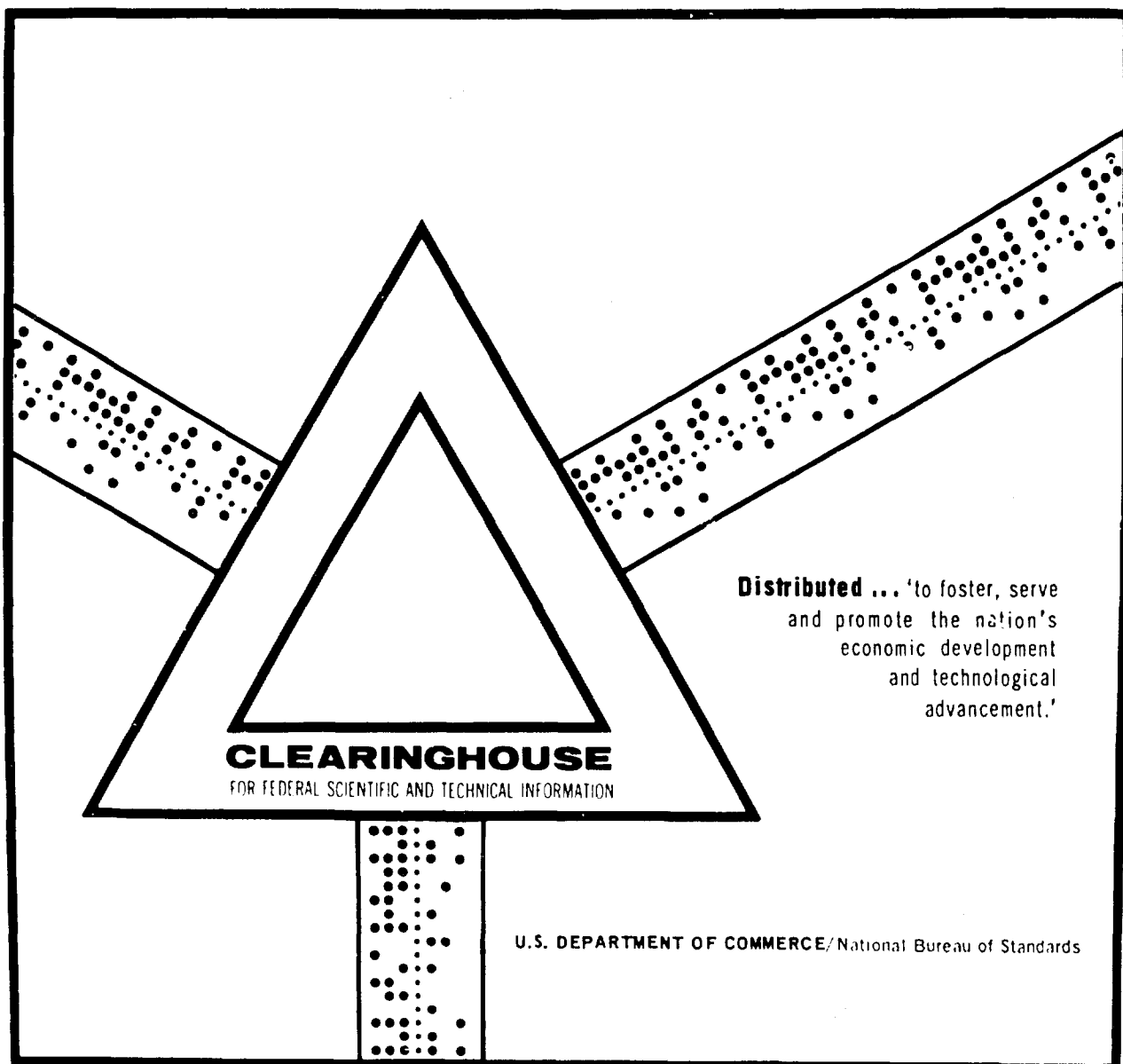


COMBUSTION RESIDUES FROM N204-MMH MOTORS

Hideyo H. Takimoto, et al

Aerospace Corporation
El Segundo, California

15 September 1969



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Combustion Residues from N_2O_4 -MMH Motors

Prepared by H. H. TAKIMOTO and G. C. DENAULT
Aerodynamics and Propulsion Research Laboratory

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Laboratory Operations
THE AEROSPACE CORPORATION

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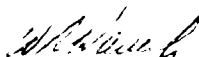
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FOREWORD


This report is published by The Aerospace Corporation, El Segundo, California, under Air Force Contract F04701-69-C-0066. This report, which documents reaserch carried out from January 1968 to March 1969, was submitted for review and approval on 17 September 1969 to Lt. H. D. Gibbs, SMTAE.

Approved



W. R. Warren, Jr., Director
Aerodynamics and Propulsion
Research Laboratory

Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.



Harold D. Gibbs, 2nd Lt, USAF
Project Officer

ABSTRACT

Motor residues from the combustion of N_2O_4 and MMH were analyzed. Under steady-state and pulse-mode firings, the major constituents of the non-volatile residues were found to be ammonium nitrate and $MMH \cdot HNO_3$, respectively. The latter material contained a heat-sensitive compound which triggered its decomposition at a lower temperature. The application of heat to $MMH \cdot HNO_3$ initially converted the salt to monomethylammonium nitrate. The infrared spectra and differential analysis curves for these materials are presented.

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I. INTRODUCTION

N_2O_4 -hydrazine bipropellant systems are used not only in large boosters but also in small rocket motors where rapid start and restart capabilities are required for carefully controlled maneuvers under high altitude conditions. Although the hypergolic propellants have been known and utilized for a number of years, the intermediate steps involved in their combustion, especially under low pressure environment, are not understood. Two major problems resulting from the use of these restartable space environmental engines are the contamination of sensitive space vehicle surfaces from bipropellant rocket exhaust products and the occurrence of large transient ignition overpressures. These problems are related in that they both are the results of the incomplete combustion of the oxidizer and the fuel.

An investigation of the combustion residues from N_2O_4 -MMH motor firings is described in this report. The analyses and identification of the residues were undertaken in order to obtain a better understanding of the oxidation process taking place in the reaction of N_2O_4 with MMH. Such knowledge will be useful in determining a possible solution to problems associated with the use of hypergolic propellants at high altitude conditions.

II. BACKGROUND AND HISTORY

Although the N_2O_4 -hydrazine propellant systems are suited for auxiliary thrust and power requirements, particularly where these propellants are employed for main propulsion, the operation of the upper stage rockets and space vehicles in vacuum environment has created a unique propulsion problem related to hypergolic ignition. In addition to high pressure ignition transients, plume contamination from partially combusted propellants can lead to undesirable coatings on the external surfaces of the vehicle. If a solution to these problems can be found, the bipropellant systems would offer advantages over mono-propellant systems, particularly for future space missions where increased power requirements are of critical importance.

The hypergolic ignition problems are basically concerned with the course of a chemical reaction in a very low pressure environment. The combustion intermediates accumulated during the non-burning cycle can undergo smooth thermal degradation or a destructive detonation during multiple restarts. Further, the accumulated products can be expelled from the rocket nozzle, resulting in plume contamination. Fundamental to the true understanding of the oxidation process taking place in a bipropellant motor system is the knowledge of the complex series of reactions which occur during the impingement of two streams of propellant as well as after shutdown of the injector valves. The problem is further complicated in the motor where the oxidizer-to-fuel-mixture ratio, temperature, and total pressure are changing during the multiple start conditions.

The earlier work on the bipropellant reactions was directed primarily toward the problem of high pressure ignition overpressures. An extensive literature survey of the N_2O_4 -hydrazine reactions was reported by Perles, et al. (Ref. 1). They have also carried out experimental studies on the analyses of motor residues from test firings. From propellant systems

utilizing N_2O_4 with either N_2H_4 or MMH, the fuel nitrates were obtained as residues. On the other hand, ammonium nitrate was found to be the major product from N_2O_4 -UDMH combustion. In a similar study, Seamans (Ref. 2) had reported that MMH nitrate was the major product in the non-volatile residue from the N_2O_4 -MMH system.

The ground test experience using low-thrust attitude control engines has shown a correlation between the pressure spike phenomena and the residue formation. A decrease in residue formation occurs when the motor is operated under conditions where ignition overpressures are eliminated. These results are consistent with the hypothesis that both the plume contamination and pressure spikes are the result of the incomplete combustion of the oxidizer and the fuel. Some factors which are believed to increase the quantity of residue formation are (1) the upfiring position of the motor, (2) multiple pulses of short duration, (3) low temperature, and (4) oxidizer leads. These observations are still qualitative in nature, and further verification of the significance of these factors is required.

Previous work (Ref. 3) at Aerospace Corporation has been concerned with the introduction of gaseous NO_2 into a vacuum chamber containing liquid or solid N_2H_4 , MMH, Aerozine-50, or UDMH. At pressures below 100 mm of Hg and temperature of $-11^\circ C$, a flameless reaction took place with a noticeable deposition of a viscous condensed phase. Oily residues thus obtained were identified by infrared analyses as nitrate salts of the fuel. The buildup of non-volatile, partially combusted products occurs under these low-pressure conditions where only a flameless reaction takes place.

In the study of the N_2O_4 - N_2H_4 reaction, the presence of hydrazoic acid as a transient species in the combustion has been reported by Dauerman, et al. (Ref. 4.). He proposed that the pressure spikes occurring during the restarting of the bipropellant motors are caused by this azide triggering the detonation of the nitrate salts which are accumulated during the pulse-mode operation of the motor. Such formation of an azide using a mono-substituted hydrazine has been demonstrated in our laboratory by the reaction of phenylhydrazine with N_2O_4 in chloroform solution.

to yield phenyl azide.¹ A similar reaction taking place in the N_2O_4 -MMH combustion would lead to methyl azide, which is also a shock and heat-sensitive energetic compound. We have also shown that the phenylhydrazine- N_2O_4 reaction in solution could lead to the formation of benzenediazorium nitrate, which has been isolated and identified.² The analogous reaction of N_2O_4 with MMH would lead to an explosive gaseous product, diazomethane. Because of the high volatility of methylazide and diazomethane, they would not readily be observed in a motor firing. Although they would not be accumulated, nevertheless, they can serve as ignitors which set off the non-volatile salts that had formed in the rocket chamber during multiple starts.

¹H. H. Takimoto and G. C. Denault, unpublished work.

²See footnote 1.

III. RESULTS AND DISCUSSION

The analyses and identification of combustion residues from N_2O_4 -MMH motor firings at Air Force Rocket Propulsion Laboratory (AFRPL) and Arnold Engineering Development Center (AEDC) were carried out. Analyses were performed on the residues primarily by infrared spectroscopy and by differential thermal analyses. Nitrate salts of MMH were prepared for the purpose of comparison.

A. AFRPL MOTOR TESTS

Combustion products from the pulse-mode firings of a Marquardt 22-lb thrust engine at AFRPL were delivered to the laboratory for analyses. This motor utilizing N_2O_4 and MMH as propellant had undergone approximately 2400 pulses varying in width from 10 ms to 100 ms under high altitude conditions. The combustion products were collected on a cryopanel and thus consisted of 80% volatile materials, which were primarily water and some unreacted fuel. The volatiles, removed from the red amber solution, were collected in a dry ice trap leaving a gold amber residue. This residue was water soluble and extremely hygroscopic, turning tacky upon exposure to air. When heated on a melting point block, it started to bubble at 45°C and finally became clear at 100°C. Trituration with carbon tetrachloride left a creme-colored crystalline solid, m.p. 75-88°C, which gave the following elemental analysis: carbon, 8.47%; hydrogen, 5.66%; nitrogen, 37.58%. The infrared spectrum (KBr disc) of this material is unlike the spectrum for MMH- HNO_3 obtained by others from the combustion of N_2O_4 and MMH. In Fig. 1 is shown the differential thermal analysis (DTA) curve for this motor residue. It exhibited exotherms at 130° and 260°C similar to the engine residue obtained by Seamans. The exotherm at the high temperature (260°C) is typical of many nitrate compounds (Ref. 5).

The volatiles removed from the red amber solution were not specifically analyzed, but water was found to be the major constituent and the odor of an amine (or MMH) was distinctly detectable. In the upper part of the cold trap,

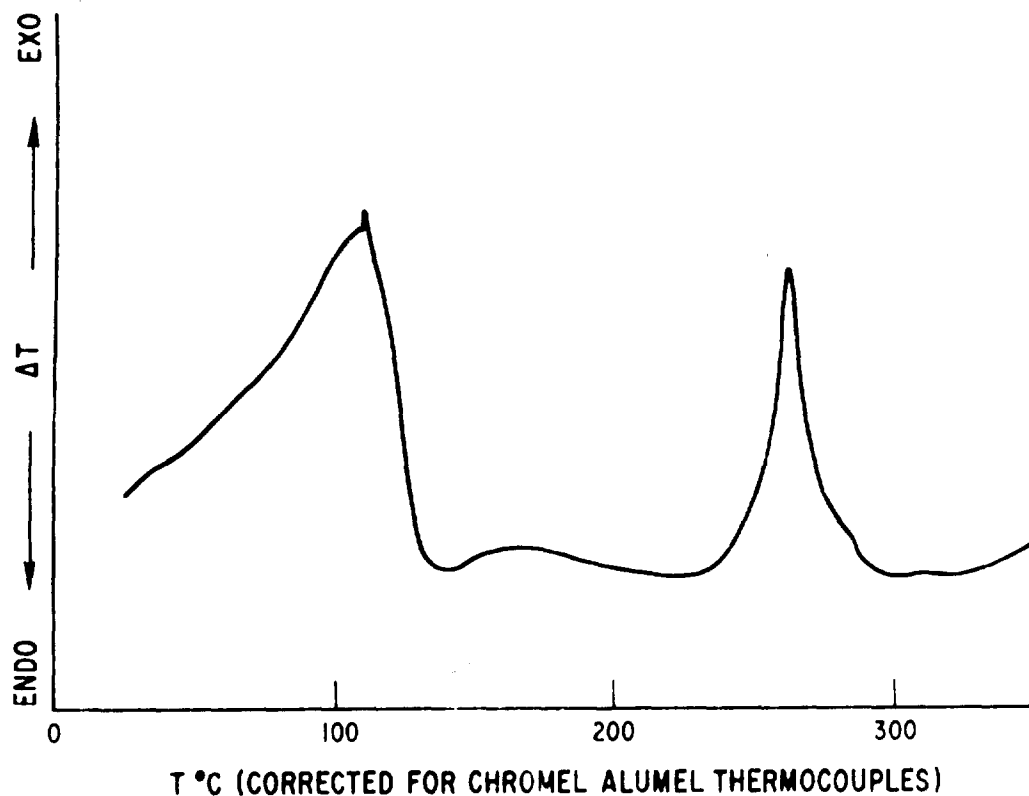


Fig. 1. Differential Thermogram of KPL Rocket Engine Residue (Marquardt 22-lb Motor)

a white crystalline solid was found which melted with violent decomposition at 94-97°C. The material disappeared by sublimation on standing at room temperature, and no further tests were made.

B. AEDC MOTOR TEST - STEADY-STATE

Engine tests were conducted in which a 1-lb thrust motor was fired in a vacuum chamber using N_2O_4 and MMH as propellants. After a steady-state firing, some residues were observed on the surface of a fence fabricated as a protective barrier. The residue on the fence was wiped off with a filter paper and was brought to our laboratory for analyses. The solids which were found on the filter paper were separated and analyzed by infrared, DTA, and x-ray diffraction. Figure 2 shows the comparison of infrared spectrum with an authentic sample of ammonium nitrate. The spectra are very similar, with major absorption being found at the same wavelength. In Fig. 3 is shown the DTA curves for the engine residue and ammonium nitrate. Again, the shapes of the curves are similar, although the small quantity of the residue available made the DTA

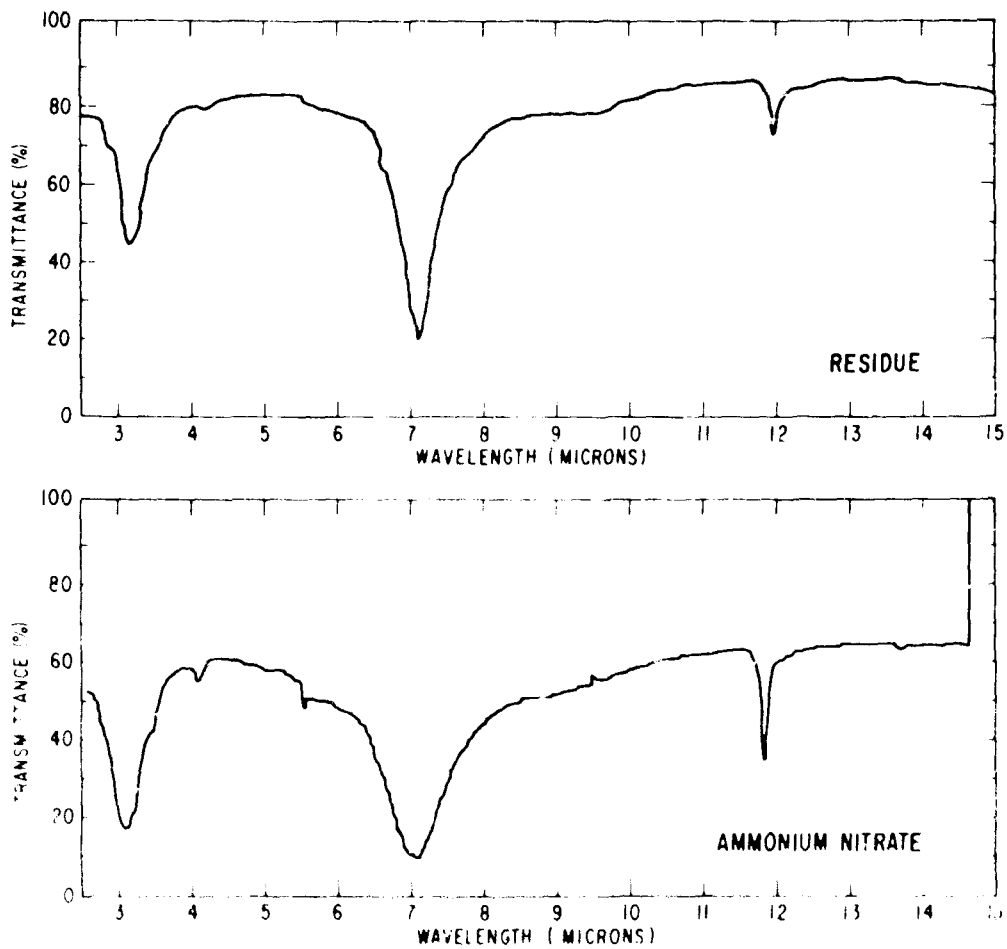


Fig. 2. Infrared Spectra of Ammonium Nitrate and Residue from N_2O_4/MMH 1-1b Motor Firings (Steady-State)

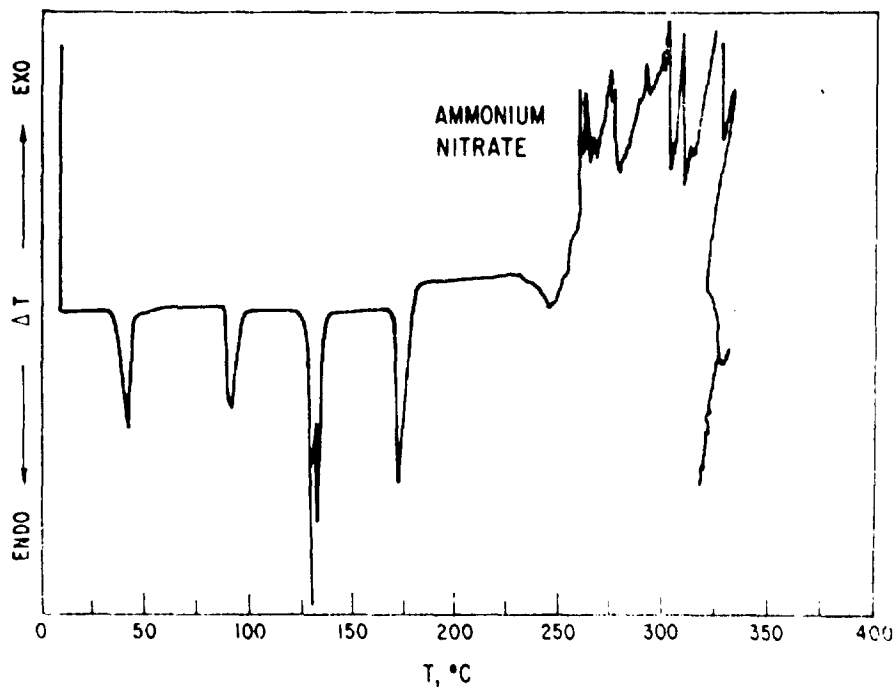
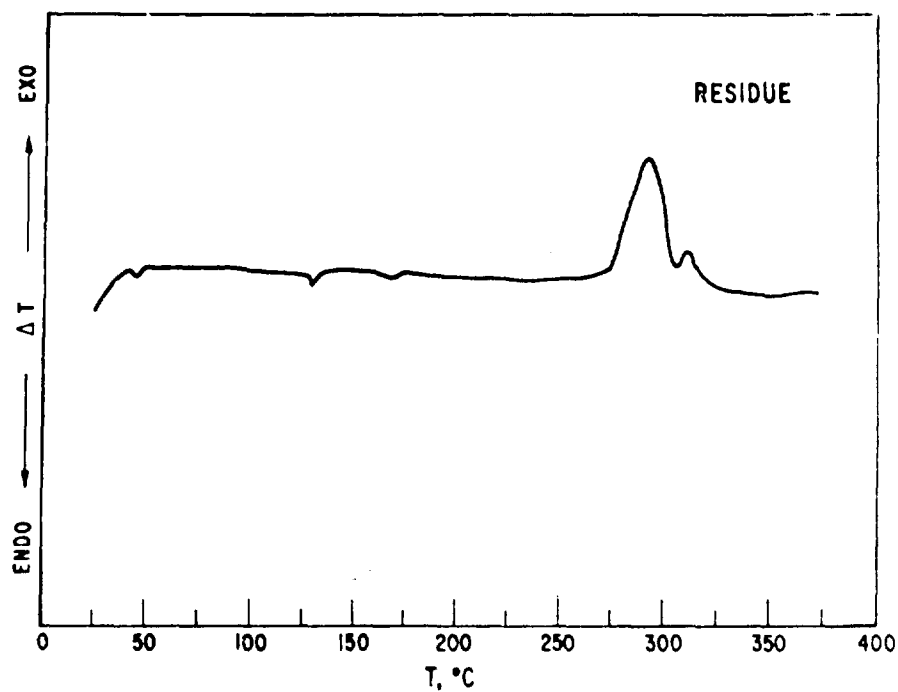


Fig. 3. Differential Thermograms of Ammonium Nitrate and Residue from N_2O_4 /MMH 1-lb Motor Firings (Steady-State)

for the unknown less definitive. Also, the melting point of the residue checked well with the value reported in the literature for ammonium nitrate. Further verification was obtained in the x-ray diffraction study. Table I shows the excellent agreement of the values for the solid with known values for ammonium nitrate. Although we feel confident that the solid on the wipings was ammonium nitrate, these results do not preclude the formation of $\text{MMH}\cdot\text{HNO}_3$ during the steady-state firing. $\text{MMH}\cdot\text{HNO}_3$, being a solid at room temperature only when very pure, may have flowed down off the vertical fence leaving only the more crystalline ammonium nitrate on the wall.

C. AEDC MOTOR TEST - PULSE-MODE

A pulse-mode firing test was carried out on the same 1-lb thrust motor at AEDC under high altitude conditions. This test was one of many that had been run to study the contamination problem from the incomplete combustion of N_2O_4 -MMH. This particular test consisted of a series of firings of 1000 pulses each with 20 ms on and 100 ms off times. The oxidizer-to-fuel-mixture ratio was 1.6 to 1. Combustion residues were obtained for analyses from this test.

In Fig. 4 is shown the schematic of the 1-lb motor in the test chamber. A cylindrically shaped shroud was placed around the nozzle during this test. One of the test objectives was to study the effect of the shroud temperature on the amount of combustion residue formed. The shroud was heated from 93°C to 379°C in an attempt to decompose the combustion residue dripping out of the nozzle into gaseous products upon contact with the hot shroud. The undecomposed material would drip off the shroud, through the pyrex tube, and flow into the flask. The results of the test by visual observation showed that a heated shroud appeared not to have a marked difference on the residue formation. The view port of the vacuum test chamber, however, did not permit the close scrutiny of the firings; thus, only a drastic difference would have been detected. A quantitative determination of the residue was not possible.

Table I. X-ray Diffraction Pattern of Ammonium Nitrate and Residue from N_2O_4 /MMH 1-lb Motor Firings (Steady-State)

<u>SAMPLE B</u>		<u>AMMONIUM NITRATE</u>	
<u>I</u>	<u>d</u>	<u>d</u>	<u>I</u>
M	4.928	4.95	45
S	3.951	3.96	67
VS	3.087	3.087	100
W	2.878	2.879	10
VS	2.721	2.722	75
W	2.487	2.485	10
W	2.382	2.380	8
VS	2.258	2.260	44
VW	2.079	2.094	2
W	1.974	1.978	4
VW	1.827	1.835	1
VW	1.779	1.786	4
W	1.724	1.730	3
W	1.623	1.631	5
W	1.571	1.578	5
VW	1.507	1.513	1
VW	1.488	1.492	2
W	1.457	1.461	?
W	1.365	1.380	1

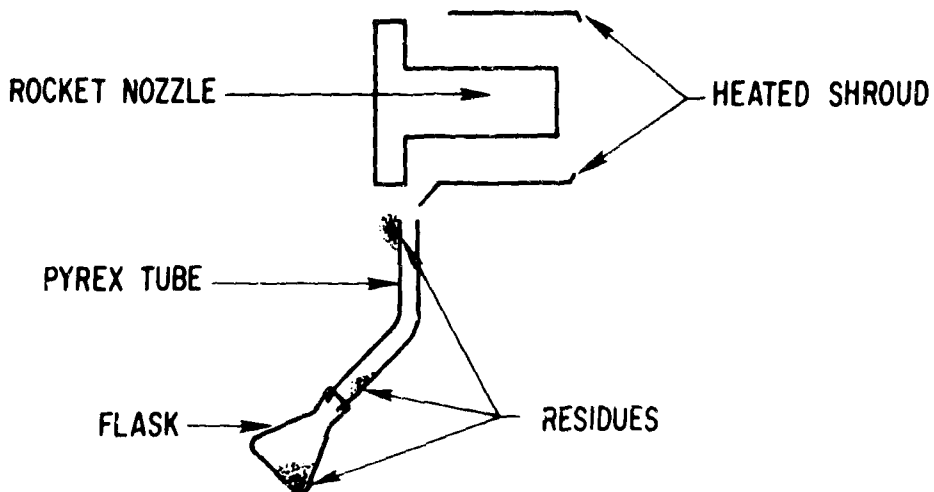


Fig. 4. Schematic of N_2O_4/MMH 1-lb Motor and Collection of Residues during Pulse Mode Firings at AEDC

At the conclusion of the test, three samples of the engine residues were obtained. The materials scraped from the engine mount and from the pyrex tube were semi-solids. The former was dark brown in color, and the latter was yellow. In the flask was collected a yellow-amber colored liquid. The semi-crystalline materials were found by analyses to be $MMH \cdot HNO_3$ and the liquid in the flask to be primarily $MMH \cdot HNO_3$ contaminated with small amounts of $CH_3NH_2 \cdot HNO_3$.

The semi-solid materials from the engine mount and from the pyrex tube turned out to be identical despite the difference in their color. An infrared spectrum of the residue is shown in Fig. 5, together with that of $MMH \cdot HNO_3$. The latter compound was prepared in the laboratory by treating 1 mole of MMH with 1 mole of dilute nitric acid and removal of the water. The two spectra are identical. The dinitrate salt of MMH was also prepared, and its spectrum is superimposable on the mono-nitrate.

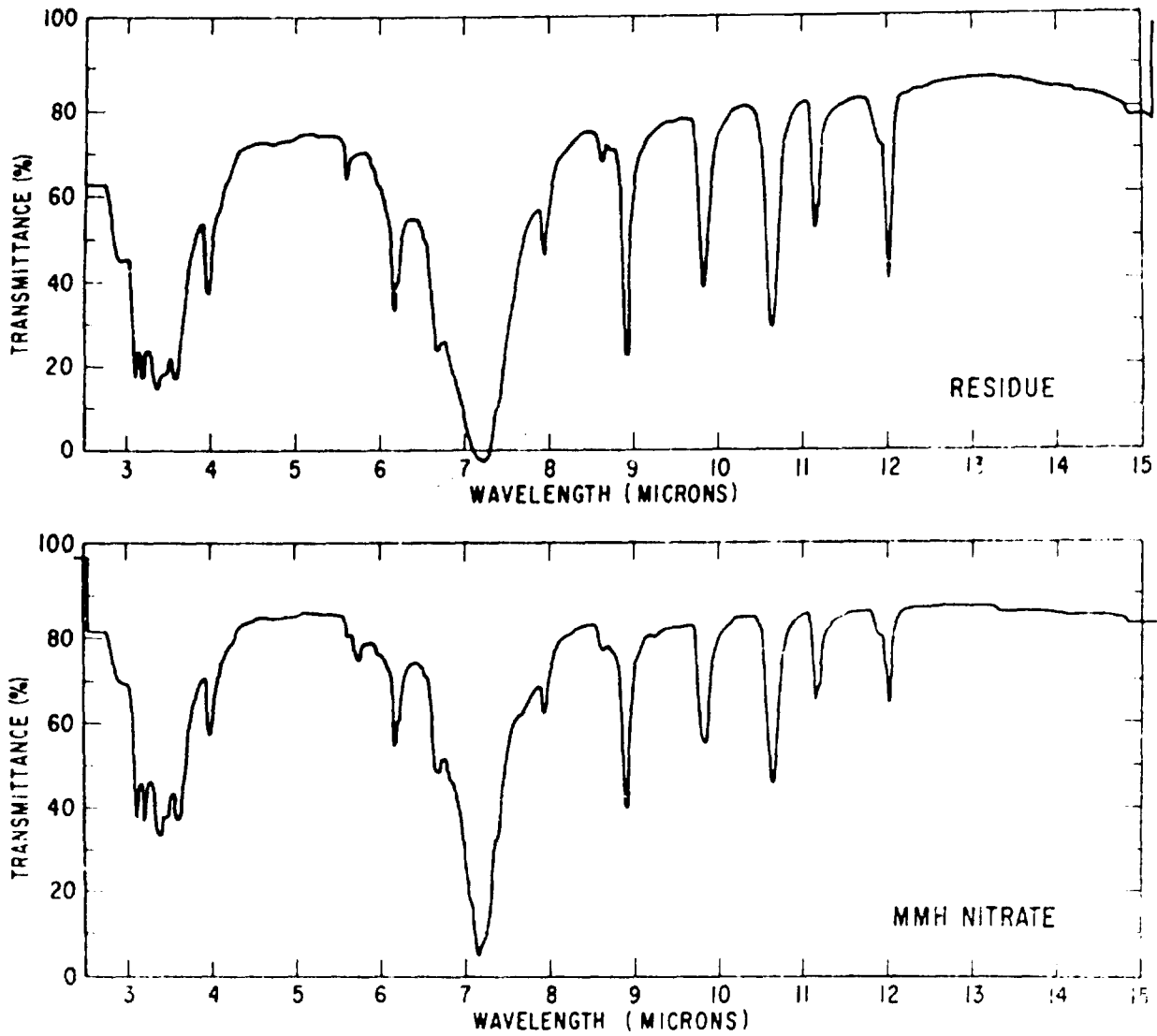


Fig. 5. Infrared Spectra of MMH Nitrate and Residue from N_2O_4 /MMH 1-lb Motor Firings (Pulse)

A discrepancy appears to exist regarding the nature of the infrared spectrum of $\text{MMH}\cdot\text{HNO}_3$. For example, the spectrum obtained in this study for the nitrate salt was similar to that reported by Perlee, et al. (Ref. 6), but differed with those obtained by Mayer, et al. (Ref. 3) and by Seamans (Ref. 2). This discrepancy was resolved when the spectrum of the identical sample of $\text{MMH}\cdot\text{HNO}_3$ used for Fig. 5 was taken as a film between sodium chloride plates as shown in Fig. 6. Two different spectra were obtained, depending on whether the spectroscopic analysis was carried out by mixing the nitrate with potassium bromide and pressing it into a disc or as a film on sodium chloride.

The elemental analyses taken on the crystalline solid from the pyrex tube are shown in Table II. The results agree well with the values calculated for $\text{MMH}\cdot\text{HNO}_3$, particularly when one considers that the analyses were carried out on the material directly from an engine firing with no purification.

Despite the fact that the infrared spectrum and elemental analyses corresponded to $\text{MMH}\cdot\text{HNO}_3$, the DTA curves (Fig. 7) were different. The thermogram of the residue at the top shows an exotherm at $\sim 160^\circ\text{C}$, whereas $\text{MMH}\cdot\text{HNO}_3$ decomposed at $\sim 260^\circ\text{C}$. On the other hand, $\text{MMH}\cdot 2\text{HNO}_3$ shows an exotherm at $\sim 110^\circ\text{C}$.

Table II. Elemental Analyses of Residue from N_2O_4 -MMH 1-lb Thrust Motor Firings (Pulse)

Elements	Found	Calc. for $\text{MMH}\cdot\text{HNO}_3$
% Carbon	11.34	11.01
% Hydrogen	6.41	6.46
% Nitrogen	40.40	38.52
% Oxygen	43.85 ^a	44.00

^aObtained by difference

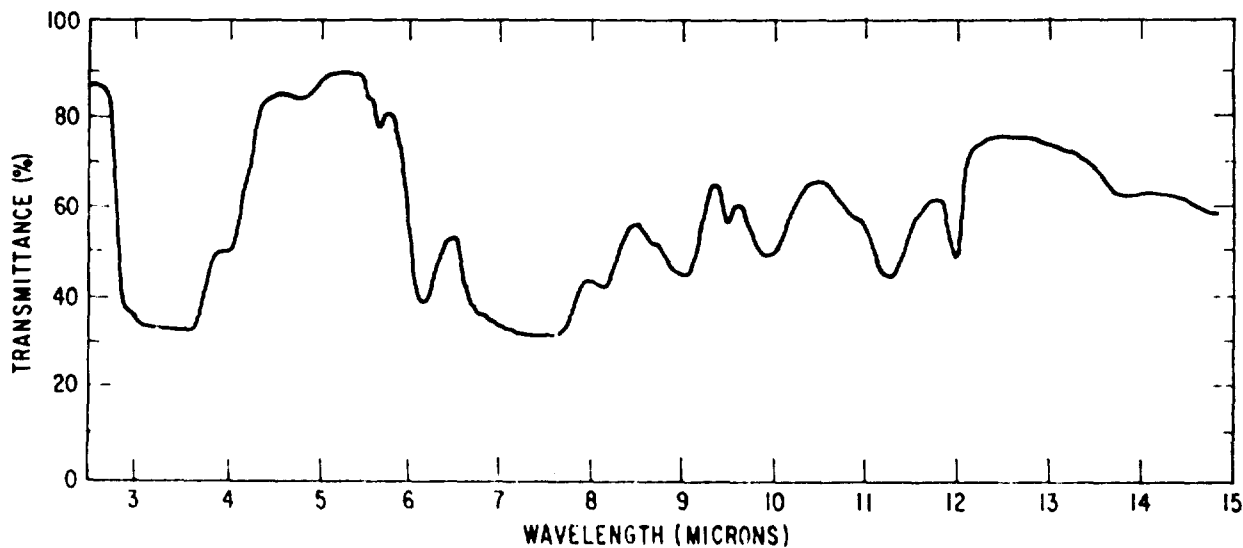


Fig. 6. Infrared Spectra of MMH Nitrate Film on NaCl Plates

Our interpretation of the results of infrared, element analyses, and DTA is that the residue is essentially all $\text{MMH}\cdot\text{HNO}_3$. However, it contained a small amount of another material, which decomposed at a lower temperature. This decomposition of the minor constituent with the simultaneous liberation of heat served to set off the $\text{MMH}\cdot\text{HNO}_3$, with the former acting as an ignitor. The nature of this less stable material is not known at this time, although it is suspected that it may be a compound such as $\text{MMH}\cdot 2\text{HNO}_3$ or possibly a nitro or nitroso derivative.

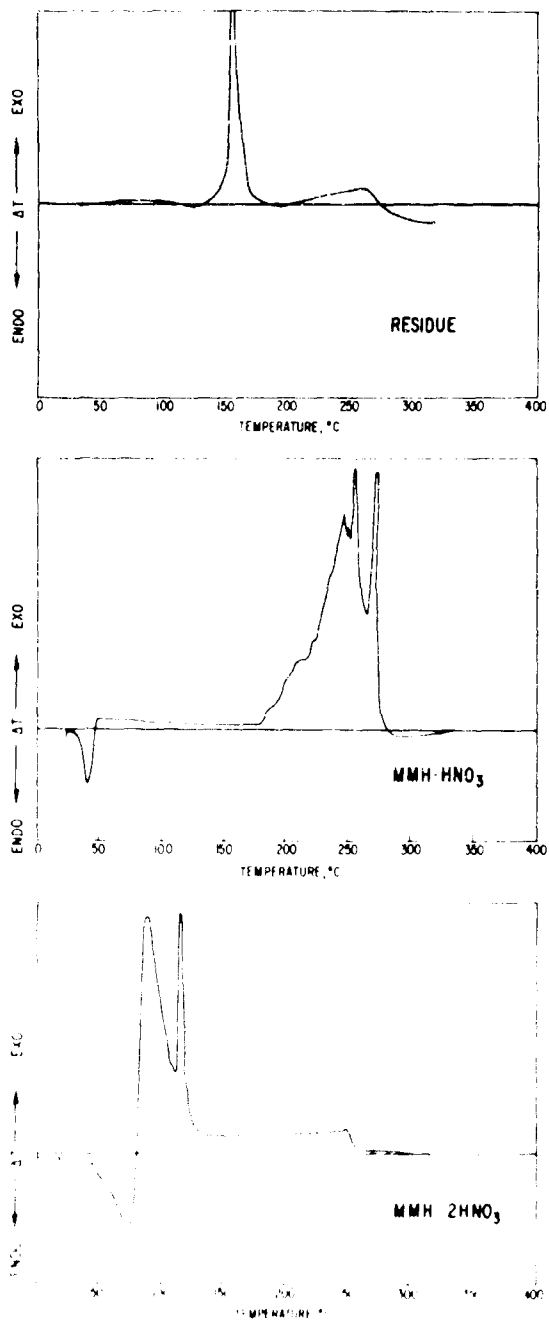
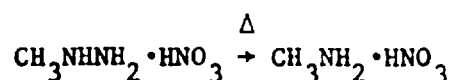


Fig. 7. Differential Thermograms of MMH Nitrates and Residue from N_2O_4 /MMH 1-lb Motor Firings (Pulse)

$1^\circ C$ (Corrected for Chromel Alumel Thermocouples)

One of the possible solutions to the plume contamination problem is devising means of entrapment or collection of non-volatile rocket exhaust products. If residue entrapment is feasible, then the problem of how to remove the collected material arises. One approach is the application of heat on the engine residue. The use of the heated shroud was based on this approach. We have studied the temperature effect on the combustion intermediate further. The semi-solid engine residue, which we have identified as primarily $\text{MMH}\cdot\text{HNO}_3$, was heated to various temperatures on a heating block. The heat caused a bubbling reaction to take place. This reaction was followed by infrared spectra and by DTA. The engine residue heated to 150°, 160°, and 170°C gave the DTA curves shown in Fig. 8. The bottom curve is the DTA of the engine residue obtained from the pyrex tube. The exotherm on the DTA curve at 160°C became smaller and the one at ~260° became larger. The analyses of the infrared spectra taken on the heated residue indicated that the material was being thermally converted to $\text{CH}_3\text{NH}_2\cdot\text{HNO}_3$ as shown:



The DTA curve for $\text{CH}_3\text{NH}_2\cdot\text{HNO}_3$ prepared in the laboratory is also shown in Fig. 8.

The analysis of the effect of temperature just described was carried out on the engine residue found in the pyrex tube leading to the collection flask. The yellow liquid collected in the flask contained, in addition to water and unreacted MMH, a mixture of nitrate salts of MMH and CH_3NH_2 . What may be concluded is that during the motor firings, pure crystalline $\text{MMH}\cdot\text{HNO}_3$ (the major product) solidified in the tube, whereas this low melting solid contaminated with small amounts of $\text{CH}_3\text{NH}_2\cdot\text{HNO}_3$ became liquid and flowed into the flask. The effect of the heated shroud was to convert some of the $\text{MMH}\cdot\text{HNO}_3$ to $\text{CH}_3\text{NH}_2\cdot\text{HNO}_3$, as we have shown in the laboratory. Thus during the pulse-mode operation, MMH is converted to $\text{MMH}\cdot\text{HNO}_3$ by N_2O_4 followed by a partial thermal decomposition to $\text{CH}_3\text{NH}_2\cdot\text{HNO}_3$.

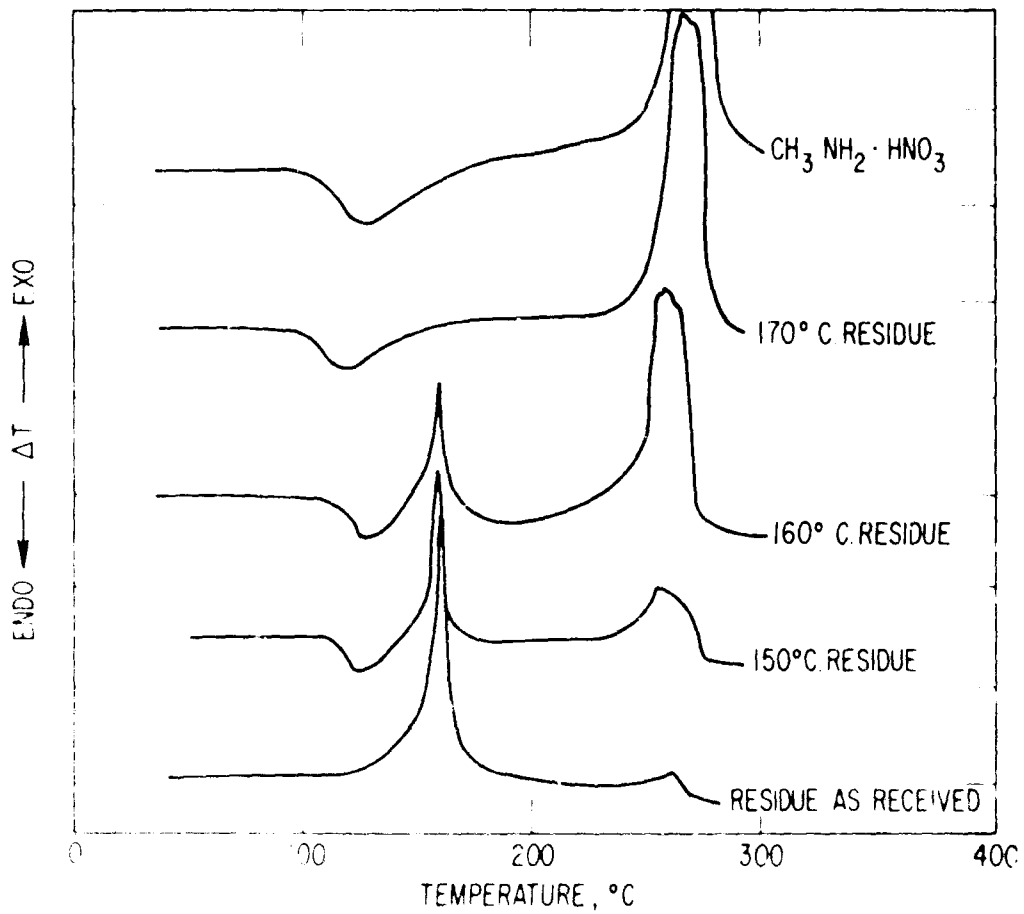


Fig. 8. DTA Effect of Heat on Residue from N₂O₄/MMH 1-lb Motor Firings (Pulse)
T°C(Corrected for Chromel Alumel Thermocouples)

Surface tension measurements were determined on the residues from the pulse-mode firings using a Natelson apparatus. This apparatus, which was calibrated with liquids of known values and corrected for density, allows a fast, convenient method for obtaining surface tensions. Values of 63.5 and 60.5 dynes/cm at 19.5°C were obtained for the residues from the motor mount and the flask, respectively. These measurements were made several months after the motor firings, and thus it is likely that further reactions of the residues had taken place on standing. On the other hand, MMH·HNO₃ prepared in the laboratory yielded surface tension approximating these values.

IV. SUMMARY AND CONCLUSION

The combustion of N_2O_4 and hydrazine derivatives under high altitude conditions leads to the formation of residue which can cause contamination. Larger quantity of residue is formed, particularly during the pulse-mode operation of the motor. Why ammonium nitrate was produced during steady state and $MMH \cdot HNO_3$ during pulse-mode firing is not clearly understood. However, it should be noted that during steady state, very high temperatures are obtained, whereas the motor remains relatively cool during pulse mode. Not only would a more complete reaction be expected to occur at the higher temperatures but also the combustion intermediates, once formed, can be thermally decomposed to yield products which can form ammonium nitrate. Another question which remains to be answered is whether the residue is generated during preignition or after shutdown of the injector valves. In the latter case, the dribble volume of the particular motors between the valves and the combustion chamber will play a major role in the quantity of the intermediates formed.

The pressure-spiking phenomenon has been observed to be eliminated in the N_2O_4 -Aerzine 50 system when the temperature of the propellants and of the hardware is kept above a certain threshold value. Under these conditions the residue formation is considerably decreased or completely eliminated. Similarly, a threshold temperature exists for the N_2O_4 -MMH system. These results have been interpreted by some workers to mean that at the higher temperatures a vapor-vapor reaction takes place between the oxidizer and the fuel, yielding no ignition overpressure and no combustion intermediate. The vapor-liquid reaction occurring at the lower temperatures leads to pressure spikes and residue formation. These hypotheses still remain to be tested and verified. It is clear that the knowledge of the step-by-step reaction occurring in the oxidation of hydrazine derivatives with N_2O_4 is lacking at this time. Such knowledge of the fundamental process of bipropellant combustion can be obtained in the laboratory only under carefully controlled conditions.

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13 ABSTRACT <p>Motor residues from the combustion of N₂O₄ and MMH were analyzed. Under steady-state and pulse-mode firings, the major constituents of the non-volatile residues were found to be ammonium nitrate and MMH·HNO₃, respectively. The latter material contained a heat-sensitive compound which triggered its decomposition at a lower temperature. The application of heat to MMH·HNO₃ initially converted the salt to monomethylammonium nitrate. The infrared³ spectra and differential analysis curves for these materials are presented.</p>			

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KEY WORDS

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Abstract (Continued)

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