2.4% potassium). These doped whiskers were then carefully mixed with various amounts of undoped AP whiskers and formulated into a series of propellant strands: 100% KW-2; 75% KW-2:25% AP; 50% KW-2:50% AP; 25% KW-2:75% AP; and 100% AP. The burning rate data are shown in Fig. 8.

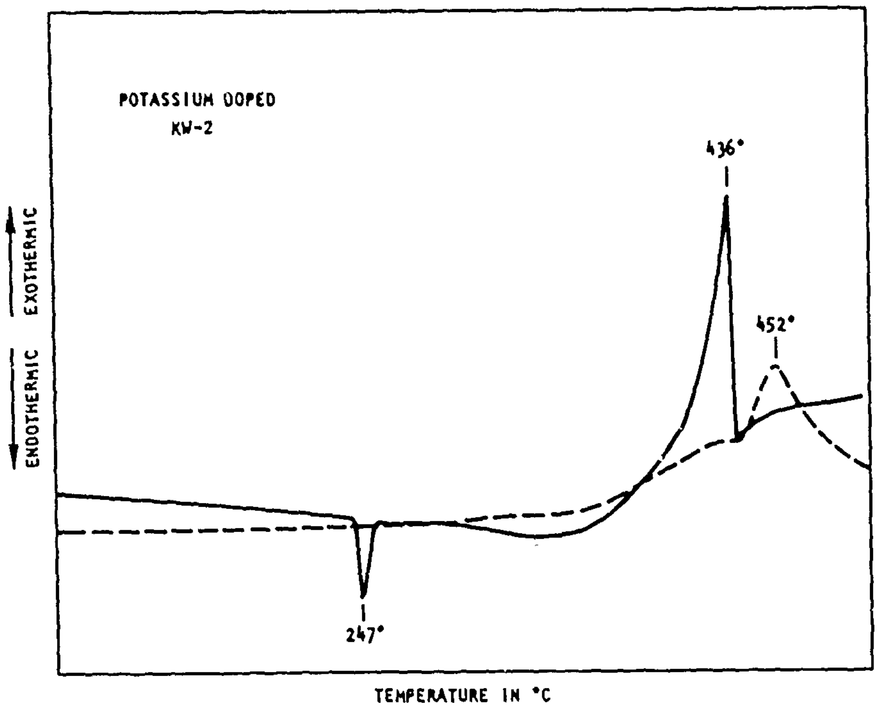
(C) The pressure exponents for the various whisker AP propellants are listed in Table 5.



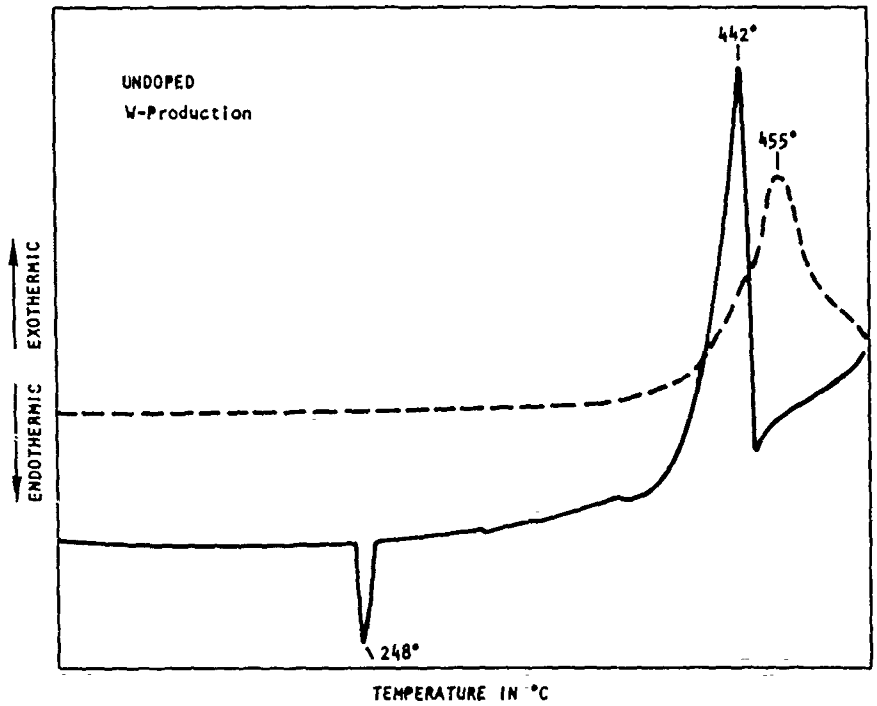
(C) FIG. 2. Differential thermal analysis of dichromate doped whisker ammonium perchlorate (solid line) and gas profile (broken line).



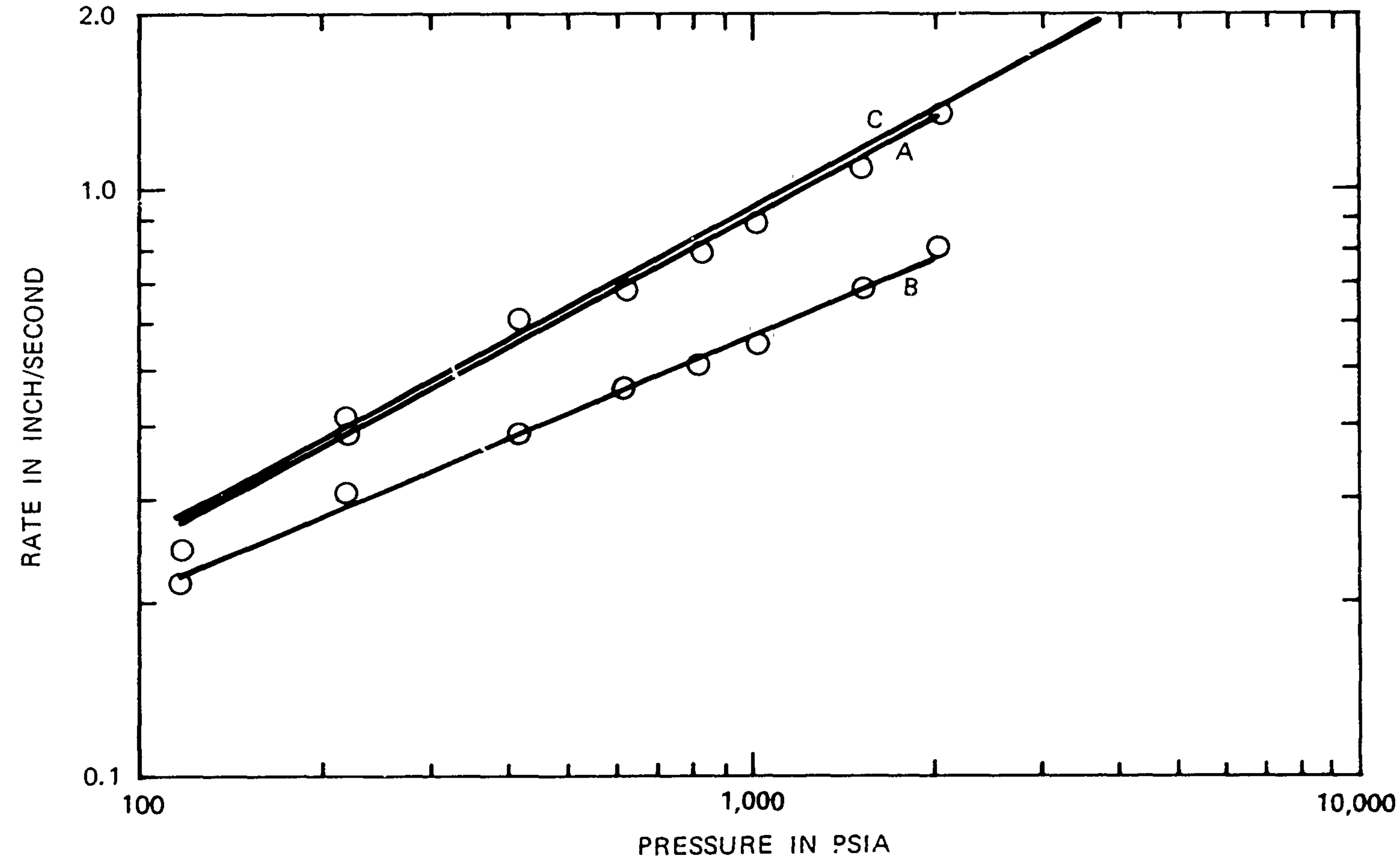
(C) FIG. 3. Differential thermal analysis of permanganate doped whisker ammonium perchlorate (solid line) and gas profile (broken line).



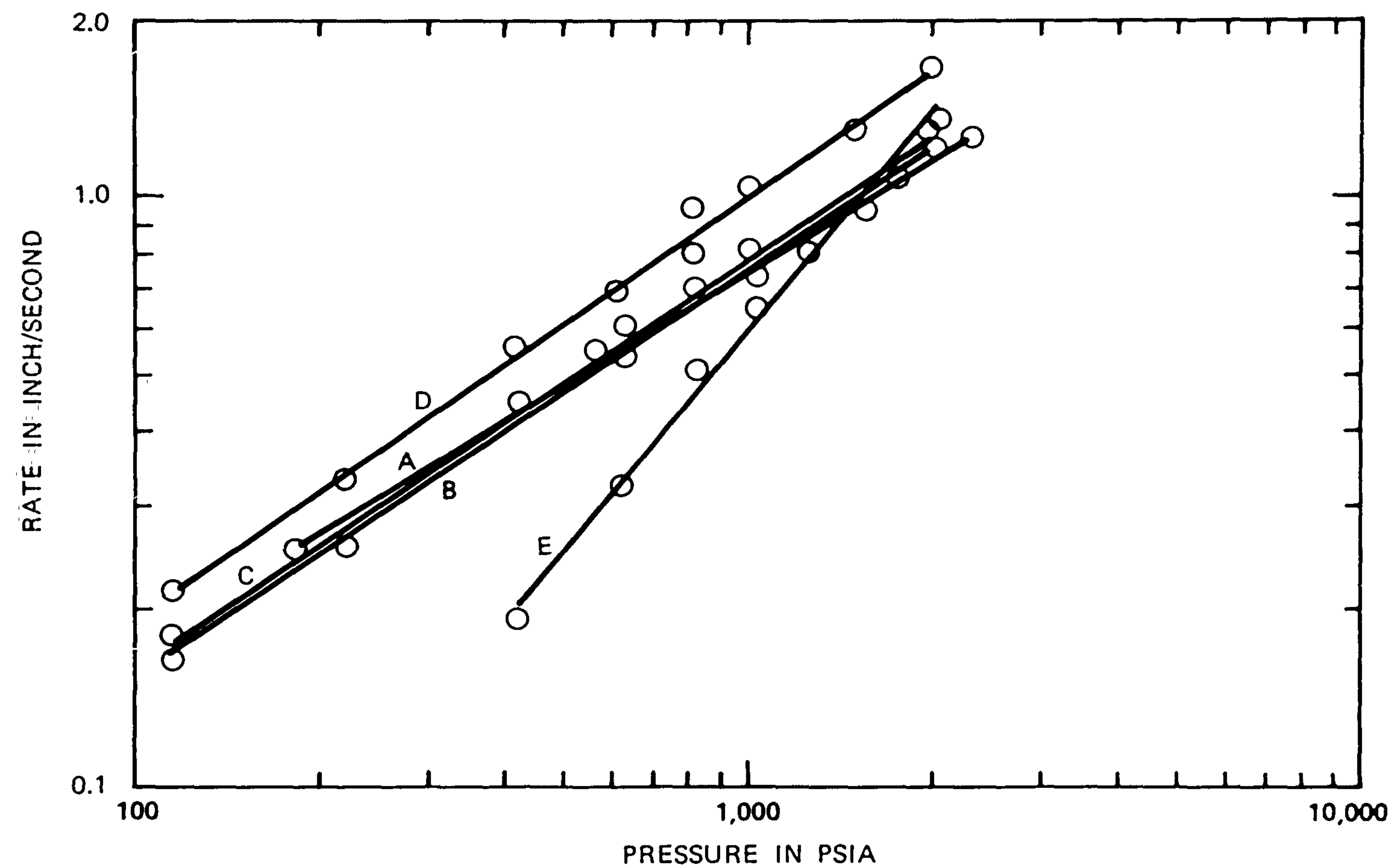
(C) FIG. 4. Differential thermal analysis of potassium doped whisker ammonium perchlorate (solid line) and gas profile (broken line).



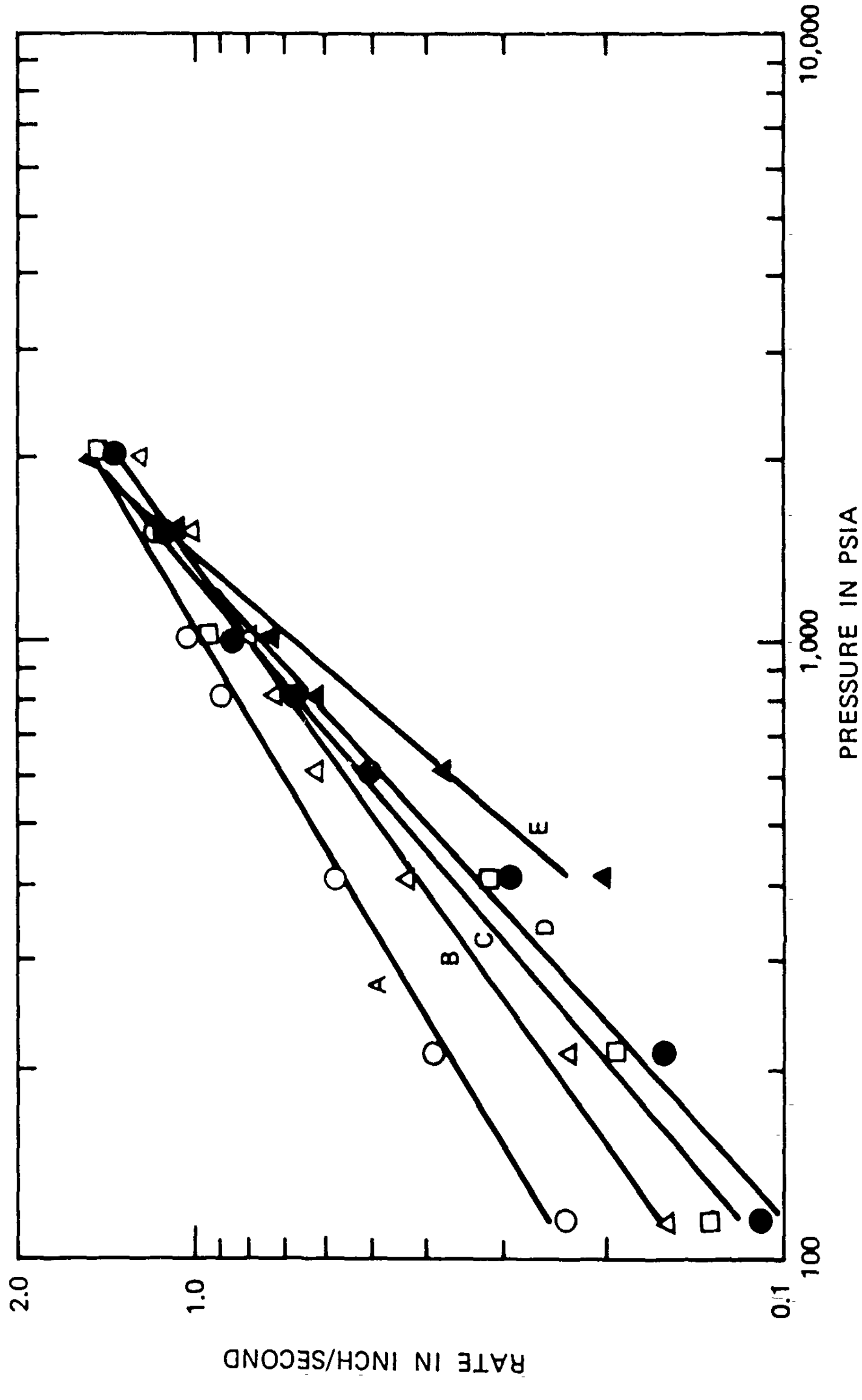
(C) FIG. 5. Differential thermal analysis of undoped whisker ammonium perchlorate (solid line) and gas profile (broken line).



(C) FIG. 6. Burning Rates for Propellant Strands. Curve A: permanganate doped whisker ammonium perchlorate (MnW-1); Curve B: dichromate doped whisker ammonium perchlorate (CrW-1A); Curve C (no data points given): undoped whisker ammonium perchlorate, nominal 3-μ diameter.



(C) FIG. 7. Burning Rates for Propellant Strands Incorporating Potassium Doped Whisker Ammonium Perchlorate. Curve A (KW-1); Curve B (KW-1A); Curve C (KW-1A); Curve D (KW-5); Curve E (KW-2).



(C) FIG. 8. Burning Rates for Propellants Made of Mixtures of Undoped Whisker Ammonium Perchlorate (AP) and Potassium Doped Whisker Ammonium Perchlorate (KW-2). Curve A (100% AP); Curve B (25% KW-2:75% AP); Curve C (50% KW-2:50% AP); Curve D (75% KW-2:25% AP); Curve E (100% KW-2).

(C) TABLE 5. Pressure Exponents for Propellants Containing Doped and Undoped Whisker Ammonium Perchlorate

Dopant Ion	Dopant Ion in Whiskers, %	Pressure Exponent	Strand Designation
MnO_4^-	0.89	0.558	MnW-1
$Cr_2O_7^{=}$	0.26	0.438	CrW-1
K^+	0.34	0.629	KW-1
K^+	1.09	0.709	KW-5
K^+	1.28	0.675 0.683	KW-1A
K^+	1.44	1.24	KW-2
K^+	2.4	1.19	100% KW-2
K^+	1.8	0.933	75% KW-2:25% AP
K^+	1.2	0.874	50% KW-2:50% AP
K^+	0.6	0.728	25% KW-2:75% AP
None	0.008	0.606	100% AP

DISCUSSION

(C) Doped whisker AP incorporating ammonium permanganate, ammonium dichromate, and potassium perchlorate were grown successfully from porous glass tubes. These three dopants are capable of isomorphic substitution within the crystal lattice of orthorhombic AP and thus represent a somewhat limited case for the direct introduction of catalysts into a propellant via the ammonium perchlorate oxidizer. The scope of the present report was to extend the whisker technique to doped AP, to characterize the resultant material, and to correlate the burning rate data of the propellants to relevant information within the literature.

(C) The distribution of the dopant between the whisker AP and the initial feed solution was qualitatively a 1:1 relationship for permanganate and potassium. The permanganate in the whisker AP analyzed slightly lower than the solution composition. This is attributed to the partial decomposition of the permanganate in solution to lower oxides of manganese. Once the permanganate is incorporated within the whisker AP, the stability of the permanganate is increased substantially. The porous glass tubes become dark red to brown depending upon the extent of decomposition that has occurred. The incorporation of the dichromate within the whisker AP was limited. The distribution of the dopant represents a rejection from the whisker AP of about 8:1. Over a period of time this factor accounts for an increased concentration of the dichromate in the feed solution. Occasionally, the formation of whiskers with different depths of color were observed. This was attributed to mixtures of dichromate doped AP and possibly AP doped ammonium dichromate. The porous glass tubes develop an intense yellow color.

(C) Although the distribution of the potassium between crystal AP and the solution favors incorporation within the AP lattice structure, the results for the AP whisker growth indicate an overall 1:1 correspondence. If one assumes that the analyses for potassium by flame emission or atomic absorption spectroscopy are correct, then an unusually large variation exists in the dopant levels of potassium within the whisker AP. The average of the analyses approaches the initial solution level but the individual results on small samples fluctuate widely. One explanation involves local depletion of potassium within the feed solution through preferential incorporation within the whisker AP followed by whisker growth of the potassium deficient AP. In support of this supposition, the observance of concentration gradients of AP solution within the porous tube have been noted frequently. Additional factors which possibly influence the concentration of dopant in the whisker AP are the extent of absorption of the potassium in the porous glass, the length of time the tube has been filled with the feed solution, the frequency of washing out the tube or shutting down whisker production, differential growth rates over the surface of the porous glass, and precision of the analyses.

(C) The average diameters of the doped whisker AP ranged from 1.77 μ for potassium (KW-1A) to 3.84 μ for permanganate (MnW-1), Table 3. The impact sensitivity of the doped whisker AP was essentially identical to the values for the undoped whisker AP, Table 4. Nominal diameter does not seem to be a factor in the impact sensitivity of whisker AP.

(C) In Fig. 5, the DTA for undoped whisker AP indicates the endothermic phase transition near 248°C and the exothermic decomposition peaking at 442°C; the profile of gas evolution shows a 455°C peak. The results for the potassium doped whisker AP, Fig. 4, are practically identical to those for the undoped whiskers. In Fig. 2, the dichromate doped whisker AP has an exothermic peak at 279°C with a corresponding gas evolution peak at 298°C in addition to the previously noted exothermic decomposition now observed near 429°C. In Fig. 3, the exothermic decomposition near 250°C for the permanganate doped whisker AP nearly masks the endothermic reaction of the phase change, 244°C. A second exothermic reaction for the DTA peaks at 297°C; corresponding gas profiles for the two DTA peaks occur at 264 and 310°C. No additional maxima are indicated in this latter thermogram.

(C) In Fig. 6, the burning rate data for the propellant strands incorporating the 0.89% permanganate doped whisker AP (Curve A) are experimentally indistinguishable from the previously reported data (Ref. 1) of the nominal 3-μ undoped whisker AP (Curve C). No data points are given for Curve C. The data for the 0.26% dichromate doped whisker AP (Curve B) establish a burning rate curve lower than that of the undoped whisker AP (Curve C) and also possess a small pressure exponent.

(C) The burning rate data for the potassium doped whisker AP (Fig. 7) indicate an increased pressure exponent with increased potassium dopant level, Table 5. The value of the pressure exponent measured from 0.629 (KW-1) to 1.24 (KW-2); the corresponding potassium dopant level in the feed solution from which the whiskers were formed increased from 0.28 to 2.02%, Table 1. Accompanying this general increase in pressure exponent with increased dopant level was a reduction in the burning rate at the lower pressures. The minimum pressure at which burning is sustained is increased to a value of approximately 400 psia for KW-2. This minimum combustion pressure is not well defined and may be related to the thermal flux available to heat the propellant. It was believed that certain inconsistencies in the burning rate data in Fig. 7 were possibly attributed to inhomogeneities of the potassium doped whisker AP. The burning rate data for the potassium doped propellants in Fig. 8 essentially removed the anomalous effects seen in Fig. 7. The initial burning rates at low pressures together with the pressure exponents are closely correlated with the overall potassium dopant level within the propellant. The burning rate data for mixtures of potassium doped whisker AP and undoped whisker AP appear to be related only to the overall potassium concentration, not to the uniformity of its dispersion within the oxidizer.

(C) The burning rate data in Fig. 8 were not continued to higher pressures because of equipment limitations. The linear equations determined by the method of least squares clearly indicate intersection of the burning rate curves near 1.00 in/sec at 2,000 psia. Projection of the linear equations to higher pressures would suggest phenomenally faster burning rates. A possibility exists, however, that the burning rates at higher pressures merely approach those values expected for the undoped whisker propellant.

(C) The present investigation of selectively doped whisker AP is augmented by related studies on ammonium perchlorate decomposition and deflagration (Ref. 4). Of particular note is the contribution made by the joint use of cinephotomicrography of burning samples and the scanning electron microscopy of quenched samples. These studies have been extended recently to single crystals of AP with controlled isomorphic substitution of potassium, permanganate, and dichromate (Ref. 2). With potassium doped AP the effect on deflagration rate showed more evidence of a liquid surface layer than with pure AP, especially at higher potassium concentrations. The burning rate was sporadic and uneven, accompanied by the surface accumulation of unreactive products. The data suggested that a mild increase of the liquid layer correspondingly increased the deflagration rate of AP. The region of decreased deflagration rate above 2,000 psia for pure AP was completely removed at all potassium concentrations. The obvious analogy between the deflagration of potassium doped whisker AP propellant suggests that the effect of potassium could be limited to the region below 2,000 psia. The evidence would certainly indicate that an upper boundary exists beyond which an increase in potassium dopant level would have no additional effect on the enhancement of propellant burning rate.

(C) It was reported (Ref. 5) that the effect of doping single crystal AP with permanganate was a large increase in the rate of formation of nucleation sites during the isothermal decomposition of AP. The limited work on the deflagration of single crystal AP doped with permanganate (Ref. 2) was characterized by failure to sustain combustion at pressures below 1,700 psia, long ignition delays at higher pressures, and deflagration rates highly dependent on initial sample temperature. The present study involving the permanganate doped whisker AP confirmed the enhancement of the low-temperature decomposition (Fig. 3) and established a burning rate for the propellant incorporating the doped whisker AP essentially identical to that of the propellant utilizing the undoped whisker AP, Fig. 6.

(C) The deflagration rates of single crystal AP doped with dichromate were insufficient to unequivocally establish burning rate curves (Ref. 2). In the present report no effort is made to correlate the deflagration rates with the propellant burning rates. In Fig. 2 the differential thermal analysis clearly indicates a low temperature exotherm nearly 30°C above the AP transition temperature. The propellant burning rate for the 0.26% dichromate doped whisker AP showed a lowering of the burning rate at low temperatures and a pressure exponent of 0.438, substantially less than that for the undoped AP propellant, Fig. 6.

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(C) The present study of ammonium perchlorate which is grown as whisker crystals from porous glass by a recently discovered process is extended to doped ammonium perchlorate. The selection of dopants in whisker ammonium perchlorate is limited initially to those materials capable of isomorphic substitution in the ammonium perchlorate lattice structure. These dopants include potassium perchlorate, ammonium

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Burning rate (U)						
Explosives (U)						
Fine particle size ammonium perchlorate (U)						
Porous glass (U)						
Ammonium perchlorate particle size control (U)						
Modified burning rate (U)						

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