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THE JOHNS HOPKINS UNIVERSITY
APPLIED PHYSICS LABORATORY
8621 GEORGIA AVE. SILVER SPRING, MD.

Operating under Contract NOrd 7386
With the Bureau of Ordnance, U. S. Navy

A SUMMARY OF THE STATUS OF RESEARCH ON THE MECHANISM OF COMBUSTION

Prepared by
Arthur D. Little, Inc.

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Arthur D. Little, Incorporated

This report is one part of a SURVEY OF THE LITERATURE ON COMBUSTION prepared by Arthur D. Little, Inc., for the Applied Physics Laboratory, The Johns Hopkins University. The Survey was conducted and the report written under the direction of Dr. W. H. Avery.

October 1946

NOTE

Attach this statement to BUMBLEBEE Report No. 47 by pasting this page to the blank page opposite the Table of Contents.

FOREWORD

Nature and Scope of

"SURVEY OF THE LITERATURE ON COMBUSTION"

as prepared by Arthur D. Little, Incorporated, for the Applied Physics Laboratory, The Johns Hopkins University.

I. Description of Project

A. Task Statement - "to provide a survey of information available on chemical reactions which may be of interest for rocket and ram-jet propulsion systems; information is particularly desired on studies leading to the elucidation of mechanisms of gas-phase oxidation reactions, including investigations of the initiation of reaction, explosion limits, flame propagation and stability, nature of reaction intermediates, and of accelerating and inhibiting agents."

B. Problem Statements

1. Experimental Methods - "to consist in the preparation of a report dealing with experimental methods which have been developed for the elucidation of reaction mechanisms; report should include a description of each method, nature of the information obtained, critical evaluation, references and suggested methods of applying new experimental techniques."

2. Reaction Studies - "to consist in the preparation of a card index giving,

   a. Chemical oxidation reactions on which significant data are available for the problems outlined in the task statement; each reference should include conditions of pressure, temperature and flow, nature of measurements, and summary of results.

   b. Short abstracts of the more significant papers in the field covered by the task statement.

   c. Short abstracts of all publications dealing with reactions which have been applied in the propulsion of rockets and ram-jets."

3. Summary of Status - "to consist in preparation of a report which summarizes briefly the material examined under Problem 2 above, showing the state of present knowledge with regard to the various aspects of the study of gas-phase oxidation reactions."

II. Scope and Preparation Procedure

A. Classified Material - the successful accomplishment of the task as outlined above required a review of both unclassified and classified information because most of the work on combustion carried out during the war - both here and abroad - was of a classified nature. Unfortunately no complete index of such material had been made nor had all of the pertinent classified reports, which numbered several thousand, been collected in a single agency. Consequently, with minor exceptions, within the time and funds at the task's disposal it was possible only to determine the sources of classified material and to indicate what general information was available from each. A description of these sources and a resume of the information obtained has been submitted as a separate part of the report to APL/JHU. Index cards were prepared for the few pertinent classified references which were disclosed in the investigation of these sources; in addition index cards and abstracts were prepared of the classified reports pertinent to this task which are located in the files of the Chemical Engineering Department of Massachusetts Institute of Technology. One copy of the classified abstracts and a set of all classified index cards are located at APL/JHU.

B. Unclassified Material - Review of unclassified material was carried out as follows:

1. Index cards carrying short abstracts were prepared for all references (approximately 3000) listed in CHEMICAL ABSTRACTS under the headings given below for the period beginning with 1936 (Volume 30) and running through August 1 1946. (Earlier work was not abstracted because of the existence of excellent summaries of combustion research, prior to 1936, by Jost, von Elbe & Lewis, and others). Subject headings abstracted were:

Calorific Value
Combustibility
Combustibles
Combustion
Decomposition
Detonation
Explosibility
Explosions
Heat of Combustion
Hydrocarbon Oils
Hydrocarbons
Igniters
Ignition
Inflammability
Inhibitors
Isotopes
Oxygen
Ozone
Para magnetism
Photochemistry
Projectiles
Promoters
Reaction Kinetics
Reactions

Explosives
Flames
Flash Point
Free Radicals (Detection of)
Fuels
Heat of Activation
Jet Propulsion
Mass Spectrograph
Mirrors
Molecular Beams
Ortho & para hydrogen
Oxidation
Rockets
Sound
Spectroscopy
Thermal conductivity
Thermodynamic properties
Thermodynamics
Turbines

2. On the basis of the short abstracts on the index cards, the references were divided in terms of relative importance into A, B, C and D groups. Articles rated A were judged to be of first importance to the general understanding of the mechanism and phenomena of combustion. Those grouped under B were judged to be either of less general excellence or more limited pertinence; C references were thought to have significance such that they would be worthy of consideration only in a thorough study of some phase of combustion research; the D group comprised references believed to be relevant only to special or derivative phases of combustion research.

3. After the references had been grouped as indicated above, abstracts of the A and B items as given in CHEMICAL ABSTRACTS were reviewed. If the importance of any given article seemed to warrant a more complete treatment than appeared in this publication, the Centralblatt reference was consulted. If neither appeared adequate the original article was studied and a more thorough review prepared.

4. Typed copies were prepared of all A and B abstracts plus those from CHEMICAL ABSTRACTS of the C articles which were not reviewed before being copied. Although these abstracts numbering some 2,000 were not requested in the task statement, it was felt that they would add greatly to the value of the survey. They, along with the card index are on file at APL/JHU.

5. With the A and B references as a basis, supplemented by the reading of selected references in whole or in part, the summary reports requested in Problems 1 and 3 were prepared.

III. Distribution of Results of the Survey

A. Problem 1 - Summary report has been issued by APL/JHU as Section T Memorandum CM-366, dated October 1946.

B. Problem 2 - The card index and abstracts described above are located at APL/JHU where they can be consulted by anyone visiting the laboratory.

C. Problem 3 - Summary report is presented herewith as BUMBLEBEE Report No. 47, dated October 1946.

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A SUMMARY OF THE STATUS OF RESEARCH
ON THE
MECHANISM OF COMBUSTION

A. Introduction.

In the preparation of this summary approximately 3000 papers, reports, and patents have been reviewed. Of these, roughly 800 were found to be of sufficient importance to merit further consideration. Unfortunately, because of time limitations, it has not been possible to read all of the pertinent papers. However, abstracts of these papers have been read and those papers, which from their abstracts seemed to be of primary importance, have been used as a basis for the preparation of this summary. Since the primary emphasis of this review has been placed on mechanism of reaction, detailed discussion of empirical phenomena is omitted. An attempt is made, however, to indicate sources of material, to aid the reader, interested in any special topic, to find the published reports on that topic. All compounds listed in Chemical Abstracts as promoters or inhibitors of reaction have been incorporated in the card index supplied with the report. No further explicit reference is made to them in this summary.

B. Fundamental Considerations.

Through the work of a large number of investigators, among whom Nernst, Bodenstein, Bonhoeffer, Polanyi, Rice, Eyring and Semenov may be mentioned particularly, the fundamental principles underlying the phenomena of gaseous oxidation reactions are rather well understood. Very briefly the salient features of the theory may be summarized as follows:

> Reaction between two molecular species, such as H_2 and O_2, proceeds through a series of bimolecular or, occasionally, termolecular collisions of molecules or atoms until final products are formed which are stable under the given experimental conditions. Usually reaction between two molecules or atoms does not take place unless the particles possess energy considerably in excess of the mean equilibrium thermal energy for the given temperature, i.e., an activation energy is required for reaction to take place. This is true even though energy is liberated in the reaction, i.e., internal energy is converted to translational, rotational, vibrational, electronic energy or radiation.*
>
> In addition to fulfilling the energy requirement, the colliding molecules must also be properly oriented if they are to react. Thus,

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* Although accurate values of the activation energy cannot usually be calculated, it is possible from thermodynamics to set lower limits to these values. Thus, the activation energy for an endothermic elementary reaction cannot be significantly less than the energy absorbed in the process.

the expression for the reaction rate includes a factor giving the collision probability of the two species which may be interpreted as representing the probability that the two colliding particles will possess sufficient energy to react, and finally, a steric factor representing the probability that the orientation requirements will be fulfilled. The first term will depend on the concentrations of the reacting particles, on their dimensions and on the forces of attraction or repulsion which act between them. The general expression for the rate will have, then, the following form:

$$\text{Rate} = Z\, s\, f\,(T)\; g\,([\text{reactants}])$$

in which Z is the number of collisions in unit volume, in unit time,
s is the steric factor,
f (T) is an undetermined function of the temperature,
g ([reactants]) is an undetermined function of the concentration of reactants, products and additives.

For a particular system reacting at constant temperature,

$$\text{Rate} = K\, g\,([\text{reactants}]).$$

The constant K is called the rate constant of the reaction.

To an approximation sufficiently close for most purposes, the product (at constant pressure) of Z and s may be considered proportional to the square root of the temperature and equated to $AT^{1/2}$ where A is a constant, and f(T) may be replaced by $e^{-E/RT}$, in which E is the activation energy (equal to the difference between the energy required for reaction of the given particles, and their average thermal energy, and R is the gas constant. With these substitutions,

$$K = AT^{1/2}\, e^{-E/RT}.$$

An understanding of the mechanism of a reaction, for our purposes, will be taken to mean the ability to estimate quantitatively the effects on the reaction of changes in temperature, concentration of reactants, nature and dimensions of the reaction chamber, and presence of additives. Both the rate of the reaction and the nature and amounts of products formed may be affected by these changes.

Investigations of a wide variety of chemical reactions have shown that only in rare instances can the mechanism of a reaction be understood without the postulation of one or more intermediate steps between the initial reaction and the formation of final products. This follows from the fact that the lowest energy process that can occur during the collision of two molecules is ordinarily one that involves the breaking of only a single bond. Thus reaction between molecules is most likely to occur by separate steps, involving first, dissociation of the least tightly bound molecule, followed by reaction of atoms or radicals so formed with other molecules. The radicals or atoms produced in this step react further, the process finally being stopped by the reactants being consumed or the intermediates being neutralized by chain breaking reactions in the gas phase or adsorption on the container walls.

One of the simplest examples of reactions of this type is that between H_2 and bromine. The reaction involves the following steps:[1]

(1) $Br_2 \longrightarrow 2\,Br - 45.2$ Kcal

(2) $Br + H = HBr + H - 16.4$ Kcal

(3) $H + Br_2 = HBr + Br - 40.5$ Kcal

(4) $Br + Br + M = Br_2 + M + 45.2$ Kcal

Inspection of the above scheme shows that steps (2) and (3) constitute a cycle or chain in which the active hydrogen and bromine atoms are regenerated, while two molecules of HBr are formed. Thus, if other considerations did not prevent it, all of the H_2 and Br_2 could react by the continued repetition of steps (2) and (3) once reaction (1) had furnished an initial bromine atom.

Two factors prevent ramification of the chains in this reaction. First, reaction (2) requires an activation energy of something over 16 Kcal so that only unusually energetic bromine atoms or hydrogen molecules can react and, secondly, bromine atoms are removed by reaction (4). The chain length is determined then by the relative probability of reactions (2) and (4).

The probability of reaction (4) is decreased by the fact that atoms can recombine only if a third body is present to carry off the energy released in the reaction. Polyatomic radicals may in some cases be able to recombine in the absence of a third body, since the possibility exists of distribution of the energy among several types of vibration and stabilization of the molecule for the period between collisions.

The hydrogen bromine reaction introduces several concepts of value in understanding the mechanism of gaseous oxidations:

(1) The importance of intermediates in bimolecular reactions.
(2) The influence of the energy requirements of the individual processes on the reaction kinetics.
(3) The possibility of chain formation.
(4) The termolecular character of most recombination or association reactions.

(5) The possibility of the course of the reaction's being affected by the reaction chamber's dimensions, because of chain breaking or initiation at the surface of the vessel.

Two further concepts are required for the elucidation of gaseous oxidation reactions: That of chain branching and that of formation of degenerate branching chains. Chain branching, originally postulated by Semenov[2], may be illustrated by the following sequence of reactions, which form part of the mechanism for the hydrogen oxygen reaction proposed by Von Elbe and Lewis[3a].

(1) $H_2O_2 + M = 2\,OH$

(2) $OH + H_2 = H_2O + H$

(3) $H + O_2 = OH + O$

(4) $O + H_2 = OH + H$

(5) $H + O_2 + M = HO_2 + M$

(6) $HO_2 + H_2 = H_2O_2 + H$

(7) $2\,HO_2 \xrightarrow{\text{surface}} H_2O_2 + O_2$

....

(12) $H, O, OH \xrightarrow{\text{surface}}$ destruction.

Consideration of the above scheme shows that each hydrogen atom reacting according to (3) leads to two particles capable of carrying on chains. A similar situation holds for reaction (4). As a consequence, if conditions are provided whereby reactions (5) and (12) become relatively unimportant, the number of chains will increase at a rate comparable to the collision number and explosion may result. Reactions in which chain branching may occur may be expected, therefore, to show great sensitivity to experimental conditions, since the effect of the branching is to raise to a high power ($\sim 10^9$) any slight change in the relative probabilities of the chain branching and chain ending processes. [(2)and(3) vs. (5)and(12)]

"Degenerate branching" of chains was postulated by Semenov[3b,3c,3d] to explain certain phenomena observed in the oxidation of hydrocarbons, such as the induction period and "cool flame". The phenomenon may be illustrated by the following hypothetical series of steps.

(i) $RH \longrightarrow R + H$

(1) $RH + H \longrightarrow R + H_2$

(2) $R \longrightarrow R' + \text{olefin}$

(3) $\text{olefin} + O_2 \longrightarrow \text{peroxide}$

(4) $\text{peroxide} \longrightarrow \text{aldehydes}$

(5) $\text{aldehydes} \longrightarrow \text{free radicals} + CO + H$

(6) $R' + RH \longrightarrow R'H + R$

This (purely illustrative) chain is propagated by reactions (1), (2) and (6). Branching can occur by (3), (4) and (5). However, since the products formed in (3) and (4) are relatively stable, the branching reactions are insignificant until an appreciable concentration of these intermediates is built up. Reaction may then proceed with rapidity or even violence.

The above review indicates that even a qualitative understanding of the mechanism of a reaction requires a knowledge of the identity, relative concentrations and method of reaction or destruction of the intermediate particles. Quantitative understanding requires, in addition, an estimation of the rate constants and activation energies of the individual step reactions, and the accommodation coefficients, collision cross-sections, diffusion rates, radiation probabilities and the rates of interchange of translational, rotational, vibrational and electronic energies among species and degrees of freedom.

In the following pages a brief attempt is made to show what progress toward these objectives has been made in the study of the oxidation of hydrogen, CO, methane and other hydrocarbons. Other oxidation processes are then reviewed and finally reference is made to papers dealing with the theory of chemical kinetics, with various physical aspects of combustion, with thermodynamic studies of fuels or combustion, and with rocket propellants.

C. The Hydrogen-Oxygen Reaction.

In common with other oxidation reactions, in which chain branching occurs, the hydrogen-oxygen reaction shows sharply defined regions of temperature and pressure in which the reaction rate increases rapidly with time so that explosion may result. Within these regions, as explained above, the probability of chain branching is greater than that of chain breaking. The boundaries of the regions are characterized by the manner in which they are observed experimentally, as the low pressure limit, the second limit and the high pressure or thermal limit. Thus, if a mixture of given composition is introduced into a vessel at a temperature of 575° C. and the pressure gradually increased, reaction will be negligible until the pressure reaches about 5 mm., when an explosion will occur. Mixtures introduced at pressures in the range 5 to about 150 mm. will explode in approaching thermal and kinetic equilibrium. Above this pressure, reaction proceeds at a finite but constant rate until a pressure of about 350 mm. is reached, when a third explosion occurs.

The effects on the hydrogen-oxygen reaction of pressure, temperature and vessel dimensions are well explained by the most recent mechanism proposed by Von Elbe and Lewis[3a].

$$(1)\quad H_2O_2 + M = 2\,OH$$

$$(2)\quad OH + H_2 = H_2O + H$$

$$(3)\quad H + O_2 = OH + O$$

$$(4)\quad O + H_2 = OH + H$$

$$(5)\quad H + O_2 + M = HO_2 + M$$

$$(6)\quad HO_2 + H_2 = H_2O_2 + H$$

$$(7)\quad 2HO_2 \xrightarrow{\text{surface}} H_2O_2 + O$$

$$(8)\quad H + O_2 + H_2O_2 = H_2O + O_2 + OH$$

$$(9)\quad HO_2 + H_2O_2 = H_2O + O_2 + OH$$

$$(10)\quad H_2O_2 \xrightarrow{\text{surface}} H_2O + 1/2\ O_2$$

$$(11)\quad H_2 + O_2 \xrightarrow{\text{surface}} H_2O_2$$

$$(12)\quad H,\ O,\ OH \xrightarrow{\text{surface}} \text{destruction}$$

In a very detailed examination of the kinetic consequences of this scheme, Von Elbe and Lewis show that it is in quantitative agreement with the observed low and high pressure explosion limits and third explosion limit, and that it correctly predicts the effect of change in vessel dimensions. The authors give values of the rate constants and activation energies of all reactions except (2) and (4). Concentrations of HO_2 and H are also presented.

The scheme has been substantiated by work of Nalbandjan[4a,b,c], who derives a theoretical expression for the dependence of the induction period on pressure and temperature, from which the activation energy of reaction (3) is deduced to be 23 K calories. (Instead of the value of 17 Kcal quoted by Von Elbe and Lewis). Zel'dovich and Semenov, however, from a study of the combustion of moist CO arrived at a value of 18 Kcal for the same reaction. The Von Elbe and Lewis scheme is also in agreement with results of photo chemical experiments by Smith[5] who postulated reactions (2), (3), (4), (5), (6), and (7) plus

$$O_2 + h\nu \longrightarrow 2O$$

$$O + O_2 + M \longrightarrow O_3 + M$$

to explain his data.

Oldenberg and Morris[6] state in a preliminary report that the Von Elbe and Lewis theory does not correctly predict their observations on the effect of Pyrex vessel size on the third explosion limit although it is strikingly successful in predicting the effect of added gases on the second explosion limit. Undoubtedly further inconsistencies in the quantitative aspects of the theory will appear as further experimental work is performed, but it seems unlikely that major changes in the basic mechanism will be required.

Studies of various phases of the hydrogen-oxygen reaction by a number of investigators are listed below under appropriate headings.

(1) Mechanism and Kinetic Theory.

An early treatment upon which most of the later work is based was given by Kassel[7]. Polyakov and co-workers[8-16] have discussed the mechanism with particular reference to the prediction of H_2O_2 formation. Frank-Kamenetskii[17] has applied his theory of thermal explosions with success to the third explosion limit. Behrens[18] and Clusius and Gutschmidt[19] have discussed the effect of hydrogen diffusion on the rate of flame propagation. The latter authors experimented with light and heavy hydrogen.

(2) Induction Period.

In addition to the work of Nalbandjan previously mentioned, Shtern, Kravets and Sokolik[20] have studied the auto-ignition of mixtures of hydrogen and air.

(3) Thermal Reaction.

The thermal reaction has been the subject of a number of studies by Oldenberg and co-workers[21-24], by Chirkov[25], who concludes from his work that the mechanism of the reaction is different at high and low pressures, and by Neimark, Kulashina and Polyakov[26].

(4) Lower Limit of Ignition or Explosion.

The effect of various experimental conditions, including the presence of metal wires of different compositions, was investigated by Kimata, Aomi and Goto[27] and Horiba and Goto[28]. Data on the lower region of ignition in quartz and pyrex vessels with and without added argon were obtained by Biron and Nalbandjan[29]. Tauzin[30] and Garner and Pugh[31] have studied the propagation of low pressure flames in tubes. Hayakawa and Goto[32] have studied the spark ignition of $H_2 - O_2$ mixtures at low pressures.

(5) Second Explosion Limit.

An extensive investigation of the explosion limit as a function of pressure was made by Dyksman[33] using various types of sparks for ignition. Von Elbe and Lewis[34] have discussed the consequences for the theoretical mechanism of the effect of water vapor on the second explosion limit. Much of the work mentioned under Section (4) is also pertinent to this topic.

(6) Reaction in the Region of the Third Explosion Limit.

The theory and kinetics of the third explosion limit have been studied by Von Elbe[35], Heiple and Lewis[36], Oldenberg and Sommers[37], Oldenberg[38] and Ziskin[39].

(7) Effect of additives.

References are listed without discussion under appropriate headings.

(a) Nitrogen Dioxide.
Von Elbe and Lewis[40,41], Norrish and Dainton[42, 5], Crist and Wertz[43].
(b) Nitrosyl Chloride.
Dainton and Norrish[44,45].
(c) Nitrous Oxide
Danby[46].

(d) Inert Gases
Von Elbe and Lewis[47], Jorrissen[48], Valkenburg[49]

(e) Hydrogen Peroxide
Dainton and Norrish[50]

(f) Silver
Nalbandjan[51,52]

(g) Carbon Monoxide
Prettre[53]

(8) Measurement or Calculation of Concentration or Reaction Rates of Intermediates.

Studies of the concentration of hydroxyl radicals in reacting hydrogen-oxygen mixtures have been made by Avramenko[54,55], by Kondrat'ev and Ziskin[56] and by Oldenberg and co-workers[57,58,59,60]. Kondrat'ev[61] has reviewed spectroscopic data on OH obtained from various sources. Rodebush[62] has studied products obtained from reactions in a glow discharge and mentions the use of the luminescence of the reaction NO + O as a test for the presence of oxygen atoms. Von Elbe and Lewis[63] discuss reactions of HO_2 and give rate constants for the reactions

$$H + O_2 + H_2 = HO_2 + H_2$$

and

$$H + O_2 \longrightarrow OH + O.$$

Semenov[64] gives rate constants for the latter reaction and

$$H_2 + O_2 = 2\ OH.$$

Baker[65], Kondrat'eva and Kondrat'ev[66], and Mackenzie and Mowbray[67] discuss the decomposition of hydrogen peroxide.

(9) Photochemical and Spectroscopic Observations.

In addition to the work of Smith[5], the photochemical oxidation of hydrogen has been studied by Nalbandjan[68] and Pavlyuchenko[69]. Kitagawa[70] has found spectroscopically, water molecules with vibrational energies as high as 50 Kcal. A chain mechanism is proposed in explanation. Kondrat'ev[70a] has computed the temperature dependence of the lower limit from spectroscopic measurements of hydroxyl concentration. Oldenberg[70c] has studied OH radicals produced by a discharge in water vapor.

(10) General

A clear and interesting review of the mechanism of the hydrogen-oxygen reaction has been given by Von Elbe[70b] who lists the following values for some of the rate constants for the reaction steps.

Absolute Values of Rate Coefficients and Activation Energies

$$k_3 = 1 \times 10^{-17} \sqrt{T/803} \; e^{-(1-T/803)17000/803R} \; \text{cm.}^3 \; \text{sec.}^{-1}$$

$$k_6 = 4.23 \times 10^{-19} \sqrt{T/803} \; e^{-(1-T/803)24000/803R} \; \text{cm.}^3 \; \text{sec.}^{-1}$$

$$k_9 = 2.0 \times 10^{-16} \sqrt{T/803} \; e^{-(1-T(803)14000/803R} \; \text{cm.}^3 \; \text{sec.}^{-1}$$

$$k_{1,H_2} = 3.39 \times 10^{-22} \sqrt{T/803} \; e^{-(1-T/803)45500/803R} \; \text{cm.}^3 \; \text{sec.}^{-1}$$

$$k_{5,H_2} = 3.7 \times 10^{-35} \sqrt{T/803} \; \text{cm.}^6 \; \text{sec.}^{-1}$$

$$k_{H_2O_2} + H + O_2 \longrightarrow H_2O + O_2 + OH = 6.8 \times 10^{-35} \sqrt{T/803} \; \text{cm.}^6 \; \text{sec.}^{-1}$$

$$k_{7,H_2} = 17.2 \times 10^{20} \sqrt{T/803} \; \text{cm.}^{-1} \; \text{sec.}^{-1}$$

D. Combustion of Carbon Monoxide.

Although the reaction of carbon monoxide and oxygen appears superficially to present less complicating features than that of hydrogen and oxygen, the details of the mechanism are less well understood. Experimentally, the reaction shows a low pressure and high pressure explosion limitsoccurring at 600°C at about 13 and 30 mm. respectively (Zyono[71]). There is some doubt, however, that explosion can occur in the absence of sources of hydrogen atoms. Buckler and Norrish[72], Lewis and Von Elbe[73] have proposed a reaction mechanism involving CO_3, ozone and oxygen atoms which satisfactorily explains the upper explosion limit as a function of pressure and added gases. This mechanism has been criticized by Jost[74] who feels the evidence favors a branching due to energy chains rather than radicals. Work of Gaydon[91] on the absorption of hot oxygen in dry $CO-O_2$ flames is of interest in this connection. Zatsiorskii, Kondrateev and Solnishkova[87b] have studied spectroscopically the reaction between CO and O_3, which is of importance in the mechanisms proposed by Von Elbe and Lewis[3] and Jost[74].

Interpretation of the reaction is rendered difficult by its extreme sensitivity to traces of hydrogen or other impurities, and most of the work since 1938 has been concentrated on mixtures of carbon monoxide and oxygen containing additives.

Buckler and Norrish[75,76] appear to have established the following mechanism for the hydrogen sensitized reaction:

$$\left.\begin{array}{l} H + O_2 = OH + O \\ O + H_2 = OH + H \end{array}\right) \text{ Chain Branching}$$

$$\left.\begin{array}{l} OH + H_2 = H_2O + H \\ OH + CO = CO_2 + H \end{array}\right) \text{ Chain Propagation}$$

$$\left.\begin{array}{l} H + O_2 + M = HO_2 + M \\ O + CO + M = CO_2 + M \end{array}\right) \text{ Chain Ending}$$

Support for the mechanism was found in experiments on the ignition limits using D_2 instead of H_2 and using inert gases. The work of Norrish is in agreement with experiments of Kondrat'eva and Kondrat'ev[77] who have measured the concentration of hydroxyl radicals present during the reaction. Their measurements show that the hydroxyl concentration is one hundred times the equilibrium thermal concentration and prove that the radical is produced by chemical means. Comparison of the rate of disappearance of OH with the rate of formation of CO_2 shows that the two rates are in agreement, substantiating the hypothesis that CO_2 is formed primarily by

$$OH + CO = CO_2 + H$$

with an activation energy of 10 Kcal. From a study of the emission of radiation of moist and dry carbon monoxide flames, Kondrat'eva and Kondrat'ev[78] concluded that the reaction mechanism was different in the two mixtures. Their measurements of the total amount of radiation [79] indicated that one molecule in ten of CO_2 was in an excited state. Kondrat'ev[80] and Zel'dovich and Semenov[81a,81b] have reviewed experimental work.

Additional work on carbon monoxide oxidation is summarized below under appropriate headings.

(1) Slow Thermal Reaction.

Korneieva[82] has studied the effect of platinum wires of various lengths on the rate of the slow oxidation. The results are interpreted to indicate a predominantly homogeneous chain reaction at temperatures above 600°C but with some evidence of catalysis or inhibition by surface effects. Below 600°C, the reaction becomes more heterogeneous.

(2) Reaction in the Region of the Upper Explosion Limit.

The effect of quartz surface on the reaction has been studied by Kondo and Toyama[83].

(3) Spectroscopic and Photochemical Studies.

Kondrat'eva and Kondrat'ev[84] have determined the chain length in the photo-oxidation of CO as a function of the temperature. The length varies from 2 at 400°C to 560 at 490°C where the dark reaction is appreciable. Faltings, Groth and Harteck[88] and Groth[88a] have studied the photochemical decomposition at room temperature using CO and CO, H_2 mixtures. In the latter case, HCO was postulated as an intermediate product to explain H_2CO production. Flame studies have been made by Kondrat'eva and Kondrat'ev[77,78,79,80,84,85,86,87,87b] Tawde[89], Bonhoeffer[90], Gaydon [91,92,93,94,95,96], who finds evidence in the last reference for a high concentration of atomic oxygen in both dry and moist CO flames, and Jorissen[97].

(4) Mixtures and Additives.

Further studies have been conducted by Walls[98], who found evidence for a change in mechanism with change in pressure in H_2, CO oxidations; Karzhavina[99] who studied the effect of water at temperatures over 1000°C; by Dijksman[100] who studied CH_4-CO-O_2 mixtures; by Drozdov and Zel'dovich[101] who investigated the effect CCl_4 on the flame propagation; and Calhoun[102], Brown and Crist[103] who studied CO-NO_2-O_2 mixtures.

E. Oxidation of Hydrocarbons.

Because of the large number of intermediate reactions possible in the oxidation of hydrocarbons, only a few general conclusions have been drawn regarding the reaction mechanisms. The oxidation of all hydrocarbons displays certain general features indicating that the reactions proceed through the formation of degenerate chains, but the specific intermediates involved have not been established. It is evident that much more extensive studies, possibly supplemented by new techniques, are required of the oxidation of the specific hydrocarbons before an approach can be made to a quantitative understanding of the processes involved.

(1) Methane.

Studies of the partial oxidation of methane by a number of investigators, e.g. Garner and Ham,[104] Slotin and Style[105], Bone and Gardner[106], and Degens[107] have shown that formaldehyde methyl alcohol, formic acid and traces of peroxides are formed, as well as water, carbon monoxide, hydrogen and carbon dioxide. The reaction shows an induction period which varies with pressure and temperature in accordance with the degenerate chain theory of Semenov (Gardner and Ham[104]). Addition of any of the intermediates shortens the induction period, indicating that they or products of their decomposition play a part in the chain reaction. Formally, these observations may be explained by a number of theories, of which, superficially at least, the most satisfactory is that of Von Elbe[70b]. The following scheme is proposed:

(1) $OH + CH_4 \longrightarrow H_2O + CH_3$

(2) $CH_3 + O_2 \longrightarrow CH_3O_2 \longrightarrow H_2CO + OH$

(3) $OH + H_2CO \longrightarrow H_2O + HCO$

(4) $HCO + O_2 \longrightarrow HO_2 + CO$

(5) $HO_2 + H_2CO \longrightarrow HCOOH + OH$

(6) $HCOOH \longrightarrow H_2O + CO$

Evidence for all of the above reactions appears to be on rather firm ground through work of Von Elbe and Lewis[108], Van Tiggelen[136b], and Harteck, Groth and Faltings[88] among others. The mechanism explains the induction period and, qualitatively the explosion limits, but additional steps would be required to explain the remaining intermediate products. A more extensive discussion of these points is given in references 70b, 74, 109, 110 and 141.

Additional work pertinent to the oxidation of methane is reviewed below.

(a) Ignition and Induction Period.

Kane[111] has compared ignition temperature as a function of pressure for a number of the simpler aliphatic hydrocarbons, alcohols and aldehydes. The purpose was to determine the relative reactivity of alcohols, aldehydes and parent hydrocarbons. For the single C-atom set, the order aldehyde > alcohol > hydrocarbon was observed. Townend[112] has tabulated ignition temperatures and inflammability limits of a wide variety of hydrocarbons. Sachsse[113] has measured induction times and ignition temperatures in methane oxygen mixtures by a flow method. Polyakov and Kuleshina[114] have studied the effect of mixture ratio and added gas. Malinovskii and Malyar[115] and Davies[116] have studied the ignition of methane by hot wires.

(b) Second Pressure Limits.

Explosion pressure as a function of mixture composition was studied by Dijksman and Brandhof[117].

(c) Reactions of Intermediates and Products.

Kushnerev and Shekter[118] obtained about 50% H_2CO by reaction of methane with oxygen atoms. Soloveichik[119] found that ozone gave only partial oxidation of methane. Staronka and Czerski[120,121] studied the variation in the amounts of methyl alcohol and formaldehyde formed as a function of time and mixture ratio. Polyakov and Stadnik[122] found that an organic peroxide is not a primary oxidation product.

(d) Photochemical and Spectroscopic Studies.

Groth and Laudenklos[123] and Groth[124] studied the photodecomposition of methane. An extensive study of flames of hydrocarbons including methane has been made by Vaidya.[125] Bands of OH, HCO, C_2, CH have been identified. The bearing of the observations on the mechanism of combustion is discussed. Similar studies have been made by Guenault,[126] Coppens,[127] and Bell.[128]

(e) Studies of Additives and Mixtures.

1. Ethylene
   Rivin[129] found that purified methane air mixtures did not detonate. On addition of .3% ethylene, the mixture could be exploded.

2. Nitrogen
   Polyakov and co-workers[130, 130a] have studied the formation of nitrogen oxides as a function of composition pressure and dimensions of the reaction vessel.

3. Methyl Nitrite

   Gimmelman and Neiman[131] found that methyl nitrite eliminated the induction period in methane and ethane combustions.

4. Surface

   Norrish and Reagh[132] have examined theoretically and experimentally the influence of surface on the reaction and the bearing of the results on the chain theory. Polyakov and co-workers[133,134] investigated the effect of platinum on the induction period. Jorissen and Lebbink[135] found that the reaction temperature of methane-oxygen mixtures varied between 170°C and 540°C depending on the salt used to coat the inside of the reaction vessel. LiF gave the lowest temperature, RbI the highest, while in the uncoated vessel, the minimum temperature for observable reaction was 250°C.

(f) Studies of Reaction Mechanism or Kinetics.

Van Tiggelen[136a,b] has proposed a mechanism for methane oxidation depending on hydroxyl radicals and HCO radicals for chain propagation. This is essentially the same process as proposed by Von Elbe. Prettre[137] proposes that two mechanisms operate in the oxidation of methane and ethane, one involving peroxidation at temperatures around 200 to 300°C and the other above 500°C, where peroxides apparently play no part. Polyakov and Korneeva[138] have investigated the kinetics of slow combustion of methane at low pressures in the presence of platinum surfaces. Norrish[139] has studied in considerable detail the kinetics of methane combustion. Among other things, his work shows that chlorine at low pressures inhibits reactions, but at high pressures catalyses it; iodine has a marked anti-catalytic effect in the neighborhood of ignition temperature. Addition of small amounts of formaldehyde removes the induction period but does not alter the maximum velocity of the reaction. Nitrogen peroxide has a strong sensitizing reaction and HCl catalyses the production of formaldehyde. The mechanism proposed involves an atom chain modified to include the phenomena of degenerate branching. Semenov[139a] has also proposed an atom chain to explain oxidation of methane and other hydrides. Newitt and Gardner[139b] have investigated the compounds formed during the induction period in the slow oxidation of methane. Ogura[141] has reviewed the oxidation of methane.

(g) Thermal Decomposition of Methane.

Patat[140,140a] has studied the reaction $CH_3 + H_2$ by means of para hydrogen. Hinshelwood and co-workers[142,143] used nitric oxide to study the chain length in the methane decomposition, and Letort and Duval[144] found evidence with tellurium mirrors that CH_2 radicals are formed in an electric discharge through methane.

(2) Ethane.

No examinations of ethane oxidation of sufficient generality to serve as a basis for a complete mechanism have been proposed. However, various aspects of the oxidation have been studied and these are summarized under the appropriate headings.

(a) Reaction Kinetics.

Sadovnikov[146] has studied ethane oxidation at pressures of 50 to 200 mm., temperatures in the range of 600 to 750°C with various reaction vessels and found that the kinetics can be explained in terms of the Semenov theory of branching chain reactions. Andreev[147] has investigated the influence of water, carbon dioxide and carbon monoxide on the kinetics. Gimmel'man, Neiman, and Sokov[148] have investigated the effect of nitrogen dioxide and methyl nitrite on the region of inflammation of ethane-oxygen mixtures. Fernandez[149] has investigated the effect of ozone on the combustion of a number of hydrocarbons including ethane. Chirkov[150] studied the action of water vapor on the kinetics of ethane oxidation.

(b) Photochemical Studies.

The photochemical decomposition of ethane has been studied by Faltings[151], who found the primary products to be methyl radicals.

(c) Thermal Decomposition.

Thermal decomposition of ethane has been investigated by Hobbs and Hinshelwood[152], Theile[153,154], Storch and Kassel[155], Steacie[156], Steacïe and Shane[157], Kuchler and Theile[158], and Dintses[159]. Rice and Herzfeld[160] have examined the bearing of new values of the heats of activation on the mechanism of the thermal decomposition of ethane, which they proposed earlier.

(3) Propane.

A tentative scheme for the oxidation of propane, proposed by Von Elbe[70b], is presented on the following page. An enormous amount of research would be required to prove or disprove its validity. In the main, the mechanism predicts correctly the products obtained by Newitt and co-workers[161,162] who have made careful studies of the intermediates formed by slow oxidation. These workers found n propyl, isopropyl, ethyl and methyl alcohols, formaldehyde and higher aldehydes, acetone and traces of peroxides in the product of the slow oxidation at pressures above ten atmospheres. Harris and Egerton[162a] found propylene and H_2O_2 also. Other investigations of propane are summarized below under appropriate headings.

(a) Inflammation Ranges.

The cool flame range of propane in air at ordinary temperatures and pressures above 12 atm. has been determined by Hsieh and Townend[163]. Van der Hoeven[164] has studied the low pressure limit.

(b) Reaction Kinetics and Physical Measurements.

The kinetics of propane oxidation have been studied by Pease[165,166,166a] and by Newitt and Thornes[167]. Van der Poll and Westerdijk[168] have measured the flame temperature of propane oxygen mixtures and Ribaud and Gaudry[169] have investigated the effect of pressure on the velocity of flame propagation.

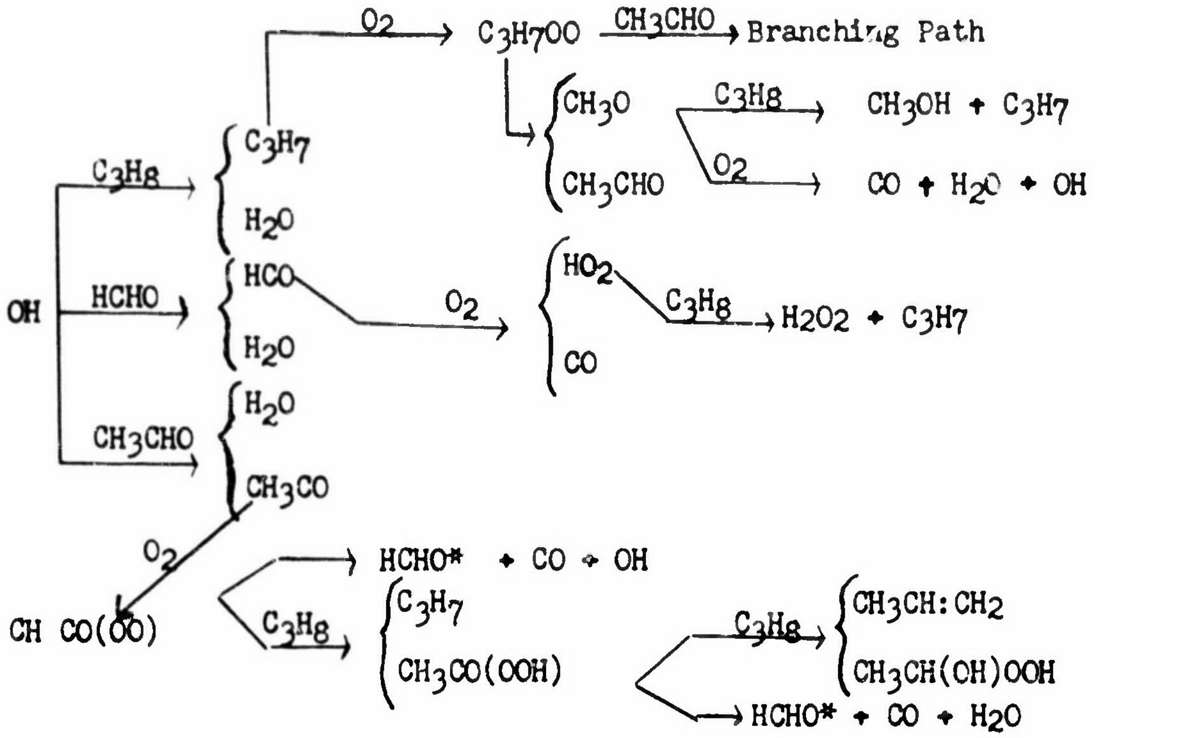
(c) Studies of Mixtures and Additives.

Fernandez[149] found that ozone accelerates propane oxidation, Kane[170] has studied the effect of nitrogen peroxide on the ignition of propane butane and acetaldehyde. His results suggest a different mechanism of oxidation at high and low temperatures depending on the stability of the intermediate hydrocarbons. From studies of the effect of surface treatment on the ignition characteristics, Day[171] concluded that peroxides were not of primary importance in the phenomena of cool flames.

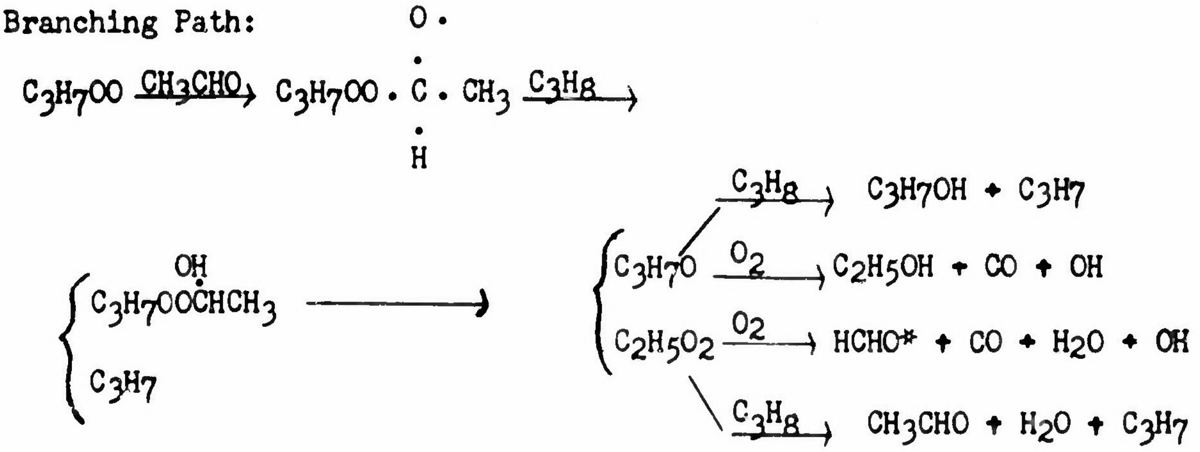
(d) Mechanism.

A mechanism of propane oxidation involving propane molecules was proposed by Pease[172] in 1935.

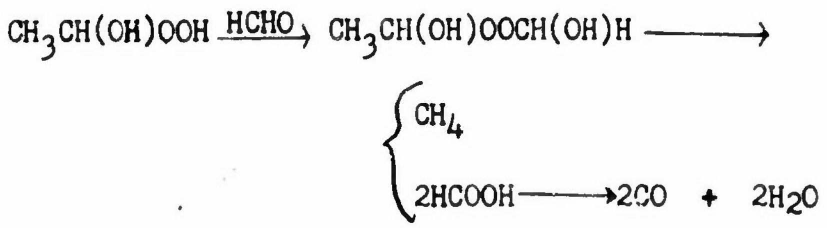
Tentative Scheme of Chain Reaction Between Propane and Oxygen



Branching Path:



Side Reactions:



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* Pease and Munro report that a test for this aldehyde was negative. J. Am. Chem. Soc., 56, 2034 (1934)

(e) Thermal Decomposition.

Burk[173] has proposed a theory for the decomposition of straight chain hydrocarbons which he feels is superior to that of Rice. He assumes that every C-C bond is equivalent and that reaction occurs when a given amount of energy is accumulated in a single bond. The rate is given then by the probability of the localization of the requisite energy.

(4) Butane.

The mechanism of the oxidation of butane and the higher straight chain paraffins is presumably similar to that of propane. No general discussion will be attempted of the mechanism of these reactions.

(a) Ignition.

Andreev[174] has investigated the effect of pressure and temperature on the "cold" and "hot" flames of butane. Neiman and Tutakin[175,176] studied the kinetics of the cold flame phenomena and concluded that the results were evidence for the chain theory of reaction. Pushlenkov[177] showed that the silent discharge reduces the induction period of the cold flame.

(b) Studies of Mixtures and Additives.

Aivazov, Neiman and Khanova[178], Avramenko and Neiman[179], and Aivazov and Neumann[179a] studied the effect of H_2, CO_2, diethyl and diisopropyl ethers, acetone, NO_2 and acetaldehyde on the induction period of the cold and hot flames. Only diethyl ether, aldehyde and NO_2 promote ignition. Blat, Gerber and Neiman[180,181] studied the effect of diethyl peroxide and methyl hydroperoxide on the induction period and derived an expression, based on the Semenov theory, which gave quantitative agreement with their results. Smittenberg and Kooijman[181a] have studied the effect of lead tetraethyl on the flame velocity and concluded that the antiknock action of the lead was caused by its effect on the preflame reaction rather than on the flame speed. The effect of ozone has been investigated by Fernandez[149] and the effect of NO_2 by Kane[170]. Schuit[181b] has made an extensive study of the effect of surface and of a large number of additives.

(c) Mechanism.

The role of peroxides in the formation of cold and hot hydrocarbon flames has been reviewed by Neiman[182].

(d) Thermal Decomposition.

The thermal decomposition of butane has been studied by Echols and Pease[183,184]. The decomposition induced by free radicals is discussed by Rice and Polly[185] and Steacie and Folkins[186], the NO inhibited reaction by Steacie[187]. An alternative to Rice's theory has been proposed by Burk[173].

(5) Pentane.

(a) Ignition.

The ignition delay and slow oxidation of pentane oxygen mixtures have been shown by Prettre[188,189] and Shtern, Kravets and Sokolik[190] to be in accord with predictions of the Semenov theory.

(b) Reaction Kinetics.

Further kinetic studies have been made by Aivazov and Neiman[191] and Prettre[192].

(c) Studies of Mixtures and Additives.

Aivazov and Neiman[179a,193] observed the effect of addition of N_2, NO_2 and CH_3CHO on the induction period of cold flames. Belov[193a] investigated the effect of diisopropyl ether and proposed an integral equation valuable in predicting the length of the induction period. Prettre[194] has studied the inhibition action of hydrogen and of the metal walls[195]. Fernandez[149] has studied the action of ozone in the slow combustion and Kane[170], the effect of NO_2. Neiman[182] reviews the role of peroxides in the formation of hydrocarbon flames.

(d) Mechanism.

Aivazov and Neiman[196] propose a two stage mechanism to explain the low temperature combustion of pentane.

(e) Thermal Decomposition.

Gray and Travers[197] have studied neopentane decomposition. Mechanisms of hydrocarbon decomposition have been proposed by Rice and Polly[185] and Burk[173].

(6) Ethylene.

Apparently the mechanism of ethylene oxidation resembles that of the saturated hydrocarbons, the principal difference in the products being the appearance of ethylene oxide. Jost[198] has found evidence from the spectrum that the reaction involves hydroxyl radicals, as reactions of saturated hydrocarbons appear to. Smith[198a] has also studied the flame spectrum.

(a) Ignition.

Forsyth and Townend[199] and Kane[200] have determined the inflammation range of ethylene at various pressures and found that NO_2 facilitates combustion.

(b) Reaction Kinetics.

The slow oxidation of ethylene in the presence of varying lengths of platinum wire has been studied by Polyakov and Vainshtein[201] and Polyakov[202]. Burning velocities and inflammability ranges were investigated by Young and Krase[203].

(c) Studies of Mixtures and Additives.

Bone[204] has studied the effect of dilution with inert gases on explosions of ethylene and oxygen.

(7) Acetylene.

The oxidation of acetylene probably proceeds through the initial formation of an acetyl radical, which reacts with oxygen to form a glyoxal radical (Lewis and Von Elbe[70b]):

$$OH + C_2H_2 \longrightarrow HC \equiv C\text{—} + H_2O$$

$$HC \equiv C\text{—} + O_2 \longrightarrow (CH \equiv C.O_2) \longrightarrow CHO \cdot CO\text{—}$$

$$CHO \cdot CO\text{—} + C_2H_2 \longrightarrow CHO \cdot CHO + HC \equiv C\text{—}$$

(a) Reaction Kinetics.

Steacie[205] investigated the oxidation of acetylene and proposed a mechanism involving direct oxidation as the first step. Bone[204] studied the effect of inert gases on the explosive combustion. Khitrin[205a] studied the effect of pressure on the rate of flame propagation.

(b) Spectroscopic Studies.

Avramenko[206,206a] measured the OH concentration and found that the number of OH radicals was proportional to the combustion rate. Jorrisen[97] studied the behavior of flames burning in a combustible atmosphere. Rossikhin and Timkovskii[207,208] studied the effect of a high frequency discharge on the flame spectrum. No significant effect was found. Geib and Vaidya[208a] studied the flame in atomic oxygen and Tominaga and Okamoto[208b] the flame in Cl_2 and Br_2.

(c) Thermal Reaction.

Frank-Kamenetskii[209] investigated the kinetics of the polymerization reaction.

(8) Aromatics.

An extensive series of investigations of the oxidation of aromatics has been conducted by Burgoyne and Newitt[210-214] and Fahlbusch[214a]. The reactions are characterized by primary oxidation of the side chains followed by phenol formation and then ring rupture. A detailed mechanism has not been proposed. Vaidya[214b] concludes from spectroscopic studies that oxygen is first incorporated in the ring.

(a) Ignition.

Maccormac and Townend[215] studied the spontaneous ignition under pressure. Liviu[216] investigated the ignition potentials of the xylenes.

(b) Reaction Kinetics.

The kinetics of the slow combustion of benzene have been studied by Amiel[217]. The products contained phenol and benzoquinone but no peroxides. Fahlbusch[214a] has reviewed (1942) the oxidation, thermal and photochemical decomposition of simple aromatic compounds.

(c) Thermal Decomposition

Hein and Mesee[218] studied free radical formation from pyrolysis of benzene and toluene at 900 - 1100°C. Unequivocal evidence for the tolyl radical was found but the product from benzene could not be identified.

(9) Studies of Miscellaneous Hydrocarbons.

(a) Ignition and Induction Phenomena.

Damkohler and Eggersgluss[219] investigated the products formed by rapid compression without ignition of gasoline air mixtures. Using a similar technique Jost et al[220,220a] obtained curves of induction period vs temperature for heptane, isooctane and benzene. Spontaneous ignition and slow combustion of paraffin and olefin hydrocarbons have been discussed by Newitt and Townend[221], Hsieh and Townend[163], Day and Pease[166a], Teichmann[222], and Townend[223]. Reutenauer[224] determined the upper and lower ignition regions of ketene, decalin and dipentene. Treer[225] has tabulated values of the constants in an equation useful for predicting ignition times.

(b) Reaction Kinetics.

Thermal studies of hydrocarbon oxidation have been made by Estradere[226]. Kroger and Kaller[227] studied the concentration of peroxides in cetane oxidized for varying time intervals. Maillard and Friedrich[228] determined the products of incomplete combustion of light hydrocarbons The preliminary reactions in a gasoline engine were studied by Peletier, van Hoogstraten, Smittenberg and Kooyman[229], and the oxidation of cracked gases by Pigulevskii and Gulyaeva[230].

(c) Mechanism.

Gervart and Frank-Kamenetskii[231] have discussed the periodic character of the cold flame of hydrocarbon-air mixtures under turbulent flow, in terms of the Frank-Kamenetskii theory. George, Rideal and Robertson[232] found support for the primary formation of hydroperoxides in oxidation through experiments on catalyzed and uncatalyzed oxidation of alkyl benzenes and long chain aliphatic hydrocarbons. Jost, Muffling and Rohrmann[233] proposed (1936) a generalized chain reaction for hydrocarbon oxidation. Prettre[234] discusses the negative temperature coefficient of hydrocarbon oxidation in terms of the formation of an intermediate which may decompose to give branching or stable products. Semenov[235] gives a preliminary theory to explain hydrocarbon oxidation. Townend[236] discusses the possible connection between "blue flames" and peroxide decomposition. Ubbelhode[237,238] has reviewed the oxidation of paraffins. Von Elbe[239] proposed (1937) a mechanism for hydrocarbon oxidation. Von Elbe and Lewis[240] have discussed the relation between hydrocarbon oxidizability and engine knock.

(d) Studies of Mixtures and Additives.

Briner, El-Djabri and Paillard[241] and Tawada[242] studied the ozonization of hexane, heptane and octanes. Maess[243] studied the accelerating effect on the oxidation of heptane, of free radicals formed from azomethane decomposition. Ubbelohde[244] discusses the effect of pro-detonant additives on hydrocarbon combustion. Vyrubov[245] studied the effect of ozone and hydrogen on oxidation in Diesel engines.

(e) Spectroscopic Studies.

Kondrat'ev and Ziskin[246] showed that the hydroxyl radical concentration in hydrocarbon flames is several hundred times the equilibrium concentration, indicating that these radicals are an important factor in hydrocarbon oxidation. Townend[247] mentions candoluminescence as a test for hydrogen atoms in flames. Gaydon[247a] discusses the spectra of chilled hydrocarbon flames.

(f) Thermal Decomposition.

Küchler[248] has found the thermal decomposition of cyclohexene to be a homogeneous first order reaction. Mayor[249] has reviewed the free radical theory of hydrocarbon decomposition. Rice and Polly[250] have explained the inhibiting effect of nitric oxide in terms of chain breaking. Burk[173] has proposed an alternative to the Rice theory.

F. Oxidation of Aldehydes.

Although aldehydes occur as intermediates in the oxidation of hydrocarbons and are themselves important as fuel constituents, few studies of their oxidation have been attempted. The theory of their oxidation has not progressed beyond the stage at which it was discussed by Jost.[74]

(1) Formaldehyde.

(a) Mechanism and Kinetics.

Snowden and Style[251] and Spence[252] postulated the intermediates CH_2O_2 and CH_2O_3 to explain their observations of the kinetics of formaldehyde oxidation. Bone and Gardner[106] measured the oxidation rate and found no evidence of an induction period.

(b) Thermal Decomposition.

The thermal decomposition has been studied by Patat and Sachsse[253] using para hydrogen as a test for free radicals. Rollefson and Faull[254] investigated the effect of iodine.

(c) Photochemistry.

Photochemical investigations of formaldehyde decomposition have been made by Gorin[255]. The photooxidation has been studied by Carruthers and Norrish[256] who obtained about 50% conversion of formaldehyde to formic acid.

(2) Acetaldehyde.

More extensive kinetic studies have been made of acetaldehyde than of any other oxygen containing hydrocarbon. Unfortunately, most of these studies have dealt with the photo or thermo decomposition and, consequently, give only indications of the manner in which the oxidation proceeds. There is some evidence to support the view that the reaction chain involves the two radicals: CH_3CO- and $CH_3CO-OO-$ (Bodenstein[257]). Aivazov[258] in a careful study of the oxidation and the effect of peracetic acid on the kinetics, (peroxides were determined by analysis and polarographic means), concluded that his data were in accord with the peroxide theory of oxidation.

(a) Ignition and Induction Period.

Aivazov[259] has studied the cold and hot flames and determined the temperature dependence of the induction periods. Baron[260] studied the effect of pressure on ignition temperature. Keyer and Neimann[261] investigated the variation of induction period with composition.

(b) Mechanism and Experimental Kinetics.

Newitt[262,263] in an extensive study of the oxidation determined the products formed at various degrees of reaction as well as the dependence of ignition on temperature and pressure. In the analysis two types of peroxides were distinguished.

(c) Studies of Mixtures and Additives.

Kane[170] showed that nitrogen peroxide catalyzed the ignition of acetaldehyde.

(d) Thermal Decomposition.

The thermal decomposition of acetaldehyde has been studied by Brenschede and Schumacher[264], Burton[265,266], Fletcher[267], Leifer and Urey[268], who analyzed the products with a mass spectrograph, Letort[269,270], Patat and Sachsse[271] and Rollefson[254].

(e) Photolysis.

Grahame[272], Gorin[273,274], Mitchell and Hinshelwood[275], Leighton[276], who reviews (1938) the status of the knowledge of aldehyde and ketone photolysis. Carruthers and Norrish[256] studied the photo-oxidation and showed that peroxides and biacetyl are formed.

(3) Propaldehyde.

The only extensive investigation of propaldehyde combustion is that of Newitt[262,263], who showed that the boundary values for the upper slow combustion is given by the Semenov equation. Newitt and Townend[277] have reviewed the combustion phenomena of the higher aldehydes.

(a) Thermal Decomposition.

The thermal decomposition has been studied by Patat and Sachsse[271] and Rollefson and Faull[254].

(b) Photolysis.

May, Taylor and Burton[278] have used the Paneth method for detection of free radicals from photolysis of propaldehyde. Mitchell and Hinshelwood[275] have studied the NO inhibition and Leighton[276] (1938) has reviewed the photochemical studies of aldehydes and ketones.

(4) Butaldehyde and Higher Aldehydes.

Newitt and Townend[277] have reviewed the processes taking place during the autoignition. Briner and co-workers[279-283] have investigated the oxidation of butaldehyde and benzaldehyde by mixtures of oxygen and ozone. Chain lengths and inhibitor effects have also been studied. Payne and Lemon[284] have studied the mechanism of aldehyde oxidation by H_2O_2. Leighton[285] has investigated the photolysis of butaldehyde and isobutaldehyde.

G. Oxidation of Ketones.

Fahlbusch[217a] has reviewed work prior to 1942 on the oxidation, and thermal and photo decomposition of ketones. No further work has been done on the direct oxidation.

(1) Photo-oxidation.

Rice and Schildknecht[286] showed the products from the photo-oxidation of acetone to be principally acetic acid and formaldehyde. Small amounts of peroxides were formed. Fugassi[287] found that the photo-oxidation of acetone produced a blue fluorescence, which changed to green when the oxygen was all consumed. This provided a test for traces of oxygen.

(2) Thermal Decomposition.

Gantz and Walters[288], Gantz[289], Smith and Hinshelwood[290], Davoud and Hinshelwood[291], Steacie and Alexander[292] have studied the thermal decomposition of acetone under various conditions.

(3) Photolysis.

The photolysis of acetone has been investigated by Feldman, Burton, Ricci and Davis[293], Gorin[255,274], Spence[294] and Glazebrook and Pearson[295]. Gorin[255,274] has studied also methyl ethyl ketone and Glazebrook and Pearson[295,296] have determined the relative amounts of radicals formed from MeEtCO, MePrCO, Me(iso-Pr)CO, MeBuCO, Pr_2CO, $(iso\text{-}Pr)_2CO$, $(tert\text{-}Bu)_2CO$ and $(sec\text{-}Bu)_2CO$.

H. Combustion of Formic Acid.

Bone and Gardner[106] showed that only CO, CO_2, H_2 and H_2O are formed in appreciable amounts from the combustion of formic acid. Patat and Sachsse[253] studied the thermal decomposition in the presence of para hydrogen and concluded that no radicals were present in the decomposing gas.

I. Oxidation of Ethers.

(1) Methyl Ether.

Ermolova and Neiman[297] have studied the reaction kinetics and induction periods for dimethyl ether and found good agreement with the predictions of the Semenov theory. Addition of diethyl peroxide decreases the induction period. Pannetier and Laffitte[298, 299] determined the inflammability limits of mixtures of methyl ether with air and oxygen using a spark. The effects of surface and nitrogen addition were also investigated.

(a) Thermal Decomposition.

The thermal decomposition has been studied by Thompson and Meissner[300], Stavely[301], and Leifer and Urey[268].

(2) Diethyl Ether.

Ignition phenomena and the kinetics of propagation of cool and hot flames have been investigated by Baron[260], Ermakova[302], Gibyanskii[303], and Townend and co-workers[304-306]. Kane and Pandit[307] by study of the sensitizing action of nitrogen dioxide on low temperature ignition found evidence that acetaldehyde is primarily involved in the formation of cool flames.

(a) Spectroscopic Observations.

Kondrat'ev[308] has pointed out that bands found by Emeleus in diethyl ether flames (C.A. <u>27</u>, 2381) appear in the fluorescence spectrum of formaldehyde. This is evidence that the cold flame of diethyl ether contains excited formaldehyde molecules. Hsieh and Townend[309] have studied the green flames produced in ether oxidation and showed that they may be ascribed to C_2.

(b) Thermal Decomposition.

The thermal decomposition of diethyl ether has been studied by Patat[310], Hobbs[311] and Fletcher[312].

(c) Electrolytic Decomposition.

Pearson[313] has studied reactions of the radicals produced by electrolysis of ether in solution.

(3) Diisopropyl Ether.

Belov[193a] studied the ignition phenomena of diisopropyl ether and obtained an integral equation expressing the dependence of the induction period on the pressure and temperature.

(4) Dioxane.

Küchler and Lambert[314] have studied the thermal decomposition of dioxane in the temperature range 723 - 808° K. at pressures from 30 to 80mm.

J. Oxidation of Alcohols.

The combustion of methyl alcohol has been investigated by Bone and Gardner[106] who showed that it was more resistant to oxidation than formaldehyde. Jorissen, Van Selms and Kreulen[315] showed that oxidation of methyl and ethyl alcohols occurs with simultaneous decomposition. Geib[316] studied the flame of CH_3OD and showed that OD bands were not emitted.

K. Peroxide Formation and Decomposition.

Evidence has accumulated during the past two decades which indicates that peroxides play an important part in the oxidation of almost all organic compounds. (See, for example, Von Elbe's scheme for propane oxidation page 18). Since it is also known that stable peroxides act as prodetonants in internal combustion engines, a considerable stimulus has been given to the study of their mode of formation and decomposition and to the development of kinetic theories in which they play an important part. It is likely that the phenomena of the induction period and of cool and hot flames can be explained in terms of an initial accumulation of peroxides followed by branching chain reactions when the peroxide concentration reaches a critical value. At high temperatures, intermediate formation of peroxides may not occur.

(1) Studies and Theories of Peroxide Formation.

Bakh[317], who was the first to propose the peroxide theory of oxidation, has reviewed its status as of 1941. Medvedev[318,319] has shown that some features of hydrocarbon oxidation can be explained in terms of the superposition of two chain reactions, one initiated by thermal activation of the hydrocarbon molecules, the second by the decomposition of the intermediate peroxides. Substantiation of the theory was found in experiments with tetralin in which the peroxide intermediates could be isolated and studied separately. Neiman[182] has discussed the role of peroxides in the formation of hydrocarbon flames and Dumanois[320], Oosterhoff[321] and Sokolik[322] have proposed explanations of the behavior of internal combustion engines based on peroxide formation in the compressed fuel air mixture.

(2) Peroxide Decomposition.

Harris and Egerton[323-327] have studied the decomposition of ethyl hydroperoxide, propyl hydroperoxide and diethyl peroxide. Neiman and Tutakin[328] have investigated the kinetics of diethyl peroxide decomposition; Ivanov, Savinova and Mikhailova[329] investigated the decomposition of tetralin, its hydroperoxide, the hydroxymethyl derivative and hydroxymethyl derivative of tetrahydronaphthyl peroxide. Walker and Wild[330] studied the thermal and photochemical decomposition of acetyl peroxide.

L. Oxidation and Decomposition of Miscellaneous Compounds.

The work in this section is too special and heterogeneous to make a detailed summary worthwhile. Pertinent references are therefore listed without comment.

(a) Ammonia.

Bodenstein[331], Tokarev and Nekrasov[332], Verbrugh[333], Atroschenko and Sedasheva[334], and Frejacques[335].

(b) Butyl Amine.

Beachell and Taylor[336].

(c) Carbon Disulfide and Carbonyl Sulfide.

Griffith and Hill[337], Kondrat'ev and co-workers[338-342], (extensive spectroscopic studies of intermediates in CS_2 flames), Nalbandjan[343], Pavlov[344,345], Semenov and Voronkov[346,347].

(d) Chlorine - Hydrogen.

Bodenstein[348] and Bodenstein and Launer[349].

(e) Chlorine Monoxide Decomposition.

Bodenstein and Szabo[350].

(f) Diacetyl Reactions.

Rice and Walters[351].

(g) Diethyl Acetal (Inflammation)

Dugleux and Laffitte[352].

(h) Ethylene Oxide (Free Radicals from)

Fletcher and Rollefson[353].

(i) Hydrazine

Bamford[354], Bamford and Bamford[355].

(j) Methyl Amine.

Travers and Hawkes[356], Emeleus and Jolley[357].

(k) Methyl Iodide.

Blake and Iredale[358], and Vaidya[359] (both studies of flame spectra).

(l) Methyl Isocyanate.

Bamford and Bamford[360].

(m) Nitrogen Oxides.

Lemon[361], Lowry[362], Stoddart[363], Murzin[364], Lewis and Hinshelwood[365], Furman[366,367], Briner[368].

(n) Nitrates and Nitrites.

Appin, Todes and Khariton[369], Carter and Travers[370], Taylor and Vesselovsky[371], Thompson and Dainton[372], Steacie and Kata[373], Steacie and Shaw[374], Purkis[375].

(o) Nitrosyl Chloride.

Natanson[376], Krauss and Saracini[377].

(p) Silanes.

Shantarovich[378], Emeleus and Welch[379].

(q) Sulfur Compounds.

Yakovlev and Shantarovich[380], Schenk and Jablonowski[381], Emanuel[382,383,384,385], Emanuel, Pavlov, Semenov[386], Kondrat'eva and Kondrat'ev[387,388], Brodovich[389].

(r) Sodium and Organic Halides.

Allen and Bawn[390].

M. Theory of Chemical Kinetics.

In this section a short survey is made of the more important developments in the kinetic theory of the processes occurring in chemical transformations.

Akulov[391-394] has extended the theory of branching chains and studied the application of the theory to the determination of induction periods, etc. He has also developed equations relating the effect of a negative catalyst (e.g. surface) to the volume of the reaction space. Dijksman[395,396] has investigated the explosion limits for spark ignition of mixtures which pass continuously into the limits for high temperature inflammation. Evans[397] derives Kassel's formula for the rate of an unmolecular reaction from transition state theory. Von Elbe[398] derives an expression for the steady state rate of a chain reaction in which there are varying efficiencies of chain breaking at the walls. Frank-Kamenestskii[399-405] has published several important papers on the theory of thermal reactions and has discussed the mechanism of two-stage inflammation and periodic processes in oxidation reactions. Gurney[406] derives a theory for reaction chains where intermediate particles react with members of preliminary and following steps. Geib[407] has studied the relation of molecular structure to reaction mechanism. Hulburt[408] derives equations for reaction rates in reacting flowing systems. Jost[409] and Landau[410] discuss the theory of thermal explosions. Meixner[411] has discussed the "thermodynamics" of irreversible processes. Prettre[412] discusses wall effects in chain reactions. O. K. Rice[413] has applied the Frank-Kamenetskii theory to explosions of azomethane, ethyl azide and methyl nitrate. The evaluation of specific rate constants in chain reactions is the subject of a review article by Rollefson[414]. Szczeniowski[415] has derived a generalized combustion equation taking into account dissociation at high temperatures. Stupochenko[416] discusses the possibility of accumulation of molecules of a certain type around an active center and its effect on the kinetics of reaction. Semenov[417-419], in an early paper (1936) gives a review of his theory. Two later papers discuss methods of determining concentration of intermediates and the effects of diffusion of chain centers to the vessel wall. Todes and co-workers[420-424] have presented several papers on the theory of thermal explosions.

The theory and mechanism of combustion in internal combustion engines has been the subject of articles by Blume[425], Broersma[426], Boyd[427,428], Boerlage and Broeze[429,430], Dreyhaupt[431], Evans[432], Isogai[433], Jost[434], Jost and Muffling[435], Kling[436], Kneule[437], Meurer[438], Mock[439], Newitt[440], E. Schmidt and Muhlner[441], F.A.F. Schmidt[442,443], Serruys[444,445], Zeise[446,447] and Zinner[448].

Studies of various experimental aspects of internal combustion have been made by Ernst[449], Funck[450], Herele[451], Laure[452], Lichtenberger and Seeber[453], Lonn[454], Rassweiler, Withrow and Cornelius[455], Rothrock , Spencer and Miller,[456] Scheuermeyer and Steigerwald[457], Shoemaker and Gadebusch[458], Sokolik and Yantovskii[459], Spencer[460], Starkman[461], Wolfer[462], Watts and Lloyd-Evans[463] and Widmaier[464].

The role of free radicals in chemical reactions has been examined by Aten[465], Groth[466], Mandala[467], Pearson and Purcell[468], Rice and Teller[469], Rice, Walters and Ruoff[470], Staveley and Hinshelwood[471], Smith[472], Ubbelohde[473] and Waters[474].

General reviews of combustion kinetics and phenomena have been published by Eucken[475], Fiock[476,477], Jost[478], Lewis[479], Prettre[480], Stevels[481,482], Steacie[483], Sokolik[484], Sachsse[485], Thiemann[486] and Wittig[487]. Berger[488] lists data on physical properties and oxidation phenomena for over 400 compounds. Vaidya[489] has reviewed the evidence for various intermediates which has been obtained from spectroscopic studies of the processes in internal combustion engines. Kondrat'ev[490,491] and Czerlinsky and Seibt[492] have have published similar reviews.

N. Studies of Physical Aspects of Combustion.

A large number of investigations have been made of phenomena, such as detonation rates, which are intimately related to combustion mechanism, but for which the physical and thermodynamic properties or state of the reactants are of more direct importance than the detailed chemical mechanism in their quantitative treatment. References to these reports are listed below under appropriate headings.

(1) Studies of Physical Aspects of Ignition.

Guest[493], Landau[494], Linnett, Raynor and Frost[495], Jorissen et al[496], Mole[497], Paterson[498,499], Schade[500], Semenov[501], Silver[502], Vaillard[503,504], Zel'dovich[505].

(2) Flame Propagation.

Coward and Payman[506], Coward, Hartwell and Georgeson[507], Dicent and Shchelkin[508], Denues[509], Damkohler[510,554], Fiock and co-workers[511,512,513], Jost and Muffling[514], Khitrin[515], Kolesnikova[516], Kolodtsev[517], Lewis and Von Elbe[518,519], Mache[520], Manson[521], Malinovskii[522], Smith[523], Townend and Maccormac[524], Van de Poll[525], Zel'dovich and Semenov[526].

(3) Detonation.

Becker[527], Broersma[528], Jost[529], Jost and Muffling[530], Laffitte[531], Manson[532], Pfriem[533], Reingold[534], Shtsholkin[535], Tokarev and Nekrasov[536], Thomas[537], Veron[538,539], Zel'dovich[540,541,542], Zouckermann and Heyberger[543].

(4) Theory and Properties of Burner Flames.

Behrens[544], Garside, Forsyth and Townend[545], Khitrin[546], Lewis and Von Elbe[547], Von Elbe and Mentser[548], Vasilesco[549], Wolfhard[550].

(5) Diffusion Effects in Shock Waves.

Cowling[551].

(6) Turbulence and Mixing.

Dreyhaupt[552,553], Damkohler[554], Shvab[555], Shchelkin[556].

(7) Latent Energy in Flames.

David and co-workers[557-570], Leah[571], Lewis and Von Elbe[572], Zeise[573].

(8) Flame Temperature Measurements.

(a) Discussions of Errors and Reviews.

Bosanquet[574], Eggert[575], Von Elbe[576], Fishenden and Saunders[577], Hase[578], Matthews[579], Meissner[580], Pirani[581], Ribaud and co-workers[582,583], Richardson (1945)[584], Schroder[585], Swallow[586] and Tewes[587].

(b) Flame temperatures from the Emission Spectrum.

Brinkman[588], Coheur and Coheur[589], Graff[590], Knauss[591].

(c) Measurement by Thermometry.

Kobayashi[592], Larsen et al[593], Schack[594], Severinghaus[595], Swart[596], Thring[597], Zhukovskii[598].

(d) Temperatures by the Na-line Reversal Method.

David[599], Van de Poll et al[600], Rosenthal[601], Yosida[602].

(e) Two Color Pyrometry.

Graff[603], Naeser[604], Uyehara et al[605].

(f) By pressure rise in closed bomb.

Leah[606,607].

O. Thermodynamic Studies.

(1) Experimental Measurements.

Mathews and Hurd[608] have determined the thermodynamic properties of methane. Rossini and Knowlton[609,610] give data on the heats of combustion of the normal alkenes-1. Wartenberg[611] determined the equilibrium constant for acetylene-hydrogen and graphite at 1750°C. Coops et al[612] have measured the heats of combustion of a number of olefinic and phenol substituted methanes and ethanes. Jessup[613] has determined the heats of combustion of the liquid paraffin hydrocarbons from hexane to decane. Pitzer[614] discusses methods of calculating free energies and heat contents of gaseous hydrocarbons and gives tables of values for the paraffins through heptane and for the simpler unsaturated hydrocarbons; Zeise[615,617] presents data on the thermodynamic functions of water, CO_2 and of some additional molecules and radicals important in combustion. Burke and Hunt[618] have determined the heat of combustion of acetone.

(2) Calculation of Thermodynamic Quantities.

Benedict[619] has derived empirical equations for the thermodynamic properties of light hydrocarbons and their mixtures. Hottel and Eberhardt[620] show how the use of Mollier diagrams simplifies combustion calculations. Pitzer[621] gives tables useful for the calculation of thermodynamic functions of molecules having restricted internal rotations. Rossin and Fehling[622] discuss the use of I-T diagrams in combustion calculations. Von Stein[623] discusses methods of calculating dissociation equilibria. Walker[624,625] derives expressions for the temperature, pressure and specific volume changes of a dissociating gas. Wiebe[626] gives Mollier diagrams calculated according to Hottel and Eberhardt for octane-air and octane-water-air mixtures. Withrow and Cornelius[627] discuss the use of thermodynamic charts in the analysis of flame picture and pressure data. Zeise[628] discusses the thermodynamic calculation of combustion temperatures and reactions in gas mixtures with precise consideration of all possible cracking phenomena.

P. Rocket Propulsion Chemicals.

Only a few unclassified reports on this subject have significance. Kuhn et al[629] summarize the chemical combinations used for jet propulsion in Germany. Monofuels included 65-85% methyl nitrate in methanol, ammonium nitrate plus ammonia, nitrous oxide plus ammonia and tetranitromethane plus 8-hydroxyquinoline in 80 to 85% H_2O_2. Bifuels were made up of an oxidizing agent and a fuel. Oxidizing agents considered were: 98 - 100% nitric acid, 98-100% nitric acid plus 5 - 10% strong sulfuric acid, liquid oxygen, 80-85% hydrogen peroxide with or without calcium permanganate, and ammonium nitrate. The fuels considered were: Methanol, ethanol, hydrazine hydrate, hydrazine hydrate plus methanol, 57% m-xylidine plus 47% triethylamine, 8-hydroxyquinoline, furfuryl alcohol, vinyl ethyl ether, gasoline, diesel oil, Optol (a hydrogenated lignite tar fraction, Ergol (a mixture of Optol, tetrahydrofurane, furfuryl alcohol and aniline). Bellinger et al[630] report on propellant systems consisting of (1) concentrated hydrogen peroxide plus permanganate, (2) red fuming nitric acid and aniline, and (3) mononitromethane plus catalyst. Lawrence[631] has discussed engineering aspects of the operation of an ethyl alcohol - oxygen propulsion unit.

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AUTHOR(S)

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* Combustion

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