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AFAPL-TR-70-71 Part I

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# Hydrocarbon Fuels for Advanced Systems

G.H. Ackerman, L.E. Faith, C.K. Heck, H.T. Henderson, A.W. Ritchie, L.B. Ryland

> Sheli Development Company A Division of Shell Oil Company Emeryville, California

Technical Report AFAPL-70-71, Part I December 1970

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#### HYDROCARBON FUELS FOR ADVANCED SYSTEMS

G. H. Ackerman, L. E. Faith, C. K. Heck, H. T. Henderson, A. W. Ritchie, and L. B. Ryland

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#### FOREWORD

The work described in this technical report was authorized under J.S. Air Force Contract No. F33615-70-C-1038, Project 3048. The program was administered by Mr. H. R. Lander and Lt. J. C. Ford, Project Engineers, Air Force Aero Propulsion Laboratory/APFF-1, Wright-Patterson Air Force Base, Ohio 45433.

This program is being conducted at Shell Development Company, Emeryville, California 94608 under the general supervision of K. D. Detling, Manager, Government Research Department. This report is the first annual technical report under Contract No. F33615-70-C-1038. It was submitted on September 17, 1970. The report describes the results of work performed during the period 1 September 1969 to 31 August 1970. This investigation represents a continuation of a previous study, "Vaporizing and Endothermic Fuels for Advanced Engine Application" under Contract No. AF 33(615)-3789, the results of which were published in Technical Report AFAPL-TR-67-114, Part I, October 1967, Part II, September 1968; Part III, February 1970.

Acknowledgment is made to A. C. Nixon for many valuable contributions to the program as principal investigator and project supervisor until his retirement on 1 June 1970.

This technical report has been reviewed and approved.

Arthur V. Churchill

ARTHUR V. CHURCHILL, Chief Fuel Branch Fuel, Lubrication and Hazards Division

AFAPL-TR-70-71

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-11-

#### ABSTRACT

The general objective of this investigation is the development of new fuels and new fuel systems which will provide the cooling and propulsion or tits of advanced sir-breathing engines. In previous studies on the utiliza ⇒ndothermic reactions of hydrocarbon fuels, the catalytic dehydrogenation thenes showed the most promise for practical applications. In continuing these studies, a large number of catalysts were prepared and tested for activity and stability for the dehydrogenetion of methylcyclohexane and decalin. The stability of supported platinum catalysts for these reactions was affected by the physical properties and chemical composition of the support and by the metal content and metal composition of the catalyst. Bicyclo(2,2,2)octane was dehydrogenated to bicyclooctene in low yield with a supported platinum catalyst. Additives were found which enhanced the rate of thermal cracking of paraffins. Improved wall catalysts were formulated for the dehydrogenation of naphthenes and a mathematical model was developed for a catalytic wall reactor. Various fuels were evaluated for thermal stability in both our standard ASTM Coker and a special Alcor JFTOT unit designed for operation up to 1000°F and 1000 psi. The high temperature thermal stability of fuels was improved by means of additives. The deposit profiles on coker tubes were reproducibly determined with a recently developed beta-ray backscatter instrument which is capable of measuring deposit thicknesses up to 2500 Å. The physical properties of JP-7 jet fuel were recalculated using improved methods. The new values compare favorably with data obtained from heat transfer experiments. The supersonic combustion of decalin, tetralin and naphthalene was investigated using the shock tube. A literature survey was made of articles and patents of interest to this and related programs.

AFAFL-TR-70-71

1.1.1

-iii-

### CONTENTS

	Page
INTRODUCTION	1
SUMMARY	2
LABORATORY STUDER OF CATALYTIC AND THERMAL REACTIONS	6
Bench-Scale Catalyst Stability Studies	6
Dehydrogenation of Decalin	7
Dehydrogenation of Methylcyclohexane	9
Dehydrogenation of Bicyclo(2,2,2)octane	11
Thermal Reaction	12
Catalytic Dehydrogenation	14
Thermal Cracking of n-Dodecane Using Additives	18
Effect of Propane on the Catalytic Dehydrogenation of Methylcyclohexane	19
CATALYST STUDIES	21
Preparation and Evaluation of Granular Catalysts	22
Preparation of Catalyst Coatings on Metal Surfaces	30
Stainless Steel	30
Aluminum Clad Stainless Steel • • • • • • • • • • • • • • • • • •	32
MEASUREMENT OF CATALYTIC REACTION KINETICS	33
Need for Intrinsic Kinetic Data	33
Design of Experimental Reactors	34
MATHEMATICAL MODEL OF CATALYTIC WALL REACTOR	35
Model Development	35
Application to Methylcyclohexane Dehydrogenation	40
Accuracy of Fredicted Reaction Rates	42

AFAPL-TR-70-71

.

Sec. Se

-v -

# CONTENTS (Contd)

NTTIMD ACTIVATION OF STRAFT THE THE PHONE AND AND AND A	
SIMULATION TEST RIG	3
THERMAL STABILITY OF FUELS	0
Measurement of Deposits by Beta-Ray Backscatter 5	1
Boeing Titanium Surips	3
Alcor JFTOT and SD/M-7 Coker Tubes	4
CRC Erdco JFTOT Preheater Tubes	5
CRC Alcor JFTOT Preheater Tubes	8
Problems in the Use of the Beta-Ray Backcastter Instrument 6	3
Fuel Additives for Improvement of Thermal Stability6	4
Hydrocrackate Jet Fuels	l
Modifications and Assembly of Thermal Stability Apparatus 7	3
SD/M-7 Fuel Coker	5
STORAGE STABILITY OF METHYLCYCLOHEXANE AND SHELLDYNE-H® FUELS	7
Elastomer Interactions With SHELLDYNE-H $^{ extsf{B}}$ Fuel $\ldots$	8
THERMAL STABILITY OF NEW BATCH OF METHYLCYCLOHEXANE	9
ESTIMATION OF PHYSICAL PROPERTIES OF FUELS	9
JP-7 Jet Fuel	9
Binary Mixtures of Methylcyclohexane and a Low	
Molecular Weight Hydrocarbon	1
SHELLDYNE-H <sup>®</sup> Hydrocarbon Fuel	5
SUPERSONIC COMBUSTION OF HYDROCARBONS	5
Experimental Equipment and Conditions $\ldots \ldots \ldots \ldots \ldots \ldots $	ł
Shock Tube Attenuation	ł
Correlation of Ignition Delay Times	7
Rate of Combustion of Tetralin	)

AFAPL-TR-70-71

Page

-vi-

# CONTENTS (Contd)

Page
FUTURE PROGRAM         93
RELATED DEVELOPMENTS AND APPLICATIONS
REFERENCES
APPENDIX
Table 55. Dehydrogenation of Decalin Over Various Catalysts . following 99
Table 56. Dehydrogenation of Methylcyclohexane Over Various         Catalysts
Calculation of Rate Constants (Bench-Scale Reactor)
Calculation of Reaction Products for the Thermal Reaction of Bicyclo(2,2,2)octane
Micro Catalyst Test Reactor
Table 57. Dehydrogenation of Methylcyclohexane With Various Catalysts in MICTR
Table 58. Dehydrogen ion of Methylcyclohexane With Various Catalysts in MICTR
Derivations of Equations for the Catalytic Wall Reactor Model 103
Description of the Beta-Ray Backscatter Apparatus for Rating Deposits
Table 59. Ignition Delay Times for Decalin-Oxygen-Argon Mixtures 113
Table 60. Ignition Delay Times for Tetralin-Oxygen-Argon Mixtures 114
Table 61. Ignition Delay Times for Naphthalene-Oxygen-Argon Mixtures
Table 62. Physical Properties of Methyleyelohexane/Ethane Mixtures
Table 63. Physical Properties of Hechyletelohessne/Propase Mixtures
Table 64. Physical Properties of Nothy Peyclohexane/n-Butane Mixtures

-vii-

AFAPL-TR-70-71

# <u>e MTENTS (Contd)</u>

やいの

Table 65. Liquid Properties of JP-7 Jet Fael at Saturation 1	.25
Gas Properties of JP-7	.25
BIBLIOGRAPHY	27
Advanced Fuels Including Endethermic and Vaporizing Fuels 12	27
Physical and Chemical Properties of Fuels	32
Thermal Stability of Fuels	37
Fuel Contaminants and Additives	41
Catalysts and Catalytic Reactions	47
Heat Transfer and Fluid Mechanics	56
Combustion	59
Advanced Engine Development	76

Page

#### 间开心出意书的现象。

Fig	ave the second	
1	Dehydrogenation of Decalin: Fifeet of Semperature of Conversion	
c)	Debydrogenation of Decalin: Effect of Temperature a Catalyst Stability	
3	Dehydrogenation of Decalin: Effect of Pemperature on Catalyst Stability	
<u>}</u> †	Dehydrogenation of Decalin: Effect of Catalyst Port Diameter on Stability	
5	Pehydrogenation of Methylcyclohexane Over One Percent Platinum Catalysts	
6	Dehydrogenation of Methylcyclohexanc Over Four Percent Platinum Catalysts	
7	Effect of Temperature on Thermal Reaction of Mesitylene and Bicyclo(2,2,2)octane	
8	Dehydrogenation of Bicyclo(2,2,2)octane With One Percent Platinum on Alumina in Pulse Reactor	
9	Dehydrogenation of Methylcyclohexane: Effect of Propane Dilution	
10	Methylcyclohexane Dehydrogenation in a Wall Catalyst	
17.	Methylcyclohexane Dehydrogenation in a Wall Catalyst	
12	Methylcyclohexane Denydrogenation in a Wall Catalyst 42	
13	Outer Wall Temperatures During Methylcyclohexane Dehydrogenation in a Wall Cacalyst	
14	Outer Wall Temperatures During Activation of Wall Catalyst $h_{\ell}$	
15	Calculated Temperature of Catalyst Layer	
ī€	ESSTR Reactor Section: 0.277-In. ID x 2-Ft Length	
17	FSSTR: Dehydrogenation of Decelin in 2-Ft Reactor, Initial Activity of Three Catalyst Formulations	
18	FSSTR: Dehydrogenation of Decalin Over HOP-R8 in 2-Ft Reactor, Decalin Conversion, Scries (0018-194	

- j x -

ILLUSTRATIONS (Contd)

Figur	efollowing	page
19	FSSTR: Dehy/rogenation of Decalin Over UOP-R8 in 2-Ft Reactor, Fluid Temperatures, Series 10018-194	46
20	FSSTR: Dehydrogenation of Decalin Over Shell 10280-113 in 2-F: Reactor, Decalin Conversion, Series 10018-198	47
21	FSSTR: Dehydrogenation of Decalin Over Shell 10280-113 in 2-Ft Reactor, Fluid Temperatures, Series 10018-198	47
22	FSSTR: Dehydrogenation of Decalin Over Shell 10860-146 in 2-Ft Reactor, Decalin Conversion, Series 11644-6	48
23	FSSTR: Dehydrogenation of Decalin Over Shell 10860-146 in 2-Ft Reactor, Fluid Temperatures, Series 11644-6	48
24	Beta-Ray Backscatter Instrument for Measuring Coke Deposits	51
25	Deposit Frofile: Sample Strip No. 2 From Boeing Aircraft Company	53
26	Deposit Profile: Sample Strip No. 3	53
27	Deposit Profile: Sample Strip No. 4	53
28	Deposit Profile: Sample Strip No. 5	53
29	Comparison of Methods of Rating Erdco JFTOT Tubes	58
30	Comparison of Methods of Rating Erdco JFTOT Tubes	<b>5</b> 8
31	Beta-Ray Backscatter Ratings vs Erdco Reflectance Ratings	58
32	Tuberator Ratings vs Beta-Ray Backscatter Ratings of Erdco JFTOT Tubes	58
33	Comparison of Methods of Rating Alcor JFTOT Tubes	62
34	Comparison of Methods of Rating Alcor JFTOT Tubes	62
35	Correlation of Beta-Ray Backscatter Ratings With Those From the Erdco Reflectance Rater	62
36	Correlation of Beta-Ray Backscatter Ratings With ASTM Tuberator Ratings of Alcor JFTOT Tubes by Shell and CRC Exchange Group Raters	62
37	Effect of Exposure of SHELLDYNE-H <sup><math>(P)</math></sup> Fuel (F-157) to Different Elastomers	<b>7</b> 8
38	Melting Points of Metnylcyclohexane-Propane Mixtures	82

AFAPL-TR-70-71

.

# ILLUSTRATIONS (Contd)

Figu	re	g page
39	Melting Points of Methylcyclohexane-Propane Mixtures	82
<b>4</b> 0	Surface Tension of SHELLDYNE-H® Fuel	83
41	System for Preparation of Gaseous Combustion Mixtures Containing Naphthalene	86
42	Combustion of Decalin Using Constant Attenuation	86
43	Combustion of Decalin Using Measured Attenuation	86
44	Combustion of Decalin Using Attenuation Correlation	86
45	Correlation of Shock Wave Velocities at Two Points in Tube	88
46	Measured Attenuation Coefficients	88
47	Decalin Data Fitted to Logarithmic Equation	88
48	Decalin Data Fitted to Exponential Equation With Equal Weighting (Constant Variance)	89
49	Decalin Data Fitted to Exponential Equation With Changing Varianco	89
50	Probability Plot of Tetralin Data for Logarithmic Equation	91
51	Probability Plot of Tetralin Data for Exponential Equation	91
52	Correlations of Ignition Delay Times	91
53	Ignition Delay Times of n-Octane	91
54	Ignition Delay Times of Methylcyclohexane	91
55	Ignition Delay Times of Toluene	91
56	Ignition Delay Times of Toluene-Hydrogen Mixtures	91
57	Ignition Delay Times of Methylcyclohexane-Toluene-Hydrogen Mixtures	91
58	Ignition Delay Times of Dimethanodecalin	91
59	Ignition Delay Times of SHELLDYNE-H <sup>®</sup> Fuel	91
60	Ignition Delay Times of SHELLDYNE <sup>®</sup> Fuel $\ldots$ $\ldots$ $\ldots$	91
61	Ignition Delay Times of Decalin	91

ArAPL-TR-70-71

۲

.

-xi-

.

# ILLUSTRATIONS (Cor. 'd)

**\$** 

۱.

Figue	efollowing	page
62	Ignition Delay Times of Tetralin	91
63	Rate of Combustion of Tetralin	92
64	Rate of Combustion of Tetralin	92
65	Combustion of Tetralin	93
66	Secondary Furnace Liner for Pulse Reactor	100
67	Schematic Diagram of Pulse Reactor	100
68	Pulse Reactor System	100

## TABLES

<u>Table</u>		Page
1	Dehydrogenation of Decalin Over Various Catalysts: First Series	g 8
2	Dehydrogenation of Decalin Over Various Catalysts: Second Series	g 8
3	Dehydrogenation of Methylcyclohexane: Summary of Catalyst Stability Test Data at Termination of Tests	10
4	Thermal Reaction of Mesitylene: Pulse Reactor	13
5	Thermal Reaction of Bicyclo(2,2,2)octane: Pulse Reactor following	g 13
6	Comparison of Thermal Reaction Rates of Bicyclo(2,2,1)heptane and Bicyclo(2,2,2)octane: Pulse Reactor	14
7	Dehydrogenation of Bicyclo(2,2,2)octane Over One Percent Pt on Al <sub>2</sub> O <sub>3</sub> : Pulse Reactor	g 14
8	Comparison of Reactivities of Bicyclo(2,2,2)octane and Bicyclo(2,2,1)heptane: Pulse Reactor	15
9	Dehydrogenation of Bicyclo(2,2,2)octane Over Catalyst 10860-1320: Pulse Reactor	g 15
10	Dehydrogenation of Bicyclo(2,2,2)octane Over Catalyst 10860-141A	17
11	Dehydrogenation of Bicyclo(2,2,2)ociane Over Catalyst 11639-190: Pulse Reactor	g 17
12	Thermal Cracking of n-Dodecane Using Additives: Pulse Reactor	g 18
13	Thermal Cracking of n-Dodecane Using Additives	g 19
14	Dehydrogenation of Propane-Methylcyclohexane Mixture	20
15	Approximate Physical Properties of Various Catalyst Supports	22
16	MICTR Screening of Various Platinum Supported Catalysts of Different Physical Properties	23
17	MICTR Evaluation of Platinized Catalysts With Newer Type 1 Supports	24
18	MICTR Evaluation and Comparison of Various Platinum Promoted Type 6 Supported Catalysts	26

-xiii-

AFAPL-TR-70-71

В

TABLES (Contd)

T <b>a</b> ble	P	age
19	Effect of Metal G on Performance of Platinum/Type 1 Supported Catalysts With Methylcyclohexanc in MICTR	27
20	Relative Activities of Catalysts Prepared From Spherical Type 1 Support With Methylcyclohexane in MICTR	28
21	MICTR Evaluation of Platinum/Type 1 Supports Containing Several Concentrations of Various Metals Intended to Improve Activity- Stability With Endothermic Fuels	29
22	MICTR Evaluation and Metal Adhesive Properties of Various Wall Coating Candidates	30
23	MICTR Evaluation and Metal Adhesive Properties of Various Preplatinized Wall Coating Candidates	30
24	MICTR Evaluation and Metal Adhesive Properties of Various Platinized Wall Coating Candidates	31
25	FSSTR: Dehydrogenation of Decalin in 2-Ft Reactor; Summary of Tests With Three Catalysts Formulations	45
26	FSSTR: Dehydrogenation of Decalin Over UOP-R8 in 2-Ft Reactor, Data Summary Series 10018-194	45
27	FSSTR: Dehydrogenation of Decalin Over UOP-R8 in 2-Ft Reactor, Product Analyses for Series 10018-194	46
28	FSSTR: Dehydrogenation of Decalin Over Shell 10280-113 in 2-Ft Reactor, Data Summary Series 10018-198 following	<b>4</b> 6
29	FSSTR: Dehydrogenation of Decalin Over Shell 10280-113 in 2-Ft Reactor, Product Analyses for Series 10018-198	47
30	FSSTR: Dehydrogenation of Decalin Over Shell 10860-146 in 2-Ft Reactor, Data Summary Series 11644-6 following	47
31	FSSTR: Dehydrogenation of Decalin Over Shell 10860-146 in 2-Ft Reactor, Product Analyses for Series 11644-6	48
32	Dehydrogenation of Decalin Over Various Catalysts	49
33	Activities of Catalysts 10280-113 and 10860-146 for Methylcyclo- hexane Dehydrogenation at Several Temperatures	49
34	Comparison of ASTM Code and Beta-Ray Backscatter Ratings for Alcor JFTOT Stainless Steel and SD/M-7 Coker Tubes	54
35	Comparison of Beta-Ray Backscatter Ratings of CRC Erdco JFTOT Tubes With Those From the ASTM Tuberator and the Erdco Reflectance Rater	56

-xiv-

# TABLES (Contd)

<u>Table</u>	·	P€	age
36	Summary Comparison of Other Rating Methods With Beta-Ray Backscatter Method for CRC Erdco JFTOT Tubes	•	59
37	Comparison of Beta-Ray Backscatter Ratings of CRC Alcor JFTOT Tubes With Those From the ASTM Tuberator and the Erdco and Alcor Mark V Raters	•	<b>6</b> 0
38	Summary Comparison of Other Rating Methods With Beta-Ray Backscatter Method for CRC Alcor JFTOT Tubes	•	62
39	Estimated Errors in Beta-Ray Backscatter Measurements of Deposit Thicknesses Due to Noncarbon Elements	•	63
40	Description of Jet Fuel RAF-159-60	•	66
41	Effectiveness of Additives in Improving the Coker katings of F-68 Jet Fuel	•	67
42	Effect of Additive A on SD/M-7 Coker Ratings of Methylcyclohexane	•	67
43	Effects of Additive A and Copper Undecylenate on the Thermal Stability of Decalin	•	68
44	Effect of Additive A on Alcor JFTOT Fuel Test Ratings of F-187 Turbine Fuel	•	70
45	SD/M-7 Coker Ratings of Hydrocrackate Jet Fuels	¢	72
46	Composition of Tube Metals for after	•	74
47	Comperison of Ratings by the JFTOT and ASTM Methods	٠	<b>7</b> 6
48	Storage Stability of SHELLDYNE-H $^{\otimes}$ Fuel	ing	77
49	Microgum and Refractive Index Analyses of SHELLDYNE-H $^{\oplus}$ Fuel Samples Soaked With Elastomers for a Period of Six Months $\ldots$ .	•	78
50	Surface Tension of SHELLDYNE-H <sup>60</sup> Fuel	•	83
51	Summary of Conditions of Shock Tube Runs	•	85
52	Errors of Different Equations of Decalin Ignition Delay Times	•	88
5 <b>3</b>	Methods of Ignition Delay Data Correlation	•	89
54	Correlations of Ignition Delay Times	•	91

AFAPL-TR-70-71

ę

\$

.

- + 44 F.

-xv-

• •

# TABLES (Contd)

Table	<u>P</u>	age
55	Dehydrogenation of Decalin Over Various Catalysts	99
56	Dehydrogenation of Methylcyclohexane Over Various Catalysts	99
5 <b>7</b>	Dehydrogenation of Methylcyclohexane With Various Catalysts in MICTR	.02
58	Dehydrogenation of Methylcyclohexane With Various Catalysts in MICTR	.02
59	Ignition Delay Times for Decalin-Oxygen-Argon Mixtures	.13
60	Ignition Delay Times for Tetralin-Oxygen Argon Mixtures	.14
61	Ignition Delay Times for Naphthalene-Oxygen-Argon Mixtures 1	.16
62	Physical Properties of Methylcyclohexane/Ethane Mixtures	17
63	Physical Properties of Methylcyclohexane/Propane Mixtures 1	19
64	Physical Froperties of Methylcyclohexane/n-Butane Mixtures 1	22
65	Liquid Properties of JP-7 Jet Fuel at Saturation	25
	Gas Properties of JP-7	25

#### HYDROCARBON FUELS FOR ADVANCED SYSTEMS

#### IN""

As vehicles propelled by airbreathing engines are being designed to fly at greater speeds, thermal problems multiply and cooling needs increase. The most critical regions which require cooling are leading edges and engines. Although thermal effects can be somewhat accommodated by improved materials and passive cooling, sustained hypersonic flight in the atmosphere requires a substantial heat sink. Mechanical refrigeration or a noncombustible coclant can be used for cooling, but the fuel remains as the most efficient source of heat sink.

The speed limit on vehicles in the range above Mach 4 will depend on the cooling capacity of the fuel. Fuels such as hydrogen, methane, and ammonia can furnish a heat sink only through sensible heating and vaporization, whereas hydrocarbon fuels can provide additional cooling through endothermic reactions. Hydrocarbons can undergo both thermal (noncatalytic) and catalytic endothermic reactions. Theoretically the total heat sink of hydrocarbon fuels range from 50 to 112% of the cooling capacity of hydrogen, based on heat sinks normalized by the heat of combustion. Laboratory proven capability has achieved 85% for catalytic reaction accompanied by sensible heating and vaporization, whereas thermal reaction, such as cracking, with heating and vaporization has been limited to 55%.

Catalytic reactions of interest include dehydrogenation, dehydrocyclization, and depolymerization. The most promising type that has been studied is the catalytic dehydrogenation of naphthenes. A typical example of this reaction is the dehydrogenation of methylcyclohexane over a platinum on alumina catalyst. Other promising reactions are the catalytic dehydrogenation of dicyclohexyl and decahydronaphthalene.

Until a few years ago the use of hydrocarbon fuels in advanced engines at speeds greater than Mach 3 was no more than a concept. Studies under contracts AF33(657)-11096 and AF33(615)-3789 investigated the possibility of using hydrocarbon fuels under the severe conditions of hypersonic flight. These programs were directed toward evaluating the capability of these fuels in advanced engines and developing the information necessary for the design of systems for the promising fuels.

The current contract is a continuation of these past programs with emphasis on extending the knowledge and improving the capability and performance of known hydrocarbon fuels. In addition, investigations of new candidate fuels and new techniques for utilizing hydrocarbon fuels are continuing.

Areas of investigation in the current program are the study of endothermic reactions and the determination of properties and characteristics of hydrocarbon fuels. The catalytic and thermal reactions of different fuels are being investigated and the kinetics of reactions with significant heat sinks are being measured and analyzed. Catalysts are being studied with the objective of optimizing the composition and geometry so as to improve catalyst activity, stability, and performance. Properties and characteristics of fuels are being determined experimentally and theoretically. Gas properties can be predicted reliably, whereas liquid properties must be measured. Thermal stability and combustion characteristics of fuels are being determined by experimental work.

-1-

AFAPL-TR-70-71

#### SUMMARY

As hypersonic aircraft and missiles are developed with increasing speed, the need for engine cooling multiplies. The fuel used in an engine is a convenient coolant for this purpose, absorbing heat as sensible heat and latent heat of vaporization. Certain hydrocarbon fuels can furnish additional heat sink in the form of endothermic reactions. Possible endothermic reactions include thermal reactions such as cracking, and catalytic reactions such as dehydrogenation, dehydrocyclization, and depolymerization. Of these, the catalytic dehydrogenation of naphthenes to aromatics is currently the most promising type of reaction. Reactions of this type are very selective and proceed rapidly to achieve high conversion. The total heat sinks for these reactions compare favorably with heat sinks for other fuels.

Over forty-five catalysts were evaluated in bench-scale studies for stability in the dehydrogenation of naphthenes without added hydrogen. Almost all of the catalysts contained platinum either alone or with other metals as bimetallic or trimetallic mixtures; these were mounted on eighteen different supports. In short tests with decalin (30 minutes) at 10 atm pressure, six catalysts showed moderate stability at 1200°F and good stability at lower temperatures. Four of these catalysts contained platinum on four different supports; the other two were bimetallic and trimetallic mixtures. In less severe tests with MCH nine of the catalysts showed moderate stability at high space velocity (LHSV = 100). Based on the tests with decalin it appeared that catalyst stability was affected by catalyst support composition, physical properties of the support, and the catalyst metal content and composition.

Exploratory studies with bicyclo(2,2,2) octane in a pulse reactor showed that this nephthene could be catalytically dehydrogenated to bicyclooctene. Yields were low (ca 26%) and the reaction was quite sensitive to catalyst properties and reaction conditions. With one catalyst bicyclooctene was formed using He carrier gas but not with H<sub>2</sub> carrier; with two other catalysts containing platinum, bicyclooctene was formed with one catalyst but not with the other.

Twenty different additives were tested in the pulse reactor as free radical initiators for enhancing the rate of the thermal cracking reaction. Of the additives tested six gave substantial increases in the rate of cracking of n-dodecane at 1100°F. The most effective additive caused an increase in conversion of about 75%.

Addition of 30% propane to methylcyclohexane enhanced the reactivity of MCH at and below 932°F but reduced reactivity at higher temperatures.

Development of granular and coating catalysts and screening of catalysts for dehydrogenation activity have continued. Many additional granular catalysts have been prepared which consist of one or more metals on various supports or mixtures of several unsupported metals. Development of catalytic coatings for metal surfaces have continued in order to improve ease of application, mechanical properties, adherence to metal surfaces, and catalytic activity. Candidate coatings in platinized granular form have been evaluated in tests in the micro catalyst test reactor. The need for obtaining intrinsic reaction kinetic data in the upsence of heat and mass transfer effects has been discussed. Packed bed reactors have a number of drawbacks and some kind of recycle reactor must be used. A catalytic continuous stirred tank reactor is appropriate and one has been designed to measure the kinetics of the dehydrogenation of methylcyclohexane and decalin.

A mathematical model of the catalytic wall reactor configuration has been developed. The model is one-dimensional in fluid flow, but accounts for radial heat fluxes. Differential equations describing temperature, pressure and conversion are integrated numerically along the reactor. The experimental data on methylcyclohexane dehydrogenation obtained in the Fuel System Simulation Test Rig (FSSTR) are discussed and compared with the model. After slight modification of the kinetic parameters, the model agreed well with the experiments. Approximations and possible modifications of the model are also considered.

Test runs were made on the dehydrogenation of decalin with three different catalysts in the FSSTR. Shell 10860-146 catalyst was far superior in activity and stability to Shell 10280-113 and UOP-R8 catalysts at conversions up to 80% and outlet fuel temperatures up to 1050°F. The two latter patalysts were comparable in initial activity, but Shell 113 catalyst was much less stable than UOP-R8 catalyst at higher temperatures.

A problem in the determination of fuel thermal stability by a heat transfer device such as the ASTM Coker is the uncertainty in evaluating both the amount and the distribution of the deposits on the tube at the conclusion of the test. While it is usually possible to rate deposits by appearance on highly polished aluminum tubes, many other metals such as stainless steels and nickel change color when heated, even in inert environments, and this complicates the visual rating. To solve this problem we have built a beta-ray backscatter instrument which is capable of evaluating deposit thicknesses up to 2500 Å. We have demonstrated that the deposit profiles on coker tubes can be reproducibly determined. The calibration of this instrument on aluminum tubes has been accomplished, and similar calibrations on stainless steels and other metals are in progress. We have measured deposits from several sources, and in many cases the geographical distribution of deposits measured by this instrument is the same as determined by the optical methods of evaluation. However, the relative values vary greatly, and in some cases even the geographical distributions are different. In fact, it appears at this time that the beta-ray backscatter instrument is capable of giving a truer picture of deposit distribution than the optical method.

One recent addition to the battery of equipment being used for evaluation of fuel thermal stability is a special Alcor JFTOT unit, designed for operation up to 1000°F and 1000 psi. Although the equipment has not been operated as yet up to its maximum capability, considerable data have been obtained with it under less severe conditions with excellent results. Good agreement has been found between data obtained with this equipment and those obtained with our standard ASTM Coker. However, some difficulty has been encountered at high temperatures due to a tendency of the thin aluminum tubes to buckle. Attempts to correct this by the use of TEFLON® 0-rings and a conducting grease at the bus-bar clamps were not successful.

AFAPL-TR-70-71

-3-

About 50 compounds have been screened on the SD/M-7 Coker for their possible use as fuel additives in improving high temperature thermal stability. Six of these have been found effective in the liquid temperature range of 675 to  $700^{\circ}$ F, and the best of them increases the thermal stability breakpoint by 125°F liquid or 160°F metal temperature.

A storage stability experiment on high purity MCH, which was initiated about 4-1/2 years ago, has been terminated with the removal of the samples from the hot room and their examination. The samples were stored in one-gallon epoxylined pails with a 2:1 gas/liquid ratio at 130°F, the gas compositions ranging from pure oxygen to pure nitrogen. The remarkable result was that all the samples appeared equivalent to their original condition; no deterioration had occurred that could be ascertained by color change, gum determinations, or thermal stability testing with the SD/M-7 Coker. Some tests have been made for the selection of the most favorable antioxidant for SHELLDYNE-H<sup>®</sup> hydrocarbon fuel. Samples were tested with various commercial inhibitors under accelerated conditions, 200°F in the presence of excess oxygen for 18 and 30 hours. Although the results were not clear because of difficulties with gum determinations, the most effective antioxidant for SHELLDYNE-H<sup>©</sup> fuel appears to be the amine inhibitor, di-sec-butyl p-phenylene diamine.

A new batch of MCH, synthesized by toluene hydrogenation, has been tested in the SD/M-7 Coker, and is now being filtered to meet thermal stability specifications.

Revised physical properties for JP-7 jet fuel were calculated by improved predictive methods. Properties were estimated for saturated gas and liquid at subcritical temperatures and for the gas at supercritical temperatures and different pressures. These revised values are considered to be better than previous properties, especially for the liquid heat capacity and thermal conductivity. The revised properties agree quite well with properties of similar mixtures, and compare favorably with data from heat transfer experiments.

Physical properties were estimated for binary mixtures of methylcyclohexane (MCH) and a low molecular weight hydrocarbon (ethane, propane and n-butane). These were prepared for use in future studies on fuel cooling of advanced engines. Mixture properties were determined from known properties of the individual constituents. Melting and freezing points of MCH-propane mixtures were measured and used to calculate melting point depressions for the desired mixtures.

The surface tension of SHELLDYNE-H $^{\odot}$  hydrocarbon fuel was measured at three temperatures. These results were correlated, and the correlating equation was used to estimate surface tension at higher temperatures.

The supersonic combustion of decalin, tetralin and naphthalene were investigated using the shock tube. Methods of accounting for attenuation were compared, and the best one was found to be the use of a constant value (0.001) for the attenuation coefficient. The ignition delay time correlation was modified by adding the effect of fuel concentration. The data obtained on decalin and tetralin and much of the data on ignition delay times obtained earlier were correlated using the new equation. A statistical analysis showed that the correlations could not be analyzed by standard techniques. The method of fitting the parameters in the equation to

the experimental data was also discussed. In general, it is seen that oxygen has the effect of reducing the ignition delay times, while increases in fuel concentration tend to increase it. The rate of combustion of tetralin was also measured. From limited data it seems that temperature, oxygen, and fuel all tend to increase the rate of combustion. As has been observed before, the activation energy is fairly low.

Pertinent articles and patents that appeared in the literature during the past year were compiled into a bibliography. This survey covered the following subjects: Advanced Fuels, Physical and Chemical Properties of Fuels, Thermal Stability of Fuels, Fuel Contaminants and Additives, Catalysts and Catalytic Reactions, Heat Transfer and Fluid Mechanics, Combustion, and Advanced Engine Development.

#### LABORATORY STUDIES OF CATALYTIC AND THERMAL REACTIONS

The bench-scale studies of candidate endothermic fuels and their catalyst systems that were initiated under the previous contract are being continued. Tests were conducted in apparatus developed under the previous contract.1/2/3)

Extending the work on the stability of catalysts for the dehydrogenation of naphthenes, over forty-five catalysts were evaluated using the dehydrogenation of decalin and of methylcyclohexane as the test reactions in our bench-scale system. The dehydrogenation of bicyclooctane was studied in a pulse reactor as part of our exploratory search for additional endothermic fuels. A number of additional additives were tested as free radical initiators for the thermal cracking of n-dodecane.

#### Bench-Scale Catalyst Stability Studies

Work under the previous contract thowed that there were considerable differences in the stabilities of supported platinum catalysts for the dehydrogenation of naphthene reactions.<sup>2</sup>)<sup>3</sup>) Further, preliminary studies with a few commercial and laboratory catalysts showed that stability was strongly affected by the catalyst pore structure.<sup>3</sup>) As the most efficient catalyst for naphthene dehydrogenation appears to be platinum, it was of interest to expand the study of the stability of this catalyst system. The purpose of the work was to study the variables that influenced stability, with the subsequent eim of optimizing the catalyst composition.

Under the present contract about forty-five catalysts have been evaluated for stability using the dehydrogenations of decalin (DHN) and methylcyclohexane (MCH) as test reactions. Three of these catalysts contained no platinum. The remainder consisted of fourteen different elements combined with platinum as bimetallic or trimetallic mixtures or compounds and mounted on twelve different supports. The variables under study were composition of catalyst support, physical properties of the support, metal composition, metal content, and metal dispersion. A detailed description of the preparation of these catalysts is given in a later section.

The tests were done in our bench-scale laboratory reactor system which was a tubular flow recetor equipped with conventional devices for measuring feed flow rates and for collecting liquid and gas products. The reactor was a stainless steel tube (No. 347, 1/2-in. IPS) 32-in. long and 5/8-in. ID., which was heated by an electric furnace. The catalyst was contained in the annular space between the thermowell and the reactor wall. In order to supply heat rapidly to the catalyst bed, the annular distance between the thermowell and the reactor wall was about 1/16 in., which was about one-pellet dismeter. The catalyst bed was about 4-1/2-in. long and had a volume of 7 ml. Prior to carrying out the experiments, the catalysts were reduced in situ with hydrogen for 30 minutes at 572°F (300°C) and then for one hour at the reaction temperature. The complete apparatus was described in detail in a previous report.<sup>3</sup>

The reactor wall temperature was measured by a thermocouple pressed against the outside reactor wall by the furnace block and located about 1 in. below the top of the catalyst bed. The catalyst bed temperatures were measured by thermocouples contained in the thermowell. The thermocouples were 1 in. apart and the top thermocouple was about 1/2 in. below the top of the catalyst bed (the flow was downward through the catalyst bed). The "effective" catalyst temperature was between the reactor wall temperature and the catalyst bed temperature.

During reaction the catalyst bed temperature (thermocouple measurements) was considerably over than the furnace block temperature due to the endothermic heat of reaction. As the catalyst deactivated the catalyst bed temperature increased and the magnitude of the temperature increase was taken as a measure of catalyst deactivation. Another quantitative indication of catalyst deactivation was the movement of the "cold spot" down the catalyst bed.

Product analyses were done by GLC from which conversions and selectivities were calculated.

#### Dehydrogenation of Decalin

The catalysts were tested at 10 atm pressure and a liquid hourly space velocity (LHSV, volume of feed per volume of catalyst per hour) of 100. Each catalyst was tested initially at 842°F, and then at successively higher temperature (in 90°F increments) through 1202°F or until the catalyst became inactive. The test period was 30 minutes at each temperature. The feed (F-113 DHN) had the following composition:

> 25.0% trans-DHN 74.6% cis-DHN 0.4% tetralin (THN)

The catalysts were tested in two groups, one of which contained catalysts prepared under the previous contract for another purpose, and the other which contained catalysts prepared under this contract. (The preparative methods are described in a later section.)

The principal reaction products were tetralin (THN) and naphthalene (N). With fresh catalyst high selectivities (90-100%) to THN and N were observed. When the catalysts became highly deactivated side reactions occurred that gave products which emerged from the GLC before and after trans-DHN, after cis-DHN, and after N. These products were not identified further, alth ugh those emerging before trans-DHN were assumed to be cracked material (i.e., lighter than DHN). Some cis to trans-DHN isomerization was observed at the lower temperatures (842° and 932°F) with all of the catalysts. The complete data, including product analyses, are shown in Table 55 in the Appendix.

In the first series of runs, fifteen catalysts were tested that had been prepared under the previous contract. These consisted of pure platinum, platinum combined with twelve other metals as bimetallic or trimetallic mixtures, and three different non-platinum bimetallic mixtures. The metals were mounted on four different supports. The pertinent data are summarized in <u>Table 1</u>, which also includes data for a few catalysts tested previously. <u>Figures 1 and 2</u> show activity (i.e., conversion) and deactivation (i.e., increase in catalyst temperature,  $\Delta T_{max}$ °F) as functions of block temperature.

AFAPL-TR-70-71

The most stable catalyst in this series was Shell 157B (10280-157B). This catalyst was a bimetallic platinum mounted on a granular Type 1 support. The stability (Figure 2) was about that of Shell 46 (granular Type 1 support) and 114C granular Type 6 support) but the activity was lower than that of the latter two catalysts (Figure 1). Another catalyst with good to moderate stability was Shell 15A (10280-15A), a trimetallic platinum mounted on a spherical Type 1 support. In fact this catalyst was the most stable of all of the catalysts containing this support that have been tested thus far. UOP-R16E, which is the UOP "high stability" platforming catalyst, did not deactivate at 1112°F, but did show a catalyst temperature increase of 104°F compared to only 36°F for Shell 15A. However, the activity of this latter catalyst was lower than that of UOP-R16E (Figure 1). Thus Shell 15A was more stable but less active than UOP-R16E.

The remainder of the catalysts in this series showed poor stability at the higher temperatures (Table 1, Figure 2).

In a second series of runs eighteen different catalysts prepared under the present contract were tested. These catalysts were pure platinum or platinum plus another element that were mounted on ten different supports. The preparation of these catalysts is described in a later section. The pertinent test data are shown in <u>Table 2</u>.

Of the catalysts tested in this series, Shell 135B and 142B were the most stalle. Thus moderate stabilities with increases in catalyst bed temperatures of 47° and 56°F, respectively, were observed at 1202°F (block) and good stabilities at lower temperatures. The other catalysts showed poor stabilities at 1202°F ranging from complete deactivation to temperature increases of 131 to 227°F (Table 2). Figure 3 shows stability as a function of block temperature for a number of these catalysts. The performance of Shell 135B and 142B was comparable to that of Shell 46 and Shell 114C (Table 2) and these four catalysts together with 157B are the most stable of these tested thus far. Actually Shell 135B and Shell 46 are nearly identical catalysts. They contained the same support and same Pt content, but the method of mounting the platinum on the support was different. One of the catalysts contained chloride and the other was chloride-free. Within the limits of our tests the performance of these catalysts was the same.

Shell 146 (10860-146) was a large batch of catalyst (500g) prepared in same manner as was Shell 46 and is intended for use in the FSSTR. In our tests the performance of this catalyst was similar to that observed with Shell 46, possibly a little more active and a little more stable than the latter, based on the test data at 1112°F (<u>Table 2</u>). Unfortunately it was not possible to complete the test at 1202°F with Shell 146 due to plugging of the liquid receiver with naphthalene. This suggests that Shell 146 was a little more active (i.e., gave higher conversion to naphthalene) than Shell 46 as the receiver did not plug in tests with this latter catalyst.

Activities (as measured by conversions) were different for the various catalysts but in general the more active catalysts were the more stable.

Tables 1 and 2 and Figures 1 through 3 follow

AFAPL-TR-70-71

-3-

Table 1. DEHYDROGENATION OF DECALIN OVER VARIOUS CATALYSTS: FIRST SERIES. Catalyst Stability Tests

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IMSV: 100 Reaction Time: 30 minutes Pressure: 10 atm

: 25.0% trans DHN 74.6% cis DHN 0.4% THN 7 ml Feed: F-115 DHN Feed Composition:

Catalyst Volume:

Catalyst	密	V Convers	sion, tw	at		Increase	in Catal	yst Temper	eture. Ar	+a [10
Number	Bho°F	3°CZO	10006	1.00 L L	-0000	-0010				K2 + 40
	₩ 5	3 266	1 720T	4 2TTT	1202°F	ዕዛደ" ፑ	932°F	1022°F	1112°F	1202°F
10280-46 <sup>4</sup>	41.9	50.0	59.2	67.9	81.2	13	٦L	۶۲	.ic	Ľ
10280-157B	2 <b>9.</b> 8	37.9	47.2	56.3	67.9		8		+	0 í
10280-1190	37.5	47.0	£.6.3	67.0	77.	2 1 1	] [	- 7	10	<u>8</u> 1
10280-124C	25.3	34.0	43.2	53.4	1.0	Ì			<u> </u>	
10280-15A	30.6	38.3	2.8 <del>1</del>	50.3	y y	) \   -		- u u u	₹,	524
AE01-4780	29.3	36.4	0.21		<u>, ,</u>		() u V -		<u>ب</u> ر ال	107
10280-13	30.3	37.0	1		:			104	5.650	1
10280-504	0				•	≠ \ -  (	ŝ	238	281 <sup>0</sup>	ł
0874-1044		- 0		•	•	07T	ð	1	1	8
	2.4.0	6.10	<b>5</b> 2.2	\$	8	18	43	256b)	1	ł
N261-126	29.0	35.2	30.4	1	1	8	157	ocob)		•
9874-186A	29.7	36.3	37.0	,	1	1 6		( q, yo	3	ł
9874-189A	27.3	33.4	23.2	1	1				•	1
9874-187A	27.8	32.0	26.6	ı			ע ר ק		1	ŧ
9874-194B	21.5	25.8	25.6	ı	1			2 (2	1	•
9874-1398	19.2	8.8	20.7	C	1			( 4,	8	,
UOP-RIGE <sup>a</sup> /	31.1	 21	0.05	د ري ري	   ,	) L	T AT	7.46T	8	1
110P-R8 <sup>a</sup> )	10.9%	ILA O	11	1.10	ł	^-	킈.	52	ð	•
(Baller ASAnt				:	1		£ <del>1</del>	160		•
		~• <u>0</u> ,	62.0	7.17	83 <b>.</b> 0	5	Q	4	<u>ب</u>	67
T0500-7/A	30.1	47.2	57.0	64.3	72.8	13	म्।	00	2 6	- 00
9874-1198	39.3	47.1	55.8	65°0	57.4	\_ <del>_</del>	- N	1.4 1.4	222	
									رر <del>+</del>	500

a) Previoualy tested. b) Catalyst completely deactivated.

DEHYDR CENATION OF DECALIN OVER VARIOUS CATALYSTS: SECOND SERIES Table 2.

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Catalyst Stability Tests

Pressure: 10 atm LHSV: 100 Reaction Time: 30 minutes Temperatures: Block

Feed: F-115 Feed Composition: 25.0% trans DHN 74.6% cis DHN 0.4% Tetralin

Catalyst	Support Pore	\$ D	EN Con	vereio	n at,	ju ju	দ্ব	°F of	Catalys	t Bed e	tt, °F
Number	Diameter, A	846	932	1022	गा	1202	842	932	1022	2111	1202
10260-44 <sup>d</sup> )	106	33.5	12.7	51.4	•		-	8	Ð	•	•
10860-132A	176	33.2	39.5	33.7	•	1	2	58	248	1	•
WHET-	Jiq	26.2	31.1	19.4	1	ł	27	ま	182 <sup>8.)</sup>	\$	1
-13#B	176	21.2	34.5	33.4	3	•	H	56	241	ŧ	ı
10860-1354	<b>9</b>	34.8	42.6	52.2	6.49	72.0	16	6	27	31	131
-1320	्म	35.5	43.7	53.5	61.7	68.8	14	50	25	94	(B_
10280-114B	1	33.1	6.44	53.2	60.6	64.1	14	16	1 <del>4</del> 3	108	ı
10860-1328	176	33.5	44.0	55.5	66.6	69.2	ŝ	6	32	95	(dr25
10860-1388	106	37.2	45.0	55.2	65.9	32.2	53	ଝ	ĸ	79	B.
-1350	20	37.2	48.0	55.2	63.7	73.2	п	16	٢	53	142
-1340	65	36.1	46.7	51.1	62.6	71.2	~	4	Ц	53	146
-1428	28	41.4	50.7	56.3	71.2	77.6	Ś	<del></del>	ź	36	56
-1378	20	29.7	39.6	48.5	56.0	ŧ	13	23	141	67	1
10280-46	2	41.9	50.0	59.2	61.9	81.2	13	14	16	54	65
1.020-1398	i	30.3	40.0	1.7	59.5	52.2	18	53	56	139	(в.
-143B	ł	28.9	33.6	20.4	•	1	47	160	(B_	8	1
10860-146	Ş	43.7	53.8	62.2	71.2	84.1 <sup>c)</sup>	ñ	5	13	18	() ()
-147	ı	30.0	36.7	43.2	44.2	ı	5	ଷ୍ପ	9	135	1
-1358	Q.	4.31	53.7	62.6	70.0	6.17	9	5	6	12	47
-1634	•	39.3	44.7	54.3	63.7	45.5	~	ц	37	84	290
a) Catalyst b) Catalyst c) Reaction d) Standard	completely de completely de time 10 minut Catalyst	activa activa es.	ted af ted at	ter 10. end 0:	15 mil	outes at	this	tempe	rature.		



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In earlier work it was shown that stability varied greatly with the catalyst supports.<sup>3)</sup> Further it appeared that the best supports were those with smaller pore diameters. <u>Figure 4</u> shows the increase in catalyst bed temperature ( $\Delta T_{max}$ ) as a function of average pore diameter of the support for catalysts with similar Pt contents and supports at block temperatures of 1022° and 1202°F. Indeed those catalysts with smaller pores appear to be more stable.

#### Dehydrogenation of Methylcyclohexane

The MCH-catalyst system was considerably more stable than the decalin system. Thus with our standard 1% Pt on Al<sub>2</sub>O<sub>3</sub> catalyst good stability was observed with MCH at 10  $\varepsilon$  tm pressure but not at 1 atm<sup>4</sup>), while with DHN good stability was observed at 30 atm but not at 10 atm pressure.<sup>1</sup>) Consequently in these studies with MCH, stability tests were made at 1 atm pressure. Each catalyst was tested at a single temperature, 852°F, in a series of successive runs with increasing space velocities of 5, 15, 30, 50, 80 and 100 LHSV. The reaction time at each LHSV was 30 minutes. The test was terminated if the catalyst became if active before reaching LHSV of 100. Fourteen catalysts were evaluated in these tests, four of which had shown good stability with DHN. The complete data are presented in <u>Table 3</u>.

Catalyst deactivation was measured by the magnitude of the increase in catalyst bed temperature during the 30 minute test period. Conversions and catalyst bed temperatures increases at the highest LHSV are shown in <u>Table 3</u>, which also includes two catalysts for comparison that were tested previously (UOP-R8 and UOP-R16E). Conversion as a function of space velocity are shown in <u>Figure 5</u> for 1% Pt and in <u>Figure 6</u> for 4% Pt catalysts.

Of the catalysts tested during this period all but five showed little or no catalyst deactivation at the highest space velocity (catalyst bed temperature change was 0  $\pm$  2, <u>Table 3</u>). This group included platinum mounted on both commercial and Shell prepared supports. Our test was not severe enough or of sufficient duration to differentiate between these ten catalysts; thus, all of them must be considered to have comparable good stability for MCH dehydrogenation. Commercial UOP-R8 appeared to be the least stable, but stability was improved greatly by UOP by adding a "metal activator" (i.e., UOP-R16E).

Activities varied greatly between catalysts, and at LHSV of 100 the most active catalysts were commercial Girdler T-309C and 10860-171E. Six of these catalysts have been tested with decalin. Further testing of the others with this naphthene are plunned, as decalin gives a more severe test of catalyst stability.

In summery, we have evaluated many catalysts for stability for the dehydrogenation of naphthenes without added hydrogen. Almost all of the catalysts contained platinum, either alone or with other metals as bimetallic or trimetallic mixtures, mounted on various ""pports. In the tests with decalin six of the laboratory prepared catalysts showed moderate stability at 1202°F and good stability at lower temperatures. All of these catalysts contained platinum; one was a bimetallic and one was a trimetallic mixture mounted on four different supports. With the less severe tests with MCH nine of the catalysts showed moderate stability at high space velocity. These catalysts will be evaluated further with decalin.

#### DEHYDROGENATION OF METHYLCYCLOHEXANE: SUMMARY OF Table 3 CATALYST STABILITY TEST DATA AT TERMINATION OF TESTS

	<del></del>		
Catalyst Number	LHSV	MCH Conversion, fou	Catalyst Bed Temperature Increase, °F
10280-44 <sup>a</sup> )d)	80	19.8	76
10280-108	100	31.6	+2
10860-1320 <sup>d)</sup>	100	30.9	0
10860-135A <sup>d)</sup>	100	32.5	-2
10860-1378 <sup>d)</sup>	100	16.1	27
10860-1428 <sup>d)</sup>	100	37.3	-2
10860-170A	100	33.3	+2
10860-170B	80	10.7	180
10860-1714	50	10.8	_b)
10860 <b>-</b> 1718	100	34.9	+2
10860-1710	100	33.2	0
10860 <b>-</b> 171D	100	34.8	0
10860-171E	100	41.4	+2
10860-196	80	2.9	<sup>29</sup> b)
UOF-R8 <sup>c</sup> )d)	30	15.3	168 <sup>b)</sup>
UOP-R16 <sup>c)d)</sup>	100	18.5	31 <sup>e)</sup>
Girdler T-309C <sup>c)</sup>	100	44.4	0

Pressure: 1 atm Block Temperature: 842°F

a) Standard Catalyst 1% Pt on Al<sub>2</sub>O<sub>3</sub>.

b) Catalyst almost completely deactivated at the end of the test.

c) Commercial catalyst.
d) Also tested with decalin.
e) Cold spot moved down the catalyst bed.

Figures 4, 5 and 6 follow





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Based on the tests with decalin it appeared that stability was affected by catalyst support composition, physical properties of the catalyst, the catalyst metal content, and composition.

This study is continuing with other supports and metal compositions. Longer tests are being considered in which the catalyst will be run for 50-100 hours at constant reaction conditions.

### Dehydrogenation of Bicyclo(2,2,2) octane

Bicyclo(2,2,2)octane (BCO) is a monocyclic naphthene with a C-C bridge across the 1,4-position. In principle it can be dehydrogenated to yield three molecules of hydrogen according to the reaction:



The endothermic heat of this reaction is about 1200 Btu/1b for the first step and possibly 1800 Btu/1b for both steps. Another possible reaction of BCO involves small ring formation, thus:



The total endothermic heat for reaction I is estimated at about 2300 Btu/lb and for II over 4000 Btu/lb. Thus BCO is potentially a very attractive fuel, even though it may be difficult to carry out the reaction beyond the first steps.

An exploratory study of the dehydrogenation of BCO was initiated using our pulse reactor. In this system a carrier gas such as helium or hydrogen flowed through the reactor. A small amount of liquid feed (i.e., 1 µl) was injected into the gas stream and was carried through the reactor as a "pulse". The exit gas was led directly into a GLC for analysis. The reactor was a 1/4" 0.D. stainless steel tube (type 304) five inches long and was heated by an electric furnace. This reactor system is described in detail in the Appendix. GLC analyses were made with an F and M Model 5754 chromatograph using a hydrogen flame detector with a 160' capillary column 0.010" I.D. coated with SF96.

BCO was tested under conditions of both thermal and catalytic reaction at 10 atm pressure. One microliter of liquid feed was injected per pulse. BCO melts at 334°F. Hence, it was dissolved in a solvent in order to inject it into the reactor. Mesitylene (1,3,5-trimethylbenzene, TMB) and n-hexane were used as solvents. Neither one was particularly satisfactory; both were catalytically

reactive and gave reaction products similar to those obtained with BCO. The solutility of BCO in the above solvents was limited to about 30% in IMB and 40% in n-hexane.

Product identification was based on GLC emergence times obtained with pure compounds. With BCC numerous side reactions are possible such as cracking and dealkylation leading to the formation of benzene, toluene, ethyl benzene, pxylene, and their corresponding naphthenes.

### Thermal Reaction

The thermal reaction was studied over the temperature range of 572° to 1202°F with both helium and hydrogen carrier gas. Apparent contact times (ACT) were 4.2 to 0.2 seconds. (Apparent contact times were calculated from the carrier gas flow rates and the void volume in the reactor tube. The void volume was assumed to be one-helf of the bulk volume of the quartz clips.) The reactor tube was filled with quartz chips having a bulk volume of 2.1 ml. Feed was 25% BCO in mesitylene (TMB). Pure TMB was tested separately. The complete data for TMB are shown in <u>Table 4</u>.

With He carrier gas TMB was reasonably stable, and at contact times of 4.0 and 3.7 seconds, conversions of only 8.0% and 2.2% were observed at  $1202^{\circ}F$  and  $1112^{\circ}F$ , respectively (Table 4). Lower conversions were observed at shorter ACT. Based on first order kinetics (see Appendix) the apparent activation energy was 37.1 k. cal/mole (1202° to 1112°F; ACT = 3.7 sec). Figure 7 is an Arrnehius plot of the data.

With H<sub>2</sub> carrier TMB was considerably more reactive than with He at  $1202^{\circ}$ F but not at 1112°F. Also the apparent activation energy was higher with H<sub>2</sub> carrier. For example with H<sub>2</sub> and an ACT of 3.7 seconds, 24.5% conversion was observed at 1202°F and only 2.4 at 1112°F, which corresponded to an apparent activation energy of 79.2 kcal/mole (Figure 7).

Products and product distributions appeared to be similar with both carrier gases, based on GLC emergence times. With this hydrocarbon the principal reaction product appeared to be ethyl benzene (peak no. 4, <u>Table 4</u>).

BCO was more reactive thermally than TMB by about a factor of 3 to  $4_{,}$  based on first order rate constants. Further, as was observed with TMB, BCO was more reactive with  $H_2$  than He at 1202°F but not at 1112°F. The complete data are given in <u>Table 5</u>, which shows the values for the product analyses in parentheses and the calculated values on a TMB-free basis. These calculated values were also corrected for any contribution to the various components by thermal reaction of TMB, using the data of <u>Table 4</u>. A typical correction calculation is shown in the Appendix. With BCO the lower molecular weight hydrocarbons were the principal reaction products (possibly benzene or toluene).

Activation energies with BCO were 38.3 kcal/mole and 74.2 kcal/mole with He and H<sub>2</sub>, respectively. The data are shown in <u>Figure 7</u>.

Table 4. THERMAL REACTION OF MESITVLENE: PULSE REACTOR

Pressure: 10 atm Pulse Volume: 1 µl Reactor Filled With Guartz Chips.

100 0.19 104-3 2.9 ŧ 105-1 200 67 0.31 1202 10.0 ຜ່ 105-2 J°7 24.5 50 8 107-4 8 0.93 ŝ Нг 1 107-3 0.74 4.0 .ೆ. ಬ 50 107-2 1022 20 4. 5 8.2 ŧ 106-2 1000 c.19 0.1 ı 106-1 ર્જુ 0.33 0,8 1208 1 105-3 2,13 8.0 3.7 20 \$ 108-1 \*-- 2111-28 66´•0 1,0 1 \_\_\_\_\_ 107-5 0.67 000400<u>00000</u>000 ŝ ù.0 ດ ດີ 107-1 88 600 Ť 0.7 ł -1023 186-3 0.6 <u>с</u> د، 1 ł Conversion, Ma Block Temperature, °F Carrier Gas Flow Rate, cc/min Product Analysis First Grder Rete Constant, sec"<sup>1</sup>(x 10<sup>3</sup>) RID ILOSSfeak No. (IMB) ACT, seconds Carrier Gas E E

-1]-

AFAPL-TR-70-71

Table 5 and Figure 7 follow

• •

Table 5. THERMAL REACTION OF BICYCLO(2,2,2) OCTANE: PUISE REACTOR

Pressure: 10 atm Reactor Filled with Quartz Chips Pulse Volume: 1 µl Feed: 23% BCO in Mesitylene

Run No. 11623-	<del>.</del> 66	2 <b>-</b> 66	100-2	100-3	100-4	102-1	100-2	100-3	1001	1	2. Bo	1.000						
Block Temperature, "F	Ĭ			-1112-	1		- छहा 		1		- ]08 - 108 - 108		(-co)	1-101	6- TO T	S	103-2	104-2
Carrier Cas		2			He			-			-			-			-221 	1
Carrier Ges Flow Mate. cc/ain	20	150	50	50	<b>20</b>	50	S	60	1000	3	8	500	- 05		5			1
ACT, seconds	4.2	7.	0. +	6; •0	0.33	3.7	16.0	0.31	0.19	4.2	FL-0	8				2 6		
Froduct Analysis, www. Peak Mc. ]	0.4 (0.1)	0.0) (0.0)	1.2 (0.4)	0.0)	0.0 (0.0)	(2.4)	2.2	0.4	0.0	1.3	0.6	4	0.0	4.0		20.7	8.0	6 6 °C
¢,	1.2 (č.3)	0.1) (0.1)		(0.1) (0.1)	0.0 0.0	12.3 (3.0)	2.7	0.9	0.0	5.6	1.0			1.7	0.1)	(7.2) 25.0	(1.1) 6.1	(0.1) 1.3
F \	4.0 (1.0)	၀ ၀ ၀ ၀ ၀	0.0)	0.0) (0.0)	0.0)	0.9 (0.6)	0.0	4.0	0.0)	4.0	1.0	100	0.0	(† - ) († - ) ()	0.0	0.0	0.0	(r
¥	0. <b>k</b> (0.1)	0.1)	0.4	0.0) (0.0)	0.0) (0.0)	0.9 (0.6)	0.0)	0.0)	0.0)	0.0	0.8	(r.o)	( ) ( )	(T-0)	(0.0) (- 0)	(6.0)	(2.0) (2.0)	(0.5) (0.5)
E) P)	0.0 0.0	0.0) (0.0)	0.0)	(0.0) (0.0)	0.0) (0.0)	0.0)	0 0.4 (0.8)	0.0) (0.0)	0.0) (0.0)	0.0) (0.0)	(0.0)	0.0)	0.0	0.0		0.0	0.0	
ŧ	3.1 (0.9)	0.8 (0.3)	3.3 (2.0)	0.0) (0.0)	0.4 (1.0)	0.4 (5.3)	0.0	0.0)	0.9 (0.2)	0.2) (0.4)	0.8)	0.1	3.0	0.0	1.4	0.0	0.02	6.0 6.0
BCC	94.5 (24.2)	98.4 (23.9)	91.7 (22.2)	99.6 (23.0)	99.6 (23.2)	76.2 (17.9)	94.2 (21.3)	98.3 (22.6)	99.1 (22.8)	95.8 (22.7)	95-9 (23-3)	98.4 (23.1)	93.69 (21.8)	0.76	97.4 2	(0.0) 28.9	(+ · · · · · · · · · · · · · · · · · · ·	6
n v	(0.1)	(6.0)	(0.1)	(0.2)	(0.2)	(1.0)	(0.2)	(0.2)	(0.0)	(c.)	([.0)	(c.o)	(0.0)	(0.0)	(0.2)	(0.2)	(0.2)	(1.0) (1.1.5)
, <b>64</b>	(277.3)	(1.27)	(0.0) (73.8)	(12.1)	(16.2)	(0.0)	(0-0) (75-3)	(0.0)	(0.0) (0.0)	(0.2) (75.0)	(0.2) (75 8)	(0.2) (75 7)	(0.0)	(0.0)	(0.0)	(0.0)	(0.0)	(0.0)
æ	(0.2)	(0.0)	(1.0)	(0.2)	(0.0)	(1.0)	(0.2)	(0.0)	(0.0)	(0.1)	(0.2)	(1.0)	(2.0)	((1.0)	(4-91) (0-0)	(0.0)	(9.22)	(75.0)
6 OI	(4.0)	(0.2)	(0.3)	(1.0)	(0.2)	(6.3)	(4.0)	(1.0)	(0.0)	(0.3)	(0.0)	(0.4)	(1.0)	(6.3)	(0.1)	(0.2)	(0.2)	(0.0)
BCO Canversian, 🚧	5.5	1.6	8.3	4.0	4.0	23.8	5.8	1.7	6.0	(T•0)	(0.1)	(1.0)	(0.1) 4 3	(0.1)	(0.0)	(0,1)	(0.1)	(0.0)
First Order Rate Constant, sec <sup>-1</sup> (x 10 <sup>3</sup> )	'	,	5' <b>J</b> 3	,	•	9.15	4	1	•	ı	•	2 1	2.13			21.40	10.0	<u>ه.</u> '



AFAPL-TR-70-71 66758

The enhanced reactivities of TMB and BCO with H<sub>2</sub> carrier gas could have been due to a hydrocracking-type reaction that occurred at a significant rate at 1202°F but not at lower temperatures. This would explain the higher activation energies with H<sub>2</sub> carrier, as the rate of disappearance of starting material at the higher temperature with H<sub>2</sub> would be due to two (main) reactions, while with He only one reaction occurred (i.e., thermal cracking). This effect of H<sub>2</sub> carrier om naphthene reactivity was observed earlier in work with dimethanodecalin and bicycloheptane.<sup>3</sup>) Based on first order rate constants the reactivity of BCO was less than that of bicycloheptane (BCH) by about a factor of 1.7 to 2.4 (Table 6).

Table 6.	COMPARISON OF	THERMAL	REACTION	RATES OF
BICYCLO(	2,2,1)HEPTANE A	ND BICY	CLO(2,2,2)	OCTANE:
گ و	PULSE	REACTOR		

	AUT = 4.0 00 0.1	seconds	rressure.	TO COM.	
	0 i	First	Order Rate	Constant,	sec <sup>-1</sup> x 10 <sup>3</sup>
°F	<u>Gas</u>		BCO	BCHa	)
1112	He		2.8	4.8	
	Hg		2.1	5.0	
1202	Не		9.2	22.0	
	H2		21.4	37.0	

a) Table 48, pg. 107, reference 3.

### Catalytic Denydrogenation

Dehydrogenation of BCO was studied over the temperature range of 572° to 1022°F with both He and H<sub>2</sub> carrier gas. Two feedstocks were used, namely, BCO dissolved in TMB and in n-hexane. Four laboratory catalysts were tested which were 1% Pt on Al<sub>2</sub>O<sub>3</sub> (standard catalyst), 10860-132C, 10860-141A, and 11637-190. In these studies 0.25 mJ of catalyst was diluted with 1.0 mJ quartz chips. One ul of liquid feed was injected per pulse.

Two of the catalysts were tested with the BCO in TMB feed (about 30% BCO) at 572-752°F using He carrier gas. These were our standard catalyst and 10860-132C; 132C was a bimetallic platinum catalyst. Liquid hourly space velocities varied from 172 to 686 based on total liquid fed or from 52 to 206 based on BCO fed.

With the standard catalyst (1% Pt), -1.5 BCO conversion was observed at the highest temperature and lowest space velocit. (<u>Table 7</u>). Lower conversions were observed at higher space velocities and lower temperatures. This is shown in <u>Figure 8</u> which shows conversion as a function of space velocity for each test

Table 7 and Figure 8 follow

AFAFL-TR-70-71

-14-

Table 7. DEHYDROGENATION OF BICYCLO(2,2,2)OCTANE OVER 1% Pt ON A1203: PUISE REACTOR

Pressure: 10 atmCatalyst No: 10280-44Carrier Gas: HeFeed: 27-30% BC0 inCatalyst Volume: 0.25 mlMesityleneCatalyst Wt: 0.2349gPulse Volume: 1 µlCatalyst Diluted With 1.0 ml Quartz Chips.

Rum 11627 -	1-56	91-5	<b>8</b> -1	₹ %	<u>%</u> -5	1-26	93-2	5-3	4-66	94-2	ŗ-;ŧ	95-2	95-3	96-J
Carrier Flow Rate, co/min	150	300	8	150	300	600	150	300	600	300	ŝ	150	8	8
Total LKSV	2	343	696 969	22	343	88	172	343	686	343	686	2.[	545	686
BCO LHSV	т. т.	8	27	<del>1</del> 45	88	172	ł13	8	22	<u>,</u> %	172	1-3	<u>,</u> %	2
Block Temperature, Fr		- 572			- 617	1		-  - - - (96	1		L01		752	
Froduct Anelysis, % (TWB-free) <sup>2)</sup>											-		<u> </u>	1
(q <sup>-1</sup> 2	(0.1)	3.2 (6.0)	1.9 (0.5)	4.7 (1.4)	3.6 (1.0)	2.6 (0.7)	7.7 (2.4)	5.8 (1.7)	3.8 (1.2)	5.9 (1,7)	4.0 (1.1)	16.5 (6.3)	10.5 (3.2)	9.9 (3.0)
Jenzene	2.6 (0.7)	6.1 (1.7)	(1.1)	7.7 (2.3)	6.0 (1.7)	6.3 (1.7)	12.5 (3.9)	12.7 (3.7)	8.6 (2.7)	13.5 (3.9)	10.3 (2.8)	27.7 (10.5)	22.2 (6.8)	23.6 (7.2)
Toluene	1.9	1.4 (0.1)	0.2) (0.2)	3.3 (0.5)	1.1 (0.3)	1.1 (0.3)	3.5 (1.1)	1.7 (0.5)	1.9	2.1 (0.6)	1.5 (0.4)	8.4 (3.2)	3.6 (1.1)	3.6 (1.1)
Ethyl Benzene plus Bicyclopotene		3.2 (0.8)	1.9 (0.5)	8.0 (2.4)	4.3 (1.2)	3.3 (0.9)	11.2 (3.5)	7.9 (2.3)	4.8 (1.5)	5.6 (1.6)	5.5 (1.5)	9.5 (3.6)	7.2 (2.2)	6.0 (1.8)
H-P-Xylene	17-8 (0-7)	ک.د (0.6)	3.3 (6.0)	11. (3.3)	6.0 (1.7)	4.0 (1.1)	16.0 (5.0)	8.2 (2.4)	5.7 (1.8)	8.5 (2.4)	5.5 (1.5)	26.4 (10.0)	13°4 (1,•1)	11.3 (3.4)
() ;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;	-(î:	غ.د (و.0)	1.5	6.3 (1.9)	4.3 (1.2)	2.6 (0.7)	6.1 (1.9)	4.8 (1.4)	2.9 (0.9)	4.5 (1.3)	2.2 (0.6)	2°4	4 5 (1.3)	2.3 (0.7)
ι Ω	66.5) (18.5)	80.6 (22.4)	86.7 (23.4)	60.7 (18.2)	74.7 (21.0)	80.1 (21.8)	42.9 (13.4)	58.8 (17.1)	72.4 (22.8)	60.1 (17.3)	71.0 (19.3)	9.0 (4.£)	38.9 (11.9)	43.0 (13.0)
Mesitylene	(L. Z.)	(20.5)	(0.67)	(0.07)	(n.8)	(12.8)	(0.89)	(6.07)	(68.3)	(T.2)	(9:21)	(0.39)	(1.69)	(69.8)
BCD Conversion, Su	еч - г 1 М\	19.4	13.3	39.3	25.3	19.9	57.1	2.[4	27.6	39.9	29.0	91.0	61.1	57.0
First Order Rate Constant, sec?	0.168	0.195	0.257	0.234	0.274	0.415	0-413	0.520	0.630	0.517	0.697	1.271	766.0	1.779
<pre># Numbers in parenthesis give tot</pre>	al produc re n-hexa r æ-p-Xylv	t anelysi ne. me.	• v											]



temperature. There was no measurement of catalyst deactivation during these tests; hence, the conversion values are minimal. The complete data are presented in <u>Table 7</u> in the order in which the tests were performed. Product analyses, calculated on a TMB-free basis, are shown in the <u>Table 7</u>, and the complete product analyses are shown in parentheses. The calculated values were obtained assuming no TMB reaction. This will introduce a slight error at the higher temperatures, as, in a separate experiment with pure TMB at 752°F (LHSV = 172, He), it was determined that 1.6% TMB was converted to m-xylene (0.8%), toluene (0.4%), and unidentified products. Thus 1.6% should be the maximum error due to TMB reaction, unless there are synergestic effects when TMB is mixed with BCO.

First order rate constants, calculated from conversion of BCO, increased with increasing space velocity. Presumably this effect was due to changes in pulse shape with varying flow rate of carrier gas. Apparent activation energies were calculated from the rate constants at the same space velocity and were 15 to 18 kcal/mole.

Product material was principally benzene and other alkyl aromatics. No dehydrogenation products with bicyclo structures were observed. This suggests that with this catalyst ring opening at one of the secondary carbon atoms is faster than dehydrogenation of the ring, or that the two reactions occur simultaneously.

Based on the first order rate constants the BCO was more reactive than BCH. A comparison of the reactivities of the two naphthenes is shown below:

Table 8	B. COMPA	RISON OF REA	CTIVITIES OF
	BICYCLO	(2,2,2) OCTAN	EAND
	BICY	CLO(2,2,1)HEP	TANE:
	1	PULSE REACTOR	
			_
Temperature, °F	LHSV	First Order H	Rate Constant, sec-1
		BCO	BCH <sup>a</sup> )
1112	43-57	0.413	0.022
	172-238	0.630	0.070

a) Table 51, pg 113, Reference 3.

Preliminary results indicated that considerably more catalyst poisoning occurred with BCH, probably due t > formation of cyclopentadiene structures.

Catalyst 132C was more active initially than our standard catalyst, but deactivated appreciably with use. For example, initially 47.6% BCO conversion was observed compared to only 31.1% with our standard catalyst (572°F, LHSV = 172). However, at 752°F (after successive tests at lower temperatures) only 58.4% conversion was observed compared to 91% with the standard catalyst. Finally, on returning to the original test conditions (572°F, LHSV = 172) only 10.1% conversion with 132C was observed. Thus, this latter catalyst had very poor stability for BCO dehydrogenation. The complete data are shown in Table 9.

Table 9 follows

AFAPL-TR-70-71

8

-15-

Product distributions were similar to those observed with the standard catalyst, namely benzene and higher aromatic homologues. No component with a bicyclo structure could be definitely identified in the dehydrogenation product.

A series of short tests were made with catalysts 10680-141A (141A) and 11637-190 (190) over the temperature range of  $662 \ 1022^{\circ}F$ . Both catalysts contained the same type of support; 141A was platinum only and 190 was a binary platinum mixture. The feed was 38% BCO in n-hexane. Tests were made at a single LHSV with H= (LHSV = 65) and H<sub>2</sub> (LHSV = 130) carrier gas.

With catalyst 141A BCO was dehydrogenated to bicyclooctene in low yield. Best results were obtained at 932°F with H<sub>2</sub> carrier, where 22% BCO was converted to bicyclooctene. Total BCO conversion was 83.5% which gave a yield of bicyclooctene of 26.4% (<u>Table 10</u>). Lower yields were obtained at lower and higher temperatures and with He carrier gas. The complete data are presented in <u>Table 10</u>.

Besides bicyclooctene the principal reaction products were two unidentified components that emerged after bicyclooctene and before BCO ( $U_2$  and  $U_3$ , <u>Table 10</u>), plus a considerable amount of material that emerged in the  $C_0$  region o. the GLC chromatograph. This latter group could not be successfully resolved by our analysis system but appeared to be hexanes, hexenes, and benzene.

The emergence times of component  $U_2$  was about that of p-xylene, but the emergence time for  $U_3$  did not correspond to any of the benzene homologues. Thus, identification of these components is pending.

BCO conversions were about the same with He and H<sub>2</sub> at 752°F and lower, but at higher temperatures (932°-1022°F) higher conversions and yields of bicyclooctene were observed with H<sub>2</sub>.

Catalyst 190 was less active, less selective for bicyclooctene, and for some reason appeared to be completely poisoned by hydrogen at  $842^{\circ}F$  and lower temperatures. For example, at  $932^{\circ}F$  BCO conversion was 77.5% with He compared to only 5% with H<sub>2</sub>. Further, at  $842^{\circ}F$  there was absolutely no reaction with H<sub>2</sub> carrier, although with the He 30.1% conversion was observed. This is interesting as this catalyst was designed primarily for dehydrocyclization of paraffins (i.e., ring closure), in which H<sub>2</sub> is part of the system. The complete data are presented in Table 11.

In summary, bicyclo(2,2,2) octane was catalytically dehydrogenated to bicyclocctene in low yield in pulse reactor tests. The reaction was quite sensitive to catalyst properties and to reaction conditions. With one catalyst bicyclooctene was formed with helium present but not with hydrogen, and with two catalysts containing platinum bicyclooctene was formed with one but not with the other. Present tests indicate that bridged-ring naphthenes can be dehydrogenated but that it may take a highly sophisticated naphthene-catalyst system to effect dehydrogenation to the corresponding dienes or trienes with good selectivity. The work is continuing and other catalysts and BCO solvents are being considered.

R.T. A.G. LINGA.	5	139-2	1-011	110-3	1-011	£-111	-311	112-4	113-1	5-č11	4-2LI	1-411	114-2	115-2	115+3	1-511	1-911	116-2	117-1
larriar Ply Bate, co/air Marti	0	35	<u>§</u>	150	<u>3</u> 8	<u>8</u>	150	300	<u>8</u>	150	150	360	600	150	300	ŝ	150	38	8
	83	5. 198	88	172 4,	<u>7</u> 8	889 22	년 <u>라</u>	££.88	88 E	51 51 51	17 54	343 86	172 172	172	545 86	172 172	81 12 12	£.%	86년 88년
1、1、1、1、1、1、1、1、1、1、1、1、1、1、1、1、1、1、1、	ļ	- 225 -	1		-617-	1	-   -	- 38	-	\$	-	202	ŧ	-	- 752	1	-	-572	1
Frosuct Analysis, M. (TG-free) <sup>37</sup> 	a)	2.6 (0.6)	1.4 (0.5)	2.7 (0.6)	2.3 (0.5)	1.0	4.7 (1.1)	2.0 (0.4)	1.0	2.2 (0.5)	5.5 (1.3)	1.9 (0.4)	0.5 (c.1)	5.0 (1.0)	2.5 (0.5)	4.0) 7.0)	0.5	0.0	0.0) (0.0)
e tester e	<u></u>	(	2.8) (0.6)	(1.1)	5-1 (1-3)	3.4 (7.0)	14.5 (3.4)	6.0 (1.2)	2.5 (0.5)	7.1 (1.6)	15.5 (3.7)	5.3	2.6 (0.5)	12.6 (2.9)	8.6 (1.7)	3.5 (0.9)	1.5		0.0)
	~	(5.6)	(1.5)	(°°)	(0.1)	(0.0)	0.9 (5.0)	0.5 (0.1)	0.5 (c.1)	0.4 (1.0)	0.8 (0.2)	0.5	(0°0)	0.9 (c.2)	1.0 (0.2)	0.4 (1.0)	0.5 (0.1)	0.0) (0.0)	0.0)
Ethyl Tersene plur Blaycla- uthru		ج. ج. (0.8)	2.4 (0.5)	3.5 (0.9)	5.5 ()	<b>5.4</b> (1.1)	4.2 (1.0)	6.0 (1.2)	6.6 (1.3)	5.4 (1.2)	5.0 (1.2)	7.7 (1.6)	6.7 (1.3)	5.7	8.1 (3.6)	5.4 (1.4)	4.0 (0.8)	2.7 (0.5)	0.0 0.0
2000 (N) + 4 + 4	a:0	6.8 (	2.6) (0.6)	(1.1)	3.7 (0.8)	2.0 (0.4)	6.0 (1.4)	4.0 (0.8)	3.0 (0.6)	4.9 (1.1)	7.6 (1.8)	7.7 (1.6)	4.6 (0.9)	10.4 (2.4)	11.1 (2.2)	5.4 (1.4)	2.5	[:1] (c-2)	1.1.0
G t	(5.5)	6.6 (1.5)	ج.بر) (۲۰۰)	8) (1.8)	5.1 (1.1)	0.1)	6.8 (1.6)	2.0 (0.4)	0.5	4.5 (1.0)	8.4 (2.0)	2.9 (0.6)	(1.0)	6.0 (6.1)	~~ ?`0	0.0	0.5	0.0)	0.0) (0.0)
94 (1) (1) (1) (1) (1)		(11.5) (11.5)	86.7 (18.3) (78.3)	75.2 (17.0) (77.4)	86.2 (17.4) (78.3)	87.3 (17.8) (79.6)	62.9 (14.7) (76.6)	79.6 (16.0) (79.9)	85.9 (17.0) (80.2)	(17.0) (17.0) (77.5)	51.7 (12.3) (71.5)	74.0 (15.4) (79.2)	85.1 (16.6) (80.5)	45.2 (10.4) (79.9)	64.6 (12.8) (80.2)	60.3 (15.8) (90.1)	89.9 (17.9) (75.4)	96.3 ([0.0) (80.7)	98.9 (18.9) (81.0)
al - Januar Car - Ma	1. Ú	6.2	€.+SE	a. 10	19.8	12.7	37.1	4.02	14.1	24-3	48.3	26.0	6.41	58.4	35.4	5.95	10.9	۳. ۲.	1.1
Teller 1 4 total actives and the	1 (J) (J)	en theses					 						1						

in lastrullisti arelised before n-hexane. Husenullisti eks eterret after p-yuhare.

**.** .

Carrier Gas: He Pulse Volume: 1 μl Catalyst Diluted With 1.0 ml Quartz Chips Pressure: 10 atm Feed: 20-25% BCO in Mesitylene Catalyst Volume: 0.25 ml Catalyst Weight: 0.2405 g

### DEHYDR. GFNATION OF BICYCLO(2,2,2) OCTANE OVER CATALYST 10860-132C: PULSE REACTOR Table 9.

AFAPL-TR-70-71 66758

2

### Table 10. DEHYDROGENATION OF BICYCLO (2,2,2)OCTANE OVER CATALYST 10860-141A

Pressure: 10 atm Feed: 38% BCO in n-Hexane Catalyst Volume: 0.25 ml Catalyst Wt: 0.0966g Pulse Volume: 1 µ1 Catalyst Diluted With 1.0 ml Quartz Chips

Run No. 11767-	71-1	71-3	72 <b>-</b> 1	72 <b>-</b> 2	72 <b>-</b> 3	73-1	73-2	74-1	74-2
Carrier Gas	He	Не	H2	He	Ha	He	H2	Не	H2
Carrier Gas Flow Rate cc/min	150	150	;00	150	300	150	300	150	300
LFSV Total BCO	172 65	172 65	343 130	172 65	343 130	172 65	3 <b>43</b> 130	172 65	343 130
Furnace Temp., °F	662	<b>←</b> 75	2	<b></b> 84	2>	<b>4</b> 932		<b>-</b> 10	22>
Product Analysis, fw C <sub>a</sub> and lighter U <sub>1</sub> Bicyclooctene U <sub>2</sub> d) U <sub>3</sub> BCO	71.0 0.0 0.8 1.0 0.7 26.5	71.3 0.6 1.9 1.2 1.4 23.6	71.2 0.0 0.8 2.8 1.7 23.5	80.4 0.5 3.0 1.1 1.7 13.3	71.6 0.8 4.0 7.5 2.5 13.6	86.9 0.6 2.4 <sup>e</sup> ) 0.7 1.0 8.4	75.7 1.5 8.4 6.2 1.9 6.3	87.0 0.4 2.4 0.8 0.9 8.5	85.4 1.7 6.5 3.4 0.8 2.2
BCO Conversion, #w	31.0	38.7	38.8	65.4	64.6	77.9	83.5	77.6	94.2
BCO Converted to BCO <sup>7</sup>	2.0	4.9	2.0	7.8 <sup>f)</sup>	10.4	6.3 <sup>f)</sup>	22.0	6.3 <sup>f)</sup>	17.0
Yield BCO <sup>=</sup>	6.4	12.8	5.1	12.0	16.0	8.0	26.4	8.1	18.0

a) Hexane, hexene, benzene, cyclohexane, and lighter than  $C_{\Theta}$ .

b) Emerged before bicyclooctene - unidentified.
c) Unidentified; emerged after bicyclooctene.
d) Unidentified; emerged after U2.

e) Two unresolved peaks; one was bicyclooctone, the other may have been bicyclooctadiene.

f) Assume this component is all bicyclooctene.

Table 11 follows

### AFAPL-TR-20-21

In this respect it is of interest to test BCO-MCH and BCO-DHN feed systems. Presumably interpretation of product analyses should be simplified as the two pure naphthenes dehydrogenate to toluene and to tetralin and naphthalene over these catalysts. These feeds would have a practical application also as successful dehydrogenation of BCO in DUN in effect would be extending the heat sink range of this latter naphthene.

### Thermal Cracking of n-Dodecane Using Additives

There is considerable interest as to the maximum amount of heat sink that can be obtained with a paratfinic type jet fuel (JP-7). The latent and sensible heat obtainable from this material is about 1000 Btu/1b when heated to 1300°F. An additional 300 Btu/1b could be obtained by thermally cracking the fuel to about 50% conversion. However, under conventional cracking reaction conditions some coke is produced, which is undesirable. Also, the rate at moderate temperatures is too low. Under the previous contract an investigation was initiated as to the possibility of enhancing the rate of thermal reaction with concurrent reduction in coke make, using free radical initiating fuel additives. This work is being continued under the present contract.

The experiments were done in the pulse reactor, which is described in detail in the Appendix. In this system a stream of carrier gas flowed through the reactor continuously. At the desired time a small amount of feed (ca 1 microliter) was injected into the carrier gas stream and subsequently passed over the catalyst as a pulse. Reaction products, or a slip-stream sample thereof, were led directly into a GLC for analysis.

In these experiments the reactor tube was filled with quartz chips (10-20 mesh). Liquid hourly space velocities were calculated based on the bulk volume of the quartz (i.e., volume of the empty tube), and the apparent contact time (ACT) was calculated based on the void volume in the tube (i.e., one-half the volume of the empty tube). This is close to the actual contact time and is different from our calculation of ACT for catalytic beds, which ignores catalyst volume.

The tests were done at 10 atm pressure and LHSV of 60 using n-dodecane as the test fluid. Helium was used as carrier gas and 1 ml of feed (n-dodecane + additive) was injected per pulse.

In the first series of experiments fourteen additives were tested at 1112°F. The feed was 2% or less additive in n-dodecane. (Some of the additives were not soluble at this concentration.) The results are tabulated in <u>Table 12</u>.

Some of the additives were effective in increasing the cracking rate and increases in conversions of 40% to 75% were observed (cf 165-7,8,5). Other additives were ineffective or acted as rate inhibitors (cf 165-2,3,13). The additives used were organic compounds containing various functional groups, and it was evident that some of these groups were more effective than others.

The reaction products were lighter than n-dodecane and presumably were cracked material (<u>Table 12</u>). From GLC emergence times the principal component appeared to be a  $C_{0}$  hydrocarbon (peak No. 1, <u>Table 11</u>) and was not identified

Table 12 follows

AFAPL-TR-70-71

-18-

## DEHYDROCENATION OF BICYCLO(2,2,2)OCTANE OVER CATALYST 11639-100: PUTSE REACTOR Table 11.

Pressure: 10 atm Feed: 38% BCO in n-Hexane Catalyst Volume: 0.25 ml Catalyst Wt: 0.0986 g Pulse Vol: 1 µl Catalyst Diluted With 1.0 ml Quartz Chips

Run No. 11767-	I-179	1-59	65-2	65-3	65-4	1-99	6-99	1-19	68-1	68-2	68-3
Ce <b>rrier Gas</b>	He	Ц2	Не	H2	He	Нг	H2	Не	He	Ч	He
Carrier Gas Flow Rate, cc/min 142V	150	300	150	300	150	300	300	150	150	300	150
Total BCG	172 65	<u>3</u> 43 130	172 65	343 130	172 65	3 <sup>4</sup> 3 130	343 130	172 65	172 65	343 130	172 65
Block Temperature, °F	8	•	in i	4	842	752	- ₩	1 9	- 6 	4 25	1022
Product Analysis, %w Ca and lighter	<b>66,</b> 2	62 <b>.</b> 8	66.0	63.9	66 <b>.</b> 2	-a)	.a)	71.9	6.17	63.2	87.2
Bigyclooctene	1.0	0.0	0.6	0.0	с. гт	,	1	, 	, 	0.3	0.4
	0.2	0.0	0.0	0.0	ۥ0	ı	1	0.0	0.5	0.0	0.7
U2 /	0.1	0.0	0.3	0.0	5.2	8	1	0.7	1.0	0.1	0.7
ECO	32.7	37.2	33.1	36.1	30.1	1		26.7	8.6	36.4	2.0
BCO Conversion, Aw	14.6	0.0	13.6	0.0	4.[2	_a)	_a)	30.1	77.5	5.0	94.7
BCO Converted to Bicyclooctene	2.6	1	1•5	1	3.1	1	I	3.7	2.9	6.0	1.0
Yield Bicyclooctene	18.0	1	11.0	ł	14.6	-n. á		12.3	3.7	17.6	1.1
a) No reaction: only n-herane a	nd RCO	These		- cmcm d			-				

oma vogram. Ŧ b) Unidentified; emerged after bicyclooctene. c) Unidentified; emerged after  $U_1$ .

Table 12. THERMAL CRACKING OF N-DODECANE USING ADDITIVES: PUISE REACTOR

Carrier Gas Flow Rete: 200 rs/mfr	LEV: 60 Contact Time: 1 sec
	Quartz Chips F
atm P ml	With 1112° He
Pressure: 10 Pulse Volume:	Reactor Filled Temperature: Carrier Gas:

Run No. 11225-	Feed	116-2	116-1	£-91-	1-701	167-2	167-3	168-1	168-1	0.041	160.3	1-021	0	T T	1.12	. 52
Additive 11325-		NONE	165-7	165-10	165-6	165-1	165-8	165-14	165-11	165-4	165-23	165-5	165-9	165-2	165-7	1-203-1
Additive Concentration, 🐝		,	<u>(</u> )	Sat (< 2%)	Sat (<2%)	Sat (<2%)	Q	Q	<u></u>	Sat K24)	Set Set	(V	Set < 2%	Set 2%	Ser .	Sat
Rescrop Wall Temperature, <sup>2</sup> F	[x.	110%-	1094-	- <del>1</del> 601 -1100	-4001 1011	1090-	1002-	-1001	10 <del>24</del> -	1098-	1094-	-1001	1001	1000-	1080	-0011
Product Analysis, Sw													*		2	3
Component No.																
et ()	0.0	10.7	15.5	11.4 1	10.2	4.1	20.1	7.6	6.6	11.0	9°1	16.0	10.8	9.0	0.0	10.0
<b>1</b> K				ເ <u>ດີ</u> ເ	~ ~	ເ ເ	3.4	้า	2.2	ດ. ຄ	1	0.0	5.1	5.	1.6	2.2
\			້		9. -	1.8	3.7	1. 8	ן• ז	1.7		3.4	1.7	1.6		
t u		+ ( -1 r 	5	J.6	7•4	1.5	2.0	ويتد	2.0	1.4	1.2	5,0	-+ 			- Le'
			^` -		1.1	۲. ۲	5°0	2,2	1.2	1,2	1.0	بر بر بر	1•3	-	C	, c,
×		5,	0. 0	0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	
-1 (		0 r -		ກ ກິ	6.0	1.1	1.7	1.0	1.3	1.2	0.0	1.6	1.1	1.0	0	)
- α			<b>a</b> 5 0	<b>.</b>	ю. С	0.5	0.6	6.0	0.3	4.0	4.0	0.6	0.3	0.3	0.2	0
) (1					0	2 0	0.2	0.1	0.2	0.1	ۥ0	0.1	0.0	0	0.3	0
		80		9°6	4.1	0.61	65.8	<i>к.</i> 89	80.5	6.61	83 <u>,</u> 9	7.6	80.3	83.3	4.48	81.3
)	<b>)</b>	0 5	<b>\$</b>	<b>0</b> 0	9.0	0.6	.न २	0.0	6•0	6.0	1.1	0.0	0.0		0.0	0.0
Dodecene Conversion, 4.	<b>1</b> <b>1</b>	19.4	26 <b>.</b> 8	20.4	18.6	21.0	34.2	17.71	19.5	20.0	16.1	€8 <b>.</b> 4	19.7	16.7	15.6	18.7
Increage in Conversions Due to Additives, \$	;	ł	38.1	5.2	Ľ. †	8.2	76.3	-8 <b>-</b> 8	0.5	3.1	-17.0	17 <b>0.1</b>	1.5	-13.9	9.61-	ja P

further. In calculating conversion it was assumed that each molecule of dodecane reacted gave one molecule of product; hence, the conversions are maximum values. Product distributions did not appear to be affected by additives, and analyses for several runs with different additives are presented in <u>Table 12</u>. No estimate of coke make could be made from the pulse reactor data.

These results were similar to those observed previously and the maximum rate enhancement was about that observed in the previous work. The best results obtained thus far for all additives tested showed that at 1112°F the rate can be enhanced by about a factor of 1.8 with about 2% additive.

In a second set of experiments a few additives were tested at 1202°F using 3% additive or less in n-dodecane as feed. These additives had been tested previously at 1112°F.<sup>3</sup>) The complete data are tabulated in <u>Table 13</u>, in which the values in parentheses are those for 1112°F.

At 1202°F considerably less enhancement of reaction rate was observed than with the corresponding additives at 1112°F. For example, with 200-1 and 119-13 the overall conversions were increased by 105% and 62%, respectively, compared to increases of 41% and 9% at 1202°F. No essential difference was observed in product material or product distributions at the two temperatures, nor was the product distribution affected by the additives.

Presumably the overall rate of the cracking reaction was due to free radicals generated by (a) thermal means and (b) the additives. As the rate enhancement by the additives declined with increased temperature, this suggests that the activation energies for the production of free radicals by purely thermal means was greater than that for the generation of free radicals by initiators. This work is continuing.

### Effect of Propane on the Catalytic Dehydrogenation of Methylcyclohexane

The sensible heat sink of the methylcyclohexane (MCH) system could be extended by mixing MCH with a light hydrocarbon such as propane, which would lower the melting point of the system. For additional heat sink MCH would undergo endothermic reaction. Consequently it was of interest to determine if propane affected the reactivity of MCH for dehydrogenation.

A mixture of 30% propand in MCH was tested for MCH dehydrogenation in our bench-scale reactor at 10 atm pressure, 842-1112°F, and an LHSV of 50 (basis MCH fed) over our standard 1% Pt on Al<sub>2</sub>O<sub>3</sub> catalyst. Reaction time was 30 minutes at each temperature. Liquid product material was analysed by GLC and the gaseous products by mass spectrometer. The data are shown in <u>Table 14</u>, which includes data for pure MCH obtained under the same reaction conditions.

Addition of propane enhanced the reactivity of MCH at the lower temperatures but not at  $1022^{\circ}$ F. This is shown more clearly by <u>Figure 9</u>, which shows conversion as a function of block temperature. This suggests that propane acted as a diluent, and as such improved the heat transfer to the catalyst resulting in higher conversions. At the higher temperatures it appeared that the catalyst was deactivating, possibly due to propane decomposition, although no propylene or lighter hydrocarbons were observed in the gas products. In earlier work with this catalyst and no propane present in the MCH feed, 95% MCH conversion was observed at LHSV of 100, 10 atm pressure, and 1112°F.<sup>5</sup>

Table 15 and Figure 9 follows

Table 13. THERMAL CRACKING OF N-DODECANE USING ADDITIVES

3

Pulse Reactor: 1202°F

Pressure: 10 atm Carrier Gas: He Puise Volume: 1 µl Carrier Gas Flow Rate: 200 cc/min Reactor Filled With Quartz Chips Contact Time: 1 sec.

Run No. 11325-	161-1	162-1	162-2	162-4	163-2	163-3	164-1	164-3
Adaitive: 11325-	NONE	199-7	199-12	199-13	ENCN	119-21	90-011	
Acditive Concentration, 🕬	;	2.7	2.1	Sat (<3.4%)	1	1.7	1.8	2.6
Reactor Wall Temperature, °F	1175-91	1175-91	1175-89	1175-89	<b>16-</b> 5/11	1177-93	1175 <b>-</b> 01	ro-2711
Product Analysis, 44 Component No.				<b>.</b>				
	41.1	46.2	47.4	45.5	41.9	42.9	44.9	49.8
()ı	5.7	0 • •	6.6	4.9	4.9	4.6	5.9	ć.7
Νγ	3.7	6.1	3.9	4.0	3.9	3.9	4.1	4.2
-1	5°0	3.1	3.8	3.1	0° 2	2.9	3.9	3.0
Ĵ	2°2	2.3	1.8	2.1	5	2.2	2.5	2.1
After 5	ł	ŧ	0.3	I	1	ı	0.2	1
9	8. 	ы 8	1.1	1.9	2.0	2.0	1,9	1.8
ţ,	2.0	0.6	0.4	0.7	0.8	0.6	0.7	0.7
Ċ,	ୟ 0	2.0	0.1	0.2	0.2	0.2	0.2	1.0
(1).	41.6	34.6	33.4	36.1	41.2	40.7	35.6	31.6
155 × 6	0.0	0.0	1.2	0.0	0.0	0.0	0.0	0.00
Dodecane Corversion, Eu	58.4 (20.1) <sup>a</sup> )	65.4) (27.6 Å)	66.6 (29.1)	63.9 <sub>9</sub> ) (32.6 <sup>9</sup> )	58.8 (20.1) <sup>e)</sup>	59.3 E) (27.3 E)	51. 5 (35.8°)	68.4 (21.2 P)
Increase in Conversion due to Additive, S	(37.3)	12.0) (44.8) <sup>a</sup> )	$(62.2)^{a}$	4.6	0.7 (35.8) <sup>a</sup> )	$(78.1)^{a}$	-6.5 ) (105.0 P)	17.1
2. Values poteinen merinisty a.	+ 1110°E					/ /	10.10+1	

AFAPI - 78-70-73 66758



AFAPL-1R-20-21 66758

### Table 14. DEHYDROGENATION OF PROPANE-METHYLCYCLOHEXANE MIXTURE

(atalyst: 1% Pt on Al<sub>2</sub>O<sub>3</sub> ) atalyst Volume: 7 ml

Pressure; 10 atm LHSV: 50 (basis MCH bed)

		11623-			11	767-	
Run	189	190	191	11-1	12-1	12-2	13
Feed		Pure MCH	>	- 3	0% С <sub>З</sub> Н	ы 8-70% МС	H >
Temperature, °F Block Wall CataLyst Bed	842 765 650	932 837 694	1022 921 752	842 734 662	932 806 671	1022 891 752	1112 1013 855
Liquid Product Amelysis, <b>%</b> MOd Toluene	42.2 57.8	26.1 73.9	13.9 86.1	<b>36.</b> 2 63.8	21.6 78.4	14.7 85.3	16.8 83.2
MCH Conversion, %	57.8	73.9	86.1	63.8	78.4	85.3	83.2
Gas Product Analysis, 4m				3			
Propan H <sub>2</sub>	-	-	- 100	29.3 70.7	26.5 73.5	25.5 74.5	26.5 73.5

### CATALYST STUDIES

We have continued the study and development of conventional granular and coating catalysts and the small scale screening of catalysts for dehydrogenation activity in the micro-test reactor (MICTR), which began under a previous contract!)2)3) Many additional granular catalysts have been prepared that consist of one or more metals on various supports or mixtures of several unsupported metals from in-house programs. Improvement in dehydrogenation activity at high selectivity still is being sought.

A number of catalysts consisting of 1 and 4% Pt on types 1 or 6 supports with various physical properties have been prepared in furtherance of the concurrent MICTR and bench-scale reactor studies on the activity-stability of dehydrogenation of endothermic fuels, namely MCH and decalin. Included in this study are many additional type 1 supports from both commercial and in-house sources prepared in quantity for bench-scale evaluation. In addition, a study has been started on stabilization of the activity of one of the best type 1 supports, at 1% Pt concentration, by adding single metals at different concentrations. On this support 4% Pt has given better activity-stability than 1% Pt in the bench-scale tests. These metals are expected to suppress the coke forming side reaction that is believed to limit catalyst life during dehydrogenation reactions at low hydrogen partial pressures. Bench-scale testing has shown that greater stability results from using a support with small pore sizes and a high platinum concentration<sup>3</sup>? Favorable information obtained from this present study probably can be utilized in creating coating formulations with extended catalyst life.

Study has also been initiated in improving the activity-stability of granular platinized type 6 support catalysts, a problem encountered in scale-up preparations, by using two nonalkaline sources of platinum to avoid attack on the surface of the support.

Study has continued of the improvement of catalytic coatings for metal surfaces, such as ease of application, mechanical properties, adherence to metal surfaces, and catalytic activity. New formulations have been studied by metal strip test evaluation and by MICTR tests of candidate coatings in mlatinized granular form. A possible substitute for the fibrous type 1 component in the best older formulation and which is no longer manufactured has been sought in a widely available natural fibrous material (type 16). Efforts have been made to reformulate coating materials with components shum separately to be activity-stable when platinized. Also, the effect of finer grinding of the wet component mixtures has been studied to obtain better metal adhesion on drying, and to simplify the formulation. Further, formulation with one or more preplatinized components rather than platinization of in situ coatings has been studied as a means of obtaining more exact control of the amount of impregnated pl. tinum.

Through July, 1970, a total of 958 catalysts have been prepared or obtained from proprietary or concretel sources; dl of these have been screened in the MICTR during the present are past contracts. Most of the catalysts have been tested in 10-20 mesh particle sizes, including many candidate coating formulations, and the table  $4^{4}$  D stainless tubes catalytically coated internally have also been tested. Screening has been for dehydrogenetion activity of MCH to toluene without

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added hydrogen at 10 atm pressure, 100 LHSV, and 662, 752, and  $842^{\circ}F$ . The same pump rate (90 ml/hr) was maintained for the coated tubes as for the usual granular catalyst charge (0.9 ml catalyst diluted to 2.0 ml with granular quartz). The coated tubes have been filled with quartz to create better mixing and heat transfor. All catalysts have been compared with reference catalysts 9874-139 or its equivalent 10860-70 (1% Pt/UOP R-8 type Al<sub>2</sub>O<sub>3</sub>). The purpose of the screening tests was to obtain a quick comparison with the reference catalyst so as to determine the more promising catalyst compositions, and to eliminate catalysts with activities too low or selectivities too poor to be of practical importance.

A sketch and photographs of the MICTR are shown in Figures 87, 88 and 89 in the Appendix of a previous report<sup>1</sup>) along with a description of the operational details. Subsequent modifications appear in a later report<sup>2</sup>; more recently the original GLC trace recorder has been replaced with a Westronics recorder. Detailed test data appear in the Appendix, Tables 57 and 58, of this report.

### Preparation and Evaluation of Granular Catalysts

Most of the particulate catalysts have been prepared by impregnation of various supports (10-20 mesh) with one or more metal salts, followed by drying at 126°C and reduction of the metal (or metals) in situ with hydrogen at 795°F prior to evaluation. Some of the catalysts were unsupported oxide mixtures of various types from several in-house catalytic programs.

Typically, quantities prepared were a few grams for MICTR evaluation with MCH, 30-40 grams for bench-scale studies with endothermic fuels, and 300-400 grams for FSSTR studies (10860-146 and 147). Metal limits were usually between 1 and 4% for the supported catalysts.

Catalysts of the 132, 154, 135, 137, 138, 139, 141, 142, 158, 165, 167, and 161 series were prepared at the 1 and 4% Pt levels on two types of supports with various physical properties. The approximate physical properties of the supports are given in <u>Table 15</u>. These catalysts were prepared primarily for activitystability studies with decalin in the bench-scale reactor, although they were first screened in the MICTR for activity with MCH (Appendix, <u>Tables 57 and 58</u>). Study of factor affecting activity-stability has been carried out primarily with granular materials, but the factors are considered to be likewise important in catalytic coatings on metals, also.

Catalysts of the 188, 189, 190, 191, and 192 series were prepared from a type 1 support previously shown to be activity-stable in the bench-scale reactor at 4% Pt concentration but much less so at 1% Pt concentration. Various metals (AA-AE) at different concentrations were incorporated into this support in an effort to improve the activity-stability at the lower Pt concentration (14) and to further investigate stability factors. This treatment has been directed toward the suppression of coke-forming side reactions.

Table 16 summarizes MICTR evaluations of a group of catalysts containing 1 or 4% Pt on type 1, 6, or 10 supports with various physical properties (also Table 57 of the Appendix). Table 17 summarizes MICTR evaluation of additional catalysts prepared from type 1 supports from never sources. The first group of

Table 15 follows

-22-

(mu1	filed at all	12°F)	

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Table 15. APPROXIMATE PHYSICAL PROPERTIES OF VARIOUS CATALYST SUPPORTS

There	Support	Bulk	Surface	Pore	Av.
Type	Original Configuration	(gm/ml)	$(m^2/gm)$	(ml/gm)	(Â)
1 <sup>c)</sup> """	Granular " " " " "	0.50 0.58 0.76 0.80 0.88 0.75 0.87	164 234 380 230 210 350 80	$\begin{array}{c} 0.719^{\text{e}} \\ 0.719^{\text{e}} \\ 0.384^{\text{a}} \\ 0.51 \\ 0.23^{\text{a}} \\ -0.37 \\ -0.25 \\ 0.29^{\text{e}} \\ 0.29^{\text{e}} \end{array}$	176 65 50 40 70 ~28 145
"C) "d) "d) "d) "d) "c) "c) "c)	"b) " " " " " Extrudate "	0.63 0.65 0.61 0.87 0.60 0.49 <sup>e</sup> ) 0.68 0.63	275 242 342 319 269 250 249 207	0.62 0.494 0.367 0.323 0.526 0.44 0.55 0.66	90 82 43 40 78 77 89 127
6 " "	Granular " Extrudate	0.72 0.38 0.30	~800 340 314	0.43 1.15 1.33	-21 135 125
10	19	0.52	600		-9
16	Granular <sup>f)</sup>	0.41 <sup>f)</sup>	125 <sup>g)</sup>	0.60 <sup>g)</sup>	500 <sup>g</sup> )

a) Does not include macro pore volume.
b) Primary particles are fibrous.
c) Commercial source.

d) In-house source. e) Bulk density of powder. f) With 20% type 6 binder. g) Urbonded powder.

### Table 16. MICTR SCREENING OF VARIOUS PLATINUM SUPPORTED CATALYSTS OF DIFFERENT PHYSICAL PROPERTIES

Conditions:	Feed Stoch MCH, LHSV 100, 10 atm pressure, no add	led
	hydrogen. Temperature variable. GLC samples tak	(en
	at 5, 8 and 13 minutes, respectively, at each tem	ıp-
	erature	

Cat. No.	\$		MICTR	wt	Conversion	of MCH to Tolue	no, <b>4</b> 4
10860-	Pt	Support	Run No.	Charged	662	752	842°F
9874-139	1	UOP R-8 type Al_203 (Ref.)	1122	0.43	24, 22, 24	50, 46, 47	70, 70, 70
1 <b>32 A</b> 179 B	1 14	Type 1 Support	1127 1118	0.41 0.45	24, 24, 24 24, 22, 24	52, 49, 49 47, 47, 47	76, 75, 75 76, 78, 78
13« ) " 134 D	1 * 4 7	ч н н <u>е)</u> м н п м п п п п п	1123 1134 1124 1135	0.54 0.53 0.53 0.56	24, 23, 24 33, 29, 23 20, 24, 21 (15), 25, 26	58, 54, 55 53, 53, 53 59, 48, 48 54, 53, 49	85, 84, 82 82, 81, 81 77, 77, 76 79, 77, 76
135 0 ** 135 0 #	1 r 4 n	Type 1 Support a) """ a) """ a) """ a) """ a)	1125 1137 1126 1138	0.52 0.53 0.57 0.54	25, 26, 26 27, 25, 27 27, 23, 24 25, 24, 24	59, 56, 54 54, 52, 52 53, 50, 51 58, 53, 53	83, 81, 80 81, 81, 80 78, 77, 77 83, 81, 81
135 A 135 B	5.4	Type 1 Support a)	1120 1121	0.78 0.81	24, 21, 20 25, 22, 22	51, 48, 49 58, 53, 55	76, 76, 76 83, 82, 83
137 A 137 B 144	1 4 4	Type 1 Support         a)           """         a)           """         c)	1135 1139 1153	0.73 0.72 0.83	28, 23, 24 26, 30, 27 35, 35, 35	53, 50, 49 57, 54, 52 62, 56, 58	79, 78, 77 82, 80, 81 87, 87, 86
138 А а 138 в #	1 • 4	Type 1 Support a) """" """"	1140 1142 1141 1145	0.74 0.75 0.76 0.77	26, 24, 24 22, 32, 30 28, 25, 31 33, 33, 28	51, 40, 49 55, 53, 53 59, 56, 55 60, 58, 58	80, 76, 76 82, 81, 80 83, 82, 81 87, 86, 85
139 A 139 B	1 4	Type 1 Support a)d)	1143 1144	0.31 0.32	28, 25, 24 26, 30, 27	44, 44, 42 55, 50, 50	66, 63, 63 75, 74, 73
141 A 141 B	1 4	Type 1 Support a)	1146 1147	0.31 0.34	20, 19, 18 27, 22, 22	40, 35, 34 49, 45, 44	56, 35, 55 70, 68, 69
142 A 142 B	14	Type 1 Support a)	1149 1150	0.67 0.72	23, 23, 22 30, 25, 24	55, 55, 51 62, 59, 58	80, 80, 79 89, 88, 88
143 A 143 B	). Նլ	Type 10 Support b)	1151 1152	0.61 0.66	24, 22, 21 25, 24, 21	38, 36, 41 44, 46, 46	60, 57, 56 70, 71, 11
147 #	<u>ъ</u> ц	Type 6 Support	1156 1157	0.33 0.33	19, 20, 18 23, 21, 20	44, 41, 41 43, 41, 41	64, 61, 58 65, 64, 65
146 *	4 77	Type 1 Support e)	1176 1177	0.81 0.89	37, 38, 33 30, 31, 35	60, 58, 59 63, 59, 59	88, 86, 86 89, 86, 86
9874-139 10360-136A 10360-1368 10860-1368 10860-154A 10860-1548	1 1 4 1	UOP R-8 type AlgOs ref. Type 6 """ ""	1136 1131 1132 1186 1191	0.435 0.586 0.598 0.638 0.657	$\begin{array}{c} 22, 24, 21 \\ 11, 12, 14 \\ 16, 15, 5 \\ 14, 17, 14 \\ 26, 21, .9 \end{array}$	50, 46, 47 27, 26, 26 25, 32, 32 35, 37, 34 38, 36, 35	64, 70, 71 41, 49, 37 48, 47, 48 47, 41, 40 50, 45, 45

a) thifled before impregnation. b) Muffled before Pt (MH3).\*\* ion exchange step. c) Um as received Uists d) Surface area 514 g2/gs. e) Muffled before impregnation.

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MICTE EVALUATION OF PLATINIZED GATALYSTS WITH NEWER TYPE I SUPPORTS Table 17.

Feed Stock MCH, LHSV 100, 10 atm., no added hydrogen (0.9 ml 10-20 mesh catalyst diluted to 2.0 ml volume with quartz) GLC Samples taken at 3, 8, and 13 minutes, respectively Test Conditions:

Cataïyst	25	Source of Type 1	MICTR	wt. charged,	Average Conve	rsion of MO	H To Tolters, Su
-0000T	1 12	troqque	HUT NO.	ณ์	<del>(</del> 62	752	942 ° 7
70 (ref)	r-!	U B	1201	0.410	22	94	
158A	r-1	connercial	1206	0.535	25	53	2.2 S
1588 <sup>a</sup>	<u>ດ</u> ເ		1207	0.542	ਹੈ		) (
153c <sup>a</sup> ,	<b>:</b> t	Au.	1208	0.567	t,	N <sup></sup>	(Д) М
165A **/	r - 1	€	1219	0.534	\0 0	1.0	100
165 <sup>a</sup> , <sup>U</sup>	t-	-	1218	0.549	59		20 00
157A	<u>ب</u> ـــز	"in house prepn."	1202	0.562	22	20	the second seco
157E	t		1203	0.596	50	00 (C V ir	) ⊮ - Ω_
170B <sup>07</sup>	ţ,	2	1231	0.597	55		8
1579	r:+	st st st	1204	0.345		¢.r	BC:
1257F	.t.	#1.4 8:0 9:- 9:-	1205	0, 562	10		) [- ) ()
170A <sup>b</sup> )	_t_	4.	1236	0.559	51		20
1570		14 - 11	1210	0.843	86	Y M	
1576	. <del></del>	11 11	1211	0.828	18	- N - N - N	), p 1
							יין איז
	-1-		1212	0.527	ŝ	40	t 
	t		1213	0.548	r C)	20	r-1
I'nc"	ŧ		1237	0.541	22	ŝ	á,
(qvrl1	4	connercial	1233	0.606	25	5	
171.B <sup>0</sup> /	-#	=	1234	0,493	5	5	
	r-1	-	1238	0.712	21	48	
	Q .	<b>8</b>	0421	0.762	S N	48	
1'TEU'	- <del>1</del>		1239	0.747	32	аl К	- 3
a) Granule	10 88	epared from fihrons	t.vne l s	unnowt.	and the second s	and a second	الم الله المحمد الم المحمد المحمد

a) uranutes prepared in unitured support. D) Prepared in larger quantities for bench-scale activity-riability tests with endothermic fuels.

AFAPL-TR-70-71

-24-

catalysts were predominantly larger scale preparations of various types, mostly with high activity, and were prepared primarily to further the study of activitystability in the bench-scale reactor with endothermic fuels.

Two general problems of scale-up were encountered with type 6 supports:

(1) High density type 6 support particles decrepitated extensively to smaller than 20 mesh on impregnation with platinous tetrammine dihydroxide, notwithstanding the prehumidification of the particles with water (10860-136A and 196B, <u>Table 16</u>). This was avoided in subsequent preparations (10860-134A and 154B) by a modified technique. Catalysts from this support have been shown in the past to be less active in the MICTR test and less stable in the bench-scale test with decalin than those prepared from low density type 6 support which does not decrepitate on contact with liquid water.

(2) Scale-up of type 6 supported preparations requires longer drying time and thus longer contact time, resulting from thicker catalyst layers during evaporative concentration of the highly alkaline platinum solution. This undoubtedly leads to greater attack of the siliceous surface with consequent dissolution of silica at the expense of surface area contributed largely by the walls of small pores. A glass-like film has been observed at the evaporating interface in the porcelain dish during concentration of impregnating solutions. The experimental preparations designed to avoid this problem are described below.

Earlier study with small batches of catalysts consisting of 1 or 4% It on low density type 6 support showed good activity for MCH dehydrogenation in the MICTR, notwithstanding the low charging weight (<u>Table 18</u>, catalysts 10860-114B and 114C). Also, catalyst 114C showed excellent activity-stability on bench-scale tests with decalin. Reproduction on a larger scale gave catalysts less active for MCH dehydrogenation (1086C-141A and 141B), and catalyst 141B showed poorer activity with decalin in the bench-scale test than catalyst 114C.

Various factors have been studied to elucidate further this difficulty. These results indicate that when platinum solution 30 is used as a source of platinum equally active catalysts are obtained regardless of whether the low density type 6 support is used on a "as received basis", or is muffled in air at  $1112^{\circ}$ F, (catalysts 10860-149A and 149B). Slightly higher activity is obtained if the platinum solution is first neutralized (catalysts 10860-149C, 149D, and 166A). This modification of the impregnating solution is expected to play a more important role in activity-stability with decalin in the bench-scale test, because surface attrack on the type 6 support by the highly alkaline platinum solution probably damages the catalytic surface more than the neutralized platinum solution. Catalysts of equal performance to those prepared by the latter solution have been obtained when an acidic source of platinum is used (No. 31). The data shown in <u>Table 18</u> indicate that highly active catalysts can be prepared from extruded how density type 6 support with good correlation of activity with platinum content (10860-128A, 128B, and 128C).

Catalyst 10860-113 (4% Pt, 9-16 mesh, spherical type 1 support), made in large quantity for FSSTR studies, was found to be less active in the MICTR with MCH than earlier counterparts (10280-107B and 91A). These latter catalysts had

AFAPL-TR-70-71

TENTE 18. MICTR EVALUATION AND COMPARISON OF VARIOUS PLATINUM PROMOTED TYPE 6 SUPPORTED CALARYSES

Conditions: MCH, IASV 100, 10 atm pressure, no added H<sub>2</sub>, temperature variable. (10-20 mesh particle size catalyst)

Catalyst No.	t t	Pt Source	Form of Type 6 Support	MICTR	Lierage Conv	ersion of MCH to	. Toluene, Ka
					662	752	342°F
9874-139	; <del>~</del> i	30	reference (spherical Al203)	1116	25	i48	
10860-114B " 114C " 141A " 141A " 141B	ten ten	05 05 05 05 05 05 05 05 05	granu ar (low density) " " " " "	2111 2111 3411 7411	1818	45 52 45 57 45	82:45
02 "		30	reference (spherical Al <sub>2</sub> O <sub>3</sub> )	1166	54	45	17
=====================================	+ + + + + + + + + + + + + + + + + + +	30 30°) 31 31 31	$ \begin{array}{c} \operatorname{granular}^{a}_{b} (: \operatorname{ow density}) \\ \  \  \  \  \  \  \  \  \  \  \  \  \$	1163 1173 1173 1173 1173	48 4 0 6 6 4	49444 495954	\$\$\$\$P\$\$B
9874-139		36	reference (spherical Al <sub>2</sub> O <sub>3</sub> )	1105	25	47	2
10360-128A " 123B " 128C	но. <del>4</del>	30 30 30	extruded, granular <sup>d)</sup> (low density) """"	1098 1106 1100	56 23 60 86 23 60	국부대	69
" 166A " 166B	**	30 <sup>c</sup> ) 31	granular <sup>b)</sup> (low density)	1220 1221	8 5	59 29	65 65
<pre>a) Support b) Support c) Noutrali d) Support</pre>	as j mufj Lzed mufi	received. Ned. impregna Ned in a	ating soluti r. air.				

AFAPL-TR-70-71

-26-

been prepared on the same support screened to 10-16 mesh, (<u>Table 20</u>, runs 1108 vs 1155 and 401). A 10-16 mesh portion of catalyst 113 had higher activity (run 1114) but less so than earlier counterparts; thus the particle size range was only partly responsible. Heating of the 9-16 mesh particles of catalyst 113 in air at 932°F improved the activity somewhat (run 1130).

A number of type 1 supported catalysts containing different amounts of metal G at a 1:1 weight ratio with platinum have been tested with MCH in the NTC 3 without added hydrogen. Suitable control catalysts with the same amounts of platinum without metal G on the same supports have also been tested (<u>Table 10</u>). Uncerpresent MICTR conditions the addition of metal G produced no appreciable improvement in activity for dehydrogenation of MCH. Platinum-metal G promoted catalysts have been examined for activity-stability with decalin in the bench-scale test apparatus and the results are given earlier in this report.

### Table 19. EFFECT OF METAL G ON PERFORMANCE OF PLATE MICHTED CATALYSTS WITH METHYLCYCLOHEXANE IN MICH

Conditions: LHSV 100, 10 atm. pressure, no added H<sub>2</sub>, temperature variable; 9874-139 reference catalyst.

Cotolwat No	Met	als	MICTR	Average Conve	rsion of MCH t	to Toluene, 9w
Catalyst NO.	% Pt	% G	Run No.	662	752	842°F
9874-139 10860-129F " 129A " 129G ' 129B " 132C " 129C " 129E " 129D 9874-139 10860-134A " 134B	1 0.5 0.5 1 1 2 4 4 1 0.5 1	0 1.5 0 1 2 0 4 0.5 1	1105 1110 1102 1111 1103 1119 1115 1109 1107 1102 1128 1129	26 23 26 24 27 21 23 25 25 25 25 25 25 25 25 25 25 25 25 25	47 52 54 53 49 51 55 56 47 42 43	72 82 79 84 78 74 80 83 87 70 65 65

Table 21 shows the MICTR evaluations with MCH of 1% platinized type 1 supports containing various amounts of several metals intended to improve activitystability on more rigorous bench scale testing. Metals AA and AB with content increasing from 1 to 4% decreases initial activity only slightly at 752°F (runs 1263-1259). Metal AB produces a much more pronounced decline of initial activity

AFAPL-TP-70-71

-27-

Table 20. RELATIVE ACTIVITIES OF CATALYSTS PREPARED FROM SPHERICAL TYPE 1 SUPPORT WITH METHYLCYCLOHEXANE IN MICTR

Conditions: Jame as for Table 18

Catalvst No.	<del>8</del> 8	Mesh Sïze of	NICTR	Average Conve	rsion of MCH t	o Toluene, 🚧
	Pt	Spherical Particles	Rin No.	662	752	842°F
10860-70 <sup>a</sup> )	-1	10	: 148	22	94	68
10280-91B	N	10-16	1,00	25	52	74
<b>A</b> IQ "	4	2	Tot	26	57	6
" 130G	*	(q"	543	23	45	01
1028c-107B	4	10-16	1155	22	61	5
" 113	4	9-16	1108	50	£#	Q
2	<del>ب</del>	10-16	4111	20	941	69
10860-1370	-7	9-16c)	1.1.30	51	47	R
a) Reference b) Catalyst 1 c) Catalyst 1	ca ta]; 0280-6 0280-1	<pre>/st on UOP R-8 type Al, 91A muffled in air 2 h( 115 muffled in air 1/2</pre>	203. ours. hour.			

AFAPL-TR-70-71

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MICTR EVALUATION OF PLATINUM/TYPE 1 S PPORTS CONTAINING SEVERAL CONCENTRATIONS OF VARIOUS METALS INTENDED TO IMPROVE ACT (VITY-STABILITY WITH ENDOTHERMIC FUELS Table 21.

State Barrier Division

Test Conditions: Feed stock MCH, LHSV 100, 10 atm., no added Fydrogen (0.9 ml 10~20 mesh catalyst diluted to 2.0 ml volume with quartz)

GLC samples taken at 3, 8, and 13 minutes, resp.

lyst	82 t	% And Type Of	MICTR	wt. Charged	Average Conve	rrsion of MCH to	Toluane, 🐖
	ч Ц	second Metal	HUID NO.	ħņ	662	752	842°F
		Ref. Cont.rol. <sup>a</sup> )	1254 1256	0.419 C.305	20	45 52	69 81
	н н н	1 AA 2.3 AA 4 AA	1263 1265 1266	0.711 0.767 0.764	24 23 23	51 51 51	81 81 76
	ннн	1 AB 2.3 AB 4 AB	1257 1258 1259	0.738 0.715 0.752	24 23 16	55 51 45	\$2\$
	нчч	1 AC 2.3 AC 4 AC	1260 1261 1262	241.0 121.0 651.0	29 19 15	51 49 385	81 68 147
	ннн	1 Al 2.3 Ad 4 Ad	1267 1268 1269	0.767 0.742 0.742	28 21 22	54 53 53	345
		1 AE 2.3 AE 4 AC	1270 1271 1272	0.756 0.737 0.769	24 25 23	53 53	87 83 79
ser	tes	10860-188, 189, 1	190, 191,	and92.			

AFAPL-TR-70-71

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with increasing content (runs 1260-1262). Metal AD at different concentrations has no appreciable effect on initial activity (runs 1267-1269). Metal AE with increasing concentration decreases initial activity only slightly at 752°F, acting in a manner similar to metals AA and AB ( uns 1270-1272).

### Preparation of Catalyst Coatings on Metal Surfaces

### Stainless Steel

A large number of new candidate metal-coating formulations have been studied. Most of these have been made by wet grinding the ingredients with a mortar and pestle. However, in certain cases ball-milling the ingredients together has shown to have certain advantages. Small portions of each candidate formulation were spread as thin coatings, usually 3-7 mils thick, onto the smooth and sandblasted sides of degreased stainless steel strips,  $1/2 \ge 2^{"}$ , or on aluminized stainless strips. These coatings were dried, calcined, and then evaluated for coating thickness, metal adhesion, and physical condition. A few coatings were stored wet in a humid desiccator and then subjected to similar tests to check their storability. A portion of each original formulation was dried and muffled, and the 10-20 mesh particle size platinized and evaluated in the MICTR with MCH. Activity at 752°F is shown in Tables 22 and 23 with coating evaluations. Complete MICTR test data are given in Tables 57 and 58 of the Appendix.

In the past, the more successful thixotropic candidate materials were applied to metal surfaces with freedom from cracks and with good adhesion after drving and muffling, and were platinized to produce active surfaces<sup>3</sup>. It was found that application of the hydrous fortulations must be made more swiftly, however, since these formulations dry out rapidly at room temperature while being applied to flat metal surfaces. Coating of tubes internally is less of a problem since the drying proceeds at a slower rate. It has been found that this difficulty can be avoided by the addition of 10 or 20% (based on dry solids) of deliquescent salt No. 22. On drying and thermal decomposition this salt becomes a binder itself, and also within certain limits can supplant part of t e No. 6 binder currently used. Examples of catalysts using No. 22 binder alone are the 10860-119A-D series impregnated with 2 or 3% platinum (runs 1084 to 1087) and tube No. 28 (run 1088, Table 57 of the Appendix). While activity of the solid catalysis is high, this type of formulation has poor metal adherence and self strips easily. This apparently accounts for the low activity of 1/4" G.D. coated tube No. 28 (run 1088). Coatings were made with 15% No. 6 binder - 5% No. 22 binder and 10% No. 6 binder - 10% No. 22 binder, which have been used to prepare the active platinized catalysts of series 125 and 126 (runs 1094 and 1101). Metal adhesion of the formulations appears satisfactory. Demonstration of activity of a platinized coated tube was not deemed necessary.

Some experimentation was carried out in which alkaline platinum solutions themselves were used as binders for a powdered support with a spherical particle shape (10860-121 and 123, runs 1091-1092). Although active catalysts resulted on platinization, achesive properties of this type of formulation were poor.

Tables 22 and 23 follow

AFAPL-TR-70-7!

-30-

Table 22. MICTR EVALUATION AND METAL ADHESIVE PROFERTIES OF VARIOUS WALL COATING CANDIDATES

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Test Conditions: Feed stock MCH, LHSV 100, 10 a+n, no added hydrogen. (0.9 ml 10-20 mesh catalyst diluted to 2.0 ml volume with quartz). GLC samples taken at 3, 8, and 13 min-

utes, respectively.

	States and a second second						and here a second s	A Supervised in the local data	🦉 🗠 مىدىغانىيەل ئۇلىنىش ئەمدۇسىيا	AND DESCRIPTION OF
Conv. of MCH	To Toluene, 752°F	52	59	53	24	51	6. .†	58	5	55 53
MICTR	Run No.	1084	1085	1086	1.067	1094	1095	IOII	1097	1092 1092
ио	% off	a)	(е	(e	С¢	30	15		0 r i	5
Adhesi	Rating	poor	=	poor	ŧ	joog	good- excellent		Excellent	(q. Jood
Muffled	( <u>4</u> °)	932	Ŧ	-	1004	1004	:		1004	824 "
mess	(s)	SM	SB	SM	SB	SM	SB			SM SB
Tr.ick	cim)	13-14	=	;	1	3-6	5-15		;	വവ
Material Commosition		HOW particulate type 1 support	20% type 22 binder (deligue cent before muffling)	45% particulate type 1 support Ltd fibrous type _ summert	10% type 22 binder ( deliguescent before nuffling)	40% particulate type 1 support 40% fibrous type 1 support	15% type 6 birder (deliguescent before muffling)	Hof particulate type 1 support	10% type 6 binder (deliguescent before muffling)	type 1 (spherical particle shape) <sup>c</sup> )
be :	, t	(V	ξ	Q	m	<u>م</u>	2	Q	m	2 () 5 ()
Catalyst	10860-	119A	1199	1190	119D	125A	1258	126A	1263	121 123

Self-stripped on dehydration.
b) Easily rubbed off (continuous film).
c) Platinized.
d) Neutralized impregnating solution.

AFAPL-TR-70-71 66'758

## Table 23. MICTR EVALUATION AND METAL ADHESIVE PROPERTIES OF VARIOUS PREPLATINIZED WALL COATING CANDIDGTES

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# Test Conditions: Feed Stock MCH, LHSV 100, 10 atm., no added hydrogen (0.9 ml 10-20 mesh catalyst diluted to 2.0 ml volume with quartz) GLC samples taken at 3, 8, and 13 minutes, respectively.

(ata)vst	4		SS Adhesive Pro	nerties of Surrout		
10860-	, <b>, ,</b>	Support Composition	Thi ckness	E Strinned	MICIR Run Ao.	Journer, 72 h
02		UDP R-E type Alada (ref)			- 55 44 1 - 4 1 - 4	
163A	3.2	80% type 1 fibrous Suppor <sup>c)</sup> -£1% type 6 binder	3-4 mils SM 2-3 mils SB	50 20 20		
151Å	3.2	80% type 1 particulate support <sup>1)</sup> -20% type ó binder	4∝5 mils 3M čč slin 2	70 35	8	
1630	1.6	40% type 1 support $(1)^{c}$ -40% ty $\approx$ 1 support (2)-20% type 6 binder	5 mils SM 5-7 mils SB	00 1-1-0 1-1-0	1.41	
168	1.6	40% type 1 support (1)-40% type 1 support (2) <sup>c</sup> ). 20% type 6 binder	2-3 mils SM 4 mils SB	90 50	0.25	D .d
1618	1.6	E E	3-4 mils SM 2-3 mils SB	φ Έγω	1200	20 1
1638	3.2	40% type : support $(1)^{c}$ , 40% type 1 support $(2)^{c}$ 20% S10-	3-5 mils SM 3-5 mils SB	45 10	1011	20
160A	г	804 type 16 fibrous support - 20% type 6 binder <sup>d</sup> )	3-7 mils SM	50	3011	47
1603	<u></u>	ت ت	5-6 mils 3B	52	1911	24
1600	#		1		3611	55
167C	3.2	80% type 16 fibrous support <sup>c)</sup> -20% type 6 binder	6 mils SM 4 mils SB	30 36	1225	5 ti
161A	3.2	80% type 1 particulate support <sup>c)</sup> -20% type 6 binder	4-5 mils SM 2 mils SB	35	9611	i45
22	1.6	40% type 16 support <sup>e)</sup> .40% type 1 support <sup>c)</sup> . 20% type 6 binder	3		1230	Ĺţ
173	1.6	tof type 16 support $^{f}$ -tof type 1 support $^{c}$ . 20% type 6 binder	3-4 mils 3M 4 mils 3B	40 5	1235	51
175	3.2	40% type 16 support <sup>c,f)</sup> -40% type 1 support <sup>c)</sup> - 20% type 6 binder	4 mils SM 3-4 mils SB	70 35£)	1242	50
SM = Smoo SB = Sand	oth de blas	greased stainless steel. a) & stripped off ted stainless steel.	when pressure s	ensitive tape press	ed ontc su	urface, and then

AFAPL-TR-70-71 66758

gently pulled off. b) Self stripped during drying. c) P1=platinized with 4\$ Pt before formulation. d) Enture support platinized. e) Umnuffled. f) Muffled. g) dn aluminized SS, 2-3 mils thick; 60\$ stripped.

SS = Stainless Steel.

In past studies various formulations have been used to coat the interior walls of metal tubes. After drying and platinizing, the exact platinum lavdown could only be determined by removing part of the coating and analyzing it for platinum content<sup>3)</sup>. Another approach has been studied, namely formulation in which one or more components has been preplatinized with an exactly known amount of platinum on powdered type 1 or type 16 support. These were then formulated with the usual type 6 binder and tested as 10-20 mesh granules. The adhesive properties and activities of 10-20 mesh granules are shown in Table 23. Generally. with a single platinized (either fibrous or particulate) type 1 support - type 6 binder adherence to smooth stainless steel is poor but adherence to sandblasted stainless steel (10860-163A and 161A) is fair. About the same results were obtained with a combination of fibrous and particulate type 1 supports (with binder) if either the fibrous support was preplatinized (10860-1630) or the particulate type 1 support was preplatinized (10860-163 and 161B). Somewhat better adhesion was obtained if both fibrous and particulate type 1 supports were preplatinized (10860-163B). Platinum contents varied from 1.6 to 3.2% overall in these catalysts, and the MCH dehydrogenation activity was as great as or greater than that of the reference catalyst. Similar experiments were conducted substituting preplatinized fibrous type 16 support for preplatinized type 1 support. The type 16 support is attractive as a possible substitute for the type 1 fibrous support no longer manufactured, a component in the ternary 40:40:20 type I formulation. For comparison, the results shown in Table 23 are obtained with a type 16 support type 6 binder (80:20) which is first dried and then metallized with three concentrations of platinum (10860-160A, 160B, and 160C). Adhesion of the coating material is fair on smooth stainless steel and better on sand blasted stainless steel. The activity of the metallized dry granules is a little better than the reference catalyst. If the type 16 support is preplatinized (10860-1670) and then borded, about the same adhesive properties to metal are obtained. Activity is a little less than for the 160 series of catalysts at the same platinum content. Premuffled fibrous type 16 support-preplatinized particulate type 1 support-type 6 binder (40:40:20, 10860-173) gives fair adherence to smooth stainless steel and good adherence to sand blasted stainless steel. The dehydrogenation activity is fairly high. If octn supports are platinized and type 6 bonded, adherence is poorer to both smooth and sand blasted stainless steel (10860 175); activity of the plotinized granules is about the same.

A recently available type 1 support with excellent physical properties has been studied as a coating candidate component and has shown excellent pecults in all respects, except for activity-stability on bench scale tests.

The data in <u>Table 24</u> show that substitution of this new type 1 support into the usual 40:40:20 formulation (type T), in place of the most commonly used particulate type 1 support, gives good adhesion to aluminized and sand blasted stainless steel but not to smooth stainless steel surfaces (10860-176A). Red milling improves the adhesion to smooth surfaces considerably (10860-178). Very high MCH dehydrogenation activities are obtained with the platinized granular counterparts (10860-177, 177B, and 184A). Equivalent results are obtained on rod milling the type I formulation containing the usual type 1 support component (10860-78B, 183, 81D, and 184E).

Table 24 tollows

AFAPL-TR-70-71

Quite divergent results are obtained on rod milling 80% newer type 1 support - 20% type 6 binder together (type II formulation). Adhesion is poor to both smooth and sand blasted stainless steel on mortar mixing but excellent after rod milling (10860-164 and 181). Catalyst activity is high and equivalent in both cases at the same platinum content (10860-164A-C, and 184C). With the previously used counterpart type 1 support included, adhesion is equally poor after either mortar mixing or rod milling with 20% type 6 binder. Again dehydrogenation activity is hi h at the same platinum content (10280-56A, 10860-182, 58A, and 184D). Thus, the use of the new type 1 support in the type I formulation appears to give a promising and simpler wall coating metal support than the previous corresponding support. However, another important factor, namely activity-stability, is yet to be determined.

Satisfactory adhesion to smooth, sandblasted and aluminized stainless steel surfaces has been obtained with fibrous type 16 instead of type 1 support formulations if mortar mixing is used, but poor adhesion to a smooth surface results with ball milling (176B and 180). A small penalty in activity is taken in both cases (10860-177C, 177D and 184B). The activity-stability of this type of cetalyst has not been studied as yet in bench scale studies.

### Aluminum Clad Stainless Steel

Sources of internally aluminum clad stainless steel or Hastelloy C tubes are being sought. The oxidized aluminum surface is expected to form an even better bonding surface than the bare stainless steel walls for coating formulations, and may itself be a suitable support for impregnation with very thin coatings of metals catalytically active for dehydrogenation reactions. Coated tubes of these types should have better resistance to thermal strains set up by differences in thermal expansion between metals and catalytic coating materials.

One type of aluminum diffusion alloyed 1/4" OD stainless tubing was obtained from Alon Processing Company. These were unsatisfactory in that about one-half of the tubes were plugged solid, and all tubes had rough, uneven, and very rusty internal surfaces. The exteriors had a very rusty appearance and popiously thed rust flakes. The ends of tubes were magnetic, more so than the centers, apparently because magnetic austenite was formed at high temperatures in the cladding treatment. Emission spectroscopy verified that the material was 304 type stainless steel, and this was later confirmed via telephonic conversation with a representative of Alon Processing Company. The poor condition of the tubes did not warrant catalytic coating studies with them.

Four stainless steel strips which had been aluminized on one side were obtained from the above company. These strips had the usual general grey metallic appearance of stainless steel and were free from rust. The aluminized sides had a dull grey, rough, pebblegrain appearance. On heating in air at 1292°F a slight white surface oxidation was observable. Coating aterial 10860-124, after drying and muffling, adhered well to the aluminized but unoxidized surface of a small strip. The coating principally filled the indentations between the pebblegrains. In view of the unsatisfactory physical condition of the above tubes, no further experimentation with coating them catalytically is contemplated.

AFAPL-TR-70-71

-32-

Table 24. MICTR EVALUATION AND METAL ADHESIVE PROPERTIES OF VARIOUS PLATINIZED WALL COATING CANDIDATES

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Test Conditions: Feed Stock MCH, LHSV 100, 10 atm, no added hydrogen, (0.9 ml 10-20 mesh catalyst diluted to 2.0 ml volume with quartz). GLC samples taken at 3, 8, and 13 minutes, respectively.

	Support	Met	al Adhesive Pr	operties					
10660-	(composited on	Stainles	s Steel	Limm li	nized SS	Catalyst 10860-	₩. <b>†</b> .	MICTR Run No.	Comv. of MCH To Toluene, 752°F
		Thickness	\$ Stripped <sup>a</sup> )	Thickness	💰 Stripped <sup>e)</sup>				
;	JJP R-8 type Al≥3 (ref)	-	1	1	:	70		1232	46
164	SOM type 1 support - 20% type 6 binder (not rod milled)	7 mils (SM) 9 mils (SB)	100		::	164A 164B 164B	t- 10 -1	1215 1216 1217	49 55 57
1Ĝ1	60% type 1 support - 20% type 6 binder ( rod milled)	7 mils (SM) 6-7 mils (SB)	50	3-4	5~10	1840	4	1250	65
1764	40% type 1 support (1) - 40% type 1 support (2)- 20% type 6 binder (not rod milled)	2 mils (SM) 5 mils (SB)	95 -	6-7	5	177A 177B	m.#	1244 1245	54
178	40% type 1 support (1) - 40% type 1 support (2) - 20% type 6 binder ( rod milled)	2-3 mils (SM) 2-3 mils (SB)	νO	5-3	0	184A	 *	1248	55
10880- 10880-	80% type i support - 20% type & binder (not rod milled)	(SM) (SB)	-100	1	;	10280- 58A	4	308	~55
Ъ.	60% type i support - 20% type 6 binder ( rod milled)	5-4 mils (SM) (SB)	<u>6</u> 6	3-4	70	184D	 	1251	<b>4</b>
0) 42) 75	40% type 1 support (1) - 40% type 1 support (2) - 20% type 5 binder (not rod milled)	(S:1) (SB)	100 <sup>b)</sup> 20	;		61D	Q	965	57
<u>]</u>	40% type i support (1) - 40% type i support (2) - 20% type & tinder ( rod milled)	3-4 mils (SM) 1-2 mils (SB)	νc	-1 <del>+</del>	0	184E		1252	12
1768	40% type 16 support - 40% type 1 support - 20% type 6 binder (not rod milled)	8 mils (SN) 7 mils (SB)	Ś	Ś	50	177C 177D	m.#	1246 1241	51 51
180	40% type 16 support - 40% type 1 support - 20% type 6 binder ( rod milled)	mils (SM) -4 mils (SB)	20 20	Q	10	1848	_7	1253	20
SN = Sm SB - Sa SC = Stu	octh degreased stainless steel. nd blast-d stainless steel. einless Steel.	<pre>٤) \$ Stripped b) Self stripp</pre>	off when press ed during dryi	ure sensitiv ng.	re tape pressed	l onto sur	face,	and the	n gently pulled off

Various stainless steel and Hastelloy C tubes of different lengths and diameters and several strips of stainless steel have been obtained that are aluminum clod on all exposed surfaces. These have been clad by a proprietary vacuum diffusion process (PNA-73) by Pratt and Whitney Aircraft Division. Preliminary experiments have been carried out by coating one side of the aluminized strips with the nore recent experimental coating formulations. These tests have been carried out at the same time as those with the smooth and sand blasted stainless steel strips, and the data are included in <u>Tables 23 and 24</u>. Generally, coating adherence is about equivalent to that of sand blasted stainless steel and superior to that of smooth stainless steel. Preoxidation of the aluminized surface in air at 1382°F appears to increase slightly the tenacity of the coating adhesion.

### MEASUREMENT OF CATALYTIC REACTION KINETICS

Quantitative data on the chemical kinetics of MCH dehydrogenation have been obtained previously in a tubular flow reactor with packed bed catalyst. A single stoichiometric reaction is involved, constituting fairly simple kinetics. Even so, difficulties were encountered with temperature and concentration gradients in the packed bed and in the catalyst pellets. The gradients in the packed bed were accounted for by calculating them with a rather involved computer program, and also by attempting to minimize these gradients by changing the reactor geometry. The gradients in the catalyst pellets were not fully taken into account and have only been fully appreciated recently.<sup>3)</sup> The result of these interfering nonkinetic effects of heat and mass transfer is that the true chemical kinetics are difficult to extract from the experimental data.

### Need For Intrinsic Kinetic Data

A legitimate question that could be raised is: Why is it necessary to determine the true chemical kinetics? Since in the full scale reactor heat transfer and mass transfer effects will be present, why not just run the experiments at the desired conditions and fit a model to the results, regardless of whether the effects are due to mass transfer, heat transfer, or the intrinsic chemical kinetics? The answer stated simply is that is is extremely difficult (often impossible) to exactly simulate the conditions of the final application. Furthermore, if one were to obtain such a model based on this completely empirical approach, extrapolation of the model to conditions other than those of the experiments would be highly questionable As a straightforward example, consider an experiment performe to measure the rate of chemical reaction using a catalyst pellet 1/4" in diameter. One could certainly measure this quantity and fit a chemical kinetic model to it. The problem wou'd arise if the measured rate of reaction was affected by the rate of diffusion of reactants and products through the pores of the catalyst. An attempt to use the kinetic expression for the same catalyst as a 1/8" diameter pellet would fail, since the diffusion would occur much more rapidly than before, resulting in a higher net rate of reaction. The kinetic model would not predict this since the pellet size and shape is not part of the kinetic expression. A similar problem could occur by changing the velocity of the fluid flowing past the catalyst. This can change the rate of heat and mass transfer to the catalyst pellet and thereby the apparent rate of reaction, whereas a kinetic model would not account for this and would miss the effect entirely. A broad experimental

-33-
study could be made of the effect of all of the important variables in the reactor such as reactor configuration, pellet shape and size, catalyst composition and structure, and fluid pressure, temperature, and velocity. But one rapidly finds that an enormous amount of experimental work would be required, and the resulting model would simply be a device for representing the data.

A more satisfactory procedure is to separate the different processes which occur, study each independently, and then recombine them to form a model which often has a sound fundamental basis and can be used fairly confidently to calculate results, even in regions where experimental data are not available. In heterogeneous catalysis, there are several processes which can often be separated. Much is known about the rate of heat and mass transfer to and from inert particles (e.g., spheres). This information can be applied to a model of the processes surrounding a spherical catalyst pellet. Models are also available for the rate of diffusion in the pores of the catalyst and the rate of heat transfer within the catalyst pellet. Also, much information is available on the mechanisms of catalytic chemical reactions. Assuming one can obtain kinetic information in the absence of the interfering effects of heat and mass transfer, mechanistic models can be proposed, tested, evaluated, and fitted. Once a good mechanistic model is found which relates the rate of reaction to concentrations, pressures, and temperatures, then the models of heat and mass transfer can be applied to result in a reasonable mathematical model with the desired characteristics.

### Design of Experimental Reactors

The problems in designing a laboratory reactor for catalytic chemical kinetics has been widely discussed in the literature.<sup>6,7)</sup> The problems are exactly those described above. A system is required in which the interfering effects of heat and mass transfer are either absent or mathematically describable. The reactor type used in the traditional approach, and the one used in this investigation in the past, has been a packed tubular reactor. The problems with this reactor are that the flow patterns are complicated, and there are invariably appreciable temperature and concentration gradients between catalyst particles as well as within particles. Also, the reactor produces integral rate data and not the rate at a particular set of conditions. Differential reactors are sometimes used to eliminate the last problem, but errors in the chemical analysis lead to large errors in rates. To remedy these problems two different kinds of reactors have been developed: the recycle reactor and the catalytic continuous stir ed tank reactor. In both the conditions of reaction are uniform and well defined, and many of the heat and mass transfer problems can be eliminated. In both reactors the reaction rate is measured directly from the conversion and the flow rates.

A recycle reactor consists of a small amount of catalyst exposed to a flowing stream of reactant, most of which is being recycled past the catalyst. During each cycle only a small amount of conversion is occurring, but the total conversion of feed to product can be as high as desired for easy analysis. Such reactors have been used by Butt et al.<sup>8</sup>) and Perkins and Rase<sup>9</sup>. About the only problem with this kind of reactor is the pump used to recycle the fluid. It must meet rigid standards of noncontamination and be able to withstand the operating conditions. Bernard and Teichner<sup>10</sup> describe a novel pump.

A catalytic continuous stirred tank reactor (CCSTR) has essentially the same advantages as the recycle reactor. In this type the catalyst is either contained in the impeller or is mounted on the wall, and the reactant enters, becomes well mixed, reacts, and leaves. Again, virtually any conversion is feasible. Reactors of this type have been used by several workers!<sup>1-13</sup> The problem here is one of agitation. Sufficient agitation must be supplied to the fluid phase to ensure complete mixing. In a high temperature, high pressure system special equipment is required. Since this type of reactor has been found to work well at high temperatures and pressures, we have chosen it for our work on the dehydrogenation kinetics of the MCH and decalin systems.

The catalytic continuous stirred tank reactor which we have designed will be a modification of one of our existing standard reactors made by Autoclave Engineers. The agitation will be supplied by a magnetic drive with speeds up to about 2000 rpm. The reactor is designed to operate to 1000 psig at 1000°F. It will be constructed of Inconel and be about 1 liter in size. The impeller will be constructed of parallel screens containing catalyst pellets.

## MATHEMATICAL MODEL OF CATALYTIC WALL REACTOR

A mathematical model has been developed for our catalytic wall reactor (CWR). The model should be useful in analyzing our experiments and in estimating the effects of changes in operating conditions, reactor geometry, and catalyst composition. The model assumes that the fuel flows turbulently through a tube which has a thin coating of catalyst on the wall. The fuel is assumed to be well mixed radially so that the bulk flow may be considered to be one-dimensional. The heat is assumed to be applied by generation in the tube wall, since that is how it is done in the experiments with the Fuel System Simulation Test Rig (FSSTR). The heat transfer between the outside of the tube and the environment is given as a boundary condition. The model then accounts for the heat transferred radially between the tube wall and the catalyst layer, and between the catalyst layer and the bulk fluid flow. It also calculates the axial temperature, pressure, and conversion profiles.

### Model Development

The equations for the bulk gas and for the tube wall were adapted from those derived previously for the regenerative heat exchanger model.<sup>2</sup>) The equations describing the bulk fluid flow result from a one-dimensional momentum balance, an energy balance, and a mass balance (see Appendix):

$$\frac{d}{\rho} \quad \frac{\partial \rho}{\partial P} \quad \left(\frac{dP}{dZ}\right) \quad + \quad \frac{d}{\rho} \quad \frac{\partial \rho}{\partial T} \quad \left(\frac{dT}{dZ}\right) \quad - \quad \frac{\rho d}{G^2} \quad \left(\frac{dP}{dZ}\right) \quad - \quad 2f = 0 \tag{1}$$

$$\frac{\rho^2 c_p d}{G^2} \left(\frac{dT}{dZ}\right) + \frac{Td}{G^2} \frac{\partial \rho}{\partial T} \left(\frac{dP}{dZ}\right) + \frac{4\rho^2 g}{G^3} - 2f = 0$$
(2)

where

inside diameter of catalyst)

Equations (1) and (2) may be solved for the derivatives of P and T with respect to Z:

$$\frac{dP}{dZ} = \frac{G^2}{\rho d} \left( \frac{a_{22}b_1 - a_{12}b_2}{a_{11}a_{22} - a_{12}a_{21}} \right)$$
(3)

$$\frac{dT}{dZ} = \frac{G^2}{\rho^2 d} \left( \frac{-a_{21}b_1 + a_{11}b_2}{a_{11}a_{22} - a_{12}a_{21}} \right)$$
(4)

where

$$a_{11} = -1 + \frac{G^2}{\rho^2} \quad \frac{\partial \rho}{\partial P}$$

$$a_{12} = \frac{G^2}{\rho^3} \quad \frac{\partial \rho}{\partial T}$$

$$a_{21} = \frac{T}{\rho} \quad \frac{\partial \rho}{\partial T}$$

$$a_{22} = c_p$$

$$b_1 = 2f$$

$$b_2 = 2f - \frac{4\rho^2 q_1'}{G^3}$$

Equations (3) and (4) are integrated numerically to give axial temperature and pressure profiles of the bulk fluid flow. The following data are required to solve these equations:

- 1. The density at each axial position.
- 2. The partial derivatives of the fluid density with respect to pressure and temperature.

-36-

- 3. The heat capacity at constant pressure.
- 4. The Fanning friction factor.
- 5. The rate of heat transfer to and from the catalyst layer.
- 6. The composition of fluid at each axial position.

The required physical properties are calculated by techniques discussed previously.<sup>3)</sup> In addition to density, density derivatives, and heat capacity, the viscosity and thermal conductivity must be calculated. Suitable subroutines word written to do this. The Fanning friction factor is calculated by the equation presented by  $K\infty^{17}$ , modified slightly:

$$f = 0.0014 + \frac{0.125}{(\text{Re'})^{0.32}} \left(\frac{T}{T_{i}}\right)^{0.5}$$
(5)

where

Re' = modified Reynolds number =  $(dG/\mu)$   $(T/T_{.})$  $\mu$  = viscosity at wall T = bulk absolute temperature T<sub>1</sub> = wall absolute temperature

The rate of heat transfer is based on film theory using the Dittus-Boelter correlation for the heat transfer coefficient:

$$h = 0.023 \frac{k}{d} (Re)^{0.8} (Pr)^{0.4}$$
(6)

where

h = heat transfer coefficient k = thermal conductivity of film Pr = Prandtl number of film Re = Reynolds number of film

The differential equation describing the conversion is derived in the Appendix. The equation is

> $\frac{\mathrm{d}\mathbf{x}}{\mathrm{d}\mathbf{Z}} = \frac{\mathbf{r}}{\mathbf{m}} \mathbf{v}_{\mathrm{c}}$ (7)

AFAPL-TR-70-71

-37-

where

- x = number of moles reactant converted per unit feed
- r = rate of reaction per unit volume of catalyst (kinetic expression)
- **m** = total mass flow rate
- v<sub>c</sub> = volume of catalyst per unit length

The temperatures in the catalyst layer and in the tube wall are calculated accuming that the rate of heat generation or absorption is uniform across each section. With this assumption analytical expressions may be derived (see Appendix) for the temperature rise across each section, and the average temperatures can be estimated. The temperature rise across the catalyst layer is found from

$$\Delta T_{cat} = \frac{q_R^{\dagger} y_2}{2k_c} \left[ \frac{1 - \left(\frac{y_2}{y_1}\right)^2 + 2 \ln \left(\frac{y_2}{y_1}\right)}{1 - \left(\frac{y_2}{y_1}\right)^2} - \frac{q_1^{\dagger} y_2}{k_c} \ln \left(\frac{y_2}{y_1}\right) \right]$$
(8)

And the temperature rise across the tube wall is

$$\Delta T_{wcll} = -\frac{q_{G}^{\dagger} y_{2}}{2k} \left[ \frac{1 - \left(\frac{y_{2}}{y_{3}}\right)^{2} + 2 \ln\left(\frac{y_{2}}{y_{3}}\right)}{1 - \left(\frac{y_{2}}{y_{3}}\right)^{2}} + \frac{c_{d} y_{2}}{k} \ln\left(\frac{y_{2}}{y_{3}}\right) \right]$$
(9)

where

 $\Delta T_{cat}$  = temperature increase from inside to outside of catalyst layer

 $q_{\mathbf{P}}^{\prime}$  = heat flux to chemical reaction, based on inside tube area

 $y_2$  = inside radius of tube

 $k_c$  = thermal conductivity of catalyst

y<sub>1</sub> = inside radius of catalyst layer

qi = heat flux from fluid to catalyst, based on inside tube area

 $\Delta T_{wall}$  = temperature increase from inside to outside of tube

- $q_{d}$  = heat flux of heat generated in tube wall, based on inside tube area
  - k = thermal conductivity of tube wall
- $y_3 = outside radius of tube$
- qj = heat flux from outside of tube to ambient conditions, based on inside tube area

-38-

The average temperatures in the catalyst and the wall are estimated by assuming that the temperature differences can be divided into a generation part (terms involving qd and qd) and a transfer part (terms containing qd and qd). Heat generation leads to approximately parabolic temperature profiles, while heat transferred leads approximately to linear profiles. The average temperatures then are found by using 1/3 of the difference due to generation and 1/2 of the difference due to transfer.

At the present time, the model assumes that there is no diffusional resistance between the bulk fluid and the interior of the catalyst. In the language of catalysis, this assumes external and internal effectiveness factors of one, and can be approached by high fluid velocities and thin catalyst layers.

The calculation of the radial heat fluxes and temperature profiles involves a trial and error procedure as follows:

- 1. The average catalyst temperature is assumed.
- 2. The rate of reaction is then calculated from the kinetic expression.
- 3. The heat absorbed by the reaction can then be calculated.
- 4. The heat flux from the tube wall to the catalyst is found as the difference between the heat generated in the tube wall and the heat lost from the outside of the tube.
- 5. The difference between the heat flux from the tube wall to the catalyst and the heat absorbed by the reaction is the heat flux between the catalyst and the fluid.
- 6. Knowing the temperature of the fluid and the heat flux to or from the fluid, the temperature of the inside catalyst surface can be found using the Dittus-Boelter equation.
- 7. Assuming that the heat absorbed by the reaction is constant radially, the radial temperature profile may be calculated by equation (8).
- 8. Then an improved value of the average reaction temperature can be found by a simple convergence procedure, and the process is repeated.

164 - 16 July 410 - Republication

The key assumptions in this calculation are that the average reaction rate takes place at the average temperature and that the heat uptake oy the reaction is uniform radially. Neither assumption is strictly true. The reaction rate is an exponential function of the temperature so that the average temperature will underestimate the average rate. The other assumption, however, tends to counteract the error. Since the reaction is endothermic, the hotter part of the catalyst, where the reaction occurs faster, is cooled more by the reaction, thereby retarding the rate.

#### Application to Methylcyclohexane Dehydrogenation

Only two experiments have been performed with catalytic wall reactors under realistic flow conditions. The runs were made in the FSSTR and were reported earlier.<sup>3</sup>) Test series 10018-167 was chosen for comparison with the model. The experiments were conducted at three power input levels: 0, 95,000, and 195,000 Btu/hr-ft<sup>2</sup>, all at 860 psig pressure and 930°F fluid inlet temperature. The feed rate was 19.7 1b MCH per hour, which gave a Reynolds number of about 100,000 in the highly turbulent flow regime.

The computer program required the heat losses to ambient conditions, which were obtained from the measured outside wall temperatures during the run and a knowledge of heat losses as a function of temperature, which had been obtained experimentally under no flow conditions. The critical constants of methylcyclohevane and toluene were supplied along with a set of pseudocritical constants for hydrogen:

> $T_{c} = 43.6^{\circ}K$   $P_{c} = 20.2 \text{ atm}$  $V_{c} = 51.5 \text{ cm}^{3}/\text{gmole}$

These constants for hydrogen yield more accurate results than the true ones for corresponding states correlations. The thermal conductivity of the catalyst was estimated to  $b = 0.15 \text{ Btu/hr-ft-}^{\circ}\text{F}$ , based on data of  $\text{Sehr}^{18}$  on similar catalysts. The thermal conductivity of the tube wall was calculated by

$$k = 7.33 + 0.00458 T_{\rm c} \tag{10}$$

where

$$\Gamma_{\rm ev}$$
 = temperature of the tube wall, °F

The kinetic expression used was that obtained from packed bed experiments:2)

$$r = \frac{k_{1}c}{1 + k_{2}c} \left(1 - \frac{p_{T} p_{H}^{3}}{p_{M} K_{e}}\right)$$
(11)

where

r = rate of reaction of MCH per unit volume of catalyst, lb mole/hr-ft<sup>3</sup>
 catalyst
 c = concentration of MCH, lb mole/ft<sup>3</sup>
k<sub>1</sub> = A<sub>1</sub> exp(B<sub>1</sub>/RT)
k<sub>2</sub> = A<sub>2</sub> exp(B<sub>2</sub>/RT)
p<sub>T</sub> = partial pressure of toluene, atm
p<sub>H</sub> = partial pressure of hydrogen, atm
K<sub>a</sub> = A<sub>3</sub> exp(B<sub>3</sub>/RT)

AFAPL-TR-70-71

-40-

R = universal gas constant = 1.987 Btu/lb mole -°R T = absolute temperature, °R  $A_1 = 5.4 \times 10^5 hr^{-1}$   $B_1 = -5000 Btu/lb mole$   $A_2 = 4.5 \times 10^{-8} ft^3/lt mole$   $B_2 = 54,000 Btu/lb mole$   $A_3 = 4.0 \times 10^{20} atm^2$  $B_3 = -92,500 Btu/lb mole$ 

It was found that 50 integration steps were sufficient to obtain an accurate solution to the equations.

The results using the packed bed kinetic parameters are summarized in Figure 10. The conversion of MCH at the three different power levels is shown, as is the outlet fluid temperatures. The experimental values are represented by the discrete points, and the calculations by solid lines. As can be seen, the conversions are calculated much lower than those found experimentally. The fluid temperatures are therefore much higher, since the transferred heat not absorbed by reaction must heat the fluid. The fact that the conversions are calculated too low indicates that either the catalyst temperatures are calculated too low or that the kinetic expression predicts a rate which is too low. The first possibility can be eliminated since the wall temperatures are calculated much higher than those observed experimentally, and as a result the catalyst temperatures are actually predicted too high. Therefore the packed bed kinetic parameters predict too low a rate of reaction. This is probably due to pore diffusional limitations, which are calculated to be appreciable in the catalyst pellets used in the packed bed. The thin layer of catalyst used in the catalytic wall experiments (3 mils) would not show the same diffusional limitations.

Several variations in the parameters  $A_1$ ,  $B_1$ ,  $A_2$ , and  $B_2$  were made to determine their effect on the results. The most reasonable results with the least effort were obtained by increasing the value of  $A_1$  only. A value of 30. x  $10^5$ , rather than 5.4 x  $10^5$ , gave the best agreement with the data. The results are shown in <u>Figure 11</u>. The conversions at the two higher power levels are calculated slightly too high. The outlet fluid temperatures are also too high, indicating a small error in the heat balance. The fluid temperature at zero power level is  $40^\circ$ F high, which is somewhat greater than the others. In general, though the results are encouraging. The lack of heat balance is easily explained by a loss of heat between the points at which the fluid temperatures are measured and the beginning and end of the reactor, which is not included in the calculations. If 910°F is used as the entering temperature, instead of 930°F, the heat balance checks out. <u>Figure 12</u> shows the result for using 910°F initial fluid temperatures. The calculated temperatures and conversions agree quite well with the experiments at the two higher power levels, and are not very far off at zero power.

A comparison between the calculated and experimental outer tube temperatures is shown in <u>Figure 13</u>. The agreement is not excellent, but except for the first few inches the results agree within 40°F. It became apparent during the experimental run that something was wrong with the first 4 inches of catalyst. The outer wall temperatures climbed steadily with time indicating that the cotalyst was deactivating. This could be explained in terms of coke formation on the

AFAPL-TR-70-71

-41-

catalyst; however, the phenomenon affected only the first four inches and apparently did not progress downstream. It was not clear why the apparent lack of activity only occurred in the initial region of the reactor. Part of the answer may lie in an indication that the catalyst was thicker in this region. Outer temperature profiles were obtained during the activation of the catalyst with hot nitrogen gas. The profiles for no input power and a low power level are shown in <u>Figure 14</u>. At no power input the initial temperatures appear to be too low. When power was applied, the temperatures appeared to be too high. This seems to indicate that the catalyst was thicker in the first four inches of length, which would cause at least part of the greater temperature differences observed. However, this apparent lack of activity may also be due to lower partial pressure of hydrogen or poisoning of the catalyst at the inlet end of the reactor. Further experiments are clearly in order to resolve this question.

The fluid pressure was measured to be 860 psig at the end of the reactor for all three power levels. This pressure was calculated by the model to be 853, 851, and 848 psig in order of increasing power input.

### Accuracy of Predicted Reaction Rates

In an effort to check the effect of the several assumptions concerning the temperature profile and rate of reaction in the catalyst layer, a detailed calculation of the radial variations was carried out. A set of conditions at the 195,000 Btu/hr-ft<sup>2</sup> heat flux during experimental run 10018-167 was used. The CWR model gave an average reaction rate of 10,600 lbmole/hr-ft<sup>3</sup> cat. and an average reaction temperature of 911°F. The inner and outer surfaces of the catalyst were calculated to be 892 and 1006°F, respectively. These conditions were determined for a position 0.037 ft from the entrance of the reactor.

The differential equation for the radial temperature profile of the catalyst is

$$\frac{d^2T}{dy^2} + \frac{1}{y} \frac{dT}{dy} = \frac{r\Delta H_R}{k_c}$$
(12)

where

T = temperature of catalyst, °F y = radial distance, ft r = reaction rate, lbmole/hr-ft<sup>3</sup>  $\Delta H_R$  = heat of reaction, Btu/lbmole k<sub>c</sub> = thermal conductivity of catalyst, Btu/hr-ft°F

This equation was integrated numerically using the inner catalyst surface temperature of 892°F and the known heat flux at the surface between the catalyst and the tube wall. The temperature, the reaction rate, and the concentration were allowed to vary during the integration. The results for the temperature profiles are

Figures 10 through 14 follow

-42-



AFAPL-TR-70-71 66758











AFAPL-TR-70-71 66758

shown in <u>Figure 15</u>. As was expected the temperatures resulting from the radially integrated equation gave a flatter profile (solid curve), reflecting the additional cooling in the region of highest temperature. The profile resulting from the analytical solution in the CWR model, assuming uniform heat sink (dashed line), gave more temperature variation and a higher outer surface temperature.

The average temperature of the catalyst calculated by the radial integration is 900°F, and the reaction rate is 9300 lbmole/hr-ft<sup>3</sup>. This corresponds to an error in the CWR model calculation of about 1.2% in the temperature and about 14% in the reaction rate and conversion. The results were obtained by comparison with the experiments at the most extreme conditions we have run. At lower heat fluxes the errors should be less. One solution to this problem would be to integrate radially as well as axially; however, the execution times probably would b ome excessive. Instead, there may be a way to obtain a better estimate of the a.erage reaction temperature without resorting to this two-dimensional approach.

### DEHYDROGENATION OF DECALIN IN THE FUEL SYSTEM SIMULATION TEST RIG

The reactor (3/8" OD x 0.049" wall x 24" long Hastelloy C) used in these tests has been used previously in this program. However, during its last use a leak developed in one of the welds and this had to be repaired. Since several of the attached thermocouples were also damaged, the ceramic coating was completely removed from the tube and all couples were replaced. A sketch of the reactor is given in Figure 16.

The following three test series have been completed using the indicated catelysts charged to the reactor section in the Fuel System Simulation Test Rig (FSSTR).

Series	10018-194	UOP-R	8 catalyst	(ca 0.8%	Pt/	1/16" Al <sub>2</sub> O <sub>3</sub> spheres)
Series	10018-198	Shell	10280-113	catalyst	(ca	4% Pt/ 1/16" Al <sub>2</sub> O <sub>3</sub> spheres)
Series	11644-6	Shell	10860-146	catalyst	(ca	4% Pt/10-20 mesh Al203

The normal operating sequence for these tests was as follows:

- 1) Charge catalyst to reactor and activate by heating in nitrogen for 1 hr at 1100°F.
- 2) Set feed rate at 25.0 lb/hr (LHSV = 545).
- 3) Set inlet pressure at 900 psig.
- 4) Establish inlet temperature at 900°F by preheating feed in two 3/8" OD x 10' long heat exchange sections.

Figures 15 and 16 follow

AFAPL-TR-70-71

-43-

5) Operate long enough with no power supplied to the reactor to establish steady state conditions. (Outlet fluid temperature was ca 720°F under these conditions.)

- 6) Supply sufficient power to reactor to raise cutlet temperature to 800°R. Maintain power constant for ca 1 hr.
- 7) Increase power to reach 900°F outlet and run for ca 1 hr at constant power.
- 8) Increase power to reach 1000°F outlet and run at constant power long enough to establish catalyst activity decline rate.
- 9) Decrease power to bring outlet temperature down to 800°F again and hold long enough to establish final catalyst activity.

When the outlet temperature is changed to a new value in this operating procedure, the initial power requirement is noted and maintained constant for that particular test period. By following this procedure it is possible to tell when the catalyst activity is decreasing, as a decrease in conversion at constant power input results in an immediate increase in fluid outlet temperature. Of course, product analyses also show the decline in activity, but these are not available until well after a run has been completed.

The three catalysts exhibited quite different characteristics. The UOP-R3 and Shell 113 catalysts had similar initial activity up to 900°F outlet fluid temperature, but the Shell 113 catalyst declined in activity so rapidly at this power input that the outlet temperature had increased to 1000°F at the end of one hour. Hence, no further power increase was made. The Shell 146 catalyst not only had much higher initial activity than either of the other two catalysts but also exhibited superior stability. <u>Figure 17</u> shows the initial conversion attained by the three catalysts at outlet fluid temperatures up to 900°F before any deactivation had occurred. <u>Table 25</u> summarizes the conversion and outlet temperature history for the three test runs. Decline in conversion and the accompanying rise in outlet fluid temperature are evident for the three catalysts at power levels sufficient to give 900°F or higher outlet temperatures.

Operating data and product analyses are tabulated and fluid temperatures and decalin conversions are plotted against run time for each of the tests in the following Tables and Figures:

Series 1	10018-194:	Tables	26	and	27.	Figures	<u>18</u>	and	<u> 19</u>
Series 1	.0018-198:	Tables	28	and	29,	Figures	20	and	21
Series 1	1644-6:	Tables	30	and	<u>31.</u>	Figures	55	and	23

Corresponding data for these three catalysts, or their simulants, are given in <u>Tables 32 and 33</u> in bench-scale and MICTR equipment. In the bench-scale runs with decalin the Shell 113 prototype catalyst appeared scalewhat more active and stable than UOP-R8 catalyst, but the Shell 146 catalyst was considerably

-44-





## Table 25. FSSTR: DEHYDROGENATION OF DECALIN IN 2-FT REACTOR, SUMMARY OF TESTS WITH THREE CATALYST FORMULATIONS

•

Reactor: 3/8"	0.D. x 0.049"	wall x 2 f	t long	Hastelloy C
Catalysts: UOP She She	-R8 11 10280-113 11 10860-146	0.8% Pt on 4% Pt on 4% Pt on	1/16" 1/16" 10 <b>-</b> 20	Al <sub>2</sub> O <sub>3</sub> Spheres Al <sub>2</sub> O <sub>3</sub> Spheres mesh Al <sub>2</sub> O <sub>3</sub>
Nominal Inlet Co	onditions	Feed:	33.6%	t-DHN
Pressure Temperature Feed Rate	900 psig 900°F 25.0 1b/hr (545	LHSV)	0.5%	C-DAN THN other

Test Series	Time,	Heat	,	Outlet	DHN	Select	ivity
(Catalyst)	Hr:Min	$\frac{Btu}{(hr/ft^2)}$	<u>Btu</u> 1b	°F	Convin, %	THN	N
10018-194 (UOP-R8)	11:15 - 11:50 $12:05 - 12:50$ $13:10$ $13:40$ $13:50$ $14:15$ $14:40$ $15:00 - 15:35$	-1,400 43,200 106,800 " 139,100 " 22,600	-8 251 620 " 807 " 131	723 801 906 924 1001 1038 1051 795	13.9 32.0 54.5 <b>52.</b> 5 62.4 59.0 57.2 20.1	85.2 63.1 31.5 29.2 17.0 16.1 16.5 67.9	14.8 36.9 68.0 70.2 81.4 82.2 81.8 32.1
10018-196 (Shell 10280-113)	10:35 - 11:05 11:25 - 12:05 12:20 13:20 14:20 14:50 - 15:45	-1,300 47,000 104,500 " 16,300	-8 273 606 " 94	720 805 927 1000 1054 800	14.0 33.6 51.3 43.7 39.1 14.5	83.8 61.4 28.7 21.7 19.6 61.4	16.2 38.6 71.3 78.3 80.4 38.6
11644-6 (Shell 10860-146)	10:35 - 10:45 11:10 - 12:15 12:30 13:30 13:50 14:50 15:50 16:10 - 16:35	-1,300 75,600 150,100 " 176,100 " 38,300	-7 439 871 " 1022 " " 222	709 800 916 942 1009 1035 1050 794	16.9 53.0 78.3 73.3 80.7 78.3 77.3 29.9	89.8 59.9 22.0 19.9 10.4 9.5 8.9 66.5	10.2 40.1 78.0 80.1 89.6 90.5 91.1 33.5

Table 26 and Figure 17 follow

AFAPL-TR-70-71

-45-

## Table 26. FSSTR: DEHYDROGENATION OF DECALIN OVER UOP-R8 IN 2-FT REACTOR, DATA SUMMARY SERIES 10018-194

Reactor No. 10018-49; 0.277" ID x 0.049" Wall x 2 Ft Long Hastelloy C

Feed: 99.5% Decalin, 0.5% Tetralin; 25.0 lb/hr, 545 LHSV, 59,740 lb/(hr · ft<sup>2</sup>)

	L.	Experimental Data							Smoothed and Calculated Data							
1001.94 .001.9.	Fiul	d Temp,,*	Pressur	e, peis	Decalin	Selec	tivity,#	Tube	Wall T.C. '		Tube	Wall Tree	ю. <sup>"</sup> ье)	Huat Fluy.d)	Gamulati	ve Heat*
194-	In	ut	In	Out	Conv'n	to Tetralin	to Naphthalene	Loca	ion	Temp.	length,	Outside	Inside	Btu 10-3	Btu hr	Btu
150	901 Freah	723 catalyat hr. in Ng	900 charge. at 1100*1	884 Activ	15.9 ted	85.2	14.6	U.5 1.5 3 6 9 12 15 18 21 22.5 23.5	Position <sup>D</sup> / B T B T B T B T B T B B T B	*F 842 830 798 769 760 745 744 732 733 730 751	0 2 6 10 14 18 22 24 (0 - 24)	(860) 813 775 753 742 735 730 (728)	(861) 814 774 754 755 756 736 731 (729)	-1.7 -1.6 -1.5 -1.4 -1.4 -1.4 -1.4 -1.4 (~1.45)	C -38 -57 -92 -126 -160 -193 -210	0 -1. -2. -3. -5. -6. -7. -8.
50	905	801	898	866	32.0	63.1	36.9	0.5 1.5 3 6 9 12 15 18 21 22.5 23.5	B T B T B T B T B T B T B	893 837 859 844 843 843 843 843 843 853 849 853	0 2 6 10 14 18 22 24 (0-24)	(905) 868 841 841 843 843 846 850 (852)	(896) 859 832 932 834 834 837 841 (845)	43.1 43.2 43.2 43.2 43.2 43.2 43.2 43.2 (43.2)	0 521 1565 2610 3654 \$699 57\$3 6265	0 20 62 104 146 188 229 250
10	900	. 90 <b>6</b>	899	853	54.5 +	31-5	<b>68.</b> 0	0.5 1.5 6 9 12 15 18 21 22.5 23.5	P B T B T B T B T B	953 950 946 944 956 969 980 980 980 980 980 997 994 999	0 2 6 10 14 18 22 24 (0 - 24)	(956) 949 958 970 983 997 (1003)	(934) 947 924 956 961 975 (981)	106.8 106.9 105.8 106.8 106.8 106.8 106.7 106.7 (106.8)	0 1291 3874 6457 9038 11620 14200 15490	0 51 155 258 361 464 568 619
ю і	90) (Coat	924 invation	8999 of Run 13	853 10)	52.5	29.2	70.2	0.5 1.5 5 6 9 12 15 18 21 22.5 23.5	B T B T B T B T B	955 950 946 743 956 968 982 986 1006 1007 1014	0 2 6 10 14 18 22 24 (0 - 24)	(956) 949 959 974 990 1006 (1015)	(934) 927 924 937 952 968 984 (993)	106.8 106.9 106.8 106.8 106.7 106.7 106.7 106.7	0 1291 3874 6457 9038 11620 11620 15590	0 51 155 258 361 464 567 619
o (	897	1001	899	845	62.4	17.0	81.4	0.5 1.5 3 6 9 12 15 18 21 22.5 25.5	B T B T B T B T B	989 984 992 995 1612 1051 1050 1061 1091 1095 1106	0 2 6 10 14 18 22 24 (0 - 24)	(987) 988 997 1016 1039 1065 1095 (1111)	(958) 959 968 968 1011 1057 1068 (1084)	139.2 139.2 139.2 139.2 139.1 139.0 138.9 138.9 (139.1)	0 1685 5048 8415 11780 15140 18500 20180	0 57 201 336 471 605 739 807
5	900 (Cant	1058 invetine	900 of Hun 35	<b>846</b> 50)	59.0	16.1	₿₽.2	0.5 1.5 3 6 5 12 15 15 18 21 22.5 25.5	8 1 9 T 8 t 8 t 8	995 987 996 996 1016 1058 1061 1078 1112 112k 1140	0 2 5 10 18 22 24 (0 - 24)	(989) 990 999 1020 1050 1082 1121 (1142)	(960) 961 970 992 1082 1055 1094 (1116)	139.2 139.2 139.2 139.1 139.1 139.1 139.0 138.9 138.8 (139.1)	0 1683 5048 8412 13780 15140 18450 20170	0 67. 201. 356 471 605 759 806
	902   (cont	1051 trueitor	900 of Num 15	848 50)	\$7.2	16.3	ð1.8	0.5 1.5 5 6 9 12 15 18 21 22.5 25.5	8 T 8 T 8 T 8 T 8 T 8 T 8 T 8 T 8 T 8 T	902 984 995 998 1014 1058 1063 1082 1120 1156 1150	0 2 10 18 22 24 (0 - 24))	(98J) 905 998 1019 1055 1087 1150 (1152)	(959) 960 969 1082 1060 1195 (1196)	199-2 199-2 199-2 199-2 199-1 199-0 196-9 190-0 (196-1)	0 1685 5088 8013 11780 15130 18650 20170	0 67 201 556 471 605 759 806
e   	910   	<b>מניד</b> . ו	890	8.×	20,1	67.9	<b>≫</b> c-1	0.5 1.5 5 9 12 15 18	6 T 5 T 5 T 5 T	876 866 832 807 801 795 801 795 805 805 811	0 2 6 10 14 18 24 24 24 (0,)	(894) 845 805 796 800 807 807 615 (824)	(86%) 840 900 795 795 802 815 (879)	22.4 22.4 22.6 22.6 22.6 22.6 22.6 22.6	0 277 818 1366 1914 2461 2461 261	0 10. 92. 94. 76. 98. 180.





# Table 27. FSSTR: DEHYDROGENATION OF DECALIN OVER SHELL 10860-146 IN 2-FT REACTOR, PRODUCT ANALYSES FOR SERIES 11644-6

Tine	Pro	Product Composition, fw									
TTWE	t-Decalin	c-Decalin	Tetralin	Naphthalene							
10:35 - 10:452)	51.6	31.8	15.0	1.5							
11:10 - 12:151)	35.7	12.5	31.6	20+5							
12:30 <sup>2</sup> ) 13:30 <sup>2</sup> )	17.7 20.8	5.1 7.1	17.8 15.1	59•4 57•0							
13:50 <sup>2</sup> ) 14:50 <sup>2</sup> ) 15:50 <sup>2</sup> )	15.1 16.3 16.8	5.3 6.5 7.1	9.0 8.0 7.4	70.6 69.2 68.7							
16:10 - 16-351)	41.6	29.5	19.5	9.5							
Feed	33.6	65.9	0.5								

1) Average values over indicated time period. 2) Smoothed data.

Figures 18 and 19 and Table 28 follow

-46-

AFAPL-TR-70-71 66504





20 P

## Table 28. FSSTR: DEHYDROGENATION OF DECALIN OVER SHELL 10280-113 IN 2-FT REACTOR DATA SUMMARY SERIES 10018-198

## Reactor No. 10018-49; 0.27" ID x 0.049" Wall x 2 Ft Long Hastelloy C

Feed: 99. Decalin, 0.5% Tetralin; 25.0 lb/lr, 545 LHSV, 59,740 lb/(hr · ft<sup>2</sup>)

	T	Exportmental Data							Seco	thed and	Calculated Data					
10018-	Fluid 1	'emp.,*F	Press	ure, psig	Decalin	Select	ivity, \$	Tube	Wall T.C.'s		Tube	Well Te	π., • p <sup>e)</sup>	Heat Flux, d)	Cumulativ	E Hant <sup>e)</sup>
198-	Lη	Out	In	Cant	s.	to Tetr_lin	to Naphthalene	Loca Inches <sup>2)</sup>	tion Position <sup>b</sup>	Teap. F	In.	Outside	Inside	(hr ft <sup>E</sup> ) x 10 <sup>-5</sup>	<u>Btu</u> hr	<u>9tu</u> 16
1100	906 (Frea 1	726 h Gataly hr in H	900 st Char g at 11	685 ge. Acti go'r.)	14.0 Ivetad	83.8	16.2	0.5 1.5 3 6 9 12 15 18 21 22.5 23.5	B T B T B T B T B	850 849 800 743 743 751 751 751 751 751 751 751	0 2 6 10 14 18 22 24 (0 - 24)	(895) 821 775 754 741 733 729 (728)	(89%) 822 77% 755 742 754 750 (789)	$\begin{array}{c} -1.6\\ -1.5\\ -1.3\\ -1.3\\ -1.3\\ -1.3\\ -1.2\\ -1.2\\ (-1.31)\end{array}$	0 -18 -52 -84 -115 -145 -175 -190	0 -0.7 -2.1 -3.4 -4.6 -5.8 -7.0 -7.6
1200	895	805	898	865	33.6	61.4	38.6	0.5 1.5 3 6 9 12 15 18 21 22.5 23.5	8 T U T 8 T 8 T 8 T 8 T 8	900 898 856 856 859 856 850 857 854 855 855 855	0 2 6 10 "4 18 22 28 (0 - 24)	(924) 883 856 849 848 850 850 854 (856)	(914) 875 846 879 838 840 844 (846)	46.9 47.0 47.1 47.1 47.1 47.1 47.1 47.1 47.1 (47.0)	0 568 1704 2842 5980 5117 6255 6824	0 22.7 68.2 113.7 159.2 204.7 250.2 272.9
1220	<b>903</b>	927	896	<b>65</b> 2	51.3	28.7	72-3	0.5 1-5 3 6 9 12 15 18 21 22.5 23.5	B T B T B T B T B T B	974 967 956 956 958 968 980 980 990 1007 1020 1023	0 2 6 10 14 18 22 24 (0 - 24)	(989) 962 956 961 974 993 1014 (1026)	(968) 940 934 939 959 952 972 993 (1005)	104.4 104.5 104.5 104.5 104.4 104.4 104.4 104.4 (104.5)	0 1265 3790 6516 8842 11370 13690 15150	0 50.5 151.6 252.6 353.7 454.7 555.7 606.1
1320	894 (Canti	1600 musiica	896 orRun	852 1270)	<b>43.</b> 7	21.7	<b>،</b> ه	0.5 1.5 5 9 12 15 18 21 22.5 23.5	BT BT BT PT PT PT PT	968 965 958 962 975 986 1008 1029 1059 1078 1087	0 5 10 14 18 22 24 (0 ~ 24)	(975) 961 962 976 1000 1092 1072 (1092)	(951) 959 940 954 979 1011 1051 (1072)	104.5 104.5 104.5 104.5 104.5 104.2 104.2 104.2 104.4	0 1253 3790 6516 8840 11360 13890 15140	0 50.5 151.6 252.6 353.6 454.5 555.4 605.8
1950	897 (Conti	2054 nixition	896 of Run	852 1290)	39-1	19.6	80.4	0.5 1.5 5 9 12 15 18 21 82.5 23.5	9 T B T B T B T B T B T B	974 969 955 970 987 100 <sup>4</sup> 1059 1059 1059 1125 1134	0 6 10 14 18 24 (0 - 24)	(981) 965 970 991 1021 1065 1115 (1140)	(959) 944 970 1000 1050 1050 1050	104.5 104.5 104.4 104.4 104.5 104.1 104.1 104.1 (104.4)	0 1263 9789 6314 8836 11360 13880 15140	0 50.5 151.6 252.6 353.5 454.4 555.2 605.5
1540	895	800	895	868	14-5	<b>6</b> ].4	<b>38.6</b>	0.5 1.5 5 6 9 12 15 18 81 22.5 23.5	5 T B T B T B T B T B T B T B T B T B T	372 868 935 810 800 795 800 001 814 817 817	0 6 10 14 18 20 24 (0 - 24)	(920) 851 610 798 797 804 816 (124)	(917) 6-8 807 795 798 301 815 (821)	16.0 16.2 16.3 16.5 16.5 16.5 16.5 16.5 (36.5)	0 195 587 981 1375 1769 2162 2359	0 7.8 23.5 59.2 55.0 70.8 66.5 94.3

a) T.C. is epobledized to outside well at indicated nonloss from inlet and bus bar.
 b) Konstian of T.C. junction on horizontal tube. B = Sotton, 7 = Yop.
 c) Othiside well tamperstares by emotioning apprimental data. Inside tamperstures by calculation.
 c) Othiside for houses. Walkers in ( ) are average over estars has a length.
 c) Not heat to fluid up to indicettal tube length.

AFAPI\_TR-70-71 66758

# Table 29. FSSTR: DEHYDROGENATION OF DECALIN OVER SHELL 10280-113 IN 2-FT REACTOR, PRODUCT ANALYSES FOR SERIES 10018-198

Time		Product Composition %									
1 Line	t-Decalin	c-Decalin	Tetralin	Naphthalene							
10:35 - 11:051)	43.0	43.2	11.7	2.1							
11:25 - 12:051)	35.7	31.6	20.5	12.2							
12:20 <sup>2</sup> ) 13:20 <sup>2</sup> ) 14:20 <sup>2</sup> )	26.1 26.8 27.2	24.0 30.9 35.0	15.0 9.8 8.0	34.9 32.5 29.8							
14:50 - 1 <b>5:</b> 45 <sup>1)</sup>	35.8	50.0	9.0	5.2							
Feed	33.6	65.9	0.5								

1) Average values over indicated time period. 2) Smoothed data.

Figures 20 and 21 and Table 30 follow

AFAFL-TR-70-71

## Table 30. FSSTR: DEHYDROGENATION OF DECALIN (WER SHELL 10860-146 IN 2-FT REACTOR, DATA SUMMARY SERIES 11644-6

Reactor No. 10018-49; 0.277" ID x 0.049" Wall x 2 Ft Long Hastelloy C

## Feed: 99.5% Decalin, 0.5% Tetralin; 25.0 lb/hr, 545 LHSV, 59,740 lb/(hr · ft<sup>2</sup>)

					Exper	Experimentel Data			Smoothed and Calculated Data				a			
Rum No.	Fluid Te	., ° 7	Prese	re, peig	Decalin	Selec	tivity, \$	Tube	Wal :		Tube	Wall Tw	anp., ⁺F <sup>℃</sup>	Heat Flux, d)	Cumulet	(ve Heat")
11644- 6-	În	Out	ໂກ	Out	¢ tonv•n	to Tetralin	to Maphthalene	Loc	ation Posit	emp. F	Length, In.	Outside	înside	Btu (hr · ft <sup>2</sup> ) x 10 <sup>-5</sup>	Btu	Bty 1b
1045	906 (Prust 1	709 Cataly hr in l	900 rst Clur te at 13	825 gw. Ast. 00°F.)	16.9 Lvæted	89.8	10.2	0-5 1-5 3 6 9 12 15 18 21 22.5 25-5	B T B T B T B T S T S	74 34 22 23 715 717 71	0 2 3 10 14 16 22 24 (-24)	(900) 782 785 730 722 718 718 716 (716)	(901) 783 746 731 725 719 717 (717)	-1.6 -1.4 -1.3 -1.2 -1.2 -1.2 -1.2 -1.2 -1.2 (-1.26)	0 -17 -49 -80 -109 -159 -159 -168 -183	0 -0.7 -3.2 -4.4 -5.6 -6.7 -7.3
1200	900	800	900	795	r.o	59.9	<u>لەر</u>	) 6 9 12 15 18 21 22.5 23.5	B 5 5 7 8 7 8 7 8 7 8	858 857 871 871 871 872	0 2 10 12 22 24 (0 - 24)	(980) 882 845 845 847 847 857 871 (878)	(965) 866 828 825 850 850 855 (862)	75.3 75.5 75.6 75.6 75.6 75.6 75.6 (75.6	0 912 2740 4568 6396 8224 10050 10970	0 36.5 109.6 182.7 255.9 329.0 402.1 438.6
1230	904	916	902	742	78.5	22.0	<b>78.</b> 0	10.5 1.5 5 6 9 12 15 18 21 22.5 23.5	B T B T B T B T B T B	1019 980 960 941 940 551 976 984 1014 1025 1031	0 2 6 10 14 18 22 24 (0-24)	(1050) 973 942 943 959 985 1019 (1053)	(1020) 942 910 921 927 954 988 (1008)	149.9 150.1 150.2 150.2 150.1 150.1 150.0 149.9 (150.1)	0 1813 5443 9075 12700 16350 19960 21770	0 72.5 217.7 362.9 508.1 653.2 798.3 870.8
1.330	599 (Conti	942 Duation	902 of Run	761 1230)	73.3	19.9	80.1	0.5 1.5 5 9 12 15 18 21 22.5 23.5	B T B T B T B T B T B	1015 979 960 942 941 955 983 998 1033 1048 1055	0 2 6 10 14 18 22 24 (0 - 24)	(1050) 973 942 946 967 1000 1041 (1064)	(1020) 942 910 914 936 969 1011 (1034)	149.9 150.1 150.2 150.1 150.0 149.9 149.9 149.9	0 1813 5443 9073 12700 16350 19950 21770	0 72.5 217.7 562.9 508.1 653.2 798.2 870.7
1350	899	1009	903	817	30.7	10.4	89.6	0.5 1.5 3 9 12 15 18 21 22.5 23.5	BTBT&TB TBTBTB TB TB TB TB TB TB	1046 1003 991 973 973 973 973 973 973 990 1027 1048 1092 1114 1114	0 2 6 10 14 18 22 24 (0-24)	(1070) 997 973 978 1005 1049 1107 (1159)	(1035) 961 936 941 963 1014 1075 (1105)	176.0 .76.2 176.2 176.2 176.1 175.9 175.8 (176.1)	0 2129 6363 10659 14910 19170 25420 25550	0 85.1 255.5 826.0 596.3 766.6 936.8 1021.8
1450	900 (Conti	1035 numition	903 of Rua	715 1350)	78.3	9•5	90.5	0.5 1.5 3 6 9 12 15 18 21 22.5 23.5	B T 9 1 8 1 8 1 8 1 8 1 8 1 8 1 8 1 8 1 8 1	1048 1005 992 975 977 977 998 1058 1058 1058 1058 1155 1148	0 2 6 10 14 18 22 24 (6 - 24)	(1070) 1001 974 984 1013 1062 1124 (1160)	(1035) 965 957 947 977 1027 1090 (1127)	176.) 176.2 176.2 176.2 176.0 175.9 175.9 (176.1)	0 2129 6388 10650 14910 19160 25406 25540	0 85.1 255.5 825.9 825.9 596.3 766.6 936.7 1021.7
1550	901 (Cansi	1050 nustion	903 of 8uan	718 1550)	π.,	<b>б.</b> 9	91.1	0.5 1.5 5 9 12 15 18 22.5 25.5	8 T 8 T 8 T 8 T 8 T 8 T 8 T 8 T 8	1049 1007 997 982 982 1005 1045 1045 1045 1045 1121 1150 1121	0 6 10 14 19 22 24 (0 - 24)	(1070) 1005 989 989 1019 1070 1140 (1179)	(1035) 967 944 952 965 1055 1106 (1146)	(76.0 176.2 176.2 176.1 176.0 175.8 175.7 (176.1)	0 - 2129 - 6588 - 10550 - 14910 - 2580 - 2580 - 2580 -	0 85,1 825,5 425,9 596,5 766,4 956,6 1001,6
1550	900	<b>4</b> 67	901	8gr,	29.0	<b>tx6.</b> 5	55.5	0.5 1.1 5 6 9 12 15 18 21 22,5 (3.5)	М Т И Т И Т И Т И Т И Т И Т И Т И Т И Т	0.48 876 176 765 765 765 765 805 805 850 850 850	0 2 14 18 29 29 29 29 20	(952) Bh5 T35 T35 T95 T95 B10 B20 (Bh5)	(444.) 255 784 778 706 802 802 821 (3%7)	581.0 2 581.0 2 581.5 5 581.5 5 581.5 1 581.5 1 581	1000 1000 1000 1000 1000 1000 1000 100	0 18.4 55.5 52.6 196.7 196.7 196.7

a) T.C.'s epothwided to outside well at indicates insumes from inlet and hum har.
 b) incestion of T.C. junction on horizontal tube. B - Botkin, T - Toy.
 a) -biddle will take returns by monthing experiments) data. for fir take writtens by "simulation" of Corrected for lasses. Values to () har every over entire heaven langth.
 e) Not heat to fiuld up to indicated tube length.



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## Table 31. FSSTR: Di HYDROGENATION OF DECALIN OVER UOP-R8 IN 2-FT REACTOR, PRC DUCT ANALYSES FOR SERIES 10018-194

Ties		Product Composition, #w										
1 HARE	t-Decalin	c-Decalin	Tetralin	Naphthalene	Unknown							
11:15 - 11:501)	43.4	42.9	11.8	1.9	0							
12:05 ~ 12:50 <sup>1)</sup>	36.7	32.1	20.1	11.1	0							
13:10 <sup>2</sup> ) 13:40 <sup>2</sup> )	26.0 26.6	20.9 22.3	17.4 15.6	35.4 35.2	0.3 0.3							
13:50 <sup>2)</sup> 14:15 <sup>2</sup> ) 14:40 <sup>2</sup> )	21.1 22.0 22.6	18.0 20.5 21.7	11.0 9.9 9.8	48.9 46.6 44.8	1.0 1.0 1.0							
15:00 - 15:35 <sup>1</sup> )	38. <u>8</u>	41.5	13.6	6.0	0							
Feed	33.6	65.9	0.5									

1) Average values over indicated time period. 2) Smoothed data.

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Migures 22 and 23 follow

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-63**-**-

AFAPL-18-70-71





## Tuble 32. DEHYDROGENATION OF DECALIN OVER VARIOUS CATALYSIS

Feed: F-115 DHN Feed Composition: 25.0% trans DHN 74:5% cis DHN

0.4% THN

Pressure: 10 atm <u>LHSV:</u> 100 <u>Reaction Period</u>: 30 minutes

Reactor: Bench Scale, 5" bed in 1/2" IFS SS tubing furnace heated.

		% DHN	Conversi	on at,	°F	$\Delta T$ ,	Fof	Cataly	st Bed	at,°F
Catalyst	842	932	1022	1112	1202	842	932	1022	1112	1202
10860-146	43.7	58.3	62.2	71.2	84.1 <sup>a</sup> )	3	5	13	18	66 <sup>a)</sup>
uop-r8	36.2	43.9	47.7b)	-	-	14	43	160 <sup>b)</sup>	<i>a</i> .	-
10280-107B <sup>c)</sup>	39.1	46.7	54.2	61.5	66.5	11	25	34	56	160
10280-91A <sup>c)</sup>	39,0	46.1	54.9	61.6	68.8	13	22	36	50	205

a) Reaction time: 10 minutes.

b) Catalyst completely deactivated at the end of this run.

c) 10280-113 prototype.

## Table 33. ACTIVITIES OF CATALYSTS 10280-113 AND 10860-146 FOR METHYLCYCLOHFXANE DEHYDROGENATION AT SEVERAL TEMPERATURES

Conditions: MCH, LHSV 100, 10 atm pressure, furnace heated Reactor: MICTR 4-1/4" bed in 1/4" O.D. S.S. Tubing, catalyst diluted 0.9/1.1 with quartz chips

Catalyst No.	% Pt	Support	,	Average Conversion of MCH to Tolmene, fw						
		Туре	Mesh Size	Run No.	662	752	842°F			
10860-70	1	UOP-R8 type A1203	~10	1166	24	45	71			
10280-113	4	1	9-16	1108	20	43	69			
10860-146	4	1	<b>10-2</b> 0	1177	<i>5</i> 3	59	86			

better than either the UOP-R8 or Shell 113 cetalysts. Thus the FSSTH data confirms these finding in general. The lesser activity/stability of the Shell 113 catalyst compared to UC -R8 catalyst in the FSSTH compared to the results with the prototypes may be due to some misadventure in the makeup of the large batch prepared for FSSTH evaluation; this will be rechecked. In the MICTH the comparison involves MCH rather than decalin, but the order of activity bears out the results found in the FSSTH with decalin.

## THERMAL STABILITY OF FUELS

Thermal stability st dies have progressed along several lines during the past year. One of the chief advances has been in the completion of the prototype beta-ray backscatter rig for measuring deposits. This device, although still undergoing evaluation and testing, has been used successfully on standard ASTM, SD/M-7, and JFTOT Coker tubes. It has also been used in measuring the deposit thicknesses on titanium strips from experimental fuel tanks at Boeing Aircraft Company.

The characterization of candidate fuels requires the development of suitable apparatus to characterize routinely the behavior of the fuel at high temperature and pressure in both the absence and presence of endothermic reaction. To provide a method of so characterizing fuels, we developed and put into operation during the last contract period a piece of equipment, the Catalyst and Fuel System Test Rig (CAFSTR), capable of evaluating a fuel/catalyst combination to a maximum temperature of about 1300°F. However, a major difficulty in the utilization of this apparatus was the determination of the amount of deposit on the heating tubes, which, because of the high temperatures involved, could not be fabricated of aluminum and rated visually as is done with tubes from the ASTM Coker. Now that the beta-ray backscatter device is available, we shall be able to start fuel evaluations with the CAFSTR, in which Incomel 600 heater tubes are used. Because of color changes of the metal itself, color rating of the Incomel tubes is impossible and deposits from this equipment could not be evaluated in the past.

Further progress in thermal stability testing has been achieved, in that the JFTCT fuel tester is now complete and operative, and a number of tests have been made. The rig has been found to be delightfully simple and rapid to use, but does seem to have a high temperature limitation. This can probably be solved, however.

No further changes have been made on the SD/M-7 fuel coker, except that a Beckman Oxygen Analyzer has now been installed and is functioning properly.

Various thermal stability investigations have been made including brief studies of hydrocrackate fuels and current commercial Jet-A type fuels. In addition, we have been exploring a new class of potential additives for improving thermal stability of fuels. Several additives have been found to be effective, although tests to date have shown that they are beneficial only in fuels containing dissolved oxygen, the benefits from the additive being substantially the same as those from nitrogen sparging of the fuel. This suggests, of course, that the additives directly interfere with the degradation mechanisms in which oxygen is a participant.

-50-

### Measurement of Deposits by Beta-Ray Backscatter

After a broad investigation of possible ways to measure fuel coker deposits quantitatively, a prototype beta-ray backscatter test rig was built for this purpose. This equipment<sup>2</sup> has been completed to an operational stage, and its characteristics and capabilities are now being evaluated. A photograph of the apparatus in its present state of development is shown in <u>Figure 24</u>. A description of the equipment appears in the Appendix.

Initially, the test rig was set up partially from ordered commercial and shop produced components, plus some borrowed components, including a vacuum pump and a scanning mechanism drive motor. These borrowed parts have now been replaced with a large capacity vacuum pump to provide rapid pumpdown, and a suitable drive motor, speed control and gear reduction arrangement to permit scanning of different sized tubes. In addition, the original temporary detector, devised by modifying one which had been used for other purposes, has been replaced by one designed specifically for this instrument, and embodying improvements based on experience with the first. A commercial detector which was ordered while the temporary one was used has finally arrived. Although this detector is no longer needed, we plan to test its operation in comparison with the newly designed one. However, we are not optimistic about the commercial unit, since it has a window thickness of approximately 2500 Å. Since it was ordered, we have determined that the maximum window thickness to avoid loss of sensitivity is about 1500 Å.

Our currently installed window has a thickness of approximately 1000 Å and has been in use 24 hours per day for five weeks without leakage. The differential pressure across the window, which is supported by a 280 line-per-inch ickel screen, is essentially one atmosphere, and the vacuum system holds readily at a few millimicrons Hg pressure. This clearly demonstrates that there are no holes in the window. However, the window has been a problem. If the window thickness is too small, the window either breaks or develops holes; if it is too thick, it cuts out the low energy electrons required for measurement of deposit thickness. We originally used a cellulose nitrate meterial which was on hand in the laboratory, but windows cast from this material, when sufficiently thin, proved too fragile and short-lived. The window material that we had planned to use (Onion Carbide PARYLENF® film) was too thick (2600 Å). We have now received a thinner film (1000 Å PARYLENE<sup>®</sup> film), which we plan to install later. While we were waiting for delivery of the thin film, we successfully cast a 1000 Å film of PARLODICN<sup>®</sup> (also a cellulose nitrate material), which we are now using. Although the PARYLENE® material is believed to be stronger, the present window is performing satisfactorily and will not be replaced until necessary. The present arrangement of window film and wire screen permits about a 70 percent transmission of electrons, which is considered adequate.

A considerable amount of effort has been expended in the evaluation of this equipment and is still continuing. The effects of vacuum level, detector time constant, rotational-translational tube speed, detector head positioning, chart speed, pen response time, and a number of other variables have been under investigation.

a) A. Telfer and R. M. Curtis of our Analytical Department were responsible for the design, construction and preliminary testing of this equipment.

Figure 24 follows

AFAPL-TR-70-71

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As was noted previously<sup>3)</sup>, it is possible to reach saturation for any given electron energy level, and hence to reach a maximum deposit thickness beyond which readings will be inaccurate. If thicker deposits are to be measur 4, a dual set of detectors and sources might be required to be assured that true deposit thicknesses are being measured. In addition, the span of the current 5 mv single channel 5-inch strip recorder is so narrow that it cannot read out the signal from all deposit levels at the same sensitivity setting. This often requires multiple runs at different sensitivities in order to measure the entire deposit profile. This could be corrected by utilizing a 1 mv 10-inch strip recorder with dual channels. This would increase the sensitivity, and the use of the two channels a decade apart would enable us to handle a greater range of thicknesses.

We have found that wobble or surface unevenness of tubes can cause baseline fluctuation, and experimentation has shown that this is observable for steps as little as 0.0001 inch. This is not due to differences in air thickness, but rather to the geometric characteristics of the beta-ray collimetor. This problem can be overcome, however, by cleaning deposit from nerrow band. spaced along the strip to provide a reference level of backscatter.

Although the beta-ray backscatter equipment has given reproducible results, a satisfactory method of calibration which relates backscatter values to the actual thickness of the deposit is still being pursued. For our purposes, such calibration, while intellectually satisfying, is not actually necessary as long as we compare deposite on tubes of the same composition. The output of the device is assumed to be proportional to the number of about of material on the tube under examination, if the average atomic number does not depart too seriously from that of carbox; this requires a low concentration of higher atomic number elements. This will generally be the case.

Collibration of the instrument thus far has been attempted with nitrocellulose films wrapped around an aluminum coker tube. These films have been separately east and then taped to the tube for rating by the backscatter equipment. The film is then removed and the thickness of it measured independently by a microscopic light diffraction technicue. Roughly, we have found by using this method that 1 inch on the strip chart corresponds to a thickness of 550 Å on an aluminum tube. However, the film so applied has always been wrinkled and nonuniform; hence, the search for an accurate method of calibrating the instrument is still underway, and an absolute calibration was not available in the earlier work. For that reason, deposit measurements are reported here in nondimensional units, but are approximately equal to thicknesses in Angstroms.

As a part of the everall calibration problem we needed a calibration method for deposits on different substrate metals. To implement this investigation, a calibration rod of 1/8-inch OD has been constructed which consists of sections of the following materials: bress, silver, eluminum, cadmium, cobalt, zinc, iron, tin, titanium, magnesium, carbon, and TEFLON<sup>®</sup> rod. By scanning this rod we obtain a series of steps on the recorder due to the different backscattering efficiencies of the materials of the rod. A 1000 Å PARYLENE<sup>®</sup> film then is wrapped onto the tube under tension, thus avoiding the previous wrinkling problem. The tube is then rescanned to obtain the calibrations for deposit thickness. An additional advantage of the multimaterial tube is that it will be available for restandardizing the tube when it becomes necessary to change windows, or to make regular calibration checks in the event of drift in the electronic equipment.



SETA-RAY BACKSCATTER INSTRUMENT FOR MEASURING COKE DEPOSITS Figure 24.

AFAPL-TR-70-71 66701

Calibration with other pure hydrocarbon films is planned, although calibration with nitrocellulose is not considered to introduce a serious error, since the difference in the beta-ray backscattering by nitrogen and oxygen and the backscattering by carbon is not expected to introduce a significant error. However, this will be checked.

#### Boeing Titanium Strips

We have completed an evaluation of the tank deposit strips that were sent to us by Mr. George Hays of Boeing Aircraft Company. Results of these surveys are plotted as deposit profiles in <u>Figures 25-28</u>, where deposit thickness in A° (approximate) are plotted versus distance along the strips. The narrow vertical dips shown are due to the fact that divider strips were welded and cemented to the flat titanium sheets, from which the fuel tanks were constructed, and were then removed. The greatest thickness of deposits was in the region of the divider strips, and this suggests a possible interaction of fuel with the cement or sealant, or an electrostatic attraction of deposit towards the dividers. The thickness of the sealant is indicated by the ends of the dips and ranged from an apparent 150 to  $800 \text{ Å}^\circ$ .

In order to compensate for the departure from flatness of the strips, the deposit was periodically removed by mild abrasive action, although this is not shown on the deposit profiles. By assuming that the minimum deposit thickness midway between divider strips represented the true unhindered depth, approximate dashed curves have been sketched in. It can be seen from the figures that apparent deposit thicknesses ranged from about 500 to 1400 A°. Calibration was made with a 1350 A° nitrocellulose film on cleaned titanium strips.

An effort was made to check these results by cutting two sections 2.5 cm in length with apparently uniform deposits from one of the strips. One of these areas measured 1200 A° and the other 1400 A° by beta-ray backscatter. These were then analyzed by combustion for carbon and hydrogen and a clean, freshly abraded sample was also analyzed for background impurities. Great care was taken to prepare these specimens so as to avoid contamination, and the edges and backs were filed clean with a fine file. The samples were then rinsed with normal heptane and dried in a vacuum oven for two hours at 150°C. The amount of carbon and hydrogen determined for the deposited samples, however, was much higher than would be expected from an oxidized hydrocarbon film of the thicknesses indicated by beta-ray backscatter, calculated densities being about 4.0 and 6.1 gm/cm<sup>3</sup>. This could have been the result of the inclusion of substantial amounts of oxides of heavy elements in the coating, since they would scatter more effectively than titanium, and would make the deposit appear thinner to the instrument. However, scanning the strips with the IEES instrument (Varian Corporation) indicated that no large concentration of elements existed in the film other than carbon, hydrogen, and oxygen. At the present time the most reasonable explanation for this phenomenon is either that the original calibration of the instrument was at fault or that the deposit retained substantial amounts of the normal heptane with which it was washed, in spite of being dried in a vacuum oven for two hours at 160°C. This will be checked further.

Figures 25, 26, 27 and 28 follow

-53-

#### Alcor JFTOT and SD/M-7 Coker Tubes

A few aluminum and stainless steel tubes used in our Alcor JFTOT unit have been examined both in the Tuberator and with the beta-ray backscatter device.

A distinct advantage of the beta-ray backscatter method is that it enables one to know when an observed color is not a true deposit, but rather the color of the metal itself. This was demonstrated with a stainless steel Alcor JFTOT tube having a faintly yellow tinted area which one might have rated a code 1-1/2. Nevertheless, the area still looked like bare metal, and indeed the beta-ray backscatter scan showed no difference between the tinted and the original metal colored areas. The beta-ray backscatter readout was constant for this entire tube.

However, a stainless steel Alcor JFTOT tube having an apperent code 2-1/2 rating, and giving the impression of being a true deposit overlay, gave a definite beta-ray backscatter count indicative of a deposit, as shown in <u>Table 34</u>. Here only the maximum visual code rating was taken, and then only on the side with the heaviest deposit. The beta-ray backscatter readout was, of course, on the entire tube, and shown in <u>Table 34</u> is the range of thicknesses for both the heaviest and lightest deposits on opposite sides of the tube. The deposited area was about 1-1/4 inches long, but no effort was made to reduce the code ratings into shorter lengths, since most of the deposited area was the maximum code color, with lighter color around the fringes. This is rather typical of JFTOT deposit appearances.

Tube Designation		AS'IM Code Tube Ratings											
	1	2	3	4	5	6	7	8	9	10	11	12	13
JFTOT Stainless Steel <sup>a)</sup>	(	5	-1/2	1		0	0	0	0			{ 	
SD/M-7 Coker Run 525 <sup>b)</sup>	1	1	1		1/2	1/2	1/2	1/2	0	0	0	0	0
	Thi	cknes	s Renge	e by	Beta-F	 юу В	achse	atter	, Å	l			
JFTOT Stainless ${ m Steel}^{a)}$		( 1	40-700	/480-	.340	)	(no	depo	sit	; )			
SD/M-7 Coker Run 525 <sup>b</sup> )		160- 175	140- 160	50- 116	<b>3</b> 5~ 50	55 50	ης β0	≏. 22		( n	o de	post	ts)

#### Table 34. COMPARISON OF ASTM CODE AND BETA-RAY BACKSCATTER RATINGS FOR ALCOR JETOT STAINLESS STEEL AND SD/M-7 COKER TUBES

a) per cm.

b) per inch.

-54-

AFAP: TR-70-21









AFAPL-TR-70-71 66504

One should not take the deposit thicknesses shown as being precise, since calibrations on stainless steel had not been made when this tube was measured. Moreover, since bare metal backscatter is a function of true metal composition, true calibration must be done on the same metal composition as that on which the deposit is measured.

The ACTM code versus the beta-ray backscatter ratings of a lightly deposited SD/M-7 coder tube deposit is also shown in <u>Table 34</u>. Actually, the areas shown do not correspond exactly, the area "1" for the backscatter ratings starting about an inch further away from the fuel outlet point than that of the ASTM code. One has to keep in mind this point in studying the other tube rating comparisons; the starting points are not necessarily the same. However, the regions in which deposits are located are in approximate agreement, the beta-backscatter results being much more discriminating than the visual code ratings.

The scanning width of the beta-ray backscatter instrument is slightly greater than 1/8 inch, since the radiation source slit is 1/8 inch wide and about 1/8 inch from the nearest point of the tube surface. The beta-ray backscatter readout is an average measurement of deposits for an area about  $1/8 \times 1/8$  inch. Actual deposit boundaries can be detected accurately, but the thickness at the boundary cannot be determined precisely because of the slit width. Alcor JFTOT tubes have a diameter of only 1/8 inch; hence, the backscatter measurement is influenced by tube diameter, or surface curvature. Consequently, for careful work calibrations would be necessary not only for every tube material, but for different tube diameters and for flat surfaces.

#### CRC Erdco JFTOT Preheater Tubes

A series of ten Erdco JFTOT preheater tubes from the CRC JFTOT evaluation program were supplied to us and rated by both the beta-ray backscatter method and our own laboratory ASTM Tuberator. The results of these ratings together with the average values of the ASTM code ratings from the panel evaluations and the results with the Erdco Reflectance Rater are tabulated in <u>Table 35</u>, and represented graphically (except for the values for the two most heavily fouled tubes) in <u>Figures 29</u> and 30.

These tubes are 5/8" OD and 8-1/2" overall length. All ratings are given as the maximum values within one-inch long sections of the tube, numbering from the fuel outlet end. Actually the beta-ray backscatter readout was in the form of a sine way, because the deposits were situated mainly on one side of each tube and the tube rotates and translates during the rating procedure. However, for the the present comparison we have simply given the maximum rating or deposit thickness in A for each inch of the tube length. In the majority of cases, the geographical distribution of the deposits are found to be about the same by all methods of rating. That is, the deposits are heaviest near the exit end, although there are a couple of exceptions to this rule as shown with Tubes 10 and 2. Probably the two most interesting cases are tubes 7 and 15, which by both our own ratings and the CRC average fall in the critical region of code 2 to 3. Here the results with the beta-ray backscatter apparatus suggests that Tube 15 should fail whereas Tube T would probably pass, while the measurements with the Erdco Reflectance Rater would lead to the opposite conclusion. Although the Erdco Rater seems

AFAP1-TR-70-71

Tube	Tuberator	Ratings	FRDCO	Beta Ray 。
Inches	CRC Ave.	Snell	Rater	Backscatter, A
	Tube No. 8	5.2, P. and 1/2" x 5/8"	W. No. 56 <u>Tubes</u>	
0-1 1-2 2-3 3-4 4-5 5-6	1.5 1 0.5 0.5 0.5	0.5 0 0 0 0	0.04 0.02 0.01 0.01 0.01 0.01	30 0 30 80 110 360
2.0	Tube No	5. 3, P. and (8 1/2" x 5	W. No. 34 /8")	5.0
1 2 3 4 5 8	2 3 1 1 0.5	3 5 4.5 0.5 0.5 0.5	0.08 0.17 0.08 0.03 0.02 0.02	820 1130 580 none none none
	Tube No	. 4, P. and	W. No. 138	
1 2 3 4 5 6	2.5 3 2.5 1 0.5	3 6.5 6.5 0 0 0	0.15 0.21 0.21 0.04 0.03 0.02	1290 1790 1210 220 0 0
	Tube No	. 7, P. and	W. No. 38	
1 2 3 4 5 6	2.5 2.5 1.5 1 1 0.5	1.5 1 0.5 0.5 0 0	0.16 0.11 0.08 0.04 0.02 0.02	680 440 220 130 30 0
1 2 3 4 5 6	<u>Tube No</u> 1 1.5 1 1 1 1	10, P. and 1.5 1.5 1.5 0.5 0 0	W. No. 80 0.06 0.07 0.07 0.06 0.04 0.04	30 0 60 140 140 80

# Table 35. COMPARISON OF BETA RAY BACKSCATTER PATINGS OF CRC ERDCO JFTOT TUBES WITH THOSE FROM THE ASTM TUBERATOR AND THE ERDCO REFLECTANCE RATER

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AFAPL-TR-70-71

~56-

Tube	Tuberator	Ratings	ERDCO	Beta Ray 。
Inches	CRC Ave.	Shell	Rater	Backscatter, A
Derys	<u>Tube No. 12</u>	, P. and W	. No. 90	n an
1 2 3 4 5 6	2 1.5 1 1 0.5 0.5	0.5 0.5 0 0 0 0	0.08 0.07 0.03 0.03 0.02 0.02	580 190 0 0 0 0
	Tube No. 14	P. and W	<u>No. 118</u>	
1 2 3 4 5 6	2.5 2.5 2 1 1 0.5	5 4 2 0.5 0 0	0.13 0.14 0.06 0.03 0.02 0.01	1400 1140 470 30 0 0
	Tube No. 15	P. and W	No. 137	
1 2 3 4 5 6	3 2.5 2 1 1 0.5	2 1.5 1.5 1 0.5 0	0.11 0.09 0.05 0.04 0.03 0.02	1620 1260 910 600 360 220
	Tube No. 17	P. and W	No. 102	
1 2 3 4 5 6	1 1 1 1 1 1	0 0 0.5 0.5 0.5	0.05 0.05 0.05 0.05 0.04 0.02	0 140 140 310 240 200
	Tube No. 18	P. and W	No. 9	
1 2 3 4 5 6	1.5 3 3 1.5 1	1 3 5.5 0.5 0 0	0.08 0.16 0.12 0.02 0.01 0.01	880 1320 1700 630 80 0

### Table 35. (Contd). COMPARISON OF BETA RAY BACKSCATTER RATE OS OF <u>CRC</u> RDCO JFTOT TUBES WITH THOSE FROM THE ASTM <u>TUBERATOR AND THE ERDCO REFLECTANCE RATER</u>

AFAPL-TR-70-71

-57-

1000年また。1990年までは、1990年によっていた。 1990年までは、1990年までは、1990年までは、1990年までは、1990年までは、1990年までは、1990年までは、1990年までは、1990年までは、1990年までは、1990年までは、1990年までは、199 to give quite consistent values within itself, the disagreement with the beta-ray backscatter apparatus is quite marked as shown in <u>Figure 31</u>. Although these data have not been analyzed statistically, the lack of concordance between the two methods of rating is evident. Of course, some of the scatter must be due to the beta-ray backscatter instrument, but we cannot assign this precisely since complete data on repreducibility will act be obtained until development work is complete. However, repeat runs on which same tube do not show deviations greater than about  $\pm 10$  A. The relation between ASTM Tuberator ratings and the beta-ray backscatter rating is shown in <u>Figure 32</u> for both the CRC average ratings and the Shell ratings. Again a great scattering of results is noted. The CRC average ratings increase with a reduced slope for higher values because of the maximum 4 code rating imposed on those values.

A summary comparison of ratings by the other methods with the maximum deposit found with the beta-ray beta seatter method is given in Table 36.

#### CRC Alcor JFTOT Preheater Tubes

We have also examined a group of ten tubes from the test program of CRC New Test Equipment Panel for the selection of a new thermal stability tester. Information received from the panel included ratings by an Alcor Mark 5 Tube Rater and Erdco Reflective Rater and values obtained by different panel members using the standard ASTM Tuberator. The latter values were averaged. The tubes were also rated on our own ASTM Tuberator equipment using our expanded scale (up to code 8), values being obtained every 1/2" along the 2-1/2" active length of the tube.

Values obtained by the various methods are given in <u>Table 37</u> and are shown graphically in <u>Figures 33 and 34</u>, except for the two tubes with the heaviest deposits. Except for the Alcor Mark 5 Rater, which was obviously not sensitive enough, values obtained by the various methods showed general geographic agreement. The CRC Average ratings, of course, were always low in tubes having heavy deposits since the ASTM scale stops at 4. In general, the best agreement with the beta-ray backscatter method was exhibited by the Erdco rater (<u>Figures 33A</u> and <u>34A</u> and <u>D</u>). However, in a number of cases (<u>Figures 33B</u>, <u>C</u> and <u>D</u> and <u>34</u> <u>B</u> and <u>C</u>) rather marked differences were observed. This is particularly true of tubes 2, 6, and 8 (<u>Figures 33C</u> and <u>D</u>, and <u>Figure 34C</u>), which would have presumably been failed by the Erdco rater instrument.

The relation between the various ratings and the beta-ray backscatter measurements are shown in <u>Table 38</u> and <u>Figures 35</u> and 36. The considerable scatter of the points is evident. However, in view of the favorable characteristics of the Erdco rater, it will be worthwhile to evaluate this instrument further using the beta-ray backscatter instrument as a standard.

With both the Erdco Reflective Rater and the beta-ray backscatter equipment, the absolute level of the values observed could very well be related to tube geometry. Certainly in the case of the backscattering principle, the calibration of the equipment with respect to actual film thicknesses was preliminary and subject to error in absolute accurecy.

Figures 29 through 32 follow

-58-



AFAPL-TR-70-71 66504

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Figure 30. COMPARISON OF METHODS OF RATING ERDCO JETOT TUBES

AFAPL-TR-70-71 66504

History Film



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# Table 36. SUMMARY MARISON OF OTHER RATING METHODS WITH BETA-RAY BACKSCATTERING METHOD FOR CL: IRDCO JETOT TUBES

Tube No.	Max	Rating o	f Beta-Ray M	tax by <sup>a)</sup>
Tube No.	Beta-Ray	ERDCO	Shell ASIM	CRC Ave.
4	1790	0.21	6.5	3
18	1700	0.12 (. 6)	5.5	3
15	1620	0.11	2	3
14	1400	0.13 (.74)	5	.5
3	1130	0.17	5	3
7	680	0 <b>.16</b>	1.5	2.5
12	550	0.08	0.5	2
2	360	0-05	0.5	1.5
17	310	0.05	0.5	1
10	140	0.06 (.07)	0.5	1 (1.5)

 A) Values in parentheses are maximum values according to specified method.

AFAPI-TR 201-71

-59-

' <u>26</u> b	le -7.	COMP	ARISC	N OF	BETA	RAY	BAC	KSC	TTER	RATINGS	<u> </u>	2
	ALCOR .	FTOT	TUBES	WITH	I THO	SE FI	ROM	THE	ASTM	TUBERA I	OF	
		AND	THE E	RDCO	AND .	ALCOR	R MA	RK V	RATI	RS		

Tobe	Inberator	Retings	ALCOR	ERDCO	Beia Ray						
Inches	CRC Ave.	Shell	MARK V	Rater	Backscatter, Å						
		Ĩ	uie No.	ITAI							
0-1/2 1/2-1 1-1 1/2 1 1/2-2 2-2 1/4	1.5 1.5 1.5 1.5 1.5	1 1/2 1/2 1 0	9.5 9.5 9.5 9.5 9.5	).19 ).18 ).12 0.05 0.07	580 480 320 40 0						
		Ţ	ube No.	11/2							
1/2 1 1 2 2 /4	1 1 1/2 1/2	<b>1</b> N D 3	9•5 9•5 9•5 9•5 9•5	0.13 0.15 0.10 0.08 0.06	20) 12) 4) 1, : 1, :						
T be No. IIA4											
2/2 1 1/2 2 2 1/4		5.5 5.5 5.5 1 0	9.0 9.0 9.5 7.5	0.24 0.23 0.19 0.08 0.03	56. 740 540 120 20						
			ule No.	<u>1 TA6</u>							
$\frac{1/2}{1}$ $\frac{1}{2}$ $\frac{1}{2}$ $\frac{1}{2}$	3 2 1 1	2.5 3 1 1/3 1/2 0	9.0 9.0 9.0 9.	0.15 0.18 0.12 0.04 0.03	95 740 40 50 10						
		<u>1</u> )	ube Mar.	11/8							
$\frac{1/2}{1/2}$ 1 1/2 2 2 1/4	3 2 - 5 - 5 - 5 - 1 - 1 - 1	3.5 6 1 1 0	9.0 9.0 9.0 9.0 9.0	0.16 0.24 0.10 0.10 0.04	200 26. 80 120 						

(Contá)

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Tube	Tuberator	Ratings	ALCOR	ERDCO	Beta Ray
Inches	CRC Ave.	Shell	MARK V	Rater	Backscatter, A
		Ţ			
0-1/2 1 1 1/2 2 2 1/4	1 1.5 1.5 1.5 1	1/2 1/2 1 1/2 0	9•5 9•0 9•0 9•0 9•0	0.14 0.18 0.16 0.07 0.03	560 420 220 60 0
		Ĩ	ube No.	IIA9	
1/2 1 1 1/2 2 2 1/4	4+ 3•5 1 1 1	6.5 7 1/2 ./2 0	6.0 5.5 9.5 9.5 9.5 9.5	0.46 0.60+ 0.06 0.03 0.01	2480 2480 0 0 0
		T.	ube <u>No</u> .	<u>0[A]</u>	
0-1/2 1 1 1/2 2 2 1/4	3 3 3 2 1	5.5 2.5 3 1/2 0	9.0 9.0 9.0 9.0 9.5	0.22 0.25 0.20 0.09 0.03	800 900 6ಕಂ ಿ40 0
		<u>1</u>	ibe No.	TTA20-18	
0-1/2 1 1 1 2 2 2 1/4	3-5- 3-5- 3-5- 6- 3-	3.5 3.5 3.5 2 0	9.0 9.0 9.5 9.5	0.21 0.22 0.15 0.06 0.02	680 800 480 80 0
		<u>T</u>	ite No.	[ <u>1455</u>	
1/2 1 1 1/2 2 2 1/4	4,5 4,5 1	$\begin{array}{c} 6.5 \\ 7 \\ 1/2 \\ 1/2 \\ 1/2 \end{array}$	6.0 5.5 5.3 0.3	0. <b>44</b> 0.60+ 0.12 0.10 0.07	1860 2460 80 0 0

# Table 37. (Contd). COMPARISON OF BETA RAY BACKSCATTER RATINGS OF ONC ALCON JETOT TUBES WITH THOSE FROM THE ASTM TUBERATOR AND THE ERDCO AND ALCOR MARK V RATERS

AFAP1-TR-70-71

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### Table 58. SUMMARY COMPARISON OF OTHER RATING METHODS WITH BETA-RAY BACKSCATTER METHOD FOR CRC ALCOR JETOT TUBES

Tuba No	Most Question	Rating at B-Ray Max <sup>8</sup> )									
Tube No.	Max peray	Erdco	Shell ASTM	CRC ASTM Ave.							
9	2480	0.60+	7 1/2	3							
22	2460	0.60+	7	4							
1 <b>1A</b> 10	900	0.25	2 1/2 (5 1/2)	3							
20 <b>-</b> 18	800	0.22	3 1/2	3 1/2							
4	700	0.23 (0.24)	5 1/2	3							
1	600	0.19	.1	1 1/2							
14-13	550	0.14 (0.18)	1/2 (1)	1 (1.6)							
8	200	0.24	6	3.5							
2	200	0.13 (0.15)	1	l							
6	140	0.18	3	3							

a) Values in parentheses are maximum values according to specified method.

Figures 55 through 50 follow

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Figure 33. COMPARISON OF METHODS OF RATING ALCOR JETOT TUBES





AFAPL-TR-70-71 66504



AFAPL-TR-70-71 66504

#### Problems in the Use of The Beta-ray Backscatter Instrument

Although it is evident from the above that the beta-ray backscattering principle can be applied to the evaluation of the amount of deposit formed on a metallic surface, there are a number of problems which could complicate the application of the principle. One is the composition of the tube metal substrate. Since the intensity of the scattered electrons is roughly proportional to the atomic number of the element, it is obvious that a calibration must be established for each type of metal used on which deposits are measured. In addition, the composition of the deposit will have an effect on the apparent thickness. This is shown in the Table 39. However, it is evident that the amount of error introduced by the presence of the usual percentages of oxygen, sulfur, and nitrogen will not be large, and it is unlikely that any significant error will be introduced if an average composition of the deposit is assumed. The presence of large amounts of heavy metals or metal oxides in the deposit, however, would seriously hamper interpretation of the measurements. Another problem is the need for a standardized calibration procedure, which requires the production of standardized films with known thicknesses. So far we have been using nitrocellulose films, the thickness of which can be measured by optical refraction. Another factor which can influence accuracy is distortion of the surface or a serious wobble in a rotating tube. However, this has been tolerable at the level usually encountered with JFTOT tubes and warped plane surfaces by providing occasional clean surfaces on the specimen for measurement references. Another definite limitation is the thickness of the deposit measured. For each source there is a limit of thickness which can be measured as a result of saturation, i.e., capture of essentially all the electrons entering the deposit. Thus, the intensity of the source was chosen so that the instrument could measure the 2,500 Å thickness that we expected to encounter. Measurements of thicker deposits would require a more intense source.

Assumed Composition of Deposit	‰ non CH	Error In Deposit Thickness, %
СН	a)	a)
CH2	7, H	+2.0
CH0.25	23.5, 0	-1.5
CHS. OS	11.0, S	-3.5
CHFe <sub>.01</sub>	4.1, Fe	-2.5
CHPb.001	1.4, Pb	-3.1

#### Table 39. ESTIMATED ERRORS IN BETA-RAY BACKSCATTER MEASUREMENTS OF DEPOSIT THICKNESSES DUE TO NONCARBON ELEMENTS

a) CH was taken as the reference composition.

AFAPL-TR-70-71

-63-

The present instrument is still under development, and it is anticipated that the above problems will be sufficiently resolved to allow the instrument to be used for evaluating the thickness of deposits on heat-exchange surfaces, as well as for other uses in which it is desired to measure the thickness of coatings.

#### Fuel Additives for Improvement of Thermal Stability

One important consideration in the development of fuels for high temperature operations is the decision as to what additives, if any, will be used with the fuels. Although the selection of the additives is best left until the conditions under which a fuel will be used are known, some preliminary investigation has to be done in order to ascertain the interaction between the fuel and different additives. One of the additives which is suspect is the metal deactivator, since it has a decomposition temperature of about 540°F in bulk.

Accordingly, a program has been initiated to screen potential additives for improving thermal stability, particularly of a variety which will function at higher temperatures. As a part of this investigation, we have taken a look at iodine, partly because of the lubricity improving characteristics of this material. It was reasoned that if iodine decreases the coefficient of friction, as has been reported under certain circumstances, then it might reduce the deposition tendency of fuels in a coker, where metal from pump wear might be  $\varepsilon$  contributing factor. However, the addition of 0.1 percent iodine to PWA-535 jet fuel caused very severe deposit formation in the SD/M-7 coker at 600°F (code 5.5/56.5 compared to 2.5/16 for the iodine-free fuel). Consequently, no further testing is planned with this additive.

Following a lead from earlier work or the influence of metal environments on thermal stability, in which metallic zinc was thought to have a beneficial effect, zinc 2,4-pentanedione was tested in decalin at 600°F. The metal was added at a concentration of 200 ppm without effect (code 2.5/16.5 versus 2.5/15 for the nonadditive fuel) in the SD/M-7 Fuel Coker. Further observations with metallic zinc in decalin indicate that the benefit originally found was characteristic of the effect of metal deactivator (MDA; N,N'-disalicylidene-1,2-propanediamine) also present. MDA was found to provide improvements in thermal stability of decalin up to 600°F, but became ineffective at higher temperatures. This suggested the need for a chelating agent with a higher thermal stability, since MDA is not stable above about 540°F.

It had been concluded in the earlier tests that Zn did not interfere with the effect of MDA on the deposition tendency of decalin as did certain other metals such as Ni, Fe, Pb, and Cr. The results with zinc 2,4-pentanedione were therefore in harmony with this conclusion.

The beneficial action of MDA was assumed to be directly related to its action in passivating dissolved, and possibly surface metals, and not necessarily to the action of a zinc chelate which had been formed. Conceivably, even where concentrations of soluble metals are extremely low (ppb level), these metals may nevertheless play a key role in oxidative thermal degradation of fuels. Since virtually all fuel handling and storage ecuipment is metallic, metal is always present, and even in glassware tests of thermal stability the fuel has been previously in contact with metals.

It was concluded that perhaps a more stable chelating agent would extend the resistance of fuels to thermal oxidative degradation to higher temperatures than does MDA, even at air saturation conditions. Starting from this premise, some fifty compounds have been screened using the SD/M-7 fuel coker, of which six definitely have been found to be effective at  $675-700^{\circ}$ F. Probably several of the others would also prove effective at lower temperatures. A highly saturated jet fuel of a JP-7 type with high thermal stability (RAF-159-60, designated F-68) was used for the screening tests. This fuel has a thermal stability breakpoint by the SD/M-7 coker method of about  $675^{\circ}$ F. Other properties for this fuel are shown in Table 40.

The simplest member and first discovered of the new class of thermal stability additives (additive A) was found to improve the coker ratings of F-68 over the temperature range of 600 to 700°F. At the latter temperature the improvement in thermal stability is equivalent to ca 125°F. That is, the coker deposit rating at 700°F is equivalent to that obtained at 575°F without the additive.

Most of the other compounds tested have involved structural variations of the basic form of additive A by substitutions of groups and atomic species on and about the functional parts of the molecule. However, in the work done to date, no one simple mechanism appears to account for the activity of all the additives. Indeed, not all of the effective compounds are believed to be chelating agents. Some of the materials tested, although having some of the structural features of additive A, are entirely different in their chemical properties. Moreover, we have no simple explanation for the maximum temperatures at which these additives are effective. Structural modifications which increase the thermal stability of the additive do not necessarily improve its effectiveness. Most of these changes have resulted in less favorable activity, some decreasing the thermal stability of the blended fuel.

In four cases with the six effective additives, group substitutions at the functional group of additive A apparently did not harm the ability of additive A to enhance thermal stability, and in addition are expected to improve the resistance of the additive to water leaching. The fifth effective compound (additive A') is a different chemical type, but the structure was suggested by that of additive A. The sixth additive is a fluorinated form of additive A  $(A_F)$ . The effectiveness of these six compounds and MDA is shown in Table 41. Although the effectiveness of the additives increases with temperature, the results shown in Table 41 demonstrate how an additive may first be beneficial and then become harmful above some critical temperature. None of the additives has been found effective at 725°F, when tested at that level, and one additive (A') that was beneficial at 675° was very harmful at 700°F. The cause for this sudden change in activity with temperature rise is due either to a thermal stability limit of the additive itself, or to a change in the thermal degradation mechanisms with which the additives interfere. The fact that MDA was not effective above 600°F in either decalin or F-ú8 jet fuel suggests limitations in the thermal stability of this additive. The more thermally stable additive AF was found to be no better and even worse than additive A.

Table 40. DESCRIPTION OF JET FUEL RAF-159-60

	F1	0.0007		1 m	a c	) <b>-</b>	- [	1 ku 1 ru 1 ru		)   	10	1	20031	18,990				- `` √ (` ``			2) ( 2) ( 7	C)T		ດູ ( ສ	г. с <b>-</b> д. х	ć	3 <b>0</b> 3 m
30	Water Tol, F-791, 3251, Interface Rating	Suliur, D1266, 🐅	Mercaptan Sulfur, D1323, 蜿	Cornosion, Cu Strip, D130, 2 hr at 212°F	Aromatic Content, 21319. Sv	Browine No. D1159	Naphthalenes, F791, 3704T, %v	Smoke Point, D1322, mm	Luminometer No., D1740	Existent Gum, D381, mg/d1	16-hr Potential Gum, D873, mg/d1	Net Heat of Combustion, Btu/15	ASTM D240 (Faar 260) Modified)	NBS 5917	Hydrogen, D1018, &	Copper, mg/liter	Nitroger. Dum	Basir Titrogen num	Percyide No. D1563	Water Senarometer Index Senarity 16	Light Transmission & at Lot we	Sedinent. mg/gal. 0.45 mfrom	Average	Lange Witcht Dotomication	Other	Faraffins. Sv Mass Sree	Naphthenes, W Mass Spec
Propertie	50.7		399 299	£0 <del>1</del>	70 <del>1</del>	410	413 C	814	621	128	436	450	124	964	533	1.5	0.5	164	-32	30+		1.81	6.75	12.9		9227	
	Grav ASTM D287, API	DISUILLETION, AN IM DOO, F		\$< 	TOP	50 K		4.0 <del>1</del>	50%	400 1	40J	00.6		<b>3</b> 2	بين . ب.	Residue, %	Loss, %	Flash Point, TCC, D55, °F	Freezing Point, D1477, °F	Color Saybolt, D15	Viscosity, D445:	at loc'F	at 0°F	at -30°F	Aniline Pt, °F	Aniline-Gravity Constant	

AFAPL-TR-70-71

-66-

		,							and the later state and the state by several
Additive	Volume Percent	Increase	e in Tem	peratur	<u>•e (°</u> F	<u>) at B</u>	gual <u>T</u>	<u>ite Co</u>	de Rating
		Liquid Temp. (°F	): 600	625	650	675	700	715	725
MDA.	0.67		0	-	-	-	-40	_	
A	0.2		25	50	75	100	125		-50
Al	0.25		-	-	-	-	125		-
Al	0.20			-	-	-	-	130	
A <sub>2</sub>	0.20		-	-	-	-	125	-	50
A3	0.20		-	-	-	90	-	-	-
Α4	0+50		-	-	-	90	-		-
A'	0.20		-	-	-	100	-125	-	-
A <sub>F</sub>	0.50			-	-	-	-	-	-140
			1		1		1		

# TABLE 41. EFFECTIVENESS OF ADDITIVES IN IMPROVING THE COKER HATINGS OF F-68 JET FUEL

Based on SD/M-7 Coker ratings at 250 psig, air saturated fuel.

# Table 42. EFFECT OF ADDITIVE A ON SD/M-7 COKER PATINGS OF METHYLCYCLOHYXANE

	(All Sabarates)		
Additive -	Percent	Temp., °F	ASTM Code, max/total
	-	450	1.0/7.5
-	-	475	1.5/9, 1/8.5, 1/5
-	-	500	8/32.5
A	0.2 + 65ppm Ionol	475	5/39.5
А	0.2 (Ionol-free)	475	6/26
A	0.2 (Silica gel treated) <sup>a)</sup>	475	0/0
٨	475 (Silica gel treated, then H <sub>2</sub> O equilibrated)	475	3/10.5
Copper <sup>b</sup> )	3130 ppm	425	1.0/9.5
Copper <sup>b)</sup>	2920 ppm	475	1.5/6.0

(Air saturated, 250 psig)

a) Silica gel treatment to remove dissolved water.

b) Copper added as copper undecylenate.

AFAPL-TR-70-71

Of course, we are most interested in thermal stability additives for endothermic fuels, and have therefore extended our investigations with additive A to MCH. The breakpoint of air-saturated MCH is about 480°F on the SD/M-7 fuel coker and rather sharp. Some of the experimental runs with MCH are shown in Table 42. The first run with MCH containing 65 ppm Ionol oxidation inhibitor was disappointing, in that it increased deposit formation. To test whether this was due to the large concentration of Ionol, we next ran a test with ionol-free MCH, but with essentially identical results (Table 42). We then passed MCH through a silica gel column and repeated the test. As shown, additive A gave a deposit-free test at 475°F, compared to a code 1 rating without the additive. Presumably, this improvement was due to dissolved water having been removed. To test this hypothesis we put equal amounts of MCH and water together in a bottle on a laboratory shaker for 10 minutes, then carefully decanted off the MCH and added 0.2 percent of additive A to it. This sample gave a 3/10.5 SD coker rating. Although this was not as bag as the untreated MCH, it certainly demonstrated the deleterious effect of water on the additive. However, other reasons may exist for the improved response of MCH treated with silica gel to additive A such as the removal of other polar dissolved substances, or resincus or metallic particles with the gel acting merely as a depth filter.

To test the hypothesis that additive A acts by effectively removing suspended metal from the fuel, copper in the form of copper undecylenate was added to MCH. However, even the rather large amount of about 3000 ppm had no harmful effects on the rating of NCH (<u>Table 42</u>). Similar additions of 300 and 3000 ppm copper undecylenate to decalin had no effect whatever on coker ratings at 600 and 650°F, and the effect of additive A was not significant at 600 and 650°F (<u>Table 43</u>).

Additive	Concentration	Temp., °F	SD Coker Tube Rating, Max/Total
A A Copper <sup>a</sup> Copper <sup>a</sup> Copper <sup>a</sup>	0.2 0.2 0.1 300 ppm 300 ppm 3000 ppm	650 600 650 700 650 650 650	1.5/6.5 1.5/6.5 1.0/10.5 5/11.5 1.5/8.5 1.5/9.5 1.5/6.5

Table 43. EFFECTS OF ADDITIVE A AND COPPER UNDECYLENATE ON THE THERMAL STABILITY OF DECALIN

a) Copper Undecylenate.

This strongly suggests that the action of additive A has nothing to do with the presence of dissolved copper, since copper appears to have no harmful effect on the thermal stability of decalin or MCH. It also suggests that the action of additive A may not be related to the catalytic activity of dissolved metals at all, but rather to the activity of metal surfaces or to some other dissolved species. There is yet the possibility that iron or nickel, perhaps present from the MCH synthesis catalyst, may be involved. Further work is needed in this area, since the A-type additives seem to provide a tool for studying thermal stability mechanisms in addition to their possible practical applications.

Since copper addition had no effect on the thermal stability of either decalin or MCH, actual tests of additive A with added copper were not tried. Instead, it is planned to try this approach in a fuel such as F-68 in which there is a marked benefit of additive A for the thermal stability.

Since these tests had been made on the SD/M-7 Coker, which operated on the recycle mode, a question arose as to whether the result we obtained with additive A was merely an artifact of the test method or perhaps of the fuels, since both fuels tested in the SD Coker were virtually 100 percent saturated. We therefore used a commercial production turbine fuel (F-187) in the Alcor JFTOT Fuel Test. Here we found the same kind and magnitude of benefits, however, without any treatment of the fuel in advance. Results of these preliminary tests are shown in Table 44.

Both the maximum tube temperature and the fluid effluent temperature are shown. In fact, as would be expected, the maximum code ratings for the nonadditive runs correlate with the tube temperatures rather than with the libuid temperatures. With reference to liquid temperatures, 0.1 percent of additive A increases the thermal stability of F-187 by about 117°F, but the motal temperature, which is more significant, is extended by 160°F. As before, additive  $A_F$  failed to give any improvement. We do not intend to screen any more compounds of the A-type at this time. Instead, the effectiveness and concentration effects of the six superior additives are being tested on the heat sink fuels from the current program and on a few pure hydrocarbon types. The improvement in the thermal stability of MCH by additive A after silica gel treatment suggests that we should look further at the effects of pre-purification treatments, and also together with antioxidants, antiicants, corrosion inhibitors, and lubricity additives in selected cases. This will be combined with storage stability studies of the most promising combinations of additives and fuels. As soon as supplies of SHELLDYNE-H<sup>®</sup> fuel are available, work will be started to select the most desirable additives for this material.

Most of the future work will be done using the Alcor JFTOT Fuel Tester, so that realistic concentration requirements can be determined. The fuel makes over a hundred passes per test in the SD/M-7 coker but only a single pass through the hot test zone in the JFTOT. Hence, testing in the JFTOT may indicate a greater effectiveness for the additive and a higher temperature or lower concentration requirement for its use, especially if the additive is limited by its own thermal stability.

Additivo		Bomaon <sup>b</sup>		Temp	erature, °F		ASTM Ratings,
AUGICIVE	-	rerdens	Max.	Tube	Liquid	Effluent	maximum code
A	-	0.16	5'	79		411	0
A	-	0.1	60	05		437	0
А	-	0.1	68	30		485	1
А	-	0.1	73	28		505	1.5
A	-	0.1	72	24		528	2
A <sub>F</sub>	-	0.1	72	24		528	8
			56	68		411	2
			58	30		408	3
			57	'5		409	2.5

# Table 44.EFFECT OF ADDITIVE A ON ALCOR JETOT FUELTEST RATINGS OF F-187 TURBINE FUEL

AFAPL-TR-70-71

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Research on additives for improving the thermal stability of fuels is continuing in several directions:

- 1. The relationship of additive concentration to temperature level will be investigated.
- 2. Suitability of additives of this type to various other endothermic fuels will be explored, together with some study of the effect of fuel hydrocarbon type composition.
- 3. Possible interaction problems with other jet fuel additives and common impurities will be examined.
- 4. Interaction effects with catalyst systems now under study will be investigated.
- 5. Possible deleterious side effects may be uncovered, particularly with respect to other fuel properties and storage stability.
- 6. Studies of the theory and mechanism of deposit formation as related to the action of A-type additives will be pursued.

#### Hydrocrackate Jet Fuels

Modern refinery processing has progressed to the point that certain processed hydrocarbon mixtures may be sufficiently high in naphthene content to be good endothermic fuel candidates. Accordingly, we have measured the thermal stability of hydrocrackate jet fuels.

A series of SD/M-7 Coker runs on these two samples are shown in <u>Table 45</u>, from which code 2.5 tube rating brea: oints of 405 and 355°F were estimated; the high aromatics sample from the first stage (F-155) was the more stable fuel, which was a surprising result. Both ratings are better than specifications for jet fuels; the 405°F breakpoint is characteristic of a good jet fuel. Filter plugging tendencies were actually more limiting than tube deposits, reaching values of 13 inches of mercury for pressure drops at 350°F for both samples. No fuel additives were included, and since these are samples from a rather small pilot plant they are merely suggestive of what would be produced from commercial scale pla ts. No further work in this area is planned at this time.

Fuel	Temperature, °F	Filter Pressure Drop, <sup>1)</sup> psi	ASIM Code Ratings <sup>1)</sup> max./total
F-155	500	40	8/35
F-155	425	12.4	5.5/18.5
F <b>-1</b> 55	425	74.4	5/20
F <b>-</b> 155	425	100	4/18
F-155	400		(2.5/8)
F-155	3 <b>7</b> 5	24.1	0.5/9.5
F <b>-</b> 155	350	7.7	0/10
F <b>-1</b> 55	<b>32</b> 5	0.5	1/6
F <b>-1</b> 55	405		(2.5/10.5)
F-156	425	35.4	8/35
F <b>-</b> 156	400	53.7	4/14
F-156	350	5.5	1/4
F <b>-</b> 156	325	1.6	2/8
F-156	355	(6.4)	(2.5/10)
1	l l		

## Table 45. SD/M-7 COKER RATINGS OF HYDRCCRACKATE JET FUELS

1) Bracketed values are interpolations to obtain breakpoint temperatures.

AFAPL-TR-70-71

-72-

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#### Modifications and Assembly of Thermal Stability Apparatus

The ancillary equipment for the special high severity "barebones" JFTOT received from the Alcor Corporation has now been installed, including a constant speed pump, watt meter, and pressure transducer. The constant speed pump consists of a Zenith No. 1/2 pump driven by a 1/50 h.p. Bodine motor. However, this motor is slightly underpowered and we have now installed a Minarik Electric speed and torque controller, which has overcome this problem. The pump normally turns at 10 rpm, but is capable of flows four times that rate.

Temperature is controlled at the maximum tube temperature point, which is 0.85 inch from the discharge end for aluminum tubes and 0.6 inch for stainless and ordinary steel. A Guardsman West Controller is be ng used satisfactorily in this application. We have also installed a liquid effluent thermocouple in the discharge hole of the preheater with readout on a Honeywell strip recorder.

Nitrogen pressure on the system is read on a Heise Gage (0-1,000 psig), while the filter pressure drop is measured by a 0-5 psi Stratham Pressure Transducer and read out on another Honeywell strip recorder. Nitrogen pressure is controlled by a simple two-stage Victor regulator.

Voltage to the pump motor and preheater Variac is supplied by a Stabiline Automatic Voltage Regulator capable of controlling to  $\pm 1/20$  volt. All other equipment is the same as supplied by Alcor Inc.

Besides the standard aluminum tubes, tubes made of stainless steel and No. 1015 steel already have been obtained and tested. The steel tubes all appear to give more severe ratings than does aluminum, but several factors complicate this comparison, such as temperature profile and metal color change, so that true comparison awaits the proper application of the beta-ray backscatter deposit analyzer. Other metals such as Inconel, nickel, and Incoloy are also being obtained for comparison studies in JFTOT tubes. A complete list of tube metals now on hand as JFTOT tubes is shown in <u>Table 46</u>. Except for Haynes 25, these and aluminum have been run on a Jet-A type fuel for evaluation of catalytic effects of metals on fuel deposition tendency. The tubes are currently being rated by the beta-ray backscatter technique, and will be rated afterwards by combustion for confignation. Calibrations on the various metals are still being made using 1000 A nitrocellulose film. Following satisfactory completion of this work, the effect of these different tube metals will be determined on MCH.

Assistance was obtained from Alcor Inc. and from Pratt and Whitney, East Hartford, Connecticut, on obtaining some of these special materials. In general, tubing with a 3/16" OD and approximately 0.68" ID for fabricating these tubes is difficult to locate. Alcor Inc. has manufactured coker tubes by attaching 3/16"end pieces of gold-plated brass to the desired 1/8" test section, and we have obtained two of these tubes for testing. We are not sure whether the brass, although gold-plated, is entirely innocuous in the deposition mechanism.

The high temperative version of the JFTOT which we obtained was designed for operation up to 1000°F and 1000 psig. So far we have only operated the rig at 300 psig, and the highest tube temperature we have tested has been 724°F (528°F liquid effluent temperature). At this temperature and even lower, the tube becomes

AFAPL-TR-70-71

Table 46. COMPOSITION OF TUBE METALS FOR JETOT

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Tube Metal				2	pical Co	mposit	ion,	R.				
Metals in Stock	ΝÌ	°C C	Сr	мо	Fe	U	Al	Сц	Æ	3	Si	Other
Hastelloy Alloy C <sup>1</sup> )	54.6	2.5	15.5	16	5	0.08		. <b>E</b>	1.0	17	1.0	0.35 V
Seamless Monel 400 <sup>2</sup>	65.	[73)	t 8	3	1.2	0.13	1	32.33	1.05	!	0.0	0.008 S
Inconel Alloy 600	72.(	( 6	15.5	1	10	0.15	1	0.5	1.0	1	0.5	0.015 S
316 Stainlees	13.28	:	16.75	2.50	65.14	0.05	1	1	1.73	ł	0.51	0.035 P + S
304 Stainless <sup>1</sup> )	6	1	19	1	72	0.08	1	1	0 8	1	1	1
446 Stainless	i 1	:	25	! 1	75	0.35	1	1	1	ł	1	8
Mickel 200 <sup>2</sup> )	.66	56 <sup>3</sup> )	1	1	0.04	0.06	5 8	0.01	0.26	;	0.04	0.005 \$
Cartridge Brass	30	8	1	ł	ł	1	ł	70	ł	;	!	
1015 Steel	1	8	1	1	99.85	0.15	1	1	1	1	8	8
TD Nickel 1)	2 perce	ent by 1	volume 1	l Thorium	n Oxide	disper	sed	in Nî				
L-605 (Haynes 25)	06.6	51.75	19.85	1	1.60	0.12	;	\$	1.65	15	0.60	0.015 P + S
<ol> <li>These tubes are al 2) Compositions for t 3) These values are t</li> </ol>	lso can he these all the combi	and in a loys are ined nie	alumini: e measum skel-col	zed fo red va alt cc	rm. Lues. Smpositi	ons.			-			

AFAPL-TR-70-71

-74--
bent during the run so that it is no longer concentric with the outer shell. Of course, this is due to the thermal expansion of the tube, which is restricted by the clamping restraints of the end seals. This event seems to cause an actual change in the temperature difference between the tube control point and the liquid effluent during the course of the run.

We have attempted to overcome this problem by substituting the rubber O-ring seals with TEFLON<sup>®</sup> seals, which are more slippery, and applying a high electrically conductive lubricant to the electrical end clamps, in the hopes that the inner tube might slip during expansion. However, to provide good electrical connection with the aluminum tubes the clamping must still be too tight to allow for slippage during thermal expansion, and the tubes still bend. Perhaps with steel tubes this approach may work, but some redesign might be required. The bending can be particularly bothersome with the beta-ray backscatter device since it alters the distance from the source to the tube. This causes a sinusoidal fluctuation in the recorder output even on bare metal and if extreme would preclude the use of this method.

Several comparisons have been made between the JFTOT and ASIM methods on the same commercial turbine fuels, as shown in <u>Table 47</u>. These are all production fuels of recent date. The two methods are within 1/2 code number of each other generally, and at least as close as the probable repeatability of either method.

Two runs on the same F-187 fuel using stainless steel JFTOT tubes are also included for comparison. However, until these tubes can be rated by the beta-ray backscatter method, the true relative effects of aluminum and stainless steel will not be known, since the steels themselves change color on elevated heating. We do know from preliminary tests that a code 2 1/2 rating on a stainless steel tube was found by beta-ray backscatter measurement to have a thickness of less than 50 Å, while a code 1/2 deposit on aluminum with a whitish appearance (normally thought to be thinner) could be readily observed.

Recent experience with the Alcor JFTOT Fuel Tester showed that we were experiencing errors in control temperatures due to worn insulation on the control thermocouple. This emphasizes the need to inspect carefully and regularly the condition of the insulation after each run and to replace the thermocouple as needed.

To improve the accuracy of our tube metal temperatures, we have installed a double thermocouple, one which actuates the West temperature controller and one which reads out on a millivolt strip chart recorder. The latter is much more accurate and readable than the West controller dial and is not sensitive to thermocouple resistance. Liquid temperatures are also read out on a strip chart recorder as before. We believe the millivolt strip chart recorder is a more accurate instrument for the measurement of temperatures than the digital Honeywell recorder used as standard equipment with the Alcor Fuel Tester.

### SD/M-7 Fuel Coker

A Beckman Oxygen Analyzer (Model 778) has been installed on the SD/M-7 Fuel Coker. However, this instrument is limited to operation at or below 50 psi maximum pressure, and hence cannot be used continuously throughout the test period.

AFAPL-TR-70-71

Fuel	Fluid Temperature, °F	Test Method	Max. Visual Code Ratings
F-185	418	JFTOT	14
F-185	417	ASTM	4
F-185	420	ASTM	4
F-187	420	JFTOT	3
F-187	411	JFTOT	2
F-187	411	JFTOT	3
F <b>-</b> 187	409	JFTOT	2.5
F-187	408	JFTOT	3
F-187	420	ASTM	3.5
F-187	415	ASTM	3.5
F-187	415	ASTM	1.5
F-187	410	ASTM	1.5
F-187	410	ASIM	0
F-187	410	JFTOT (stainless)	6
F-187	411	JFTOT (stainless)	4

# Table 47. COMPARISON OF RATINGS BY THE JFTOT AND ASTM METHODS

AFAPL-TR-70-71

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Since the test method depends upon diffusion of oxygen through a semipermeable membrane, the flow velocity of the test fuel past the detector head must be mintained at a minimum of 1.8 ft/sec. To accomplish this a special fitting with a restricted passage was designed and built. The analyzer actually measures oxygen partial pressure, and therefore the pressure at the pickup mus known to permit calculation of  $O_2$  in prov. Static pressure at the pi measured with a Statham pressure transducer readout system. Pressure drop cooss the restricted passage has been determined to be about 0.4 psi, and so the pressure at the pickup is simply taken to be 0.2 psi less than the upstream pressure reading. This is a trivial correction.

Currently the oxygen can only be measured at the beginning and end of the test, that is, before and after operating at high pressure. After measurement of  $O_2$  concentration, the detector, which is located on a by-pass leg at the discharge end of the pump, is isolated from the rest of the system for the remainder of the coker run. This is an undesirable condition, but it will suffice until a detector is designed and built which will withstand greater pressures.

## STORAGE STABILITY OF MCH AND SHELLDYNE-H® FUELS

Storage stability tests which were started over four years ago have been completed and the results evaluated. At that time samples of pure MCH were stored in the 130°F hot room. Samples were packaged in one gallon epoxy lined pails with a 2:1 ullage ratio with oxygen concentrations from pure  $O_2$  to pure  $N_2$ . Several phenolic and diamine type oxidation inhibitors were included, as well as MDA, in some of the samples, while control samples contained no additives at all.

Examination of the samples for soluble and insoluble gum gave no indication of detericration in any of the samples during this period of storage. No insolubles were observed in any case and steam jet gums were less than 1 milligram per deciliter. No indication of discoloration was observed. Finally, coker tests showed no loss whatever of thermal stability. In fact, the actual ratings were better than those obtained on the fresh material prior to this severe exposure.

The storage stability of SHELLDYNE-H<sup>®</sup> fuel over a shorter period has also been investigated in accelerated tests. Three different batches of SHELLDYNE-H<sup>®</sup> fuel with minor differences in bromine number have been exposed to 200°F constant temperature for 18 and 30 hours. Three different oxidation inhibitors were tested in comparison with the nonadditive material, and all samples were blanketed with pure oxygen. Exposed samples were examined for both soluble and insoluble gums.

Unfortunately, results of these tests showed no consistency, either with respect to exposure time or to the presence of inhibitors (<u>Table 48</u>). Apparently an unknown factor was influencing the results; the 450°F temperature of the standard steam jet was probably too low to drive off all the SHELLDYNE-H<sup>®</sup> fuel. Accordingly, the steam jet temperature was raised to 500°F and the results were more consistent. However, even on this basis the results are quite erratic and it is possible that some other factor is affecting the results.

On the basis of our present results, it appears that the phenolic inhibitor 24M6B (dupont 22) is the most efficacious antioxidant for this fuel.

Table 48 follows

AFAPL-TR-70-71

-77-

Table 18. STORAGE STABILITY OF SHELLDYNE-H® FUEL

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Oven Tests at 200°F Under Oz Blanket

	0	- Hours			18 -	Hours				он - С₂	su	
Shelldyne-ll <sup>®</sup> Sample Designation	Insolubles ex Solvent	Solubles ex Filtered Fuel-450°F	Total Gum	Insolubles ex Solvent	Solubl ex Filt Fuel, m	es ered g/dl	Tot: Gun ng/ć	격특적	Insolubles ex Solvent	Solubl er Filt Fuel, m	es ered g/dl	Total Gum,
	10/2	ng/dl	TD/3mm	TD/Am	450°F	500°F	450°F	500°F	TD/Bm	450°F	500°F	To/Sm
F-151										-		(500°F)
	40	14.6 <sup>8</sup> )	15.0 <sup>a</sup> )	0.0	3.5	5.4		5.4	0.0	16.2 <sup>8)</sup>	6.4	6.4
durant 22 durant 30	0.0	ر م م	0.0 0.0	0.0	5.8 16.6 <sup>a</sup> )	4 5		5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	ಂ ಸ ಂ ರ	5.5 0.9	0.0	00
	0.2	20.8 <sup>8</sup> )	21.0 <sup>8</sup> )	0.6	15.2 <sup>a</sup> )	6.0		6.6	0.8	10.6	11.4	12.2
F-158												
Ionol duPont 22	4.00	13.2 <sup>a</sup> )	15.6 <sup>8</sup> )	5.8 4.0	12.2 <sup>a</sup> )	1	18.0		0.6	C L		
duPoint 30	0.0	4.6	0.4 •	13.3 <sup>a</sup> )	1 C1 C2 C2 C2 C2 C2 C2 C2 C2 C2 C2 C2 C2 C2		21.5			8		2 Q 0 O
	5.2	0.2	5.4	0.0	7.8		7.8		0.2	2.0	7.0	7.2
F-157												
Ionol	0.4	1.0	ч. Ч.	0•0	6 <b>.</b> 4	1	6.4		1.2	5.2	3.0	4.5
duPoint 22	0 <b>.</b> 6		3.0	5.6	10.4	1.0		6.6	0.4	1.0	2,4	2•8
duPoint 30	0.2	<b>5</b> .6	ۍ ۳	9.3	¥.8		14.1			5.t	1	5.8
	0.2	1.6	1,8	6.7	7.0		13.7		0.8	3.4	5.0	5.8
a) Highly sug	pect data:	values unrea	listical	ly high.								

## Elastomer Interactions With SHELLDINE-H® Fuel

Three SHELLDYNE-H<sup>®</sup> fuel samples, which were exposed to different elastomers by Atlantic Research Corporation (contract F33615-69-C-1849), have now been tested further to determine any possible solution of elastomer or leaching of plasticizer during this exposure. In <u>Figure 37</u> it is shown that the U.S. 941 Viton/Nomex nylon had the maximum effect on the light transmission properties of SHELLDYNE-H<sup>®</sup> fuel in the 335-375 millimicron wavelength range, although curiously the U.S. 3094 Viton gave the only significant change in refractive index as compared to the original stock (see <u>Table 49</u>). Results of microgum analyses, which indicate the amount of high boiling materials present, are shown in <u>Table 49</u>. These were run at 260°C under vacuum, at which conditions about 0.8 mg. carbon/100 g sample was obtained with the unexposed fuel. Fuel exposed to either U.S. 941 Viton/Nomex nylon or U.S. 3094 Viton gave about double this amount, but only the sample in contact with U.S. 566 Nitrile Nylon gave markedly higher results (20 times as much).

### Table 49. MICROGUM AND REFRACTIVE INDEX ANALYSES OF SHELLDYNE-H<sup>®</sup> FUEL SAMPLES SOAKED WITH ELASTOMERS FOR A PERIOD OF SIX MONTHS

Elastomer	Microgum (expressed as carbon) mg/100g SHELLDYNE-H® Fuel	nđ <sup>o</sup>
none	0.8, 0.9	1.5396
U.S. 941 Viton/Nomex Nylon	1.6, 1.8	1.5394
U.S. 3094 Viton	1.6, 1.9	1.5385
U.S. 566 Nitrile Nylon	15.6, 16.6	1.5394

Thus, three different methods have each selected a different elastomer as the one having the most interaction with SHELLDYNE-H<sup>®</sup> fuel. Nitrile nylon apparently contributes the most heavy material to SHELLDYNE-H<sup>®</sup> fuel, although this material apparently has a smaller effect upon light transmission and refractive index than do the impurities from the other two elastomers. This suggests the possibility of micelle or suspended solids formation in the Nitrile nylon sample, which visual inspection in room light confirmed. Both a yellow tint and a slight turbidity were observed. The other three samples were all water white and free of apparent solids content. Since neither micelles nor solids affect transmission of light of short wavelength appreciably, the more minor effect of the Nitrile nylon on refractive index and light ebsorption is at least partially explained.

In a final series of tests, the four samples of SHELLDYNE-H<sup>®</sup> fuel of <u>Table 49</u> were run by GLC analysis. The three samples which had been soaked with elastomers all showed a small unidentified peak representing about a 0.1 percent impurity, which was not present in the original stock. Other than that, the elastomer soaked SHELLDYNE-H<sup>®</sup> fuel samples all gave similar GLC analyses. The impurity could either have been introduced to the original material in handling prior to soaking, or might have come from the polyme: I lm (which appeared to be polyethylene or polypropylene) which had been placed over  $u_{12}$  shooping bottles prior to capping.

Figure 37 follows

AFAPL-TR-70-71

-78-



Whatever effect this film might have had, it probably would have been the same for all three elastomer soaked samples. The fresh SHELLDYNE-H<sup>®</sup> fuel came from our own retainer stock and was not exposed to the film. For the GLC procedure used, in which the sample size was 1 microliter, the detection limit is about 0.01 percent, unless the small impurity peaks are close to one of the larger peaks, in which case sensitivity would be much less.

In summary, we feel that the microgum test gives the more significant results with regard to compatibility of these elastomers with SHELLDYNE-H<sup>®</sup> fuel. From previous experience with SHELLDYNE-H<sup>®</sup> fuel it has been shown that light transmission properties do not necessarily correlate with coker test results. The reduction in light transmission indicates the presence of trace contamination, but thermal stability tests would be required to demonstrate any adverse effect on the fuel.

### THERMAL STABILITY OF NEW BATCH OF METHYLCYCLOHEXANE

A new batch of 1200 gallons of MCH has been made by hydrogenation of toluene, and on checking this material for thermal stability it was found to be defective when compared to the MCH remaining from the last production run. It appeared that the cause of the poor thermal stability ratings was due to presence of suspended nickel catalyst particles, although this is surprising since the MCH had been distilled. Refractive index determinations of the twelve drums of product showed no variations whatever. SD/M-7 coker tests at 475°F demonstrated that silica gel treatment brought the fuel up to standard and that filtration through a 0.45micron membrane filter gave even greater improvement. Thus, two methods for upgrading were available. We are now, therefore, filtering the MCH through a 0.2micron membrane filter, and will blend the new material with the MCH now in the storage tank as soon as coker tests confirm a high thermal stability. This method of upgrading is comparatively simpler and cheaper than that of silica gel treatment.

#### ESTIMATION OF PHYSICAL PROPERTIES OF FUELS

#### JP-7 Jet Fuel

Revised physical properties for JP-7 jet fuel (F-71) have been obtained using improved predictive methods and are given in the Appendix. These techniques were used in estimating physical properties for decalin and JP-5 jet fuel.<sup>3)</sup> The PVT behavior of the gas was represented by the Redlich-Kwong-Ackerman reduced equation of state:<sup>19)</sup>

$$Z = Z_{RK} + Z_1 + \omega Z_2$$

Z = compressibility factor

where

 $Z_{RK}$  = compressibility factor calculated by the Redlich-Kwong equation of state<sup>20</sup>)

(13)

 $\omega$  = acentric factor

 $Z_1$ ,  $Z_2$  = generalized functions of reduced pressure and temperature

AFAPL 18-70-71

-79-

The ideal gas heat capacity was represented by the group contribution method of Rihani-Doraiswamy:<sup>21</sup>)

 $c_p^{\circ} \approx a + bT + cT^2 + dT^3$ (14)

where c p = ideal gas heat capacity
 a,b,c,d = parameters determined by group contributions

Most of the other gas properties were obtained from these two properties using thermodynamic relationships.

Chemical analyses indicated that JP-7 is composed primarily of paraffins in the  $C_{11}$  to  $C_{16}$  range. Hence, the critical properties of the normal paraffins in this molecular weight range were used to obtain pseudocritical properties for the jet fuel. Pseudocritical temperature and pressure were defined as

$$\mathbf{r}_{\mathbf{c}} = \sum_{\mathbf{i}} \mathbf{y}_{\mathbf{i}} \mathbf{T}_{\mathbf{c}\mathbf{i}}$$
(15)

$$P_{c} = \sum_{i} y_{i}P_{ci}$$
(16)

where y = mole fraction of component i

 $T_{ci}, P_{ci}$  = individual component critical properties

In addition, the acentric factor was obtained from experimental vapor pressure data, and the Rihani-Doraiswamy coefficients for the fuel were taken as molar averages of the coefficients for the individual normal paraffin components.

Liquid properties were revised in the following manner. Previous estimates of density were corrected to agree with an available experimental density at 60°F and the critical density as predicted by the ecuation of state. Liquid enthalpy and heat capacity, being based on the revised gas properties, were also revised:

$$H_{1} = H_{g} - \Delta H_{v}$$
(17)

$$c_{pl} = c_{pg} - \frac{d(\Delta H_v)}{dT}$$
(18)

 $\Delta H_{y}$  = enthalpy of vaporization

AFAPL-TR-70-71

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-80-

Also, revised values of liquid thermal conductivity were obtained by the Robbins-Kingrea correlation.<sup>22</sup>) Previous estimates of vapor pressure, enthalpy of vaporization, and liquid viscosity were left unchanged.

The revised properties are considered to be better than previous values,2) because the most recently proven correlations have been used and experimental data have been applied where possible. They agree well with published or predicted values 'or similar petroleum fractions. The greatest improvement has been made with liquid heat capacity and thermal conductivity. Comparison with heat transfer experiments made previously in the FSSTR<sup>3)</sup> showed that the new heat capacity values are far superior to the old values.

### Binary Mixtures of Methylcyclohexane and a Low Molecular Weight Hydrocarbon

Physical properties were estimated for binary mixtures of methylcyclohexane (MCH) and a low molecular weight hydrocarbon. These properties are tabulated in the Appendix for mixtures containing ethane, propane, and n-butane. Mixture properties were estimated from known properties of the individual consti-tuents. 3,23)

The following properties were calculated as molar averages of the pure component properties:

> Pseudocritical Temperature Pseudocritical Volume Pseudocritical Compressibility Factor Acentric Factor Heat of Formation Net Heat of Combustion Liquid Specific Gravity Vapor Pressure

The above pseudocritical properties were used to calculate the pseudocritical pressure by the equation of state

$$P_{c} = \frac{RT_{c}Z_{c}}{V_{c}}$$
(19)

where

 $P_c$  = pseudocritical pressure

 $T_c = pseudocritical temperature$ 

 $Z_{c}$  = pseudocritical compressibility factor

- V<sub>c</sub> = pseudocritical volume R = universal gas constant
- = universal gas constant

The liquid viscosity was calculated by a molar average of the logarithimic function of viscosity:

$\ln \mu = \sum x_i \ln \mu_i$	(20)
i	

AFAPL-TR-70-71

-81-

where

e μ = viscosity of mixture μ<sub>i</sub> = viscosity of pure component i x<sub>i</sub> = mole fraction of component i

The mixture enthalpy was estimated as the molar average of the pure component enthalpies at those conditions where both components are gases or liquids in their pure state. Nonideal mixing effects are not appreciable in these systems and were neglected in the calculations. At those conditions where the mixture is a gas dissolved in MCH, the calculated enthalpy was reduced to account for the heat of absorption of the gas.

The melting points for the mixtures are based on experimental data for mixtures of MCH and propane. Melting and freezing points were measured for MCHpropane mixtures of three different compositions (Figure 38). An equation of the Clausius-Clapeyron type can be used to relate composition and melting point:

$$\frac{1}{T_{f}} - \frac{1}{T_{fo}} = \frac{R}{\Delta H_{f}} \ln x_{MCH}$$
(21)

where

 $T_{f} = \text{melting point of mixture}$  $T_{fo} = \text{melting point of MCH (-126.6°C = 146.6°K)}$  $\Delta H_{f} = \text{heat of fusion of MCH}$  $x_{MCH} = \text{mole fraction of MCH}$  $K_{f} = \frac{RT_{fo}^{2}}{\Delta H_{f}} = \text{melting point depression constant}$ (22)

Equation (21) can be approximated by

$$T_{f} - T_{fo} = -\frac{RT_{fo}^{2}}{\Delta H_{f}} (1 - x_{MCH})$$
(23)

Linear regression of the data was used to determine empirical coefficients for Equations (21) and (23) with the following results. For Equation (21)

$$\frac{1}{T_{f}} - \frac{1}{T_{fo}} = -\frac{K_{f}}{T_{fo}^{2}} \ln x_{MCH}$$
(24)

where  $K_{f} = 35.14^{\circ} K$ 

For Equation (22)

 $T_{f} - T_{fo} = -K_{f}^{T} (1 - x_{MCH})$  (25)

where  $K_{c}^{1} = 40.86^{\circ}K$ 

If the known heat of fusion  $(16.43 \text{ cal/gm})^{24}$  is used in Equation (22), the theoretical melting point depression constant is found to be 26.47°K, quite different from the above values. Melting points predicted with this theoretical constant differ significantly from the data (Figures 38 and 39). Both Equations (24) and

Figures 38 and 39 follow

-82-





AFAPL-TR-70-71 66758

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758



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(25) with the empirical coefficients lie within the accuracy of the data; they fit the data better than the theoretical predictions. Since Equation (25) is simpler, it was chosen to represent the melting points of low molecular weight hydrocarbons mixed with MCH. Calculated melting points are listed with the other physical properties of these mixtures in the Appendix.

### SHELLDYNE-H® Hydrocarbon Fuel

The surface tension of SHELLDYNE-H<sup>®</sup> fuel has been measured at three temperatures by the du Nouy ring method. These data correlated quite well to fit

$$\gamma = a \left(T_{0} - T\right)^{n}$$
(26)

where  $\gamma = \text{surface tension} (dyne/cm)$ 

T = temperature (°C) $T_c = critical temperature (530°C)$ 

a = 0.010

n = 1.33

Measured and extrapolated values of the surface tension are given in <u>Table 50</u> and <u>Figure 40</u>.

	Surface Tension (dyne/cm)							
T, °C	Measured	Predicted						
0 25 50 75 100 200 300 400 500 530	39.4 36.7 34.3	42.0 39.4 36.8 34.3 31.8 22.4 13.8 6.5 0.9 0						

Table 50. SURFACE TENSION OF SHELLDYNE-H<sup>®</sup> FUEL

#### SUPERSONIC COMBUSTION OF HYDROCARBONS

The methods used in obtaining and analyzing our supersonic combustion data have been re-evaluated. Some improvements in the equipment have been made. A new equation has been used to correlate the ignition delay times. Some new experimental data have been obtained. The rate of combustion of tetralin has been investigated.

Figure 40 follows

AFAPL-TR-70-71

-83-

## Experimental Equipment and Conditions

Several problems have been encountered in operating the shock tube with heavier hydrocarbon fuels. The tube had to be heated to 40-80°C. The resulting thermal stresses were hard on the resistance gauges which were used to measure the velocity of the shock wave. As constructed, the gauges were somewhat fragile. They consisted of a thin layer of platinum which had been sputtered onto a quartz disc encased in Bakelite. The differences in thermal expansion and the structural weaknesses led to frequent cracking and shorting. A sturdier device was designed and built. A similar design was used but the Bakelite body was replaced with aluminum. The quartz disc was replaced with Grade A Lava ceramic glued into the body with an epoxy resin. The platinum film was painted on as Liquid Bright Platinum, Hanovia No. 05-X. The improved resistance gauges have proved to be more reliable and have about the same dynamic response as the more fragile gauges used previously.

The mechanism for puncturing the shock tube diaphragms had to be modified by replacing the gasket with one able to withstand higher temperatures. A nylon tip is now used on the plunger which has improved the life and reliability of the plunger.

In order to prepare gas mixtures containing naphthalene, the mixture preparation system was modified. The hydrocarbons investigated to date have been gaseous or liquid at ambient conditions. Naphthalene, being a solid, presented a problem. The solution was to mount a tube, which could be isolated and removed, above the mixture cylinder. A schematic diagram of the system is shown in <u>Figure 41</u>. With the cylinder heated and evacuated, and the tube cold and detached at valve No. 1, a weighed amount of naphthalene crystals is added to the tube. The tube is then fastened to valve No. 1 and is evacuated by opening valve No. 1 to vacuum. When the air has been withdrawn, valve No. 1 is closed, valve No. 2 is opened, and the tube is heated to vaporize the naphthalene and drive the vapors into the cylinder. The other gases are added to the cylinder through valves No. 1 and No. 2 in the normal way.

The experimental data obtained have been on those hydrocarbons involved in the dehydrogenation of decalin, namely decalin, tetralin, and naphthalene. A summary of the conditions of the experiments is presented in <u>Table 51</u>. The complete data on ignition delay times may be found in the Appendix. The data have been obtained primarily at 9 psia and 15 psia and temperatures from  $1140^{\circ}$  to  $1760^{\circ}$ K, resulting in ignition delay times from 40 to 4000 µsec.

## Shock Tube Attenuation

An attempt was made to improve the shock tube results by accounting for velocity attenuation by measurement. The results have shown that assuming an attenuation coefficient of  $1 \times 10^{-3}$ , constant for all conditions, is the most reasonable procedure.

There are many near lealities in shock tube flow. One of these is the decrease in the velocity of the shock wave as it moves down the tube, caused by the build-up of a boundary layer at the wall of the tube. A review of wall effects has been made by Enrich and Wheeler, 25 where they present the attenuation equation:



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Fuel	Equivalence Ratio*	Percent Argon
decalin	0.2	90
decalin	0.5	99
tetralin	0.1	99
tetralin	0.1	80
tetralin	0.11	90
tetralin	0.5	99
tetralin	0.57	90
tetralin	1.0	95
tetralin	1.0	99
tetralin	1.21	90
naphthalene	0.1	90

## Table 51. SUMMARY OF CONDITIONS OF SHOCK TUBE RUNS

\*Equivalence ratio is defined as the actual fuel to oxygen ratio divided by the stoichiometric fuel to oxygen ratio.

AFAPL-TR-70-71

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-85-

$$(P_{21} - 1) = (P_{21} - 1)_{ideal} \exp(-A \frac{x}{r})$$

where

P<sub>21</sub> = ratio of pressures before and after shock wave x = distance from diaphragm

r = hydraulic radius

A = attenuation coefficient

This equation is said to be accurate enough for most work. We have presented the same equation in a slightly different form:<sup>3)</sup>

 $(M_s^2 - 1)_2 = (M_s^2 - 1)_1 \exp(-A \frac{X}{T})$  (28)

where

M<sub>s</sub> = Mach number x = distance from point 1 to point 2.

Several experiments were performed to determine experimental values of attenuation coefficients. To date, values of  $0.5 \times 10^{-3}$  and  $1 \times 10^{-3}$  have been used for A. These values were obtained from measurements in the literature on similar shock tubes. The attenuation coefficients determined experimentally on our shock tube were correlated with initial channel pressure and the ratio of driver pressure to channel pressure. The resulting correlation w<sup>p</sup> 3:<sup>a</sup>

$$A = \frac{1}{4} (0.00436 - 0.0000457 P_1 + 0.0000698 \frac{P_4}{P_1}).$$
(29)

where

 $P_4$  = initial pressure of driver gas  $P_1$  = initial pressure of reaction mixture

A set of experiments has been performed measuring ignition delay times and velocity attenuation simultaneously. We are now in a position to compare three different ways of estimating attenuation and its effect on ignition delay time measurements: (1) using a constant value of the attenuation coefficient as in the past ( $A = 1 \times 10^{-3}$ ), (2) using the correlation for attenuation coefficient, and (3) using the measured velocity attenuation. The ignition delay times as determined by the three methods for the combustion of a decalin-oxygen-argon mixture are shown in Figures 42 to 44. Figure 44 shows that using the correlation is clearly unsatisfactory. A comparison of Figures 42 and 43 reveal that the other two methods are approximately equivalent. The general scatter in Figure 43 is slightly less, but three points are badly misplaced. Overall, using the constant value of attenuation coefficient gives the best results. The reason for this is not immediately clear. One would think that measuring the variations in attenuation and allowing the coefficient to change should improve the results and decrease the data scatter, nct increase it.

a) We previously used the hydraulic diameter rather than radius. This introduces a factor of 4 into the correlation, i.e.,  $\Lambda' = 4A$ .

Figures 41 through 44 follow

-86-

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(27)



# Figure 41. SYSTEM FOR PREPARATION OF GASEOUS COMBUSTION MIXTURES CONTAINING NAPHTHALENE







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The explanation for this behavior appears to lie with the measurement of the shock wave velocity. Figure 45 shows a plot of average shock wave velocities at two points in the tube, in terms of the times of wave passage between point A and point B (18 inches) and between point B and point C (24 inches). The results do not fall precisely on a smooth curve, indicating either that the shock wave velocity fluctuates as it moves down the tube, or that there is appreciable error in the time measurements. A fluctuating shock wave velocity is fairly unlikely, although some variations can be caused by imperfect diaphragm breakage. The resolution of the time measurements is  $1 \mu sec$ , but the errors in measurement could be higher. Typical scatter of the data is 2-3  $\mu sec$ . The large effect of this apparently small scatter is shown by a plot of the attenuation coefficients calculated from the same data (see Figure 46). The values of A scatter between -0.09 x  $10^{-3}$  and  $1.7 \times 10^{-3}$ , and do not correlate with anything, including shock velocity. This is the reason for the failure of the correlation for attenuation. It is interesting that the mean value of all of the coefficients is  $0.99 \times 10^{-3}$ , which is remarkably close to the value of 1 x 10<sup>-3</sup> that was estimated from literature data. If the measurement of shock velocity was accurate, one would expect that using the measured values of A would reduce the scatter of ignition delay times. Since this improvement was not found, the problem must lie in the measurement of shock velocity. Efforts to improve velocity measurements would involve the purchase of more accurate electronic instrumentation and more instruments to measure the velocity at several points in the shock tube. This is probably not worth the expenditure of time and money required. Therefore, the best that can be done is to take the average value of A, determined experimentally to equal 1 x  $10^{-3}$ , and use it as a constant in all calculations.

Correlation of Ignition Delay Times

A new correlating equation has been used to represent the experimental data on ignition delay times:

$$\ln \tau = b_0 + b_1 \ln (c_{0_2}) + \frac{E}{RT} + b_2 \ln (c_{Fuel})$$
(30)

where

re  $\tau$  = ignition delay time, µ sec b<sub>0</sub>, b<sub>1</sub>, b<sub>2</sub>, E = correlating parameters c<sub>0</sub> = oxygen concentration, gmole/liter R = gas constant, 1.987 x 10<sup>-3</sup> kcal/gmole-°K T = absolute temperature, °K c<sub>Fuel</sub> = fuel concentration, gmole/liter

The methods of estimating the parameters have been investigated, as have the statistical techniques which are used to interpret the results.

Le equation used to correlate the data up to now has been the same as Equation (30) without the last term, which describes the effect of the fuel composition. There was some question about the correlation for decalin, since it appeared that fuel concentration had some effect on the ignition delay times. So the decalin data were fit using Equation (30) plus an additional term describing

-87-

the effect of pressure. The standard errors of the equation obtained by fitting with the addition of one term at a time are shown in <u>Table 52</u>. As can be seen, the improvement by including the effect of fuel concentration is substantial, while only slight improvement is found by including the pressure effect. Therefore, it was decided to drop the pressure effect from the correlation but retain the fuel effect, resulting in Equation (30) as the final correlating equation.

Table 52	ERRORS	OF DI	FERENT	EQUATIONS	OF I	DECALIN
	IGI	VITION	DELAY	TIMES		

Equation	Standard Error of ln τ
$\ln \tau = b_0$	0.87
$\ln \tau = b_0 + \frac{E}{RT}$	0.81
$\ln \tau = b_0 + \frac{E}{RT} + b_1 \ln(c_{0_2})$	0.68
$\ln \tau = b_0 + \frac{E}{RT} + b_1 \ln(c_{0_2}) + b_2 \ln(c_{Fuel})$	0.50
$\ln \tau = b_0 + \frac{E}{RT} + b_1 \ln(c_{0_2}) + b_2 \ln(c_{Fuel}) + b_3 \ln(P)$	0.48

In addition to the equation to be used, there is some cuestion as to the best way to fit the data and the best way to weight the data. Equation (30) could also be written as

$$\tau = \exp(b_0)(c_{0_2})^{b_1}(c_{Fuel})^{b_2}\exp(\frac{E}{RT})$$
(31)

the obvious difference being that in this form the parameters cannot be determined by linear regression. However, there is another difference. The fitting techniques used here seek the least sum of souared deviations between the observed values and the calculated values. Using Equation (31) and equal weighting to fit the data would minimize the sum of  $(\tau_{obs} - \tau_{calc})^2$  for all the data. Using Equation (30) (logarithmic form) would minimize the sum of  $[\ln(\tau_{obs}) - \ln(\tau_{calc})]^2$ for all the data. The resulting equations will be different. The logarithmic form will tend to emphasize short ignition delay times, whereas Equation (31) treats all of the data as being equally imports t and equally accurate. It is not obvious which method is the proper one. The ignition delay data for decalin were fit using the logarithmic equation, Equation (30), using standard linear regression techniques. The results are shown in Figure 47 as the observed ignition

Figures 45 through 47 follow

-88-







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AFAPL-TR-70-71 66758

delay times plotted against the calculated ignition delay times. The regression programs were set up to make these kinds of plots automatically. The correlation using equation (31) determined by nonlinear regression<sup>a</sup>) is shown in <u>Figure 48</u>. The graphs show the expected result; the linear regression gives a better fit at short times and a poorer fit at long times. Also apparent from both plots is that the errors increase as the delay times increase. The problem was rerun using Equation (31) assuming that the variance of the data increased as the times increased. The results are shown in Figure 49. The plot shows that the short times are fit better and the long times much poorer than the other two methods. Since we are primarily interested in short delay times for supersonic engine applications, one of the good fits at short times should be used. On the other hand, the correlation should not almost totally disregard the long delay time data as the increasing variance method does. Therefore, the logarithmic form, Equation (30), using linear regression was chosen as a compromise method for correlating the data. The values of the parameters found by the three methods are given in Table 53.

Re ression Method	bo	bı	₽2	$\frac{E}{\left(\frac{kcal}{gmole}\right)}$
Linear, Logarithmic Form	-16.9	-1.27	0.79	55•7
Nonlinear, Constant Variance	-10.7	<b>-1.</b> 05	0.74	44.2
Nonlinear, Changing Variance	-20.6	-1.18	0.49	55.0

#### Table 53. METHODS OF IGNITION DELAY DATA CORRELATION

Correlating eouations and experimental data can be analyzed statistically with certain restrictions on the behavior of the errors. The most common approach is to assume that the errors are normally distributed and have a constant variance. Based on these assumptions, standardized techniques are available to determine the significance of the parameters, the significance of the correlation, the confidence limits of the parameters, and the confidence limits of the calculated values. As has already been mentioned, however, the errors in the correlation of the decalin data did not have constant variance. The other assumption was checked by performing a simple test to see if the errors were normally distributed. A probability plot was made of the residuals (errors) of the correlation of the data for tetralin. The residuals were plotted as  $\left(\frac{r_1 - r}{n}\right)$ , where  $r_1$  is the residual of the i-th

data point,  $\overline{r}$  is the mean residual, and n is the total number of points. The

a) A proprietary computer program using a modified form of the damped least squares method of nonlinear estimation was used.

Figures 48 and 49 follow

AFAPL-TR-70-71

-89-

values of this quantity were arranged in decreasing order and plotted at equal percentage intervals on a normal distribution scale. If the result is a straight line, then the errors are normally distributed. The result for the tetralin data using Equation (30) is shown in Figure 50. Clearly, the errors are not distributed normally. The results are similar for other fuels. This means that the standard statistical tests do not strictly apply. To see if the form of Equation (30), which involves a logarithmic transformation, caused the un-normality, the data were refit by nonlinear regression to Equation (31). The probability plot of the residuals is shown in Figure 51. The errors still are not normally distributed, so that the problem is not with the fitting method or form of the equation. Since the normality assumption does not hold and the variance is not constant, the standard statistical tests cannot be used. The shape of the probability plots also do not resemble any other common distribution, such as logarithmic, exponential, or chisquare type. As a result, no estimate of confidence limits or significance will be meaningful. Strictly as an approximation one can calculate the standard deviation (standard error) of the calculated values assuming that the errors are normally distributed. This quantity is defined as the square root of the ratio found by dividing the sum of squared residuals by the number of degrees of freedom (number of data minus the number of parameters).

The correlating equation was fit to the ignition delay data for many of the systems studied previously. Table 54 summarizes the results. The parameters for the improved correlation are presented, along with the range of delay times studied experimentally and the standard error of each correlation. The new data on decalin and tetralin have been included in the correlations. The new decalin data were not available when the various fitting procedures were investigated; therefore, the parameters for decalin presented in Table 54 are not the same as those in Table 53. Sufficient data have not been obtained with naphthalene to enable correlation. The numerical values of the parameters probably have little significance. The parameters bo and E are highly correlated with each other so that larger values of  $b_0$  are associated with smaller values of E and vice versa. It does appear that since all of the values of b1 are negative, oxygen has a beneficial effect by reducing ignition delay time, while for the most part, fuel seems to have a detrimental effect. Oxygen concentration seems to have a larger effect than fuel concentration, too. The values of E tend to fall into two ranges. Most of the fuels have values of about 40-50 kcal/gmole, while methylcyclohexane and its dehydrogenation products have values of about 20-30 kcal/gmole. The significance of this, if any, is not known. Figure 52 shows a plot of the correlations for a typical oxygen concentration and a stoichiometric fuel concentration. The uncertainty is typified by the standard error for the decalin correlation, which is shown graphically as the arrows in the margin of the figure. Figures 53 through 62 show plots of the fits to the data. The observed ignition delay times are shown against the calculated times.

#### Rate of Combustion of Tetralin

The data on the combustion of tetralin have been analyzed to obtain rates of combustion. Some trends seem evident but more experiments are indicated. Tetralin is an intermediate in the dehydrogenation of d. alin to naphthalene. It would generally be found in the presence of hydrogen, but its combustion behavior is of interest both with and without hydrogen. The data on the combustion of tetra-

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Fuel	bo	bl	b2	E	Range of τ (μ sec)	Standard Error of ln τ
n-octane	-14.75	-0.76	0.15	41.1	70-4700	0.64
methylcyclohexane	- 2.89	-0.61	0.29	20.1	50 <b>-410</b> 0	0.61
toluene	- 5.84	-0.69	0.32	28.7	100-3400	0.39
toluene + H <sub>2</sub>	- 7.03	-0.68	0.16	26.2	100-4000	0.38
MCH + toluene + H2	- 5.36	-0.64	0.14	21.4	70-4400	0.58
dimethanodecalin	-19.56	-0.68	-0.33	45.8	80-4100	0.62
SHELLDYNE-H®	-13.80	-0.44	-0.28	35.6	60-3600	0.66
SHELLDYNE®	- 8.64	-0.94	0.60	41.0	80-3500	0.43
decalin	-10.94	-1.27	0.93	48.1	604300	0.50
tetralin	-15.99	-1.31	0.41	47.2	40-3800	0.68
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# Table 54. CORRELATIONS OF IGNITION DELAY TIMES

Figures 50 through 62 follow

AFAPL-TR-70-71

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-91-

lin for ignition delay time measurements were analyzed to obtain combustion rates. The rate of combustion can be approximated by

$$r \sim \frac{dc}{dt} = k (c^* - c)$$
 (32)

where

r =the rate of appearance of  $CO_2$ 

 $c^*$  = the ultimate concentration of  $C\Omega_2$ 

c = the current concentration of CO<sub>2</sub>

 $\mathbf{k}$  = the first order rate constant for combustion

Numetion (32) can be integrated to give the reduced concentration of  $CO_2$  as a function of time:

$$\ln \frac{c}{c^*} = -kt \tag{33}$$

It is convenient to estimate k by finding the time required for c to reach half of the final value,  $c^*/2$ . Then k is found simply by

 $k = \ln 2/t_{\downarrow} \tag{34}$ 

Since the mixtures which were burned at lower temperatures encountered the reflected rerefaction shock wave before combustion was complete, much of the data on tetralin combustion could not be used, and values of  $c^*$  could not be determined. However, the acceptable data were analyzed using Equation (34). The results showed much scatter and did not seem to correlate with anything, including temperature. In fact, it seemed that the rate of combustion tended to decrease with increased temperature, which is not logical. In an effort to check this result, an experiment was conducted with the specific aim of finding the temperature dependence of the rate of combustion.

A mixture of 99% argon with tetralin and oxygen representing an equivalence ratio of 0.5 was prepared. Experiments were run, repeating the same conditions until the rate of combustion at a particular temperature could be estimated with confidence. Figure 63 shows the results for 9 psia. The temperatures were spread as far as was consistent with obtaining good combustion measurements. If the temperature was too high, there was no measureable ignition delay and often the mixture defonated. If the temperature was too low, the reflected rarefaction shock wave interfered with the combustion. Nevertheless, there appears to be a definite trend of increasing rate with increasing temperature, in spite of considerable scatter in the data. Figure 64 shows the results for 15 psia. The rates of combustion are somewhat higher but show about the same temperature dependence.

With the knowledge that the proper temperature dependence is found (increasing rates with increasing temperature) and that pressure has a beneficial effect on the combustion rate, the tetralin data were re-examined. There are too few points at each set of conditions to analyze the results statictically, but it

Figures 63 and 64 follow

-92-



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Figure 56. IGNITION DELAY TIMES OF TOLUENE-HYDROGEN MIXTURES

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appears that the results correlate fairly well. Figure 65 shows all of the tetralin combustion data, including the new data, with approximate correlating lines. The results show that fuel concentration has a beneficial effect on the rate, since the data for 99% argon with ER = 0.5 are higher than those for 99% argon with ER = 0.1. Also, as the amount of inert gas is decreased (at ER = 0.1) the rate of combustion increases.

This work contrasts somewhat with the previous work done of the combustion of n-octane and SHELLDYNE-H<sup>6</sup> fuel<sup>3</sup>. At that time no effect of fuel-oxygen ratio or pressure was noted. Further work will be done to clarify and mathematically describe the results, and other systems will be investigated.

### FUTURE PROGRAM

The study of the stability of platinum catalysts for the dehydrogenation of naphthenes is continuing. Future work will include investigations of the effects of metal dispersion and certain metal additives on catalyst stability. The most promising catalysts will be tested in 50 to 100 hour runs at constant reaction conditions. Screening of granular catalysts for dehydrogenation activity will continue on a limited scale as new knowledge indicates any promising compositions.

The search will continue for possible second generation endothermic fuels. Dehydrogenation of bridged-ring naphthenes such as bicyclooctane will yield 1500 to 1800 Btu/lb heat sink when dehydrogenated to bicyclooctatriene. Subsequent double bond isomerization to cyclopropane rings would increase the heat sink to about 2300 Btu/lb. Dehydrocyclization of alkyl naphthenes to the corresponding aromatics, such as 1,2-diethylcyclohexane to naphthalene, would give about 1500 Btu/lb. These studies will be done initially in a pulse reactor. In the near future we expect to examine the possibility of dehydrogenating a highly naphthenic jet fuel and to prepare and test a high molecular weight naphthenic type fuel in the light gas oil range.

We shall continue to search for additives that will enhance the rate of thermal cracking of hydrocarbons. The aim of this study is to reduce both the reaction temperature and the coke made during the cracking reaction.

As a possible method for reducing the pressure drop in the reactor tube, we have been searching for a dispersed-type catalyst. Such a catalyst could be either dissolved in the liquid feed, added as a vapor to the feed vapor, or added as a finely divided solid to the feed vapor. In our previous contract we examined additives that were dissolved in the liquid feed. Some encouraging results were obtained and this study is continuing.

Development will continue on the catalytic wall reactor. The most immediate problem is the measurement of the stability and life of a wall catalyst for dehydrogenation of naphthenes. Catalyst formulations which were found to be most promising in activity screening studies will be used in preparing catalytic coatings for longer stability tests in the bench-scale reactor. If results are favorable, tests will be made in the Fuel System Simulation Test Rig to assess fully the capability of wall catalysts for dehydrogenating naphthenes. These tests will

Figure 65 follows

be made in the Fuel System Simulation Test Rig to assess fully the capability of vall catalysts for dehydrogenating naphthenes. These tests will be of sufficient duration to determine catalyst activity and stability under various conditions. Concurrently, the search will continue for wall catalysts with even more improved mechanical properties, activity, and stability.

The mathematical model of the Catalytic Wall Reactor will be improved. The current model, which includes a kinetic model for methylcyclohexane dehydrogenation, will be used to design the experiments for the Fuel System Simulation Test Rig so as to obtain data at an optimum set of conditions. These data will help to improve the mathematical model. The reaction model can be used to study the effects of geometry, flow rate, heat flux, and fuel conditions on the performance of a wall catalyst. After further development of the reactor model, it will be possible to calculate the upper limits of heat flux, conversion, and temperature of each reaction system as determined by the stability and activity of the catalyst and fuel of that system.

A Catalytic Continuous Stirred Tank Reactor has been designed for use in measuring reaction kinetics and catalyst stability. Equipment modification will begin soon and the reactor will be available for experiments in a short time. The reactor will be used to measure the intrinsic kinetics of dehydrogenation reactions on the best catalyst candidates. Data will be used to formulate kinetic models for the reaction systems. Dehydrogenation of MCH will be studied first to check the reaction kinetics that are currently used. Later experiments will be run to determine an acceptable model for the decalin dehydrogenation system.

Further calibration and evaluation of the beta-ray backscatter instrument on tubes of different dimensions and on tubes made of other alloys will be made. Now that the beta-ray backscatter instrument is available for rating tubes, the Catalyst and Fuel System Test Rig will be used in evaluating the deposits formed by fuels in various physical and chemical states: liquid, gas, vaporizing mixture, reacting mixture, and product mixture. Studies will continue on the use of additives to enhance the thermal and storage stability of fuels. Tests on thermal stability will be conducted in the recently modified JFTOT in order to determine if results from a one-cycle test such as this are better than measurements from a recycle test in the SD/M-7 coker.

Combustion studies on the shock tube will continue with the decalin dehydrogenation system. Ignition delay times will be measured for mixtures of components from the system: decalin, tetralin, naphthalene, and hydrogen, in proportions representative of products from the dehydrogenation reaction. Further experiments will be made on the combustion of tetralin and other fuels to define more accurately the relation between the combustion rate, pressure, oxygen concentration, and fuel concentration.

### RELATED DEVELOPMENTS AND APPLICATIONS

An interesting paper was presented by D. C. Thomas and P. H. Hayes<sup>26</sup>) entitled 'High Performance Heat Transfer Surfaces." They showed that heat transfer coefficients through tube walls could be increased by combining rectangular fins on the outside with twisted tape or coiled wire on the inside. Although the

-94-

AFAPL-TR-70-71



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system studied involved condensing or evaporating water, the results showed benefits in heat transfer coefficients from two- to ten-fold and should be applicable to reacting systems with suitable modifications.

An article by Watkins and Jacobs<sup>27)</sup> details the use of hydrocracking to upgrade vacuum bottoms, low quality straight run, and catalytically cracked kerosene to commercial jet fuel specifications. Although the article applies only to the manufacture of commercial jet fuel, this method could be applied to selected petroleum feed stocks to produce naphthene concentrates which might serve as cheap endothermic fuels.

An article of interest by Whisman and Ward<sup>28</sup>) on the storage stability of high temperature fuels showed by means of fuel components labelled by  $C^{14}$  that the contribution to coker deposits in different thermally stable jet fuels was highest from aromatic compounds containing a  $C_5$ -ring, but the effect of any component was strongly dependent on the gross hydrocarbon environment. It also showed that an amine type antioxidant formed more filterable deposits in a high temperature environment than did a phenolic type.

An important paper delineating the problems inherent in extending flight speeds up to Mach 6 appeared in Aeronautics and Astronautics by M. P. Dunnam and H. I. Bush.<sup>29)</sup> The authors outlined the "broad range of technical advances which will enable turbomachinery to power demanding new missions and will pave the way for exotic new engines that may supplant rockets almost up to orbital speeds." An accompanying article by W. E. Lanar<sup>30)</sup> is also valuable.

Efforts to develop high temperature materials for advanced gas-turbine engines was reported by John C. Freche and Robert W. Hall<sup>31</sup>) of Lewis Research Center, NASA. "NASA Programs for Development of High Temperature Alloys for Advanced Engines" dealt chiefly with work on the development of nickel- and cobaltbase alloys, chromium-base alloys, materials strengthened by dispersion, composite materials, and protective coatings. The development of an advanced cast nickelbase alloy, NASA-TRW VI-A, was particularly significant. This was reported to have a high temperature life of 1000 hr at 1890°F.

Another interesting paper was "The Active Cooling of a Hydrogen Fueled Scram-Jet Engine" by L. L. Pagel and W. R. Warmbold<sup>32)</sup>, McDonnell-Douglas Company, St. Louis. The results of this study indicate that Mach 12 flight at equivalence ratios of less than 1 can be achieved with a regenerative system using the hydrogen fuel as coolant in heat exchangers constructed from super alloys. Heat exchanger designs were based on TD Nickel-chromium construction. The authors suggest that this is a satisfactory material for use as a refractory metal in heat exchangers and that the use of ceramic coatings appears less urgent.

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# APPENDIX

Table 55. Dehydrogenation of Decalin Over Various Catalystsfollowing 99
Table 56. Dehydrogenation of Methylcyclohexane Over VariousCatalysts
Description of the Pulse Reactor
Calculation of Rate Constants (Bench-Scale Reactor) 101
Calculation of Reaction Products for the Thermal Reaction of Bicyclo(2,2,2)octane
Micro Catalyst Test Reactor
Table 57. Dehydrogenation of Methylcyclohexane With Various         Catalysts in MICTR
Table 58. Dehydrogenation of Methylcyclohexane With Various Catalysts in MICTR
Derivations of Equations for the Catalytic Wall Reactor Model 103
Description of the Beta-Ray Backscatter Apparatus for Rating Deposite
Table 59. Ignition Delay Times for Decalin-Oxygen-Argon Mixtures 113
Table 60. Ignition Delay Times for Tetralin-Oxygen-Argon Mixtures . 114
Table 61. Ignition Delay Times for Naphthalene-Oxygen-ArgonMixtures116
Table 62. Physical Properties of Methylcyclohexane/Ethane         Mixtures
Table 63. Physical Properties of Methylcyclohexane/Propane         Mixtures
Table 64. Physical Properties of Methylcyclohexane/n-ButaneMixtures122
Table 65. Liquid Properties of JP-7 Jet Fuel at Saturation 125
Gas Properties of JP-7

Tables 55 and 56 follow

Page

AFAPL-TR-70-71

-99-

# Description of the Pulse Reactor

The pulse reactor was a 1/4-in. OD tube of stainless steel type 304 with 9-1/4 in. length and 0.035 in. wall thickness. Swagelok Tees were fastened at each end and one arm of the Tee served as an injection port. A rubber septum (GLC type) was held in place by the fitting nut and the feed was injected through this septum from a syringe. A 5-in. length of the reactor tube was surrounded by a secondary furnace liner and the whole was heated by an electric furnace. The secondary liner had seven radial drilled holes for thermocouples, and the holes were located as shown in Figure 66. A schematic diagram of the pulse reactor is shown in Figure 67.

All lines were 1/4-in. OD tubing of stainless steel type 304. About 28 in. of line just prior to the reactor was wrapped with heating tape and constituted a gas preheater. About 8 in. of the preheater section was filled with quartz chips (10-20 mesh size).

In the pulse reactor system the carrier gas was metered through a rotameter (Figure 67) and passed through the preheater section into the reactor. The exit gas passed into a manifold and then into the GLC. The purpose of the manifold was to maintain the exit gas pressure slightly greater than the gas pressure in the GLC. This was done by adjusting the pressure control valve and the vent valve. The manifold was wrapped with heating tape and was maintained at 302° to 356°F. The injection port temperature was about 450°F. The pressure control and the vent valves were needle valves (Hoke No. 1315) and the GLC valve was a lever operated valve (Hoke No. 490).

To carry out an experiment the reactor was brought to temperature, and the carrier gas flow rate, reactor pressure and manifold pressure were adjusted by means of the appropriate flow control valves. Then with inert gas flowing to the GLC a pulse was injected through the lower injection port and subsequently analyzed. This gave an analysis of the starting material. A pulse was then injected in the top injection port, passed over the catalyst, and analyzed.

In this system the space velocity was obtained from the inert gas flow rate. Figure 68 shows the pulse reactor system with the secondary furnace liner in place.

Figures 66, 67 and 68 follow

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hun 45, 115,5-	:71	1.1%	1.75	1 1 10 .	: 177	170-1	1178-2	170	- 1-2-4 1-2-4	181-5	 -52	14.4	1894	185	18
Gatalyst No.		$(1,1,\alpha_1)\in A_1$		1		1.000-11	dy.	۰.		194	- 1 S				108
Temperature, "F				1											
Block	73421	952	1 102	1 (J41)	637	1.520	1310	12.51	73.1		1.00	102	1202	800	9
Wail	750	793-97	1214	1 707-04	650-63	$E_{\rm S}$ (b)	257	$-\alpha \beta \gamma + \alpha \gamma$	7 55 - 4	15 int	1.5.1	distant.	10.28-44	741-47	812
Catalyst Bed	019-30	(shi='j')	711-53	6-4-15	しかいやい	-73-80	714Kh	and the second	7525-54	States -	1, 18	1110 M	1156-118	614-21	- 589
Profile	1619-26	- 653-60	1014+7.15	1.629-10	- 1947-19 <b>4</b>	- 575-75	121-11	e par la Ke	$\{r_i, i_i\} = \{r_i\}$	$c_{\rm H}$ ( $c_{\rm M}$ )	1.1.1.1.44	711-29	- 912 - LUSB	- 517-24	650
	- neto - 55		711-12	610-15	- 646=53	000-07	2 Krace	80.000	1 (K (m ) ()	6.95-15	ord.	716	812~64	650-55	662
	19121-46	- 1947- <b>•</b> )) '	74 s	1.4646-40	671-75	700	YTT = t + t	iv. S. S	1.37	61.1.4.7	7.57+ 4	747-60	910-12	hó4-ó6	536
Product Analysis, fw															
Cracked, 11q.	1 0.0 G	0.0 .	0,0	0.05	· · · · · ·	2.11	$-2.0^{h}$	-1.7			0.1	18.	6.2%	6.01	a
trans DHN	( <u>א</u> גא	37.8"	22.9	413 (7 <sup>51</sup> )	$-99.7^{a}$	24 K	- 10.6 -		, L (), ()	- 51.00	20.1	25.3	12 94)	34.6	30
ets DHN	34.9	29.4	25.5	29.8	24.0	10.8	17.717	15.5	28.3	÷4	199	10.1	15.8	34.9	20
THN	11.8	9.2	5.9	16.4	12.7	8.5	5 e .	4.7	11.4	$1 \in A$	7.6	5.5	4.1	12.7	9
N	22.0	55.7	45.7	21.1	35.4	47.1	58.5	67.7	1.1.1	53.5	44.6	14.2	57.70	80.8	30
thers	0.0	0.0	0.0	L 0.0	0.0	0.0	0.0	5.1	0.0	·•••)		· · ·	3.117	0.0	0
DHN Conversion, %	33.5	42.7	. 51.4	37.0	48.0	55.0	63.7	75.5	26	1.1		12.6	71.2	33.2	39
Sclectivity for THN + N 1	100	100	100	100	100	100	99.6	96.9	- 39.4	09.7	up.8		86.2	100	100
∆T °E	.,	00	1.7												
, ·		60			11	ſ	83	144	Υ I	4	11	1.1	140	Ĩ	20
a) cis to trans isomerizat	ion durin	g the ru	n				·			h) emeri	red afte	r cis DHN	1		

n/ emerged atter fis DHN
 n) catalyst almost completely deactivated at
 n) emerged after trans DHN
 thelve components
 me component merged after trans DHN (7.4 r) five components

a) cis to trans iSSuerization during the run
b) two components
c) four components
d) appeared to be more than one component
e) eight components
f) two components that emerged after cid DHN and one component that energed efter trans DHN

Run No. 11623-	- <u>1</u> ń	17-1	17-2	18	50	21-1	21-2	55	23	24	25	~6 <b>-</b> 1	26-2	27
Catalyst No.	·<	1086	-1378	• >	<		19869-138	<del>зв</del>		<u> </u> <		1 /860-13	9B	
Temperature, °F Block Wall Catalyst Bed Profile	9110 763-65 795-97 650-62 622-26 628-30	932 835-42 887 698-72 660-62 668	1022 914-20 972-77 750-92 704-09 709-11	1.112 <sup>b)</sup> 990-100 1060-46 820-87 752-94 766-90	842 4705-11 783-95 621-44 603-08 614	932 768-77 880-84 679-94 635-40 640-50	1022 840-46 977-74 727-58 675 689-87	1112 918-39 1060-67 790-887 712-07 724-29	1202 1144-75 1155-53 1132-55 1028-1155 1028-1155	842 1745-48 748 1650-68 1650-68 1633-39 01.0-42	932 804-15 873-75 694-718 676-78 684-86	1022 878-94 959-68 740-95 712-20 729-30	1112 956-99 1053-67 826-64 770-97	1202 1118-59 1154 1099-1139 916-1008 896-932
Product Analysis, % Cracked, Liq, trans DHN cis Dhd THN N Others	1.7 32.8 37.2 9.4 16.8 2.1	0.0 <sub>8</sub> ) 28.2 32.0 8.7 31.1 0.0	0.0 24.2 27.1 5.4 43.3 0.0	0.0 16.8 27.0 3.2 55.0 0.0	0.0 <sub>a</sub> ) 34.1 28.4 15.7 21.8 0.0	0.0 31.3 23.0 12.8 32.9 0.0	0.0 25.1 10.5 8.5 46.9 0.0	0.2 18.9 15.1 4.1 61.7 0.0	6.1 <sup>d)</sup> 20.5 47.0 4.4 7.7 14.3	0.0a) 29.2 40.2 9.5 21.1 1.0	0.02) 26.42 35.2 7.1 31.2 0.0	14 54 54 54 54 54 54 54 54 54 54 54 54 54	0.2 14.9 25.4 4.6 54.8 0.1	1.8 <sup>9)</sup> 15.0 32.6 7.8 40.7 0.0
DHN Conversion, fw	29.7	39.6	48.5	56.0 <sup>b)</sup>	37.2	45.5	55-2	65.9	- 32.2 <sup>f)</sup>	30.3	40.0	44.7	59.5	52.2 <sup>h)</sup>
Selectivity for THN + N, 164	86.9	100	10)	100	100	100	100	100	67.0	100	100	101	99.5	96.9
∆r, °F	13	23	41	67	23	50	31	97	<sup>f)</sup>	18	23	50	139	<sup>h)</sup>
a) cis to trans isomerization b) resection time 15 minutes c) are component merged aft d) eight components c) ane component emerged aft f) reaction time 15 minutes g) four components	in during ter trans ter trans	g the run s DHN (1 s DHN (4	n •9%) and •8%) and	1 7 () r 1	<ul> <li>a) reaction</li> <li>a) five on</li> <li>b) emerged</li> <li>c) tem com</li> <li>c) six com</li> <li>c) me com</li> </ul>	n time mponent i after nponents i after nponents mponent	10 minutes s trans DHM cis DHN emerged a	es N after tr	ans DHN ((	0.7%) and <b>4</b>				

21

Run No. 11623-	46	47	48-1	46-2	49	51	52	53-1	53-2	54	56-1	56-2	57	58
Catalyst No.	\		10860-146			<		10860-1	46	·>	s		10860-14	<b>·</b> ۲
Temperature, °F Block Wall Catalyst Bed Profile	842 748-52 689-98 603-10 601-04 603-05	932 819 748-63 635-44 635-53 635-57	1022 887 802-30 665-76 665-67 665-69	1112 945-39 864-920 707-22 707-05	1202 <sup>0)</sup> 1040-44: 1112 892-930 783-99 783-813	842 776-79 765 599-604 597-99 597-601	932 856-54 830-28 630-35 628-30 628-30 626	1022 942-40 909-72 658-71 069-75 668	1112 982-79 977-95 700-11 700-696 702-35	1202 <sup>c)</sup> 1059-80 1061-62 763-806 765-831 767-833	842 727-32 701-74 648-68 648-66 660-66	932 79 <b>4-99</b> 853 <b>-58</b> 994-716 687-93 711	1022 862-74 943-56 748-88 734-43 765-66	1112 945 <b>-100</b> 1049 <b>-61</b> 873-100 802 <b>-69</b> 831 <b>-51</b>
Product Analysis , %w Cracked, liq trans #NN cis DHN TMA N Others	0.0 36.2 19.9 18.9 25.0 0.0	0.0 31.0 16.6 15.9 36.5 0.0	0.0 25.0 12.7 10.6 57.7 0.0	0.1 18.3 9.8 5.9 65.9 0.0	3.7 <sup>b)</sup> 10.5 7.8 2.4 75.6 0.0	0.0) 36.7 1 <b>9.4</b> 17.2 26.7 0.0	0.0 29.9 <sup>a</sup> 16.1 14.5 (39.5 0.0	0.0 (4.7 12.9 0.8 52.4 0.0	1948 1848 1944 1944 1944 1944 1944	3.5 <sup>0)</sup> 10.2 5.6 2.3 78.4 5.0	(* )4) 26.14) 4*.6 72.1 22.1 0.0	0.0 23.7 39.3 4.8 4.2 1.0	0.1 20.5 36.1 5.5 37.8 0.0	0.7 18.1 57.5 5.9 37.8 0.0
DHN Conversion, Sw	43.7	52.2	62.1	71.8	82.3°)	43.7	53.8	62.2	71.?	84.1°)	۰	36.7	43.2	44.2
Selectivity for THN + N, fw	100	100	100	; 99.8	o4.3	100	noo	1.89	99.7	95.8	105	1.00	99.8	98.0
Δ*, °F	9	1	28	; 56		,	÷	18	18	66	14	17	40	135

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a) cis to trans isomerization during the run
b) seven components
c) reaction time 10 minutes
d) six components
e) four components
f) emerged after THN

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			1.88.93	50A	1.	1.06-0-1.54	A S		10000-13	ha .		1	10869-1	દુ:ભ	``		~ 10	860-1429		``	
$\begin{bmatrix} 1 & 1 \\ 804 & 92 \\ 1070 & 96 \\ 711 & 29 \\ 710 \\ 747 & 40 \end{bmatrix}$	117-117 1-1178 - Arka 117-1179 - 117 117-1179 - 117 117-1179 - 117 117-117		- 98.0 (310) - 294 (286) - 796 (286) - 795 (286) - 79 (298) - 74	$\frac{1}{7}\frac{675}{67} + \frac{1}{26}\frac{675}{67} + \frac{1}{26}\frac{675}{67} + \frac{1}{26}\frac{1}{7}\frac{1}{12} + \frac{1}{26}\frac{1}{12} + \frac{1}{26}\frac{1}{12}\frac{1}{12} + \frac{1}{26}\frac{1}{12}\frac{1}{12}\frac{1}{12} + \frac{1}{26}\frac{1}{12}\frac{1}{12}\frac{1}{12} + \frac{1}{26}\frac{1}{12}$	946) 797-19 797-34 849-68 839-53 844-35	700-200 700-200 700-8884 1001-2884 1001-2884 1005-708	1000 985-100 981-86 906-77 737-970 772-950	545-445 745-445 745-445 745-445 745-445 745-445	100-340 766-340 636-340 636-340 636-36 676-56	1 1027 016-84 020-84 020-84 5520-84 720-861 729-856	842 767-48 767-48 70-54 822-28 604-88 608-10	050 808-15 808-55 608-17 637-59 644	1020 876-92 911-46 695-125 662-75 671-80	1133   948-77   948-77   997-1054   736-831   707-82   785-23	1202 1125-66 1150-55 1085-1139 885-1202 878-56	:   842   754-58   808-02   640-48   597-99   604-08	932 792-90 896-91 675-78 626 639-35	1022 856 982-77 712-20 651 662-66	11.12 938-41 1061-98 734-48 682-85 797	1202 1006-18 1141-44 848-903 752 788-76	1
0.5 <sup>h</sup> ) 20.9 26.1 5.5 57.2 0.0	0+0 12+9 15+8 4+1 57+7 <sub>2</sub> 5 3+1	- 55 <b>h</b> 54-6 51-9 12-7 25-9 25-9	0,0 50,6 29,6 9,2 30,6 30,6 30,6	5.42) .8.637 37.4 5.5 27.7 1.1	0.0 07.0 46.1 7.1 10.4 1.0	) 24.3 44.3 5.1 26.3 0.0	0,50) 25,48) 54,9 3.0 10,2 2,44)	0.0 28,8ª 45.7 6.9 20.6 0.0	9.0 25.7a) 59.5 5.0 29.8 29.8 3.0	0.9 24,4 41.9 2.2 20.0 1.84)	0.5 40,4%) 25.8 15.6 18.2 0.0	0.9 34,88) 21.0 12.5 51.7 0.0	$ \begin{array}{c} 0.8 \\ -26.4^{a}) \\ 17.9 \\ 6.8 \\ 48.8 \\ 0.0 \end{array} $	0.6 20.5 13.0 4.2 62.5 0.0	7.6 <sup>p)</sup> 12.3 18.4 6.3 39.6 15.5 <sup>q</sup> )	0.0 25.3 <sup>n</sup> ) 23.0 17.8 23.9 C.0	0.0 30.9 <sup>a</sup> ) 18.2 10.1 34.8 0.0	0.0 03-7 13-5 13-3 13-3 19-8 0.0	0.1 17.1 11.6 8.2 63.0 0.0	2.5 <sup>r</sup> ) 11.6 10.7 3.6 71.3 0.2	:
62.6	79.21	53.2	57.5	53.7 <sup>m)</sup>	26+5	51.1	19.4 <sup>m</sup> )	27.2	34.5	33.4 <sup>m)</sup>	33+5	44.0	25+5	66.6	69.2 <sup>m)</sup>	41,4	50.7	56.3	71.•2	77.6	
99.5	56.0	1:1	100	95+5	100	100	79.4	100	1.00	92.2	100	100	100	100	65.8	100	100	100	99.8	95.6	
el 25 - 1	140	7	- 94 - 19	248	27	-04	182	13	56	241	5	ò	32	95	227	5	4	7	36	56	I
		<u>.</u>			<u> </u>				1				! ·			ł	1_				)
r cis DHN pst comple r trans N mente t emerged nts	etely dea HN after tr	ictivated Nuns DHN	i at the (7 <b>.6%</b> ) a	end of thi nd one com	srun ponent s	fter dis D	DHN (7.9%)														

26-1		27 50-1	1 30-2 31	32-1 32-2	34-1 34-2	35 3	7 38-1	38-2 39	40	42-1 4;	2-2 43	44-1 44-2
10860-13	73	· > . <	10860-1	.35A>	< 10860 <b>-1</b>	43B > <	·····	10860-1320	>	<	10860-14	6>
1022 878-94 959-68 740-95 112-20 729-30	1112 956 <b>-</b> 99 1053 <b>-67</b> 826 <b>-6</b> 4 770 <b>-</b> 97 780 <b>-</b> 97	1200 842 1118-59 747-50 1154 512-79 1054 1159 640-57 9105400, 017-24 596-932 622-26	932 1022 812-13 874-78 5 884-76 973-51 687-96 716-43 555-57 1003-31 662-60 702-636	1112 1202 946-52 1036-54 1049-56 1134-48 770-801 869-1000 (22-34 /95-315 3 743-45 813-15	842 1932 727-40 788-884 806-05 910-03 675-722 767-07 633-40 689-727	1022         842           982-1004         716           1006-995         666           1000         614           959-997         615           869-808         633	932 23 788 -81 720-40 28 651-53 -21 658 -35 680-78	1022 1112 860 937 770-95 831-71 689 729-32 702-698 743 734-25 784-81	1202 1040-44 1004-49 819-22 822-19 873-62	842 932 750-32 81 615-28 65 586-91 62 597-601 635 612-14 651	2 1022 7-10 846-49 7-71 693-712 -22 651 6 673-69 1-49 694-91	1112 1262 <sup>h</sup> ) 954-57 1040-46 2 738-74 873-92 684 759-72 714-11 792-817 747-43 533-73
0.0 25.2 29.9 4.1 40.8 0.0	0.2 14.9 25.4 4.6 54.8 0.1	$\begin{array}{c} 1.8^{a} \\ 15.0 \\ 32.6 \\ 7.8 \\ 7.8 \\ 15.7 \\ 13.7 \\ 13.7 \\ 13.7 \\ 10.7 \\ 21.4 \\ 0.0 \\ 0.0 \end{array}$	$\begin{array}{c c c} 0.0 \\ 30.9 \\ 30.9 \\ 26.3 \\ 26.3 \\ 21.6 \\ 32.9 \\ 32.9 \\ 46.2 \\ 30.0 \\ 0.0 \\ 0.0 \end{array}$	$\begin{array}{cccc} 0.2 & 1.4^{m} \\ 18.5 & 13.0 \\ 16.5 & 14.9 \\ 4.2 & 3.6 \\ 60.6 & 67.1 \\ 0.0 & 0.0 \end{array}$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{c c} 2,1^{p} & 0 \\ 24.4 & 28 \\ 54.9 & 36 \\ 5.0 & 11 \\ 4.2 & 24 \\ 9.4^{q} & 0 \end{array}$	.0 0.0 .2 <sup>e</sup> ) 24.8 .0 31.3 .8 8.7 .0 34.2 .0 0.0	$\begin{array}{cccc} 0.0 & 0.4^{g} \\ 19.7 & 16.8 \\ 26.6 & 21.3 \\ 6.0 & 3.7 \\ 47.7 & 57.8 \\ 0.0 & 0.0 \end{array}$	2.6 <sup>r)</sup> 12.8 18.3 3.6 62.5 0.1	0.0 36.4 <sup>a</sup> ) 31 20.2 16 18.5 15 24.9 36 0.0 0	$\begin{array}{c} 0.0 \\ 0.1^{a} \\ 24.8 \\ 7 \\ 12.8 \\ 4 \\ 10.2 \\ 58 \\ 52.2 \\ 0.0 \\ 0.0 \end{array}$	$\begin{array}{cccc} 1.2 & 4.3 p^{)} \\ 19.0 & 9.5 \\ 9.8 & 6.5 \\ 4.8 & 2.3 \\ 64.5 & 76.4 \\ 0.7^{\circ} & 1.0^{3} \end{array}$
44.7 100 56	59+5 99+5 139	52.2 <sup>h)</sup> 34.8 96.5 0.0 <sup>h)</sup> 16	42.6 52.2 0.0 0.0 9 27	64.9 72.0 99.7 97.9 31 131	28.9 33.6 90.7 100 47 160	20 <sup>+h)</sup> 35 43-1 100 h) 14	.5 43.7 100 29	53.5         61.7           100         99.3           25         40	68.3 95.5 47	43.2 52 100 100 13 14	100 20	71.1 83.9 96.9 93.3 54 - <sup>h)</sup>

### after trans DHN (0.7%) and one after cis DHN (0.3%)

-1	56-2	57	58	64	65-1	65-2	66	67	69	70-1	70-2	711	71-2
		10860-147	>	<	1	0660-1358-		>	<	108	60-163a		>
-32 -74 -68 -51	932 794-99 853-58 694-716 687-93 711	1022 862-74 945-56 748-88 734-43 765-66	1112 945-1000 1049-67 873-1008 802-69 831-51	842 761-63 624 698-14 014-17 630-55	932 835-33 648-53 644-46 657-53 578-74	1022 905-07 680-89 680 698-94 729-25	-112 975-71 722-34 722 745-32 784-79	1202 1058-62 781-828 788 820-12 878-62	842 766-64 745-52 619-24 620-22 626-24	932 842-38 822-33 651-60 651-58 651-49	1022 912 905-42 656-704 689-93 698-94	1112 984-75 101 <b>3-42</b> 729-77 725 734-25	1202 1148-79 1143-50 1011-1144 869-1139 842-1132
0.0 11) 0.2 11 0.2	0.0 23.7 39.3 6.8 30.2 0.0	0.1 20.5 36.1 5.5 37.8 0.6	0.7 <sup>e)</sup> 18.1 37.5 5.9 17.8 0.0	31 26.0 16.4 26.2 9.0	0.0 25.6 20.4 11.9 38.1 0.0	0.0 21.9 15.4 7.3 55.4 0.0	0.2 16.6 13.2 4.2 65.5 0.0	3.0 <sup>d</sup> ) 12.6 10.9 3.1 70.4 9.0	0.0 34.18) 26.4 14.1 25.4 0.0	0.0 33.8 <sup>a</sup> ) 21.3 12.1 32.8 0.0	0.0 24.0 21.5 9.0 45.4 0.0	0.3 17.4 18.9 7.4 55.1 0.0	6.8 19.0 35.3 7.5 27.1 4.31)
<b>.</b>	36.7	43.2	44.2	42.4	53-7	62.6	70.0	77.9	39.3	44.7	54.3	63.7	45.5
•	100	99.8	<b>98.</b> 0	100	100	100	100	9 <b>4.</b> 4	1 <b>0</b> 0	100	100	99.5	75-2
	22 22	40	135	6	5	9	12	47	7	11	37	48	290

Table 55.

# Table 55 (Contd). DENYDROCENATION O

Run No. 115-5-	146	147	148-2	148-2	149	99	100	101	105	106	107	102	1-1-05	104	111
Catalyst No.	×		9874~1198		>	•	9874-1864		•,	9874-187A			9874-189 <b>A</b>	,	<
Temperature, "F Block Wall Catalyst Bed Profile	842 704-07 617-32 603-09 609-15 626-30	932 759-65 657-78 635-44 544-50 664-68	1022 824-30 698-743 671-80 682-89 711 14	1112 905-18 744-88; 712-34 734-38 774-72	1202 1015-1175 962-1156 815-1130 8,3-1017 1015-1175	842 725-29 650-67 632-41 637-44 651-55	932 788-99 707-81 675+94 686-87 696-700	1022 858-997 806-995 723-984 725-905 745-797	842 727-37 669-89 6114-95 648-53 657-60	932 190-813 123-824 687-723 689-700 702-05	1022 873-1011 685-997 752-997 740-997 750-987	842 736-41 667-84 644-51 649-53 662-64	932 799-820 750-810 684-723 691-702 707-09	1022 878-1008 844-5 77 750-995 743-992 758-988	842 761-828 770-817 691-815 658-799 656-763
Product Analysis, fw Cracked, liq trans DHN cis DHN THN N Others	0.0a) 30.9 29.4 17.9 21.8 0.0	0.0 27.7 <sup>a)</sup> 24.8 14.2 33.3 0.0	0.1 23.5 20.3 9.3 45.8 0.0	0.6 18.8 18.5 5.1 56.9 0.1	4.2 15.2 27.1 5.2 46.9 1.5	0.0 29.0 <sup>a</sup> ) 40.9 9.9 20.1 <sub>b</sub> ) 0.1	0.0 26.3 <sup>e</sup> ) 37.0 7.7 28.8 0.2 <sup>b</sup> )	0.2 22.2 40.4 6.5 28.9 1.8 <sup>d</sup> )	0.0 26.6 45.2 8.2 20.0 0.0	0.0 25.6 42.0 6.1 26.3 0.0	0.1 22.5 50.5 5.1 19.4 2.4	0.0 26.4ª) 45.9 7.8 19.9 0.0	0.0 24.6 41.6 6.0 27.8 0.0	0.3 22.5 53.a 4.4 15.0 <sub>f</sub> ) 3.9	$\begin{array}{c} 0.3\\ 28.3\\ 52.1\\ 5.3\\ 11.0\\ 3.0\\ \end{array}$
DHN Conversion, 164	39.3	47.1	55.8	62.6	57.4	29.7	36.3	37.0	27.8	32.0	26.6	27.3	33.4	23.2	19.2
Selectivity for THN + N, 4w	100	100	99.8	98.4	90.1	99.3	99.4	97.0	1.00	100	90.6	100	100	81.9	82.8
ΔΙ, °F	14	22	45	133	389	20	74	261	20	101	257	16	92	248	140
				1		1	1	4	1		1	-			1

a) cis to trans isomerization during run b) emerged after cis DHN c) one component emerged after trans DHN (0.7%) and one after cis DHN (0.7%) d) one component emerged after trans DHN (0.9%), and one after cis DHN (0.9%) e) one component emerged after trans DHN (1.0%), and one after cis DHN (0.9%) i) one component emerged after trans DHN (1.0%), and one after cis DHN (1.4%) i) one component emerged after trans DHN (1.5%), and one after cis DHN (2.4%) g) emerged after cis and trans DHN

Run No. 11325- Catalyst No.	138 <	139	140-1 280-15A	140-2	141 >	118 < 1	i 119 0280 <b>-</b> 50A -	120	<u>126</u>	127	128 <b>-1</b> 10280-77	128 <b>-</b> 2	129 ····>	130 ~	131
Traperature, °F Block Well Gatalyst Bed Profile	842 736-41 666-82 635-42 635 644	932 799-802 711-34 669-78 669-71 684	1022 874-78 759-84 711-16 711-08 730-25	1112 948-46 806-42 748-52 750-43 748-46	1202 1044-49 903-1010 822-44 822-17 1044-49	8k2 802-26 797 <del>-</del> 802 770-808 686-81.2 802-26	932 885-920 873-900 838-902 806-903 788-903	1022 972-1008 950-88 918-88 894-988 889-988	942 711-14 617-28 604-17 615-26 633-42	932 766-70 650-64 635-40 646-57 671-74	1022 824-26 687-7.59 667-75 682-86 714-16	1112 891-94 770-840 716-22 732-29 770-65	1202 977-1035 592-1121 770-73 797-804 853-33	842 640-42 630-42 621-26 628-32 640-42	932 680 662 <b>-78</b> 651 <b>-55</b> 662 680
Product Analysis, Sw Cracked, liq trans DHN cis DHN THN N Others PHN Conversion, Sw Selectivity for THN+N, Sw AT, F	0.0 29.9 39.1 11.0 20.0 0.0 30.6 100 16	0.0 26-4 35.0 9.2 29.5 0.0 38.3 100 23	0.0 2).8 29.6 8.1 40.5 0.0 48.2 100 25	0.4 16.4 24.3 7.2 51.4 0.2 59.3 98.1 36	1.9 11.2 21.5 5.6 59.4 0.4 66.9 96.6 107	1.5 27.0 63.0 2.1 1.7 4.7 35.4 126	1.5a) 25.6a) 67.7 1.7 0.9 1.6 <sup>a</sup> ) 5.7 40.4	2.5) 26.8 63.0 1.8 1.0 4.9 10.2 25.5 	0.0 34.5 26.5 17.5 21.3 0.0 38.1 100 13	0.0 30.8 21.5 14.3 33.5 0.0 47.2 100 14	0.0 25.3 17.3 10.5 46.9 0.0 57.0 100 22	0.5 18.3 17.0 6.7 57.2 0.1 64.3 98.8 170	3.2 12.7 14.1 4.1 64.7e) 1.2 72.8 94.0 229	0.3 34.5 <sup>a</sup> ) 27.4 14.6 23.4 0.0 37.6 99.7 13	0.3 31.0 21.6 11.5 35.5 0.0 47.0 99.4 14

a) cis to trens isomerization during run b) emerged after naphthalene c) two components - one emerged after tra d) emerged after cis DEN e) three components, one of which emerged f) two compenents that emerged after cis one to trans isomerization ouring run emerged after naphthalene emerged after naphthalene emerged after cis DHN three components, one of which emerged after trans DHN, cis DHN and naphthalene respectively two components that emerged after cis DHN and naphthalene respectively

Comments .

AFAPL-TR-70-71 66504

# HYDROGENATION OF DECALIN OVER VARIOUS CATALYSTS

105	3.4	111	112	115	96	- 97	98	55 1	ijh	95	142	143	144-1	144-11	108	109	110	114	13.5	716-1	116-2
9874-189	Α.	ولما ا	74-180H	·•	· /	7874-192/	N >	isa g	874-1044	. >	s	9874-1	oji <b>A</b>		· · 9	874 - 194B	· · · · >	ا بند معدم -	··· 1038	0-13	· ····································
932 799-220 718-817 684-723 691-702 707-09	1.322 878-1.008 714 - 97 750-565 743-968	842 761-828 770-817 691-815 658-700 656-765	-132 826-918 838-907 754-905 696-887 696-852	1022 966-95 966-95 907-95 828-992 788-982	342 750-36 657-78 639-48 644-48 644-48 655	030 797-830 707-864 682-03 682-03 091-93	022 891-1011 878-999 756-997 754-986 743-956	842 725-50 655-71 633-42 637-44 651-57	952 705-97 705-18 671-87 676-86 694-700	1022 855-988 774-995 716-972 721-85t 745-83	840 734 651-68 632-35 655-39 651+53	432 797-802 700-45 660-80 671+76 669-91	1022 869-92 774-928 709-5 712-25 730-28	1112 965-1085 970-1074 794-1064 763-1040 784-923	842 740-815 743-806 671-799 650-761 653-700	932 797-909 784-900 704-894 689-860 648-804	1022 912-1004 927-990 851-990 765-986 761-966	842 727-32 651-66 628-37 633-37 644-50	932 797-801 698-756 668-80 673-80 687-91	1022 869-950 748-986 707-904 707-904 705-768 736-769	1112 968-1094 965-1076 812-1072 788-1069 806-1069
0.0 24.6 41.6 6.0 27.8 0.0	0. * 22.5 53.9 4.4 15. 7 3.9	$(38.5^{n})$ $(38.5^{n})$ (52.1) (5.3) (11.3) (5.3) (3.3) (3.3)	$\begin{array}{c} 0.6\\ 26.2\\ 50.6\\ 6.0\\ 12.3\\ 4.3^{g} \end{array}$	1.4 23.3 55.6 5.6 7.9 7.1g)	0.0 29.1 41.5 9.4 20.0 0.0	0.0 24.6 7.7 7.4 28.0 0.2	0.4 22.6 46.5 5.8 20.6 4.0	0.0 27.5%) 42.3 9.5 20.6) 0.18)	0.0 25.3 37.2 7.3 30.0 0.2 37.0	0.2 22.1 37.0 5.5 33.6 1.6	0.0 28.7 <sup>a</sup> ) 21.5 9.7 20.0 0.0	0.0) 26.44) 36.8 7.9 28.9 0.0	0.0 22.7 34.7 7.2 36.1 0.0	1.1 17.4 39.0 7.4 30.2 4.9 <sup>g</sup> )	0.3 $30.0^{a}$ ) 40.1 5.9 13.9 $1.8^{b}$ ) 21.5	9.7 27.00 45.3 6.1 16.0 3.4g) 25.8	1.4 22.5 51.5 6.2 11.6 6.8 <sup>g</sup> )	0.0 27.2 42.1 10.2 20.5 0.0	6.0 25.2 36.5 7.9 30.4 0,0	0.2 22.8 35-3 36-2 0.0	4.0 21.5 54.1 3.5 7.6 9.3 <sup>K</sup> ) 24.0
100	81.9	H2.8	78.5	63.3	100	29.4	85.5	99.7	99.2	95.6	100	1.00	100	86.1	90.2	84.1	68.0	100	100	99.8	43.8
92	248	140	191	194	ed.	i⇔7 L	252	18	43	256	17	45	154	272	123	315		14	38	238	281

129	130	131	132-1	132-2	133-1	1.21	122	123	124-1	124-2	1 1 34	135	130-1	136-0	1220 2	_
		ł j	0280-1190		•>	<	10	390-124C		· · >	/ ·	10280	-1578	190-2	>	
1202 977-1035 892-1121 770-73 797-804 853-33	842 640-42 630-42 621-26 628-32 640-42	932 680 662-76 651-55 662 680	1062 729-27 700-16 684-87 700-698 729-27	1112 786-79 758-61 725 747-43 785-73	1202 860-38 842-1013 7 <b>89-</b> 903 812-01 860-38	842 732-38 662-82 651-62 664-71 662-87	932 797-802 704-31 691-702 709-14 797-802	1022 567-69 748-776 736-47 763-66 799	1112 948-50 799-838 790-801 630-31 880-76	1202 1058-1179 966-1157 896-1150 925-1146 981-1146	842 925-27 637-53 635-40 644-51 725-27	932 786-90 676-98 673-80 684-89 707-12	1022 851-55 716-43 709-18 725-29 756-58	1112 923 763-94 759-61 779-77 817-1,	1202 100602 833-91 817-24 842-38 885-78	
3.2 12.7 14.1 4.1 64.7 1.2	0,1 34,5 <b>e</b> ) 27,4 14,6 23,4 0,0	0.5 31.0 <sup>a</sup> ) 21.6 11.6 35.5 0.0	0.5 26.7 <sup>a</sup> ) 16.6 7.9 48.0 0.1	1.2 20.8 11.6 5.2 60.7 0.3	3.6 13.1 9.4 3.9 69.0 0.3	0.6 27.3ª) 47.0 5.6 19.5 0.2	1.2 25.9 39.7 4.7 28.3 0.0	2.2 23.3 33.5 4.6 35.8 0.6 <sup>r</sup> )	3.2 19.1 27.5 5.4 42.5 2.3	7.6 16.y 36.6 7.9 25.4 5.6 <sup>e</sup> )	0.0 30.3a) 59.5 7.3 22.9 0.0	0.0 27.9ª) 33.8 6.1 32.2 0.0	0.2 24.3 28.1 4.1 42.8 0.2 <sup>d</sup> )	0.3 19.6 23.7 3.2 53.0 0.2 <sup>()</sup>	1.9 13.9 17.8 2.5 63.4 0.5	
94.0 229	99+1 13	47.0 99.4 14	56.3 98.6 16	<b>67.</b> 0 97.6 23	77•1 94.0 171	25.3 97.6 20	34.0 95.9 29	43.2 92.6 27	53.4 89.0 40	46.5 70.8 234	29.8 100 16	37.9 100 22	47.2 99-2 31	56.3 98.1 58	ri7.9 96.5	

Table 55 (Contd).

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Table 56. DEHYDROGENATIONS OF METHYLCYCLOHEXANE OVER VARIOUS CATALYSIS

Cetalyst Stability Tests

Feed: Catalyst Volume: Pressure: Block Temperature: Reaction Time at Fech LHSV: 30 minutes

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Figure 66. SECONDARY FURNACE LINER FOR PULSE REACTOR

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# Figure 67. SCHEMATIC DIAGRAM OF PULSE REACTOR

AFAPL-TR-70-71 64458



Figure 68. PULSE REACTOR SYSTEM

AFAPL-TR-70-71 64286-5

# Calculation of Rate Constants (Bench-Scale Reactor)

First order rate constants were calculated based on the rate of disappearence of the starting material according to the following equation:

$$k = \frac{LHSV}{3600} \times \frac{\rho \times 22, 412}{MW \times P} \times \frac{T}{273} \times 2.3 \log\left(\frac{1}{1-f}\right)$$
(35)

where:  $k = first-order rate constant in sec^{-1}$ 

LHSV = liquid hourly space velocity (i.e., volumes of feed/volume of catalyst bed per hour)

MW = molecular weight

- P = reactor pressure in atmospheres
- T = reaction temperature in °K (reactor wall temperature)
- $\rho =$ liquid density
- f = fraction component reacted

# Calculation of Reaction Products for the Thermal Reaction of Bicyclo (2,2,2) octane

Both BCO and TMB react thermally to form reaction products 1, 2, 3 and 4 (Tables 4 and 5). With BCO in TMB feed the amount of component A formed from TMB only is given by

$$X_{A} = \frac{C_{r} X_{A}^{\circ}}{C_{T}^{\circ}}$$
(36)

where

 $C_T$  = fraction TMB reacted with BCO-TMB feed  $X_A^O$  = amount component A formed with pure TMB feed  $C_m^O$  = fraction TMB reacted with pure TMB feed

 $X_{\Lambda}$  = amount component A formed with BCO-TMB feed

 $C_T$  and  $C_T^O$  are obtained at the same temperature and space velocity.  $X_A^O$  and  $C_T^O$  are obtained from Table 4.

As an example, consider component 4 in Runs 102-5 (<u>Table 4</u>) and 105-2 (<u>Table 5</u>):

 $X_4^0 = 18.7$  $C_T^0 = 24.5$  $C_T = 11.1$ 

AFAPL-TR-70-71

-101-

$$X_{4} = \frac{11.1(18.7)}{24.5} = 8.5$$

Since the total amount of component 4 found by CLC was only 8.0%, all of it was formed from TMB. Hence, none of component 4 was formed from BCO.

## Micro Catalyst Test Reactor

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The Micro Catalyst Test Reactor (MICTR) and the operational techniques used for screening candidate catalysts have been described in previous reports.<sup>1,2</sup>) No further changes nave been made. <u>Figures 87 through 89</u> of reference ] show the apparatus in detail, except for changes noted in reference 2. Catalysts have been tested with MCH at LHSV 100, at 662, 752, and 842°F, and at 10 atm pressure without added hydrogen. It has been found that more consistent results are obtained if a fresh loading of the reference catalyst 9874-139, or its replacement catalyst 10860-70, is tested each week as a base point for calibration, rather than using the same reference tube over and over again, since the activity gradually declines. Also, prepared catalysts have been rescreened to 10-20 mesh to remove fines after impregnation and drying of the supports, and this gives more reproducible results. <u>Tables 57 and 58</u> give the MICTR test data in chronological order.

Tables 57 and 58 follow

AFAPL-TR-70-71

# Table 57. DEHYDROGENATION OF METHYLCYCLOHEXANE WITH VARIOUS CATALYSTS IN MICTR

# Runs 1061-1191

Conditions: 100 IHSV, 10 atm pressure, no added  $H_{z}$ , temperature variable. 0.9 ml catalysts diluted with 1:1 ml quartz chips (10-20 mesh) reduced in  $H_{z}$  at 796°F. GLC samples normally taken at 3-, and 8-, and 13 minutes operation at each temperature.

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SPR. 0.5 Mail 0' Vire 1 amport 18 Ft. 18 meil 0' Vire 1 amport 18 Ft. 18 meil 0' Vire 1 amport 18 Ft. 18 meil 0' Vire 1 amport 18 Ft. 8 meil 0' Vire 1 amport 18 Ft.Virte 1 amport (centril)	.% rv/type 1 support 1 Prv/type 1 support 5 rv/type 6 support 6 rv/type 6 support	<pre>% Pt/type 1 muggort (sphures, 9-16 main) % Pt/type 1 muggort (sphures, 10-16 main) 0000-113, muffield 92°1, 1/2 hr in air (9-16 main)</pre>	Fr/UR F-8 type Algob Fr/UR F-3 type Algob Fr/UR F-3 type Algob Fr/UR F-8 type Algob	Pu/type 1 augmont Pu/type 1 augmont Pu/type 1 augmont Pu/type 1 augmont	Pu/type 1 support Pu/type 1 support Pu/type 1 support	5% Pt, 0.5% metal G/type 1 aupport Pt, 1% metal G/type 1 aupport	Pt/type 1 support Pt/type 1 support	Pt/type 1 support Pt/type 1 support Pt/type 1 support	Pr/UTP R-8 type AlgOs 17/1791 1 emport Pr/UTP 1 emport Pr/UTP 1 emport Pr/UTP 1 emport	Pt/extruded type 6 support (low density) Pt/extruded type 6 support (low density)	Pt/type 6 support (low demaity) Pt/type 6 support (low demaity)	i Pulyane 6 augmant (nigh dennity) http://gove.thingh dennity) f.Pulyane 6 augmant (nigh dennity) i.Pulyane 6 augmant (nigh dennity)	Ft/type 1 support ft/type 1 support	Pu/tape 10 emport Pt/tape 10 emport	. Pt/102 R-8 type Al_0. (ref.) . Pt/102 R-8 type Al_0. (nev ref.)	Privys 6 support (low density) Privys 6 support (low density)	Pt/type 1 support Pt/type 1 support (suffled2'F in air) Pt/type 1 support (suffled2'F in air)	Pr/Yrys 6 support (as received) Pr/Yrys 6 support (as received) Pr/Yrys 6 support (112* surfned) Pr/Yrys 6 support (112* surfned) Pr/Yrys 6 support (as received) Pr/Yrys 6 support (as received) Pr/Yrys 6 support (112* surfned) Pr/Yrys 1 support (112* suppor	Pres/Vape 1 augoart Pres/Vape 1 augoart Pr/Vape 1 augoart Pr/Vape 1 augoart (control) Pr/Vape 1 augoart( Pr/Vape 1 augoart( Pr/Vape 6 augoart( Pr/Vape 8 5 augoart() Pr/Vape 8 5 augoart()
REE E	0.716 0.710 0.110 0.110	0.6153	0.434 0.435 0.450 0.455	0.526 0.526 0.558	0.526 0.526 0.577 0.577		0.777 0.809	669.0 52.0	564.6E	0.317	0.315	0.586 0.598 0.658 0.657	0.671 0.718	0.603 0.603	0.434 0.52	0.332 9.532	0.808 0.810 0.711	0.201 0.2010	04500 45100 45100 45100 45100 1400 1400
x x x x x x x x x x x x x x x x x x x x	8, 8	21, 20, 19 21, 20, 19 20, 21, 20	23,25 24, 25, 24 24, 22, 24	23, 23, 24 23, 29, 23 25, 28, 23 25, 28, 23	22 22 22 22 22 22 22 22 22 22 22 22 22	21, 25 21, 20, 22	25, 22, 20 25, 22, 22	28, 23, 24 26, 25, 24 24, 35, 33	888855 888855 888555	23, 25, 24 26, 30, 21	20, 19, 18 27, 22, 21	11, 12, 14 16, 15, 15 14, 17, 14 26, 21, 19	23, 23, 22 30, 25, 24	24, 22, 21 25, 24, 21	26. 21, 22 19, 22, 25	19, 20, 18 23, 21, 20	37, 38, 33 26, 23, 33 63, 23	ຄ. ສ.	ភ្លេសនិង មួយ ស្លេសន៍ មួយ ស្លេសន៍ ស្លេស ស្លេសន៍ ស្លេស ស្លេសន៍ ស្លេស ស្លេសន៍ ស្លេស
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፟ፙዿ፞፞፞ <del>ኇ</del> ፞ዿኇዿቔ ፟ፙዸፘዸኇቜቔ ቔ፟፟፟፟፟ዸዸኯ፝ቔቔቔ	8835 8835 8838	555 555 555 555 555 555 555 555 555 55	<b>LXX</b> <b>LXX</b> LXX LXX LXX LXX LXX LXX LXX LXX LXX	85, 94. 88 86, 81, 81 (TT), (TT), (T6) 79, TT, T6	83, 81, 88 81, 81, 88 83, 81, 88 83, 81, 81	55, 63, 66 65, 65, 64	76, 76, 76 83, 32, 83	88. 88. 88. 88. 88. 88. 88. 11. 11. 11.	ඇතු හු හු හු සුතු හු හු හු සුතු හු හු හු සුතු හු හු හු	8, 9, 9 7, 4, 5	56, 55, 55 71, 72, 70	41, 39, 37 48, 47, 46 47, 41, 40 50, 45, 45	80, 80, 73 89, 88, 89	60, 57, 56 70, 71, 75	11, 72, 73 53, 68, 67	64, 61, 58 65, 64, 63	\$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$	ୡ୳ୡୄୄୄୄଽ୳ୢଽୡଽୢଽଽ ଌୄଽୡୄୄୄୄୄୄୄୄୄୄୄୄଽୡୡୢୢଽୢୢ ଌୡୢଌଌ୳ଌୡଽଽୡୡଌୄ	සුසුසුසුදුදු අදුසුදුසුදු පුදුසුදුසුදු පුදුසුදුසුදු

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Table 57.

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### Table 58. DEHYDROGENATION OF METHYLCYCLOHEXANE WITH VARIOUS CATALYSTS IN MICTR Runs 1192-1276

Conditions: 100 LHSV, 10 atm pressure, no added H<sub>2</sub>, temperature variable. 0.9 ml catalysts diluted with 1.1 ml quartz chips (10-20 mesh) reduced in H2 at 796°F. GLC samples normally taken at 3-, and 8-, and 13 minutes operation at each temperature.

Run		Catalyst		% Convers	ion of MCH to	Toluene, 🚧
No.	No. 10860-	Description	wt., g.	662	752	842°F
1192	70	1% Pt/UOP R-8 type Al <sub>2</sub> O <sub>3</sub> (ref)	0.411	23, 27, 25	51, 48, 48	73, 72, 72
11.93	1 <b>63A</b>	3.2, 7t/80% type 1 support <sup>a</sup> ) 20% type 6 binder	0.346	, 26, 25	50, 50, 50	82, 81, 80
1194	163B	3.2% Pt/40% type 1 support (1) <sup>a</sup> ) 40% type 1 support (2) <sup>a</sup> ) 20% type 6 binder	0.371	26, 23, 23	54, 50, 49	81, 77, 77
1195	1630	1.6% Pt/40% type 1 support (1) <sup>a)</sup> 40% type 1 support (2) 20% type 6 binder	0.403	23, 23, 23	55, 53, 49	77, 76, 75
1196	160A	1% Pt/80% type 16 support 20% type 6 binder	0.346	23, 21, 19	50, 47, 47	74, 74, 73
1197	160B	2% Pt/ " "	0.399	25, 24, 23	50, 47, 47	74, 72, 72
1198	1600	4% Pt/ " " "	0.371	25, 25, 25	57, 54, 55	83, 82, 82
1199	161A	3.2% Pt/80% type 1 support <sup>a)</sup> 20% type 6 binder	0.594	23, 20, 21	52, 45, 45	75, 73, 73
1200	161B	1.6% Pt/40% type 1 support (1) 40% type 1 support (2) <sup>n</sup> ) 20% type 6 binder	0.442	21, 23, 22	50, 46, 46	77, 75, 74
1201 1202 1203 1204 1205	70 157A 157E 157B 157F	1% Pt/UOP R-8 type AlgO3 (ref) 1% Pt/type 1 support <sup>1</sup> ) 4% Pt/ " " i 1% Pt/type 1 support <sup>1</sup> 4% Pt/ " " i)	0.410 0.562 0.596 0.545 0.562	26, 21, 22 24, 22, 22 26, 25, 25 25, 21, 23 28, 26, 27	48, 46, 46 55, 50, 50 60, 58, 58 53, 52, 52 60, 59, 56	72, 71, 70 79, 78, 78 85, 82, 83 81, 81, 79 87, 87, 87
1206 1207 1208	158A 158B 158C	1% Pt/type 1 support 2% Pt/ " " 4% Pt/ " "	0.535 0.542 0.567	27, 25, 24 29, 2 <sup>1</sup> , 24 32, 24, 24	56, 53, 53 54, 53, 51 57, 53, 53	84, 83, 83 81, 80, 82 83, 83, 83
120¢)	160в	2% Pi/80% type 16 support 20% type 6 binder	0.338	23, 23, 19	45, 43, 43	67, 65, 66
1210 1211 1212 1213	157C 157G 157D 157H	1% Pt/type 1 support <sup>1</sup> ) 4% Pt/ " " <u>i</u> ] 1% Pt/type 1 support <sup>1</sup> ) 4% Pt/ " " i)	0.843 0.828 0.527 0.548	19, 18, 17 17, 18, 18 25, 22, 22 23, 23, 22	36, 35, 37 36, 35, 35 48, 46, 46 53, 50, 49	46, 48, 50 46, 46, 47 74, 74, 74 78, 77, 77
1214	70	1\$ Pt/UOP E-8 type Al203 (ref)	0.412	24, 20, 23	50, 45, 46	70, 69, 70
1215	1644.	1% Pt/80% type 1 support 20% type 6 binder	0.430	22, 21, 22	50, 49, 48	76, 75, 76
1216 1217	164B 164C	2% Pt/ " " 3% Pt/ " "	0.431 0.458	30, 28, 26 30, 27, 27	53, 54, 56 59, 56, 57	84, 83, 84 85, 85, 85
1218 1219 1220 1221	165 <sup>c)</sup> 165A <sup>c)</sup> 166A 166B	45 Pt/type 1 support 15 Pt/ " " 45 Pt/type 6 support 45 Pt <sup>2</sup> /type 6 support	0.549 0.534 0.321 0.325	25, 29, 29 24, 27, 25 28, 22, 22 20, 22, 20	48, 51, 51 59, 59, 54 47, 43, 42 45, 43, 43	80, 79, 80 83, 81, 81 65, 72, 65 67, 67, 65

a) Platinized with 4% metal before formulation.

ь)

Repeat of run 1197. Repeat preparations in quantity of 10860-1580 and 158A, for bench scale tests. Acetate neutralised  $Pt(NH_5)_4(OH)_B$ . с

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Inpregnate type 51. Repeat of run 1219.

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Repet of run 1218. Repet of run 1198.

n) 1)

In-nouse support.

(Continued)
Run		Catalyst		% Conversio	on of MCH to	Toluene, 🚧
No.	No. 10860-	Description	wt., g	662	752	842°F
1555	167A	4% Pt/80% type 16 support 20% type 6 binder	0.461	24, 25, 23	50, 41, 41	61, 59, 60
1223 1224 1225	165 <b>A<sup>(*)</sup></b> 167B 167C	1% Pt/type 1 Support 4% Pt/type 16 support 3.2% Pt/80% type 16 support 20% type 6 support	0.528 0.384 0.412	23, 28, 22 24, 25, 23 25, 20, 21	57, 51, 53 50, 41, 41 47, 43, 43	82, 81, 81 61, 59, 60 71, 69, 70
1226	165g)	4% Pt/Type 1 support	0.555	25, 25, 22	56, 55, 54	85, 84, 82
1554	70	1% Pt/UOP R-8 type Al <sub>2</sub> O <sub>3</sub> (ref)	0.413	21, 19, 23	48, 45, 45	71, 69, 69
1228	160C <sup>h)</sup>	4% Pt/80% type 16 support 20% type 6 binder	0.363	25, 22, 23	51, 48, 48	74, 72, 71
1229	168	1.6% Pt/40% type 1 support 40% type 1 support <sup>a</sup> ) 20% type 1 binder	0.484	23, 22, 22	52, 49, 47	77, 74, 71
1230	172	1.6% Pt/40% type 16 support 40% type 1 support <sup>5</sup> ) 20% type 6 binder	0.444	24, 20, 20	49, 47, 46	72, 71, 71
1231	170B	4% Pt/type 1 support <sup>1)</sup>	0.597	25, 24, 22	54, 51, 51	82, 81, 79
1232	70	1% Pt/UOP R-8 type Al <sub>2</sub> C <sub>3</sub> (ref)	c.416	20, 22, 22	48, 45, 46	71, 69, 68
1233 1234	171A 171B	4% Pt/type 1 support	0.603 0.606	27, 23, 24 25, 25, 25	51, 49, 51 58, 56, 57	77, 77, 76 86, 85, 83
1235	173	1.6% Pt/41% type 16 support 41% type 1 support <sup>a</sup> ) 18% type 6 binder	0.498	27, 25, 24	55, 52, 50	79, 77, 77
1236 1237 1238 1239 1240	170A 171C 171D 171E 171F	4% Pt/type 1 support 4% Pt/type 1 support 1% Pt/type 1 support 4% Pt/type 1 support 2% Pt/type 1 support	0.559 0.541 0.711 0.747 0.762	23, 24, 23 30, 27, 27 21, 21, 21 21, 22, 22 22, 20, 20	59, 57, 57 55, 55, 54 50, 47, 49 52, 51, 52 52, 48, 48	88, 87, 86 82, 82, 82 72, 69, 72 82, 82, 82 77, 76, 76
1241	175	3.2% Pt/40% type 16 support <sup>a</sup> ) 40% type 1 support <sup>a</sup> )	0.492	27, 22, 22	49, 45, 44	74, 74, 72
1242	"	20% type 6 binder	0.505	31, 22, 21	55, 50, 50	80, 77, 77
1243	70	1%Pt/UDP R-8 type Al2D3(ref)	0.420	25, 20, 20	48, 45, 46	71, 71, 70
1244	1774	3% Pt/40% type 1 support (1) 40% type 1 support (2)	0.405	23, 23, 24	53, 53, 55	82, 82, 82
1245	177B	20% type 6 binder 4% Pt/ " " "	0.401	24, 24, 26	54, 53, 53	84, 82, 82
1246	1770	3% Pt/40% type 16 support 20% type 1 support 20% type 6 binder	0.478	29, 25, 27	54, 51, 50	79, 77, 77
1247	177D	4% Pt/ " " "	0.476	26, 24, 26	54, 51, 51	84, 80, 50
1248	1844	4% Pt/40% type 1 support (1) 40% type 1 support (2) 20% type 6 binder	0.404	30, 26, 25	58, 54, 55	85, 86, 83
1250	184C	4\$ Pt/80\$ type 1 support 20\$ type 6 binder	0.478	30, 25, 25	<b>63, 6</b> 0, 58	88, 89, 91

# Table 58. (Contd-1) DEHYDROGENATION OF METHYLCYCLOHEXANE WITH VARIOUS CATALYSTS IN MICTR

(Continued)

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Run		Catalyst		% Conversion	of MCH to '	Foluero 🚧
No.	No. 10860-	Description	wt., g.	662	752	842°F
1251	184D	4% Pt/80% type 1 support 20% type 6 binder	0.664	26, 25, 29	57, 54, 54	84, 84, 63
1050	1911	40% type 1 support (1) 40% type 1 support (2) 20% type 6 binder	n. <u>48</u> 0	25, 27, 30	56, 53, 52	86, 85, 84
1253	184B	4% Pt/40% type 16 support 40% type 1 support 20% type 6 binder	0.422	22, 22, 25	51, 49, 50	79, 79, 78
1254 1255	'70 70	1% Pt/UOP R-8 type Al203 (ref)	0.419 0.422	24, 20, 20 21, 20, 20	48, 44, 46 47, 44, 44	71, 69, 68 69, 68, <b>6</b> 8
1256 1257 1258 1259	188A 188B 188C 188D	1% Pt/type 1 support (purified) <sup>j)</sup> 1% Pt, 1% metal AB/type 1 support ", 2.3% " " " " " 4% " " " "	0.803 0.738 0.713 0.752	31, 27, 26 21, 23, 24 28, 23, 22 16, 16, 16	58, 52, 51 60, 55, 55 56, 51, 51 48, 44, 45	84, 81, 80 06, 84, 84 80, 79, 79 71, 68, 68
1260 1261 1262	189A 189B 189C	1% Pt, 1% metal AC/type 1 support " " 2.3% " " " " " 45 " " "	0.759 0.731 0.747	31, 28, 30 17, 18, 20 16, 15, 15	57, 51, 51 51, 48, 49 37, 37, 32	82, 81, 81 73, 68, 67 52, 48, 45
1264	70	1% Pt/UOP R-8 type Al <sub>2</sub> O <sub>3</sub> (ref)	0.417	24, 22, 20	49, 46, 46	72, 70, 69
1263 1265 1266	190A 190B 190C	1% P%, 1% metal AA/type 1 support " " 2.3% " " " " " " 4% " " "	0.711 0.767 0.764	25, 22, 25 20, 21, 24 22, 21, 19	59, 54, 53 55, 51, 50 53, 47, 47	84, 81, 81 81, 81, 80 78, 77, 75
1267 1268 1269	191A 191B 191C	1% Pt, 1% metal AD/type 1 support " " 2.3% " " " " " 4% " " "	0.767 0.746 0.742	34, 29, 26 24, 20, 21 22, 21, 23	59, 54, 54 59, 52, 54 51, 54, 51	83, 82, 81 85, 85, 83 73, 73, 72
1270 1271 1272	192 <b>A</b> 1928 1920	1% Pt, 1% metal AE/type 1 support " " 2.3% " " " " " 4% " " "	0.736 0.737 0.769	26, 23, 25 26, 25, 24 26, 23, 22	62, 58, 56 59, 53, 52 55, 50, 50	87, 87, 86 85, 83, 83 81, 79, 79
1273	1950	Formulation for 10860-184C, stored wet 53 days, before drying, cal- cining, and 4% Pt impreg.	0.440	22, 23, 23	56, 55, 56	84, 82, 80
1274	195A	Formulation for 10860-184A, stored wet 55 days, before drying, cal- cining, and 4% Pt impreg.	0.409	24, 23, 25	54, 52, 51	84, 82, 79
1275	195E	Formulation for 10860-184E, stored wet 48 days, before drying, cal- cining and 4% Pt impreg.	0.503	24, 23, 23	58, 54, 53	84, 84, 83
1276	195D	Formulation for 10860-184D, stored wet 54 days, before drying, cal- cining, and 4% Pt impreg.	0.975	22, 22, 23	58, 54, 54	86, 84, 83

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# Table 58. (Contd-2) DEHYDROGENATION OF METHYLCYCLOHEXANE WITH VARIOUS CATALYSTS IN MICTR

j) Control for series 10860-188, 189, 190, 191, and 192.

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AFAPL-TR-70-71 66758



#### Derivations of Equations for the Catalytic Wall Reactor Model

#### Fluid Flow Equations

The fluid flow in the reactor is described by a mass balance, a momentum balance, and an energy balance. The differential equations are formed by writing the balances over a differential cylindrical element:



length,  $\triangle z$ 

Mass Balance:

Since the flow is assumed to be at steady state,

rate of mass flow in = rate of mass flow out

$$\left. \rho v A_{c} \right|_{z} = \left. \rho v A_{c} \right|_{z + \Delta z}$$

$$(37)$$

where

 $\rho = \text{fluid density}$  v = fluid velocity  $A_c = \text{cross-sectional area} = \frac{\pi d^2}{4}$  d = diameter of flowing fluid

Since  $A_c$  is a constant, then  $\rho v$  is also a constant, which is the mass flux:

$$G = \rho v$$
(38)

AFAPL-TR-70-71

-103-

Momentum Balance:

rate of momentum in - rate of momentum out + sum of forces on system = 0 rate in =  $(\rho v A_c) v |_{z}$ rate out =  $(\rho v A_c) v |_{z + \Delta z}$ pressure force =  $PA_c |_{z} - PA_c |_{z + \Delta z}$ drag force =  $-(\frac{1}{2} \bar{\rho} \bar{v}^2) A_s f$ 

where

F

 $\begin{array}{l} P = \mbox{fluid pressure} \\ \bar{\rho} = \mbox{average value of } \rho \\ \bar{v} = \mbox{average value of } v \\ A_{\rm S} = \mbox{surface area of differential element} \\ f = \mbox{Fanning friction factor (drag force equation defines f)} \end{array}$ 

$$GA_{c}(\mathbf{v}|_{z+\Delta z} - \mathbf{v}|_{z}) + A_{c}(\mathbf{P}|_{z+\Delta z} - \mathbf{P}|_{z}) + \frac{1}{2}\bar{\rho}\bar{\mathbf{v}}^{2}A_{g}f = 0$$
(39)

Dividing by the volume of the differential element and taking the limit as  $\Delta z$  approaches zero gives the desired differential equation:

$$\frac{G\left(\frac{\pi d^2}{4}\right)\left(v_{|_{z}+\Delta z}-v_{|_{z}}\right)-\frac{\pi d^2}{4}\left(P_{|_{z}+\Delta z}-P_{|_{z}}\right)+\frac{1}{2}\overline{\rho v^2}\pi d\Delta zf}{\frac{\pi d^2}{4}\Delta z}=0 \quad (40)$$

$$\lim_{\Delta z \to 0} G \frac{\frac{v|_{z} + \Delta z^{-v}|_{z}}{\Delta z} + \frac{P|_{z} + \Delta z^{-P}|_{z}}{\Delta z} + \frac{2\bar{\rho}\bar{v}^{2}f}{d} = 0 \quad (41)$$

$$G \frac{dv}{dz} + \frac{dP}{dz} + \frac{2\rho v^2 i}{d} = 0$$
 (42)

Note that the average values of  $\rho$  and v become the same as the point values as  $\Delta z$  approaches zero. The only forces considered here are pressure and drag or frictional force. Other forces such as gravitational, nuclear, radiative, and electromagnetic forces have been neglected. The gravitational force might be important in a vertical reactor at very low flow rates.

AFAPL-TR-70-71

#### Energy Balance:

rate of kinetic energy in + rate of internal energy in -rate of kinetic energy out - rate of internal energy out + not rate of heat added - net rate of work done by system = 0

rate of kinetic energy in =  $\frac{1}{2}GA_{c}v^{2}|_{z}$ rate of internal energy in =  $GA_{c}u|_{z}$ rate of kinetic energy out =  $\frac{1}{2}GA_{c}v^{2}|_{z+\Delta z}$ rate of internal energy out =  $GA_{c}u|_{z+\Delta z}$ rate of heat added =  $-q_{1}A_{s}$ 

rate of jork done by system = 
$$PA_c v \Big|_{z + \Delta z} - \frac{PA_c v}{z} \Big|_{z}$$

where U = specific internal energy $q_1' = \text{heat flux from fluid to wall}$ 

Substituting H for U + P/p, and dividing by the volume element  $\pi d^2 \Delta z/4$  gives

$$\frac{\frac{1}{2}G\left(v^{2}\right)}{\Delta z} - \frac{v^{2}}{2} + \frac{G\left(H_{z + \Delta z} - H_{z}\right)}{\Delta z} + \frac{4q_{z}}{d} = 0 \quad (45)$$

Taking the limit as  $\triangle z$  approaches zero and dividing by G gives

$$v \frac{dv}{dz} + \frac{dH}{dz} + \frac{4q_1'}{Gd} = 0$$
(44)

We can obtain H in terms of P, T, and  $\rho$ . For an ideal gas, H is a function of T only:

$$\frac{dH}{dz} = \left(\frac{\partial H}{\partial T}\right)_{P} \quad \frac{dT}{dz} = c_{P} \frac{dT}{dz} \tag{45}$$

The pressure effect can be added to give:

$$\frac{dH}{dz} = \left(\frac{\partial H}{\partial T}\right)_{P} \frac{dT}{dz} + \left(\frac{\partial H}{\partial P}\right)_{T} \frac{dP}{dz}$$
(46)

AFAPL-TR-70-71

-105-

The value of  $\left(\frac{\partial H}{\partial T_p}\right)$  is known as the heat capacity at constant pressure,  $c_p$ , and may be calculated. From he well known equation

$$dH = TdS + VdP$$
(47)

we can obtai

$$\left(\frac{\partial H}{\partial P}\right)_{T} = T \left(\frac{\partial S}{\partial P}\right)_{T} + V$$
 (48)

Substituting in one of Maxwell's Relations, gives

$$\left(\frac{\partial H}{\partial P}\right)_{T} = -T\left(\frac{\partial V}{\partial T}\right)_{P} + V$$
(49)

or written in terms of  $\rho$  instead of V,

$$\left(\frac{\partial H}{\partial P}\right)_{T} = -T \left(\frac{\partial (b)}{\partial T}\right)_{P} + \frac{1}{p}$$
(50)

$$= \frac{T}{\rho^2} \left( \frac{\partial \rho}{\partial T} \right)_{\rm P} + \frac{1}{\rho}$$
(51)

So, the final equation is

$$\mathbf{v} \frac{\mathrm{d}\mathbf{v}}{\mathrm{d}z} + c_{\mathrm{p}} \frac{\mathrm{d}\mathbf{T}}{\mathrm{d}z} + \left[\frac{1}{\rho} + \frac{\mathrm{T}}{\rho^{2}} \left(\frac{\partial\rho}{\partial\mathrm{T}}\right)_{\mathrm{p}}\right] \frac{\mathrm{d}P}{\mathrm{d}z} + \frac{4\sigma_{1}^{\prime}}{3\mathrm{d}} = 0$$
(52)

We can rewrite the momentum balance by dividing by  $\rho$  to give

$$\mathbf{v} \frac{\mathrm{d}\mathbf{v}}{\mathrm{d}z} + \frac{1}{\rho} \frac{\mathrm{d}\mathbf{P}}{\mathrm{d}z} + \frac{2}{\sigma} \frac{\mathbf{v}^2 \mathbf{f}}{\mathrm{d}z} = 0$$
 (53)

Subtracting this from Equation (52) gives

$$c_{\rm P} \frac{dT}{dz} + \frac{T}{\rho^2} \left( \frac{\partial \rho}{\partial T} \right) \frac{dP}{dz} + \frac{4q_1'}{Gd} - \frac{2v^2f}{d} = 0$$
 (54)

-106-

AFAPL-TR-70-71

Substituting  $G/\rho$  for v gives the final two equations for the fluid flow:

$$\frac{d}{\rho} \frac{\partial \rho}{\partial F} \frac{dP}{dz} + \frac{d}{\rho} \frac{\partial \rho}{\partial T} \frac{dT}{dz} - \frac{\rho d}{G^2} \frac{dP}{dz} - 2f = 0$$
(55)

$$\frac{\rho^2 c_p d}{G^2} \frac{dT}{dz} + \frac{T d}{G^2} \frac{\partial \rho}{\partial T} \frac{dP}{dz} + \frac{4\rho^2 q_1'}{G^3} - 2f = 0$$
(56)

#### Chemical Conversion

The model is presently based on a single chemical reaction. Again, the shell balance method is used to obtain a differential equation.

rate reactant in = rate reactant out + rate reacted

$$\left[ \frac{GA_{c}n}{z} \right]_{z} = \left[ \frac{GA_{c}n}{z} \right]_{z + \Delta z} + r v_{c} \Delta z$$
(57)

where

n = number of moles of reactant per unit weight feed r = rate of reaction per unit volume of catalyst v<sub>c</sub> = volume of catalyst per unit length reactor

giving

$$-\frac{\mathrm{dn}}{\mathrm{dz}} = \frac{4\mathrm{rv}_{\mathrm{c}}}{\mathrm{Gmd}^2}$$
(58)

The volume of catalyst per unit length for a cylindrical layer on the inside of a tube is

$$v_c = \frac{\pi}{4} (D^2 - d^2)$$
 (59)

where

D = inside diameter of tube d = inside diameter of catalyst

Rewriting Equation (58) in terms of x, number of moles converted per unit weight feed, gives

$$\frac{\mathrm{d}\mathbf{x}}{\mathrm{d}\mathbf{z}} = \frac{\mathbf{r} \left(\mathbf{D}^2 - \mathrm{d}^2\right)}{\mathrm{Gd}^2} \tag{60}$$

AFAPL-TR-TJ-71

-107-

#### Wall Temperature Profiles

The temperature profiles in the catalyst layer and in the tube wall may be calculated knowing the heat fluxes and assuming that the heat generation is uniform across the section. This is probably a good assumption for the tube wall, since the heat generated depends on the electrical resistance, which depends on the temperature. The changes in temperature across the tube wall are generally small enough to neglect for this purpose. The assumption of uniform heat absorption in the catalyst layer is less acceptable, since the amount of heat is related directly to the rate of reaction, which is an exponential function of temperature. The temperature drops across the catalyst layer are also larger than those across the tube wall.

The temperature in a cylindrical layer may be found by making a shell balance over an cylindrical element of inside radius y, thickness  $\Delta y$ , and length L:



The heat balance consists of

rate of heat in + rate of heat generated = rate of heat cut rate of heat in =  $2\pi y I q |_{y}$ rate of heat out =  $2\pi y I q |_{y + \Delta y}$ rate of heat generated =  $S(2\pi y I \Delta y)$ 

where

y = radius of element Δy = thickness of element L = length of element q = heat flux in + y direction S = rate of heat generated per unit volume

$$2\pi I \left[ \left( y + \Delta y \right) q \right]_{y + \Delta y} - y q = 2\pi I \bar{y} \Delta y S$$
(61)

-108-

AFAPL-TR-70-71

Divide by  $2\pi L\Delta y$  and take the limit as  $\Delta y$  approaches zero,

$$\lim_{\Delta y \to 0} \frac{\left[ (y + \Delta y)q \right]_{y + \Delta y} - yq}{\Delta y} = yS$$
(62)

$$\frac{d(yq)}{dy} = yS \tag{63}$$

Integration gives

$$yq = \frac{Sy^2}{2} + c_1$$
 (64)

or 
$$q = \frac{Sy}{2} + \frac{c_1}{y}$$
 (65)

where  $c_1$  is a constant of integration. This equation applies to both layers. The boundary conditions are slightly different. In both cases, let  $y_0$  and  $y_i$  be the outer and inner radii of the layers, catalyst and tube. Then at the outer surface, the boundary condition is a heat flux at that surface:

 $q = q_0$  at  $y = y_0$ 

Then from Equation (65)

 $q_0 = \frac{Sy_0}{2} + \frac{c_1}{y_0} \tag{66}$ 

and 
$$c_1 = q_0 y_0 - \frac{S y_0^2}{2}$$
 (67)

So

$$q = \frac{Sy}{2} + \frac{1}{y} \left( q_0 y_0 - \frac{Sy_0^2}{2} \right)$$
 (68)

Introducing the Fourier equation for heat conduction

$$q = -k \frac{dT}{dy}$$
(69)

gives 
$$\frac{d\mathbf{T}}{d\mathbf{y}} = \frac{S\mathbf{y}}{2\mathbf{k}} - \frac{1}{\mathbf{ky}} \left( q_0 \mathbf{y}_0 - \frac{S\mathbf{y}_0^2}{2} \right)$$
 (70)

AFAPL-TR-70-71

-109-

Integration gives

$$T = \frac{Sy^2}{4k} - \frac{1}{k} \ln y \left( q_0 y_0 - \frac{Sy_0^2}{2} \right) + c_2$$
(71)

where

T = temperature in layer k = thermal conductivity c<sub>2</sub> = constant of integration

The boundary condition for each layer is that at the inner surface,  $y_i$ ; the temperature is  $T_i$ .

So

$$T_{i} = -\frac{Sy_{i}^{2}}{4k} - \frac{1}{k} \ln y_{i} \left(q_{0}y_{0} - \frac{Sy_{0}^{2}}{2}\right) + c_{2}$$
(72)

$$c_2 = T_1 + \frac{Sy_1^2}{4k} + \frac{1}{k} \ln y_1 \left(q_0 y_0 - \frac{Sy_0^2}{2}\right)$$
 (73)

giving 
$$T = -\frac{Sy^2}{4k} - \frac{(q_0y_0 - \frac{Sy_0^2}{2})}{k} \ln y + T_i + \frac{Sy_i^2}{4k} + \frac{1}{k} \ln y_i (q_0y_0 - \frac{Sy_0^2}{2})$$
 (74)

for each layer.

Equation (74) may now be applied to each layer. The subscripts refer to the surfaces shown:



AFAPL-TR-70-71

The heat generated in the tube wall is written as  $q_G^{\prime}$ , the heat flux of generated heat, based on inner tube surface area:

$$S = q_{G}' \frac{Btu}{hr ft^{2}(inner area)} \times \frac{ft^{2}(inner area)}{ft^{3}(wall)}$$
$$= q_{G}' \frac{2\pi y_{2}L}{\pi(y_{3}^{2} - y_{2}^{2}) L}$$
(75)

$$S = \frac{2y_2}{y_3^2 - y_2^2} q'_G$$
(76)

The heat flux at the outer surface (3) is  $q_3$  and is corrected to be based on the inner surface:'

$$q_3 = \frac{y_2}{y_3} q_3'$$
 (77)

Then Equation (74) after some rearrangement becomes

$$T - T_{2} = \frac{1}{k} \left[ q_{G}^{\prime} \left( \frac{\frac{y_{2}}{2y_{3}} (y_{2}^{2} - y^{2}) + y_{2} \ln \left(\frac{y}{y_{2}}\right)}{1 - \left(\frac{y_{2}}{y_{3}}\right)^{2}} \right) - q_{3}^{\prime} y_{2} \ln \left(\frac{y}{y_{2}}\right) \right] (78)$$

To get the temperature drop,  $y_3$  and  $T_3$  are substituted for y and T, and rearranged to give

$$T_{3} - T_{2} = - \frac{q_{G}' y_{2}}{2k} \left[ \frac{1 - (\frac{y_{2}}{y_{3}})^{2} + 2 \ln (\frac{y_{2}}{y_{3}})}{1 - (\frac{y_{2}}{y_{3}})^{2}} \right] + \frac{q_{3}}{k} y_{2} \ln (\frac{y_{2}}{y_{3}})$$
(79)

for the tube wall. By a similar procedure, the temperature in the catalyst layer is shown to be

$$\mathbf{T} - \mathbf{T}_{1} = + \frac{q_{\mathbf{R}}^{\dagger} \mathbf{y}_{2}}{2k} \left[ \frac{1 - (\frac{y}{y_{1}})^{2} + 2 \ln(\frac{y}{y_{1}})}{1 - (\frac{y_{2}}{y_{1}})^{2}} \right] - \frac{q_{1}y_{2}}{k} \ln(\frac{y}{y_{1}})$$
(80)

and the temperature difference across the catalyst layer is

$$\mathbf{T}_{2} - \mathbf{T}_{1} = \frac{q_{R}^{\dagger} \mathbf{y}_{2}}{2k} \left[ \frac{1 - (\frac{\mathbf{y}_{2}}{\mathbf{y}_{1}})^{2} + 2 \ln (\frac{\mathbf{y}_{2}}{\mathbf{y}_{1}})^{2}}{1 - (\frac{\mathbf{y}_{2}}{\mathbf{y}_{1}})^{2}} \right] - \frac{q_{1}^{\dagger} \mathbf{y}_{2}}{k} \ln (\frac{\mathbf{y}_{2}}{\mathbf{y}_{1}})$$
(81)

AFAPL-TR-70-71

-111-

#### Description of the Beta-Ray Backscatter Apparatus for Rating Deposits

The instrument consists of the following functional blocks: The vacuum system, the scanning mechanism, the beta-ray source, and the detection system.

The vacuum system consists of a chamber made from a three-foot length of 6-inch sch 40 steel pipe with end plates sealed by 0-rings. One end provides access for loading the coker tubes and the other holds the driving mechanism. A Welch 1402B fore pump is connected by rubber tubing to a pipe nipple welded to the vacuum chamber.

The rest of the vacuum system consists of a thermocouple gauge and a bleed valve.

<u>The scanning mechanism</u> serves to translate and rotate the coker tube past the source and detector. It consists of a track fastened to the inside of the vacuum chamber on which a carriage rides. The carriage holds the coker rod and is driven by a shaft and lead screw arrangement attached to the end plate of the vacuum system. The drive shaft in the test chamber connects through a vacuum seal to a variable speed reversible motor.

<u>The beta-ray source</u> is contained in a block which is mounted to the face of the detector. The source material was obtained from the target of a Texas Nuclear neutron generator. The block consists of layers of brass with two milled slots to direct the beta-ray beam to the coker tube, which is located at the intersection of the beams. Between the two slots for the beta-rays is a third slot which is aligned with the opening in the detector. This allows the backscattered radiation to enter the detector and be counted.

<u>The detection system</u> counts the radiation that is backscattered from the coker tube and presents the count rate on a strip chart recorder. The detector is a flow proportional counter with an ultra thin window operating on P-10 gas (90% Argon, 10% methane). The window is made of a thin film of cellulose nitrate. It is supported by a Buckbee Meers Co. nickel screen of 70% transmission. The detector is attached to a flange that provides a vacuum seal and can be moved while under vacuum to position the detector with the spout to the coker tube to focus the radiation.

The rest of the detection system is outside the vacuum chamber and consists of a high voltage power supply for the proportional counter, a low noise preamplifier, a linear amplifier, a count rate meter, and a strip chart recorder with an offset zero provision.

AFAPL-TR-70-71

-112-

Press, (atm)	Тетр, (°К)	Delay, (µ sec)	Press, (atm)	Тетр, (°К)	Delay, (µ sec)	Press, (atm)	Temp, (°K)	Delay, (µ sec)
			90% n A	rgon, E	$\mathbf{R} = 0.2$			
0.60 0.61 0.67 0.61 0.63 0.64 0.60	1440 1390 1340 1230 1270 1290 1220	110 200 430 2680 1230 210 3790	1.08 1.20 1.06 1.28 1.44 1.07	1170 1340 1190 1380 1500 1190	3380 180 3240 150 60 2980			
			9994m A	rgon, E	$\mathbf{R} = 0.5$			
0.55 0.53 0.54 0.53 0.51 0.49	1480 1520 1490 1530 1640 1600	3040 1570 2220 1040 300 490	0.91 0.94 0.91 0.92 0.91 0.90 0.89 0.88 0.90 0.92 0.91	1380 1470 1480 1520 1510 1550 1550 1550 1550 1560 1600 160	3450 1740 1830 740 1160 660 560 770 530 540 270	1.56 1.61 1.50 1.47	1430 1430 1450 1460	2010 2360 1400 1330

# Table 59, IGNITION DELAY TIMES FOR DECALIN-OXYGEN-ARGON MIXTURES

AFAPL-TR-70-71

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-113-

#### Table 60. IGNITION DELAY TIMES FOR TETRALIN-OXYGEN-ARGON MIXTURES

Press, (atm)	Temp, (°K)	Delay, (µ sec)	Press, (atm)	Temp, (°K)	Delay, (µ sec)		
	99%m Argon, ER = 0.1						
0.61 0.59 0.61 0.60 0.57	1510 1540 1650 1700 1760	650 540 210 350 170	$ \begin{array}{c} 1.00\\ 1.02\\ 1.05\\ 0.97\\ 0.98\\ 0.95\\ \end{array} $	1440 1420 1420 1480 1530 1470	1310 1480 2570 330 120 1070		
		Wall HI gol	1, 21 - 0.	· 4			
0.62 0.78 0.61 0.62 0.62 0.62 0.61	1210 1470 1210 1210 1210 1210 1210 1200	3180 40 880 3110 3130 2140 3390	1.06 1.08 1.04 1.02 1.06 1.08	1140 1180 1160 1160 1180 1190	3570 770 1970 2730 460 370		
	9	Ofm Argon,	ER = 0.1	.1			
0.62 0.62 0.59 0.63 0.61 0.61 0.68 0.60	1320 1300 1320 1370 1320 1390 1430 1300	3470 3520 1060 630 1530 250 810 2540	1.04 1.03 1.07 1.07 1.11 1.14 1.11	1280 1300 1370 1280 1280 1270 1210	1050 650 290 1040 1810 2960 3890		
		99% Argan	$\mathbf{FR}=0.$	5			
0.66 0.63 0.65 0.65 0.65 0.64 0.63 0.61 0.59 0.60 0.59 0.60 0.59 0.60 0.61 0.60	1520 1540 1540 1520 1570 1560 1570 1560 1570 1630 1720 1720 1720 1720 1740 1710	3350 1120 3070 1620 950 900 1150 820 160 240 220 120 210 180	1.04 1.06 1.05 1.05 1.02 1.04 1.05 1.04 1.04 1.04 1.10 1.05 1.13 1.05	1490 1530 1530 1520 1560 1680 1690 1690 1690 1750 1690 1700 1600	2610 930 850 790 410 260 260 260 140 80 150 240 110 310		

(Contd)

AFAPL-TR-70-71

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-114-

Press, (atm)	Temp, (°K)	Dels, (µ sec)	Press,	Тетр, (°К)	Delay, (µ sec)
0.64	1560 1550	890 1380			
0.62	1520 1530	1170 1520			
		90% Argon	, ER = 0.	57	
0.66 0.62 0.61 0.60 0.59	1390 1270 1280 1270 1270	60 3310 3470 2660 2070	1.14 1.14 1.13 1.09 1.08 1.06	1250 1260 1260 1230 1250 1240	3580 2700 3650 2590 2620 2180
		95% a Argon	, ER = 1.	0	
0.58 0.62 0.61 0.67 0.62 0.63 0.63	1420 1470 1410 1650 1470 1470 1480	2880 3040 3340 110 2630 2900 2480	1.08 1.03 1.03 1.09 1.09 1.04	1430 1360 1410 1450 1440 1390	2280 3130 1640 190 2360 3130
		99% m Argon	, ER = 1.	0	
0.59 0.59 0.61 0.62 0.61 0.59 0.62	1530 1560 1640 1730 1750 1630 1650	1650 760 450 250 230 370 560	1.01 0.97 1.01 1.03 0.99 1.02 0.95 1.03	1580 1600 1740 1630 1520 1540 1510 1530	620 530 90 370 690 1520 1110 2890
		90% Argon	, $ER = 1$ .	21	
0.64	1280	<b>297</b> 0	1.14 1.14 1.12 1.12 1.10	1230 1230 1230 1250 1250	3050 3290 2730 3450 3000

## Table 60. (Contd). IGNITION DELAY TIMES FOR TETRALIN-OXYGEN-ARGON MIXTURES

AFAPL-TR-70-71

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-115-

Press, (atm)	Temp, (°K)	Delay, (µ sec)	Press, (etm)	Temp, (°K)	Delay, (µ sec)
		90% Argo	h, ER = 0.	.1	
0.63 0.60 0.63 0.62 0.60 0.61	1430 147C 1370 1360 1330 1300 1310	400 100 730 890 3020 3550 3790	1.04 1.05 1.04 1.08 1.06 1.09	1400 1430 1370 1350 1270 1280	400 240 530 690 2650 2910

#### Table 61. IGNITION DELAY TIMES FOR NAPHTHALENE-OXYGEN-ARGON MIXTURES

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# Table 62. PHYSICAL PROPERTIES OF METHYLCYCLOHEXANE/ETHANE MIXTURES

	MCH	Ethane
Weight %	90	10
Mole %	73•4	26.6

Average Molecular Weight	80.1
Melting Point	-215°F
Heat of Formation (Liquid at 25°C)	-885 Btu/1b
Net Heat of Combustion (25°C)	18,820 Btu/1b

Pseudocritical Properties Pressure

Temperature Compressibility Factor Acentric Factor

538 Dsia	
11.20 5	
0.260	
0.207	

Temp.,	Vapor Pre <b>ss</b> ure.	por Liquid Liquid sure, Specific Viscosity.	Liquid Viscosity.	Enthal	nthalpy, Btu/lb	
<u> </u>	psia	Gravity	cp	<u>O psia</u>	<u>1000 psia</u>	
-200	0.24	0.856	12.9	123	-62	
-100	8.4	0.803	2.02	139	-36	
0	59•	0.746	0.723	164	0	
100				197	45	
200				237	97	
300				283	156	
400				334	222	
500				391	296	
600				453	384	
700				520	467	
800				591	555	
900				667	640	
1000				746	725	

AFAPL-TR-70-71

-117-

# Table 62 (Contd). PHYSICAL PROPERTIES OF METHYLOYOLCHEXANE/ETHANE MIXTURES

	MCH	Ethane
Weight %	80	20
Mol %	55.1	44.9

Average Molecular Weight	67.6
Melting Point	-550°F
Heat of Formation (Liquid at 25°C)	-936 Btu/2b
Net Reat of Combustion (25°C)	18,980 Itu/Ii

#### Pseudocritical Properties Pressure

Temperature	
Compressibility	Factor
Acentric Factor	

567	pais
3	
0.00	Ĉ
0.20	X)
- 22	2

Temp.,	Vepor Pressure.	Liquid Liquid Specific Viscosity.	Enthelpy. Btu/lo		
	psia	Gravity	CD	<u>C psia</u>	<u>1000 psia</u>
-200	0.40	0.816	5.04	1:2	-60
-100	14.2	0.759	1.07	139	~39
0	99.	0.696	0.470	165	C
100				200	48
200				540	102
300				565	164
400				339	521
500				397	305
600				459	393
700				527	****7
900				598	565
900				615	649
100				754	735

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# Table 63. PHYSICAL PROPERTIES OF METHYLCYCLOHEXANE/PROPANE MIXTURES

	MCH	Propane
Weight %	90	10
Mole %	80.2	19.8

Average Molecula, Weight Melting Point Heat of Formation (Liquid at 25°C) Net Heat of Combustion (25°C)

<u>Pseudocritical Properties</u> Pressure Temperature Compressibility Factor Acentric Factor

18,770 Btu/1b 520 psia 498°F 0.256 0.226

87.5 -210°F

-867 Btu/1b

Vapor Tama Pavagouro		Liquid Liquid Specific Visconity	Enthalpy, Btu/1b		
entro.	psia_	Gravity		<u>O psia</u>	<u>1000 psia</u>
-200		0.871	22.0	124	-60
-100	0.57	0.821	2.91	139	-35
0	7.5	0.769	0.898	164	0
100	38.1	0.713	0.430	197	44
200	124.	0.620	0.257	237	96
300				282	155
40C				334	250
500				390	294
600				452	382
700				519	465
800				5¢	554
<b>90</b> 0				665	638
1000				744	723
1100				827	808
1500				913	805

AFAPL-TR-70-71

-119-

# Table 63 (Contd-1). PHYSICAL PROPERTIES OF METHYLCYCLOHEXANE/PROPANE MIXTURES

	MiH	Propene
Weight %	80	20
Mole %	64.2	35.8

536 psia 440°F 0.260 0.211

Average Molecular Weight	78.8
Melting Foint	-222°r
Heat of Formation (Liquid at 25°C)	-902 Btu/1b
Net Heat of Combustion (25°C)	18,880 Btn/15

Pseudocritical Prop	erties
Pressure	
Temperature	
Compressibility	Factor
Acentric Factor	

Tom.	Vapor	Liquid	Liquid	Enthalpy	. Btu/15
°F	psia	Gravity	<u></u>	<u>O psia</u>	<u>1000 psia</u>
~200		0.843	11.3	123	-64
-100	1.04	0.792	1.85	140	-37
Ċ,	13.5	0.737	0.646	166	0
100	67.6	0.675	0.340	199	46
200	23,5.	0.552	0.217	240	100
300				286	260
<b>4CN</b> O				338	227
500				395	302
600				457	389
<b>70</b> 0				524	473
800				596	561
900				671	646
1000				751	7DI.
1100				85,4	317
1500				981	905

 $\mathrm{ord}\left( \bigcap_{\substack{i=1,\dots,n\\m\neq i=1,\dots,m}}^{n-1} \sum_{i=1}^{n-1} \mathrm{ord}_{i} \right)$ 

# Table 63. (Contd-2). PHASICAL PROPERTIES OF METHYLCYCLOHEXANE/PROPANE MIXTURES

	MCH	Propane
Weight %	70	30
Mole \$	51.2	48.8

Average Molecular Weight	71.8
Melting Foint	-232°F
Heat of Formation (Liquid at 25°C)	-936 Btu/1b
Net Heat of Combustion (25°C)	18,990 Btu/1b

#### <u>Pseudocritical Properties</u> Pressure Temperature Compressibility Factor Acentric Factor

550 psia 392°F 0.264
0.199

Temp.,	Vapor Fressure.	Liquid Specific	Liquid Viscosity.	Enthalp	y, Btu/1b
F	psia	Gravity	cp	<u>O psia</u>	<u>1000 psia</u>
-500		0.818	6.55	123	-69
-100	1.42	0.755	1.28	141	-39
0	18.4	0.707	0.494	167	0
100	91.6	0.641	0.281	202	49
500	289.	0.498	0.189	242	1.04
300				289	166
400				342	235
500				399	310
600				462	397
700				530	481
800				602	569
900				678	654
1000				758	739
1100				842	825
1200				989	914

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-121-

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## Table 54. PHYSICAL PROPERTIES OF METHYLCYCLOHEXANE/n-BUTANE MIXTURE

	<u>MCH</u>	<u>n-Butane</u>
Weight \$	90	10
Mole \$	84.2	15.8

Average Molecular Weight Melting Point Heat of Formation (Liquid at 25°C) Not Mast of Combustion (05°C)	91.9 -207°F -859 Btu/1b
Net Heat of Combustion (25°C)	18,740 Btu/1b

Pseudocritical Properties	
Pressure	512 psia
Temperature	528°F
Compressibility Factor	0.255
Acentric Factor	0.237

Tem.	Vapor Pressure	Liquid Specific	Liquid Viscosity	Enthalpy, Btu/1b		
°F	pgia	Gravity	<u>cp</u>	<u>O psia</u>	<u>1000 psia</u>	
-500		0.879	29.2	123	-59	
-100		0.830	3.52	139	-35	
0	1.21	0.781	1.034	164	0	
100	9.5	0.730	0.473	196	44	
200	40.6	0.675	0.277	236	95	
300	123.	0 <b>.583</b>	0.192	281	153	
400				333	219	
500				389	293	
600				451	380	
700				518	463	
800				589	552	
900				664	636	
1000				743	721	
1100				826	807	

-155-

AFAPL-TR-70-71

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#### Table 64 (Contd-1). PHYSICAL PROPERTIES OF METHYLCYCLOHEXANE/n-BUTANE MIXTURE

	MCH	n-Butane
Weight % Mole %	80 70 - 3	20
ular Weight	101)	86.3

Average Molecula g Melting Point Heat of Formation (Liquid at 25°C) Net Heat of Combustion (25°C)

∞.3 -218°F -886 Btu/1b 18,820 Btu/1b

#### Pseudocritical Properties Pressure Temperature

States and s

Compressibility Factor Acentric Factor

517 psia 492°F	
0.258 0.231	

Temp.,	Vapor Pre <b>ss</b> ure,	Liquid Specific	Liquid Viscosity.	Enthalpy, Btu/1b		
F	psia	Gravity	Cp	<u>O psia</u>	<u>1000 psia</u>	
-200		0 <b>.860</b>	18.1	122	-63	
-100		0.810	2.54	139	-37	
O	2.22	0.758	0.817	164	0	
100	16.5	0.706	0.399	198	45	
200	65.9	0.646	0.245	237	98	
300	188.	0.528	0.175	284	158	
400				336	224	
500				393	299	
600				455	386	
700				522	470	
800				594	558	
900				670	643	
1000				749	<b>7</b> 28	
.100				831	813	

AFAPL-TR-70-71

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-123-

# Table 64 (Contd-2). PHYSICAL PROPERTIES OF METHYLCYCLOHEXANE/n-BUTANE MIXTURE

	MCH	<u>n-Butane</u>
Weight %	70	30
Mole %	58.0	42.0

Average Molecular Weight	81.4
Melting Point	-227°F
Heat of Formation (Liquid at 25°C)	-912 Btu/1b
Net Heat of Combustion (25°C)	18,910 Btu/1b
	20,920 000,20

#### Fseudocritical Properties Pressure

Temperature Compressibility Factor Acentric Factor 523 psia 459°F 0.261 0.226

Temp.	Vapor Pressure.	Liquid Specific	Liquid Viscosity.	Enthalp	y, Btu/1b
°F	psia	Gravity		<u>O psia</u>	<u>1000 psia</u>
-200		0.841	11.8	121	-67
-100		0.790	1.90	138	-39
0	3.11	0.737	0.664	165	0
100	22.8	0.683	0.343	199	47
200	88.3	0.620	0.221	239	100
300	246.	0.482	0.161	286	162
400				339	230
500				397	306
600				459	392
700				526	476
800				598	565
900				675	649
1000				754	734
1100				837	820

AFAPL-TR-70-71

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-124-

Table 65. LIQUID PROPERTIES OF JP-7 JET FUEL AT SATURATION

PICT FILE COND, BTU/FT-HR-R	0.07756	0.06899	0.06313	0.05496	0.04670	0.04210	0.03695	
NISCOSITY VISCOSITY	2.50	1.23	0°. 734	10.494	0.354	0.259	0.175	
DENSITY, LB/CU FT	47.4	44.8	1.3	39.5	36.0	31.9	55.5	
SPEC HEAT AT CONST P, BTU/LB-R	0.5143	0.5810	0.6646	0.7193	0.7916	0.9243	1. <sup>409</sup>	
ENTROPY, BTU/LB-R	-0.1287	0.0	0.1139	0.2163	0.3091	0.3963	0.4875	
ENTHALPY, BTU/LB	-54.7	0.0	61.83	129.3	200.9	277.9	369.8	
ALPY VAR J/LB	148.7	138.8	127.7	114.6	98.8	78.1	1.4.1 1	
ENE								_
VAPOR ENTI PRESS, OF PSIA BT	°. 8. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1.	0.147	1.59	3.76	5	54.4	136.	

Gas Properties of JP-7 follows

AFAPL-TR-20-71

-125-

GAS PROPERTIES OF JP-7

# COMPRESSIBILITY FACTOR

800     900     1060       800     900     1060       1	6,5619+00 9.6441+00 1.0%86+01
860 900 800 900 1 5.00957-01 4.7537-01 5.0165-01 5.1395-01 7.02217-01 5.1395-01 7.02217-01 5.1395-01 7.02217-01 5.1395-01 8.7580-01 8.7472-01 8.7580-01 8.7472-01 1.05285+01 1.6564+01 1.2504+01 1.5564+01 1.2504+01 1.5564+01 1.5504+0000000000000000000000000000000000	6,5613+00 9.6441+00
800 800 800 800 800 800 800 800 800 800	6°5619400
700 700 700 700 700 700 700 700 700 700	7.4522+00
600 600 600 600 600 600 600 600	6.3254+00 1600 2.6522401 2.5522401 2.5522401 2.5522401 2.011401 1.7801401 1.5705401
SURE, PSIA 500 3.4099001 4.8457001 5.62247001 7.5225001 9.0193001 5.019300 1.2103001 7.210300 1.210300 1.2517001 8.753900 5.0551001 8.7939400 6.9539400 5.95234000 5.95234000 5.95234000 5.95234000 5.95234000 5.95234000	5.1962+00 Y+LB/CUFT 54E. PSIA 1500 2.7981+01 2.5028+01 1.9535+01 1.7085+01 1.7085+01 1.4992+01
400 PRE 58274 01 PRE 58274 01 058274 01 058274 01 01 058274 01 01 058274 01 01 01 01 01 01 01 01 01 01 01 01 01	4.0820+00 DENSIT DENSIT 1400 2.4439401 2.4439401 2.4439401 2.4439401 1.62852401 1.62852401 1.6285401 1.4238401
กมารถอง จอการอง จองการร	
<ul> <li>E E M O B A A B B A A B B A A B B A A B B A A B B A A B B B B A A B B B B A B B B A B B B B A B B B A B B B A B B B B A B B B A B B B B A B B B B A B B B B A B B B B A B B B B A B B B B B A B B B B A B B B B B A B</li></ul>	2.9986+00 1300 2.3727+01 2.3842+01 1.7952+01 1.5412+01 1.3439+01
200 200 306 306 306 306 306 306 306 3	1.9559+00 2.9986+00 1200 2.6705+01 2.3927+01 2.973+01 2.3927+01 2.9595+01 1.7952+01 1.8996+01 1.7952+01 1.8433+01 1.5412+31 1.2587+01 1.3439+61
100 200 300 300 300 300 300 300 300 300 3	9.5692-01 1.9559+00 2.9986+00 1100 1200 2.9986+00 2.6515+01 2.7177+01 2.8515+01 2.5796+01 2.3927+01 1.9918*01 1.6996+01 1.7962+01 1.5412+01 1.6996+01 1.7962+01 1.3491+01 1.2587+01 1.3439+61 1.1671+01 1.2587+01 1.3439+61
100       200       300         100000000       10000000       20000000         100000000       8.2964+01       300         100000000       8.2964+01       3.00         100000000       8.2964+01       3.00         10000000       8.2964+01       3.00         10000000       8.2964+01       7.83300-01       4.995         10000000       9.77300-01       9.05644-01       7.83330-01         10000000       9.77300-01       9.05644-01       7.83330-01         10000000       9.77300-01       9.05644-01       7.83330-01         10000000       9.7730-01       9.05644-01       7.83330-01         10000000       9.79550-01       9.79550-01       9.7750-01         10000000       9.79550-01       9.77550-01       9.7750-01         1000000       9.7950-01       9.0564-01       9.0564-01         1000000       9.7731-01       9.9567-01       9.9567-01         1000000       1.00000       9.0564-01       9.05657-01         1000000       1.00000       1.00000       9.0564-01         1000000       1.7131-01       9.9567-01       9.9567-01         1000000       1.71443+00       1.900000       1.	0.0000         9.5692-01         1.9559+00         2.9986+00           1000         1100         1.9559+01         2.9986+00           2.5549+01         2.100         1200         1300           2.5549+01         2.5545+01         2.7177+01         2.7777           2.5549+01         2.55455+01         2.7927+01         2.7927+01           2.1544511         2.52904-01         2.3927+01         2.7777+01           2.1544501         1.55455+01         2.3927+01         2.3927+01           2.44511         1.59955+01         2.3927+01         2.3927+01           2.445501         1.59955+01         1.95452+01         1.7952+01           2.6419+01         1.59955+01         1.95452+01         1.7952+01           2.6419+01         1.59955+01         1.54435+01         1.7952+01           2.6419+01         1.59915+01         1.54435+01         1.79435+01           2.6419+01         1.55412+01         1.5442+01         1.3452+01           2.6485+01         1.55412+01         1.54435+01         1.3452+01

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		006	3.0544+02 4.0695+02 5.0898+02 6.0467+02 6.395402 7.5915+02 7.5915+02				006	4,4977402 5,3805402 6,2757402 7,1808402 8,0941402 9,0158402				
		008	2.9035402 3.8292402 4.7364422 5.5657402 5.2657402 6.2739402 5.8480402				800 8	4.5051+02 5.2338+02 6.2347+02 6.2347+02 7.2026+02 8.1155+02 9.0354+02				
		700	2.7446+02 3.5844+02 4.3747+02 4.3747+02 5.6389+02 5.6389+02 5.0986+02				700	4.5158+02 5.4132+02 6.3218+02 7.2312+02 8.1416+02 8.1416+02 9.0583+02				
<u>L</u>		<b>600</b>	2.5850+02 3.3295+02 3.9385+02 4.5377+02 4.9759+02 4.9759+02		1600 4.3145+02 5.8543+02 7.5544+02 9.3583+02 1.1105+03 1.2748+03		0000	4.5318+02 5.4438+02 6.3613+02 7.2678+02 8.1722+02 9.0840+02		1600 4.4561+02 5.3514+02 6.2295+02 7.1212+02 8.0270+02 8.9459+02		
RTIES OF JP	CITY, PSIA	SSURE, PS1A 300	2.4227402 3.0524402 3.560845 3.9639402 3.9639402 4.2760402 4.5138402	CITY+PSIA	SSURE: PSIA 1500 4.1184+02 5.5800+62 7.1832+62 8.8535+02 1.1993+03 1.1993+03	PY: BTU/LB	SSURE, PSIA Son	4.5580+02 5.4974+02 6.4169+02 7.3112+02 8.2063+02 9.1119+02	PY, BTU/LB	SSURE, PSIA 1500 4.4853+02 5.3521+62 6.2316+02 7.1245+02 8.9509+02 8.9509+02		
BADHA SVA	FUGA	400 PRE	2,2492+02 2,7207+02 3,0631+02 3,3289+02 3,53311+02 3,6341+02	FUGA	PRE 1400 3.9289402 5.3133462 5.3133462 5.8208402 5.8378402 5.8879402 5.1248402	ENTHALI	PR5	4.6117402 5.5935402 6.4823402 7.3580402 8.2422402 9.1411402	ENTHAL	1400 4.4852402 5.3535402 6.2346402 7.1289402 8.9571402 8.9571402		
		- 905	2.0146+02 2.2732+62 2.4638+02 2.4638+02 2.45402 2.45402 2.8206+02 2.8206+02		1300 3.7455+02 5.0537+02 6.4666+02 7.9107+02 9.2632+02 9.2632+02 1.0511+03	÷	0 9 9	4.8168+02 5.6994+02 6.5470+402 7.4042+72 8.2782+02 9.1708+32		1 1 1 1 1 1 1 1 1 1 1 1 1 1		
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	803	888 888 888 888 888 888 888 888 888 898 808 80				609	88.5% 490.5% 490.5% 490.5% 490.5% 490.5% 401.001 601.401 601.001 601.001 601.001 601.001 601.001 601.001 601.001 888.0001 888.0001 888.0000000000		
Q	0024	6.4922-01 9.0486-01 9.1007-01 9.1007-01 5.1293-01 5.1293-01 5.1293-01	84 1		ι <b>χ</b>	004	8.27225.01 8.3497.01 8.43251.01 8.543251.01 8.55436101 8.703940101 8.7036101	Č,	
-7 105 - 011/2 0	600	9.0148-01 9.1964-01 9.1186-01 9.0326-01 9.0326-01 9.175-01	URE, BTU/LE	8.7160 8.71610 8.71610 8.71610 8.71610 8.71610 8.74650 8.74650 9.12864 9.12864 9.12864 9.12864 9.12864 9.1201 9.2684 9.101 9.2684 9.101 9.2684 9.101 9.2684 9.101 9.2684 9.101000 9.100000 9.10000000000	ME, BTU/LB-	600	8.2855~01 8.3493~01 8.44023~01 8.51753~01 2.66224~01 2.66224~01 2.65175701 3.01	ME. BTUAB	<ul> <li>L660</li> <li>L660</li> <li>L441-01</li> <li>L4441-01</li> <li>L4444-01</li> <li>L44444-01</li> <li>L44444-01</li> <li>L444444-01</li> <li>L444444-01</li> <li>L4444444</li> <li>L444444</li> <li>L4444444</li> <li>L4444444</li> <li>L4444444</li> <li>L4444444</li> <li>L444444</li> <li>L4444444</li> <li>L444444</li> <li>L444444</li> <li>L444444</li> <li>L4444444</li> <li>L4444444</li> <li>L44444444</li> <li>L44444444</li> <li>L44444444</li> <li>L444444444</li> <li>L44444444</li> <li>L44444444</li> <li>L444444444</li> <li>L444444444</li> <li>L4444444444</li> <li>L44444444444</li> <li>L444444444444444444444444444444444444</li></ul>
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GAS PROPT	PRE B	9.5221-01 9.1887-01 8.7525-01 8.7507-01 8.9127-01 9.0727-01	HEAT AT CON	1460 6.6242-01 6.6242-01 8.6764-01 9.6764-01 9.1457-01 9.2737-01	HEAT AT CO	PRE 400	8884 4888 4888 4888 4888 4988 4988 4988	HEAT AT CO	8 1400 9 8 8 8 9 9 9 9 9 10 10 10 10 10 10 10 10 10 10
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GAS PROPERTIES OF JP-7

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GAS PROPERTIES OF UP-7

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GAS PROPERTIES OF UP-7 AT SATURATION

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	-2°0430402	-2.4016402 -2.4016402 -8.1540203	-5.0504402	-0,9868402 -6.9868402
ENTROP :: Aturi R-d	10-02-00	2.8170-01 2.8170-01		5.2966-01
ENTHALPY. BTU/LE	9.3332401 1.3874402	2.6502402	3.0296+02	4.1365+02
FUGACITY. FSIA	4 . 42.58 - 03 1 . 4854 - 03	1.5%25+00 8.4292+90	2.8305+01	20+87L2 * 1
DENSITY. Levicuet	1.4699.94 3.7816-03	3.4435-02 1.7238-01	5°5°,03 1.6552≿∕33	4.5387+00
COMP Factor	949695-0 9-3686-01	9,6818+01 9,6818+01	5.1082×01 3.0731-31	6.0220-03
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EAS PROPERTIES OF UP-7 AT SATURATION

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5	VISCOSITY: Levente 1.2214-418 1.4262402 1.4262402	2,8535-02 2,0799-02	2.3789-02 2.9283-02
	Jet COEF, R/PSI 1.4473+00 8.4068+01 5.1346+01	2.3790-01	2,0653-01 2,6652-01
	Sék1C VEL FT/SEC 4.5183+82 4.53514+02 4.6335402	4-0010-02	4 <b>.3768+02</b> 3.1849+02
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AFAPL-TR-70-71

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AFAPL-TR-70-71

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# KEY WORDS

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Endothermic Fuels Thermally Stable Fuels Storage Stability of Fuels Catalytic Dehydrogenation Supported Platinum Catalysts Superscnic Combustion Shock Tube Studies SFELLDYNE-H<sup>©</sup> Fuel