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AFRPL-TR-67-141

(U) INVESTIGATION OF DECOMPOSITION CATALYSTS
FOR 98% HYDROGEN PEROXIDE
FIRST QUARTERLY REPORT

Contract F 04(611)-67-C-0068

T. C. F. Munday

FMC CORPORATION
Chemical Research and Development Center
May 1967

GROUP 4

Downgraded at 3 year intervals
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This report contains part of the subject matter of Patent Application 473,328, which describes the composition of a Decomposition Catalyst for high strength hydrogen peroxide. A Patent Office Secrecy Order has been issued covering this Patent Application.

[REDACTED]

(U) INVESTIGATION OF DECOMPOSITION CATALYSTS

FOR 98% HYDROGEN PEROXIDE

FIRST QUARTERLY REPORT

Contract FO4611-67-C-0068

T. C. F. Munday

Technical Report AFRPL-TR-67-141

GROUP 4

Downgraded at 3 year intervals;
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FOREWORD

This program is being conducted under Contract FO4611-67-C-0068 (Project 3148) by FMC Corporation, Princeton, New Jersey, for the Air Force Rocket Propulsion Laboratory, Edwards Air Force Base, California. The engineering tests will be carried out under a subcontract by Walter Kidde and Company, Belleville, New Jersey. Lt. Ralph Fagnoli, USAF/RFCL, is the program monitor for the Air Force.

These investigations were carried out between February and April, 1967 and the report submitted in May, 1967. The program was administered by Dr. L. R. Darbee, Project Director for FMC, with Dr. T. C. F. Munday as principal investigator. Mr. J. C. McCormick served as rocket engineer and general consultant.

This report is classified Confidential according to DD Form 254, Security Requirements Check List, dated 19 December 1966.

Classified information has been extracted from asterisked documents listed under References.

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.

WILLIAM H. EBELKE, Colonel, USAF
Chief, Propellant Division

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CONFIDENTIAL ABSTRACT

Complex oxides containing barium, manganese, lead, and cobalt have been prepared and tested as decomposition catalysts for 98% H_2O_2 . Twenty selected compositions from the $BaO-Mn_2O_3-PbO$ and $BaO-Mn_3O_4-PbO$ systems were mixed and compressed at 33,000 psi into 7/16" diameter pellets. These were then heated to temperatures up to 1000°C. The pellets were tested for catalytic activity by a standard flood test with 10 ml. of 98% H_2O_2 . Compositions low in PbO and high in Mn_2O_3 or Mn_3O_4 exhibited the best combination of catalytic activity and resistance to leaching or mechanical breakdown. Several additional compositions containing Co_3O_4 also showed high catalytic activity and good mechanical strength. Catalyst materials and a modified 40 pound thrust motor were prepared for the motor test program. The new motor is designed to permit temperature measurements within the catalyst pack.

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SECTION I

INTRODUCTION

The catalytic decomposition of 98% hydrogen peroxide for propulsion system applications has been limited by the lack of suitable catalysts. In contrast, 90% H_2O_2 has seen widespread use mainly because systems employing silver screens as the decomposition catalyst have worked so effectively. These silver catalysts have not been used with 98% H_2O_2 because they melt at the decomposition temperature of 98% H_2O_2 , which is some 375°F higher than that for 90% H_2O_2 .

Recent investigations (1,2,3,4) have lead to a catalyst pack for 98% H_2O_2 which is suitable for many applications. This pack is based on a silver-30% palladium catalyst which does not melt when used with 98% H_2O_2 . The catalyst has performed well at flowrates up to 110 pounds of 98% H_2O_2 per minute per square inch of catalyst frontal area, and at chamber pressures above 1500 psia.

However, the cold starting capability of the pack declined as additional operating time was accumulated. This did not affect performance during subsequent parts of a run when the pack was hot, and the start response of the heated catalyst pack also remained rapid. Thus the pack is presently useful for applications which can use a programmed start or require few cold starts.

The current program was planned to develop new catalyst materials and to further characterize and improve the existing catalyst pack. During the previous contract several materials were designated as the best possibilities for the development of new catalysts which would have both thermal stability and rapid starting response. These included complex oxides containing manganese, cobalt, lead, and barium and alloys containing manganese and cobalt. Further studies were required to determine the best compositions and to incorporate these into forms useful for catalyst packs.

Three areas of further study with the existing catalyst pack were planned. Since the start response of the heated catalyst pack remained rapid, some method of heating the pack before initial contact with H_2O_2 to avoid "cold" starts could be used. Therefore, a part of the program was to measure the temperature to which the pack must be heated for consistently good starts.

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For some applications of the catalyst, it may be desirable to use H_2O_2 to which stabilizers have been added. However, studies with 90% H_2O_2 and silver catalysts have shown that many stabilizers cause plugging or poisoning of the catalyst. Thus another objective was to determine the effect of stabilizers on the performance of the existing pack when used with 98% H_2O_2 .

Lastly, the program included investigation of the reduction of pressure drop across the pack by the use of catalyst screens with larger open area.

SECTION II

LABORATORY INVESTIGATION OF CATALYST MATERIALS

1. COMPLEX OXIDE CATALYSTS

a. Previous Studies

During the previous contract (1), catalysts containing manganese, cobalt, lead, and barium oxides were prepared and tested with 98% H_2O_2 . The oxides were used primarily as components of coatings applied to inert support screens. Both the individual oxides and various combinations of the oxides were investigated. The screen coatings were not sufficiently adherent to the support screens for practical motor applications unless non-catalytic components which could serve as binders were included in the coating. Of several binders used, boric oxide appeared best.

The catalytic activity of these materials was sometimes as high or higher than that obtained with the silver or silver-30% palladium catalyst screens. However, the same approximate compositions often had very low activity. Therefore, further investigation of these complex oxide materials was needed. It was suggested that the activity of the complex oxides varied with composition. Since the particular phase produced was probably also a function of the firing temperature, this variable required examination as well.

b. Experimental

Reagent grade BaO , PbO , MnO_2 , $Mn(C_2H_3O_2)_2$, and $Co(NO_3)_2 \cdot 6H_2O$ were used as starting materials. The oxides Mn_2O_3 , Mn_3O_4 , and Co_3O_4 were prepared by thermal decomposition of MnO_2 and $Co(NO_3)_2 \cdot 6H_2O$. The MnO_2 was heated at $700^\circ C$ for 15 minutes to produce Mn_2O_3 and at 1000° for 15 minutes to prepare Mn_3O_4 . Powder x-ray diffraction patterns verified the formation of the lower manganese oxides. The transition from Mn_2O_3 to Mn_3O_4 was also shown by weight loss measurements. Decomposition of the cobalt nitrate was carried out by heating to $1000^\circ C$. Platinum or porcelain crucibles were used as containers for the decompositions.

Pellets were prepared from the appropriate oxides weighed out in selected proportions. Two gram samples of the oxides were thoroughly mixed and then compressed to 7/16" diameter pellets on a laboratory bench press. A compression of 33,000 psi was used.

The resulting pellets were fired in a resistance furnace which was proportionally controlled. Initially the pellets were placed directly in the furnace which was already at 1000°C. Since a large percentage of the compositions of interest melted or flowed when heated so rapidly, a procedure of slow heating was adopted. This involved raising the temperature in 50° increments from 700 to 1000°C, with a 5 minute hold-time at each temperature level. In this way sufficient time was allowed for reaction of the components and for the decomposition gas (mainly oxygen) to be expelled from the pellet. By this method cohesive pellets were produced.

The pellets were tested for catalytic activity by the standard decomposition test with 10 ml. of 98% H₂O₂. This test involves adding the catalyst to the H₂O₂ and measuring the time required for complete decomposition of the H₂O₂ to dryness. Additional characteristics such as starting activity and residues left after the H₂O₂ decomposition, were also noted. Since many materials have previously been checked by this test, a good measure of the relative activity of the catalyst was obtained.

c. Results

Pellets were prepared from various combinations of Mn₂O₃, Mn₃O₄, MnCO₃, BaO, PbO, and Co₃O₄. Since combinations of manganese, barium, and lead oxides had previously shown good activity, initial efforts were concentrated on a systematic study of the BaO-Mn₂O₃-PbO and BaO-Mn₃O₄-PbO systems. Other combinations were chosen primarily to indicate which general compositions merited further work.

(1) The BaO-Mn₂O₃-PbO System

Compositions were selected to give a general picture of the variation of catalytic activity and physical strength with composition, so that additional effort could be concentrated in promising sections of the system. Details concerning the preparation and testing are shown in Table I. In some cases where the pellet disintegrated or melted and decomposed, the resultant material was ground and recompressed to form a new pellet. Slow heating of the pellets resulted in greater strength, probably because more time was allowed for decomposition gases to escape. Repeated activity tests were carried out for those pellets which showed high activity and at least some cohesive strength.

Catalyst pellets for use in a motor would probably be 1/8" in diameter. Pellets of this size prepared from cobalt

Table I
Complex Oxide Catalyst Pellets

NaO-MnO-Ca-FeO System

2 Gram Samples Compressed at 3,000 PSI to 1/4" Diam. Pellets

Sample	Composition Mole Ratio			Heat Treatment		Remarks	Color	Activity Test				
	NaO	MnO ₂	PbO	Maximum Temp. (°C)	Heated Slowly			ml/min	Response	Activity		
1	1	--	1	1000	Yes	Pellet hard with small cracks in one side	Gray- Black	22	Extensive	Slow		
2	1	1	3	1000	No	Pellet hard and porous with two large cracks	Glassy- gray	16	Moderate	--		
								19	Slight	--		
								6	--	Immediate		
								16	--	Immediate		
3	--	1	1	1000	No	Pellet hard with one large crack	Glassy- gray	22	Extensive	Slow		
				1000	Yes			17	Slight	Slow		
				1000	No			6	Slight	--		
								5	Slight	--		
4	3	1	1	1000	No	Pellet hard; edges easily abraded	Gray- black	21	Extensive	Slow		
				1000	No			86	Extensive	Violent		
				1000	Yes			100	Pellet fractured	Slow		
				1000	Yes			100	Pellet fractured	Slow		
5	1	1	1	700	No	Pellet hard; somewhat porous One large fissure	Black	14	Moderate	Immediate		
				1000	No			60	Slight	Violent		
				1000	No			17	Slight	--		
								4	Slight	--		
6-A	1	3	1	700	No	Pellet frac- tured in two; easily abraded; edges strong	Brown, black	27	None	Slow		
								27	None	Slow		
								27	None	Slow		
								27	None	Slow		
7-A	2	1	--	700	No	Pellet very porous, frac- tured into pieces and easily abraded	Green	5 ⁶	--	--		
								5 ⁶	--	--		
								5 ⁶	--	--		
								5 ⁶	--	--		
8	1	1	--	1000	Yes	Pellet moder- ately hard al- though very porous	Gray-black	35	None	--		
								2	None	--		
								14	Slight	--		
								24	None	Immediate		
9	1	2	--	1000	No	Porous solid formed from melt.	--	50	Extensive	--		
								50	Extensive	--		
								50	Extensive	--		
								50	Extensive	--		
10	1	3	--	700	No	Pellet hard	Green	3 ⁹	--	--		
								3 ⁹	--	--		
								3 ⁹	--	--		
								3 ⁹	--	--		

¹After 15 minutes at 700°C, held 5 minutes at each succeeding 50° higher interval and 15 minutes at 1000°.

²Milliliters of 98% H₂O₂ decomposed per minute. The test used 10 ml. of H₂O₂ initially at about 20°C. Repeated activity tests are listed in order for each sample.

³Deposit left in container after activity test.

⁴Response at beginning of decomposition test.

⁵Part of the 2 gram pellet used for the test.

metal and manganese dioxide were successfully tested in the previous program. However, as with the silver-30% palladium catalyst packs, motor start-up with these metal-oxide pellets was slow. Activity tests on the individual pellets showed decomposition rates of about 0.5 ml./min., with extremely poor starting activity. In the current program, a 1/8" diameter pellet of Sample 5 composition was prepared and tested for comparison. A decomposition rate of 5 ml/min. was measured. The starting response was not as rapid as new silver-30% palladium screens, but much better than either the cobalt metal-manganese oxide pellets or used silver-palladium screens.

Approximately 2 to 3 catalyst pellets of 1/8" diameter can be included in a catalyst pack in place of each catalyst screen. New catalyst screens (silver-30% palladium) exhibit decomposition rates up to 30 ml./min., but after several minutes of motor operation, rates are usually 0.5 ml./min. or even lower. Therefore, the BaO-Mn₂O₃-PbO pellets appear to show catalytic activities which are high enough for good motor performance.

Most of these BaO-Mn₂O₃-PbO pellets showed a decline in activity with repeated testing. This decline was not evident for Samples 2, 3, 8, and 9 when they were heated to motor operation temperatures (950°C), cooled, and then retested. Further such tests are now required to show whether the high temperatures of motor operation will continue to activate the pellets for subsequent cold starts.

The catalytic activities for various compositions of BaO-Mn₂O₃-PbO pellets are shown in Figure 1. The rates of decomposition are given in milliliters of 98% H₂O₂ decomposed per minute. All results refer to data for the first decomposition tests after the pellets had been heated to 1000°C and cooled.

The resistance of the pellets to erosion, leaching, or disintegration is important for successful operation in catalyst packs. As shown in Figure 1 and Table I, a residue was often left after the decomposition tests. Compositions containing large percentages of Mn₂O₃ and low percentages of PbO (Samples 6-9) tended not to leave residues. These four pellets are indicated to the lower-right section of Figure 1. Sample 6 (with a decomposition rate of 31 ml/min.) had the best physical strength.

Powder X-ray diffraction patterns were made for Samples 7-A and 10. These indicated that about 50%

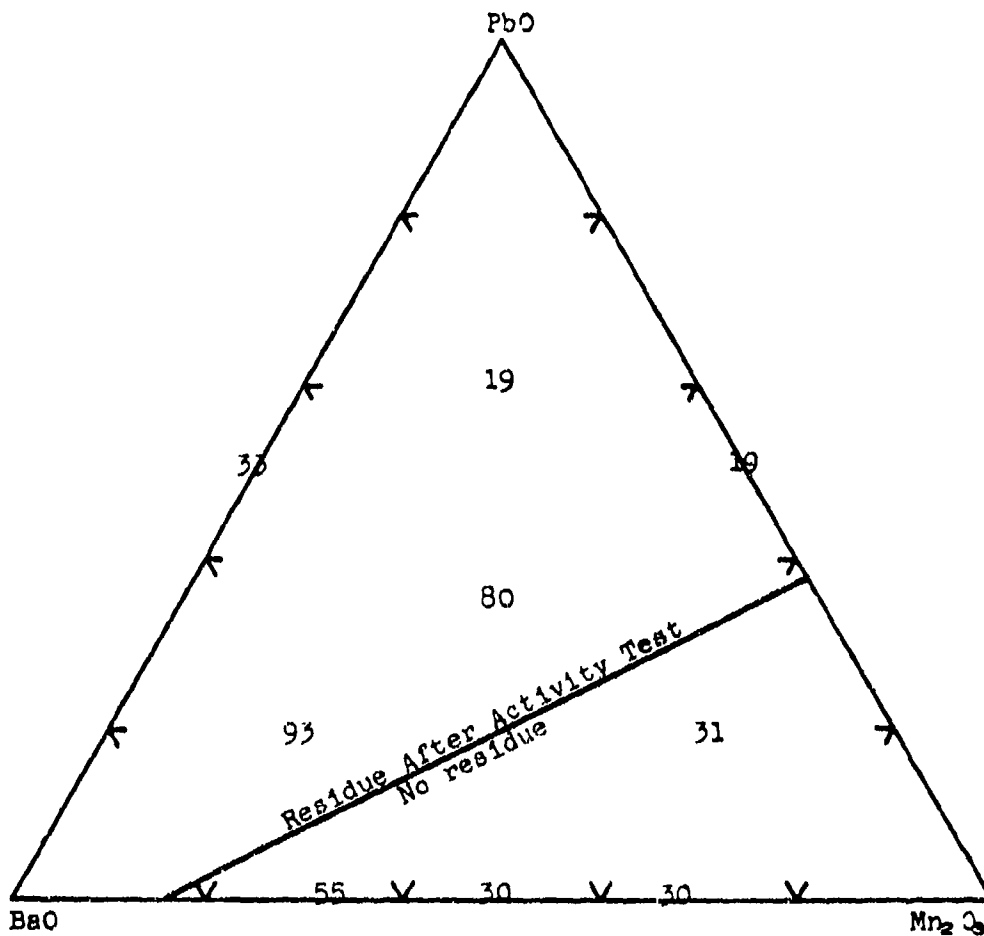
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Figure 1

Rates of Decomposition of 98% H_2O_2 by $BaO-Mn_2O_3-PbO$

Catalyst Pellets

Decomposition rates are given in ml/min. for pellets tested in 10 ml. of 98% H_2O_2 initially at 20°C. Tests used 7/16" diameter pellets which had been heated to 1000°C and then cooled. Compositions are on a molar basis.



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of the Mn_2O_3 in those pellets was unreacted. Similar determinations are likely to show that more complete reaction has occurred for samples heated to 1000° . If not, some advantage in strength or catalytic activity may be produced by grinding, repelletizing, and heat treating the material until reaction is complete.

Of the pellets containing higher fractions of PbO, Samples 2 and 3 exhibited good hardness. Though some fractures were produced during heat treatment of these samples, further fracturing did not occur during testing. However, in each case a brown residue remained after testing, indicating that lead and manganese oxides were leached from the pellet during the tests.

(2) The BaO- Mn_3O_4 -PbO System

The compositions prepared and the results obtained are shown in Table II. Like the previous system, the best overall compositions were those low in PbO and high in the manganese oxide as shown in Figure 2. The catalytic activity of these samples fluctuated. The pellet with the best physical strength had a decomposition rate of only 9 ml./min. The other compositions showed higher activities but would be more subject to erosion under motor operating conditions. Reheating of the pellets to motor operation temperatures between repeated tests has not yet been tried, but more consistent high activities will probably be produced by this method.

Pellets containing larger percentages of PbO were again very hard (Samples 1-5). Samples 1 and 3 left a residue after testing, but the others showed good resistance to leaching or mechanical breakdown.

The extent of reaction between the components was measured by x-ray diffraction patterns for Samples 7-A, 9-A, and 10. The patterns indicated essentially complete reaction of the Mn_3O_4 for Samples 7-A and 9-A. Approximately half of the Mn_3O_4 was unreacted in Sample 10.

(3) Alternative Systems

The compositions and test results for additional complex oxides are given in Table III. A consideration of the physical strength as indicated by 1) the condition after heating and 2) the residue after activity testing shows that Samples 3 and 5 are good catalyst candidates. Sample 5 showed the greatest abrasion resistance of any composition

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Table II

Complex Oxide Catalyst Pellets

PbO-Mn₂O₃-PbO System

2 Gram Samples Compressed at 11,000 PSI to 7/16" Diam. Pellets

Sample	Composition Mole Ratio			Heat Treatment		Remarks	Color	Activity Test		
	PbO	Mn ₂ O ₃	PbO	Temp. (°C)	Heated slowly			NI/Mn ²	Residue ³	Start time ⁴
1	1	--	--	1000	Yes	Pellet hard with small cracks in one side	Gray-black	55	Extensive	Slow
2	1	1	1	1000	No	Porous solid formed from melt	Gray-black	18	Slight	Slow
	Sample 2 ground and recompressed			1000	Yes	Pellet very hard	Gray-black	14	None	--
								4	None	Slow
								17	None	Slow
								17	None	Very slow
3	--	1	1	1000	Yes	Pellet very hard; fractured in two	Glassy-gray	25	Slight	--
								17	Slight	Slow
								21	Moderate	Slow
4	3	1	1	1000	No	Porous solid formed from melt	Gray-black	33	Slight	--
	Sample 4 ground and recompressed			1000	Yes	Pellet hard, edges strong	Gray-black	22	Slight	--
								6	None	--
								21	None, pellet split	Slow
								7	None	Immediate
5	1	1	1	700	No	Pellet fractured into pieces	Gray-black	16	Moderate	--
				1000	No	Pellet hard	--	75	--	Violent
	Sample 5 ground and recompressed			1000	Yes	Pellet hard; edges abraded with difficulty	Gray-black	5	Slight	--
								8	None	--
								8	None	Slow
								6	None	--
6	1	3	1	1000	No	Pellet hard and porous; one small fissure	Gray-black	9	None	--
								4	None	--
								23	None	--
7-A	2	1	--	1000	No	Pellet porous, weak	Green-black	7 ⁵	--	--
7-B	2	1	--	1000	No	Porous solid formed from melt	Black	58 ⁵	--	--
8	1	1	--	1000	Yes	Pellet moderately hard	Black	37	Slight	--
								7	None	--
								27	None	Immediate
9-A	1	2	--	1000	No	Pellet hard, edges easily abraded	Olive	2 ⁵	--	--
9-B	1	2	--	1000	No	Fractured into pieces	Olive	21	None	--
	Sample 9-B ground and recompressed			1000	Yes	Pellet hard abraded with difficulty	Olive	43	None	Immediate
								26	None	Immediate
10	1	5	--	1000	No	Pellet hard, edges easily abraded	Purple-black	6 ⁵	--	--

¹ After 15 minutes at 700°C, held 5 minutes at each succeeding 50° higher interval and 15 minutes at 1000°.

² Milliliters of 98% H₂O₂ decomposed per minute. The test used 10 ml. of H₂O₂ initially at about 20°C. Repeated activity tests are listed in order for each sample.

³ Deposit left in container after activity test.

⁴ Response at beginning of decomposition test.

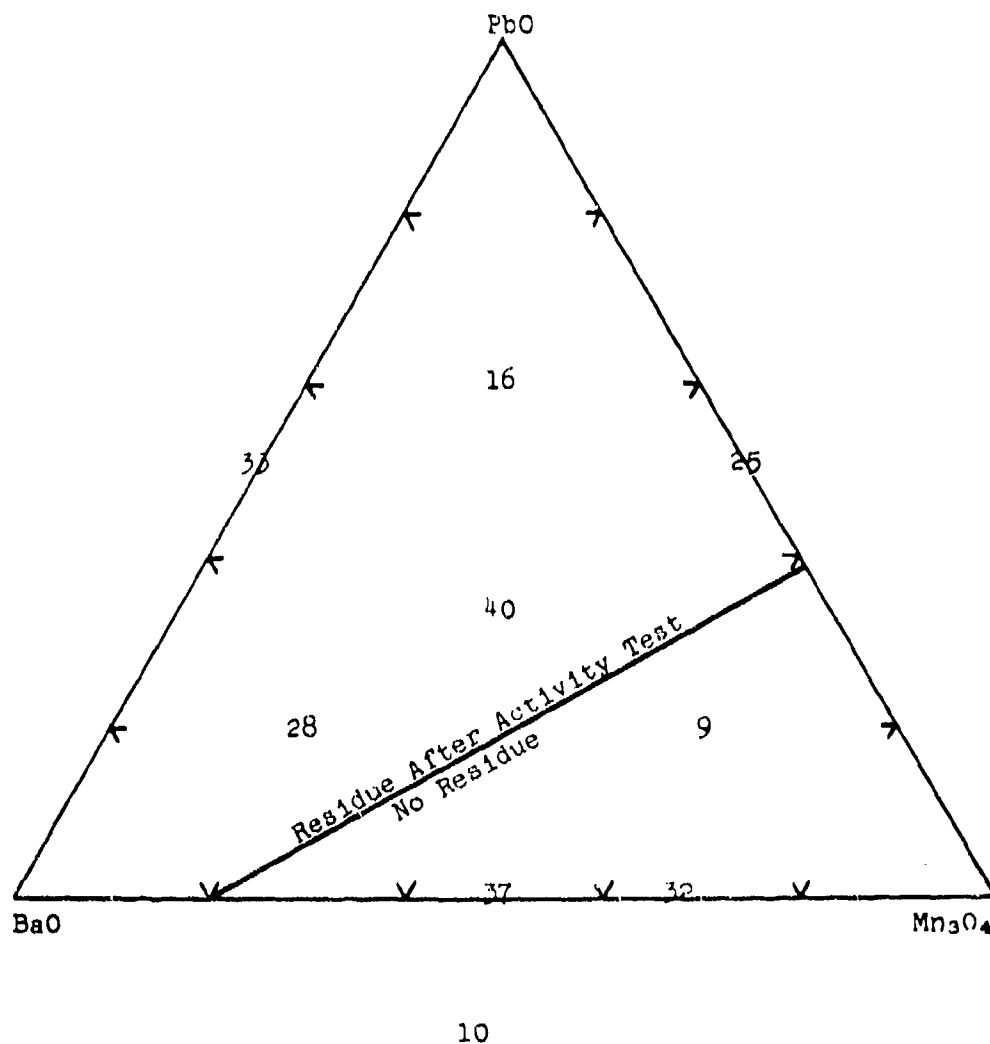
⁵ Part of the 2 gram pellet used for the test.

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Figure 2

Rates of Decomposition of 98% H_2O_2 by $BaO-Mn_2O_4-PbO$
Catalyst Pellets

Decomposition rates are given in ml./min. for pellets tested in 10 ml. of 98% H_2O_2 initially at 20°C. Tests used 7/16" diameter pellets which had been heated to 1000°C and then cooled. Compositions are on a molar basis.



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Table III
Complex Oxide Catalyst Pellets
Alternative Systems

2 Gram Samples Compressed at 33,000 PSI to 7/16" Diam. Pellets

Sample	Composition Mole Ratio	Remarks	Color	Activity Test		
				Ml./Min. ¹	Residue ²	Starting ³
1	1 BaO-1MnCO ₃	Pellet extensively fractured	Brown-black	150	Slight pellet fractured	Violent
	Sample 1 ground and recompressed	Pellet moderately hard, easily abraded	Gray	29	None	Rapid
2	1 BaO-1 Co ₃ O ₄	Pellet very porous and expanded	Black	133	Extensive, pellet fractured	Slow
3	1 BaO-1Mn ₂ O ₃ -1Ca ₂ O ₄	Pellet hard, one large fissure	Brown-black	11	None	--
4	1 BaO-1Mn ₂ O ₃ -1Co ₃ O ₄	Pellet very hard, one large fissure	Brown-black	10	Slight	--
5	1 BaO-1PbO-1Co ₃ O ₄	Pellet very hard, porous	Gray-black	15	Slight	Slow
6	1 PbO-1Mn ₂ O ₃ -1Co ₃ O ₄	Pellet soft, easily abraded	Gray-black	22	Slight	Slow
7	1 PbO-1Mn ₂ O ₃ -1Co ₃ O ₄	Pellet moderately hard, some large fissures	Gray-black	15	Slight	--
8	1 Mn ₂ O ₃ -1Co ₃ O ₄	Pellet soft, easily abraded	Black	60	Extensive, pellet crumbling	Slow
9	1 Mn ₂ O ₃ -1Co ₃ O ₄	Pellet soft, easily abraded	Gray-black	11	None	--
10	1 Mn ₂ O ₃ -1Mn ₂ O ₄ -1Co ₃ O ₄	Pellet soft, easily abraded	Gray-black	43	Moderate	Slow

Note: All samples heated 15 minutes at 700°C, then held for 5 minutes at each succeeding 50° higher interval and finally heated 15 minutes at 1000°.

¹Milliliters of 98% H₂O₂ decomposed per minute. The test used 10 ml. of H₂O₂ initially at about 20°C. Repeated activity tests are listed in order for each sample.

²Deposit left in container after activity test.

³Response at beginning of decomposition test.

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tested. Repeated tests of these samples should be carried out. Testing of various compositions in these systems may lead to other possibilities. The Samples 1 and 4 also rate well above the remaining compositions.

The other samples shown in Table III are clearly unsuitable in their present form. Some of these compositions which are very active could be of interest if binders were added. However, it is likely that the high activity was due to the large amounts of catalyst surface exposed when the pellets broke apart.

2. LABORATORY PROGRAM

The laboratory program will include continued investigation of complex oxide catalysts containing barium, manganese, lead and cobalt. In addition, alloys containing manganese and cobalt will be examined for catalytic activity and thermal stability. Development work will then proceed on incorporating the best of the complex oxides and/or alloys into forms which are appropriate for catalyst packs used in rocket motors.

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SECTION III

ROCKET MOTOR TESTS

Motor tests for the evaluation of new catalysts and for further characterization and improvement of the existing catalyst are being carried out under subcontract with the Walter Kidde and Company of Belleville, New Jersey.

1. MOTOR AND CATALYST SCREENING TESTS

Motor tests were planned for the evaluations required in the four objectives of the program. These are the standardization of the modified test motor, the reduction of pressure drop across the existing catalyst pack, the determination of catalyst response with stabilized H_2O_2 , and the motor screening of the best new catalyst materials developed in the laboratory studies.

a. Modified Thrust Motor

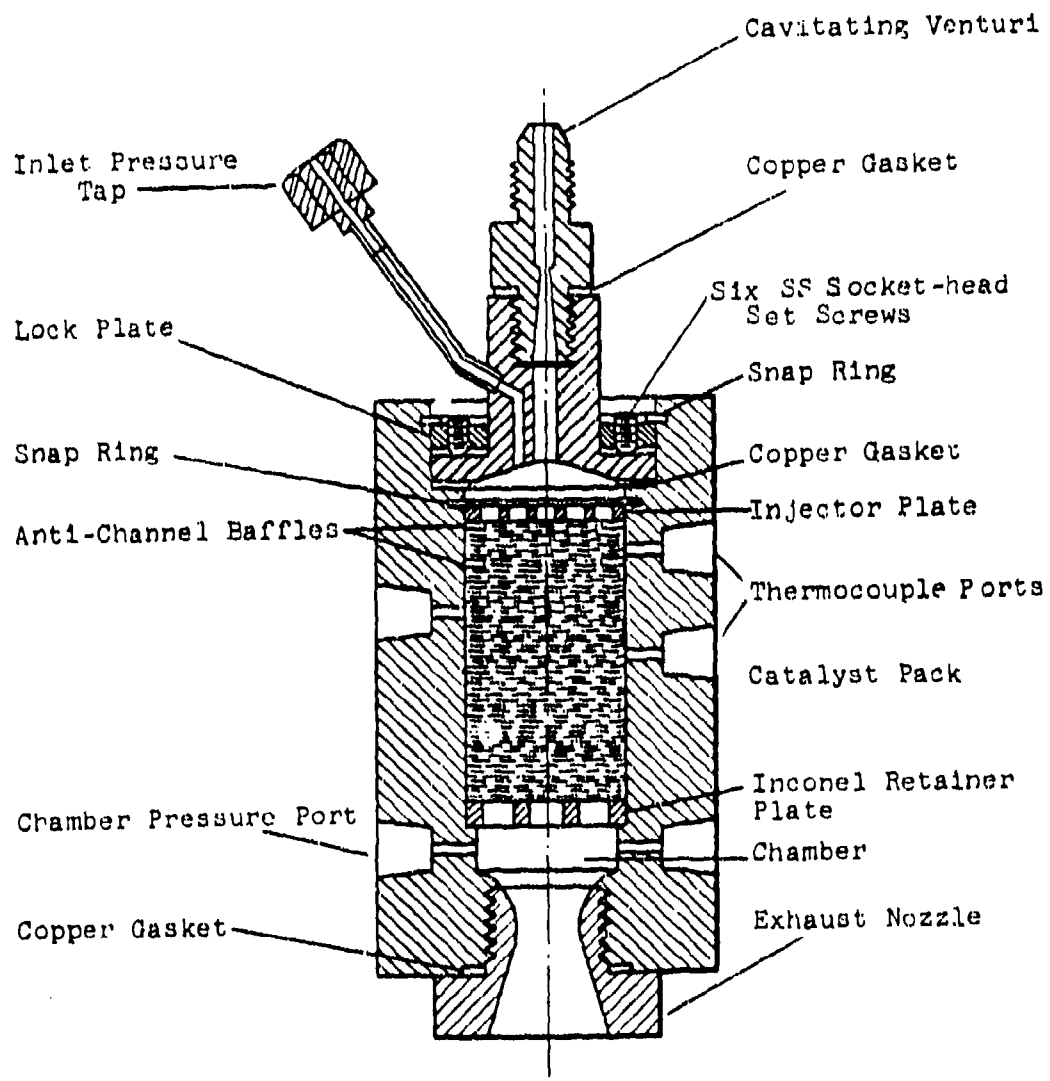
Results obtained during the previous program showed the value of temperature measurements made at various locations within the catalyst pack during motor operation. For two sections of the current program, the regulated temperature start tests and the stabilized H_2O_2 tests, such measurements were clearly warranted. Therefore, it was decided to use a motor equipped for temperature measurements for all motor tests.

A basic 40-pound thrust motor design was modified as shown in Figure 3. The body of the motor was fabricated from 2-inch hexagonal 347 stainless steel stock with a 1" diameter cavity for the catalyst pack. A total of seven thermocouple ports were distributed along the catalyst pack cavity. These ports entered at 1/8" intervals beginning 1/8" below the inlet plate and ending 7/8" down the catalyst pack. The ports were arranged around the circumference of the pack to minimize interference with H_2O_2 flow in any one sector of the pack. Two additional ports were included for temperature and pressure measurements in the chamber and one for pressure measurement just ahead of the pack inlet.

The motor was provided with a cavitating venturi to control the flow at 20 pounds of H_2O_2 per minute per square inch of cross sectional area of catalyst. A corresponding exhaust nozzle was selected to give about a 300 psi chamber pressure. The retainer plate was constructed from inconel for extra high temperature strength. The

[REDACTED]

Figure 4
Modified Thrust Motor



Scale: Full Size

[REDACTED]

remaining parts except for the copper gaskets are stainless steel. Cylindrical sleeves of 1 inch diameter, 1/16" thickness, and various lengths were fabricated to take up the excess space when short catalyst packs are used.

b. Catalyst Preparation

The silver-30% palladium 20 and 16 mesh catalyst screens were treated prior to use according to the procedure listed below.

A. Preparation of Screen

1. The screen was degreased in a vapor degreaser containing perchloroethylene.
2. The screen was then sandblasted with a 54 grit aluminum oxide at 60-80 psi.

B. Acid Treatment of Screen

1. A 1" square piece of 20 mesh, 0.014 mil, 70% Ag-30% Pd alloy, wire screen was dropped into 500 cc concentrated nitric acid and allowed to dissolve. The sandblasted screen was dipped into the nitric acid solution containing the dissolved screen and moved about in the liquid for approximately 30 seconds. It was then removed and allowed to drain as thoroughly as possible.
2. The screen was then placed in the oven at a temperature of 400°F for 20 minutes; upon its removal the screen was quite dark and rather heavily coated. It was then dipped a second time following the same procedure as above and again heated for 20 minutes at 400°F.

C. Samarium Nitrate Treatment of Screen

1. The screen was next immersed into a solution of 20% by weight of samarium nitrate until thoroughly wet, removed from the solution and allowed to drain thoroughly.
2. The material was then heated for 20 minutes for 720°F. This procedure was repeated until seven (7) dips and seven (7) heatings had been completed.

3. Since some areas of the screen openings were generally blocked, the screen was brushed with a stiff brush.

The silver catalyst screens to be used in this program were also cleaned, sandblasted, acid treated, and coated with samarium oxide. The nickel-5% manganese filler screens will be used without further treatment. Both treated catalyst screens and untreated filler screens were punched to one inch diameter discs for use in the motor.

c. Catalyst Packing

The first step in loading the motor with catalyst screens was to place the retainer plate inside the motor flat against the lip at the bottom of the catalyst cavity. Next the catalyst screens were added one-by-one on top of the retainer plate. Each screen was rotated somewhat from the orientation of the previous screen to provide a uniform distribution of catalyst within the cartridge. After one-third of the screens had been loaded, a 4" long, 1" diameter packing ram was inserted in the cartridge and the screens were compressed to 4000 psi on a laboratory bench press. The ram was removed and additional screens were added. The pack was again compressed to 4000 psi after 2/3 of the screens had been loaded into the cartridge. After all the screens were loaded, the anti-channel baffle and injection plate were placed above the catalyst screens. Then while the pack was compressed to 4000 psi with a packing ram only 1/2" in diameter, the snap ring was inserted into the slot just inside the front of the catalyst chamber.

d. Catalyst Pack Configuration

The catalyst configuration for the motor standardization test was selected to match closely with the best configuration tested under the previous contract. The inlet section contained 15 silver catalyst screens of 20 mesh. These were followed by 38 silver-30% palladium screens of 20 mesh and 32 nickel-5% manganese screens of 14 mesh. The total length of the pack was 1 3/8 inches.

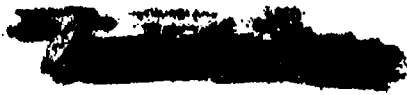
e. Motor Standardization

The initial motor checkout will provide reference data for comparison with other tests in both the current and previous programs. It will also be used to check the calibration of the venturi for use in the remaining tests. This will insure at the outset that the motor itself is not the cause of effects which might later be attributed to the catalysts.



2. MOTOR TEST PROGRAM

The motor testing after the initial standardization will consist of three basic types of test procedures. The first will be identical to that used for the standardization and for the initial screening tests of the previous program. The results can, therefore, be compared directly with earlier results. These tests will be performed to measure the effect of larger open area catalyst screens and to evaluate new catalyst materials derived from the laboratory investigations. The second and third test procedures are designed especially to test starting at regulated temperatures and performance with stabilized H_2O_2 .



SECTION IV

CONCLUSIONS AND RECOMMENDATIONS

Catalytic activity tests show that catalyst pellets containing various combinations of barium, lead, manganese, and cobalt oxides exhibit high activity. In the $\text{BaO-Mn}_2\text{O}_3\text{-PbO}$ and $\text{BaO-Mn}_3\text{O}_4\text{-PbO}$ systems the best resistance to leaching and erosion occurs for compositions low in PbO and high in the manganese oxide. Greater physical strength resulted when the samples were initially heated slowly to 1000°C . Tests also indicate that catalytic activity for cold starts is enhanced by subjecting the samples to high temperatures such as those present during motor operation.

Of other combinations tested, samples containing equimolar amounts of BaO , Mn_2O_3 , and Co_3O_4 or BaO , PbO , and Co_3O_4 were the best candidates for good catalysts. The latter composition exhibited the greatest abrasion resistance. Samples prepared from BaO , Mn_3O_4 and Co_3O_4 or BaO and MnCO_3 are also of further interest.

Additional testing is recommended for both the best barium-manganese-lead complex oxides tested to date and for closely related compositions which contain the same components. The effect on catalytic activity and mechanical strength of repeated exposure to high temperatures and extended contact with H_2O_2 should be determined. Further study of complex oxides containing cobalt is required. The best oxides should then be modified as required to produce catalysts suitable for motor tests.

[REDACTED]

SECTION V

REFERENCES

- *1. T. C. F. Munday, L. R. Darbee, and J. C. McCormick, "(U) Investigation of Decomposition Catalysts for 98% Hydrogen Peroxide. Final Report," FMC Corporation, Princeton, New Jersey. Report Number AFRPL-TR-67-80 for Air Force Contract AF 04(611)11208, March 1967. CONFIDENTIAL.
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A series of materials were prepared and tested as decomposition catalysts for 98% H ₂ O ₂ . Thirty different compositions were pelletized at high pressure and heat treated. Extent of reaction between the components was indicated by x-ray diffraction patterns. Catalytic activity was measured by a standard flood test with 10 ml. of 98% H ₂ O ₂ . Mechanical strength and susceptibility to leaching and erosion were observed. Catalyst materials and a modified 40 pound thrust motor were prepared for the motor test program.		

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
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