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(14)

REPORT NO. 23

(6) SELF-IGNITING FUELS,

EXPLORATORY WORK WITH
HYDROGEN PEROXIDE AS AN OXIDANT,

Prepared for the
Office of Naval Research
Contract No. N5ORI-78
T. O. XIX

(15) N5ori-78(11)

(11) 15 Oct 47

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(12) 34 p.

(16) DIC-6552

AD-896017

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[redacted]

DECLASSIFIED
DOD DIR 5200.9

D. I. C. 6552

(407 145) October 15, 1947

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A portion of the work described in this report was carried out under a Navy Bureau of Ordnance Contract, NOrd 9107 Task C, which expired June 30, 1947. In the interest of utility, the study is presented as a whole rather than in parts, disconnected by reason of change of sponsor.

[redacted]

ACKNOWLEDGMENT

The collaboration of John E. Leffler and Bradford Darling in a portion of the experimental work is gratefully acknowledged.

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I. INTRODUCTION

The German program in self-igniting fuels for use with hydrogen peroxide resulted in the application of several fuel-compositions operationally, as well as the study of other fuel mixtures, some quite complicated, which were in various stages of development at the end of the war. There have been no detailed descriptions available of how these combinations were arrived at, or of other combinations which could be used. The work in this country naturally started on the basis of the reported German fuels and for some time proceeded with no attempt being made to very the composition of fuel or catalyst.

It has been considered desirable to place the knowledge of fuels which spontaneously ignite with hydrogen peroxide on a sounder basis by investigating a wide range of possible fuels and studying their reactions with hydrogen peroxide. The present report is a summary of an empirical program whose object was to determine, on as broad a base as possible, what compounds merited further consideration as possible fuel components. As such, the work may have been, at least in part, a repetition of German investigations, the records of which have been lost or destroyed. This view is supported by statements in the German literature, and by the fact that the results make the German choice of fuels for development seen reasonable.

The program of experimental work has consisted of applying a simple ignitability test, first to a wide variety of different types of compounds, and then to a bariety of compounds or combinations taken from chosen groups.

The work is reported in two parts. The first is an account of tests for ignitability on a variety of systems without regard to mutual solubility or physical condition. The second part is concerned with attempts to form homogeneous solutions while preserving the ignitability.

In evaluating the results reported it is emphasized that these tests are exploratory. A positive result indicates the desirability of further work on the combination, but is not to be taken as an unqualified recommendation. Much further work is required on such characteristics as storage stability, and ignitability under rocket or power plant conditions before the fuel can be recommended for operational use.

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II. SUMMARY

Results are reported on a large number of exploratory tests made to investigate the suitability of various compounds for use as self-igniting fuels with hydrogen peroxide. The technique involved the mixing of drop quantities of the prospective fuel with 90 per cent hydrogen peroxide. Ignition was observed visually. The first part of the report is concerned with attempts to find ignitable compounds and mixtures. The second part concerns attempts to form homogeneous fuels from compounds and mixtures found to be active.

Part I.

Tests were first made on one hundred and three single compounds. Eight of these ignited. They were hydrazine hydrate, two complex cobalt compounds (salcomine and fluomine), iron β-naphthalene sulfonate, and four organic thioamides.

Combinations of a single fuel with an inorganic catalyst were then studied. In most cases the catalyst was present in the solid phase. One hundred and ten fuel compounds were tests with each of six catalysts containing Cu^{+1}, Cu^{+2}, Ni^{+2}, Co^{+2}, Fe^{+2}, and Fe^{+3}, respectively. Fifty of the compounds showed ignition with one or more of the catalysts. The activity of amines and aldehydes was pronounced. Of thirty-two amines tested, twenty-three ignited; and of twenty-seven aldehydes, twenty ignited. Tests using cobalt catalyst dissolved in the hydrogen peroxide agreed in general with the above series.

In a further series of studies, three vanadium compounds were used as cacalysts for tests with fifty-one fuel compounds. Ignition was observed in eighteen cases. All the aldehydes ignited with one or more of the catalysts, and several amines ignited.

Various solid complex compounds have been prepared from cobaltous chloride and organic bases. Twenty-two of these were tested for ignitability with hydrogen peroxide. Ignition was observed in thirteen cases.

Part II.

In general the procedure used in attempting to form homogeneous solutions which were to be ignitable was to try to dissolve the ignitable compounds either in one another or in a mutual solvent.

Attempts were made to dissolve solid inorganic catalysts in the single fuels with which they caused ignition. In eight cases, ignitable solutions were obtained.

The single solid compounds, salcomine and thio-acetamide, had been found to ignite with hydrogen peroxide. Tests were made to determine the feasibility of dissolving these solid compounds in solvents to form ignitable solutions. The attempts with salcomine were unsuccessful.

[redacted]

Thio-acetamide was tried in combination with thirty-one solvents, and ignitable solutions were obtained only with annisaldehyde and cinnamaldehyde.

Ferrous beta-naphthalene sulphonate has been found to give consistent ignition when dissolved in methyl alcohol. If pyrocatechol is added to this solution the fuel approximates the German "Ergol 56", and has good ignition characteristics.

Attempts to dissolve a self-igniting O-toluidine-cobalt complex in various solvents to produce ignitable mixtures were in general unsuccessful. When dissolved in hydrazine hydrate the complex did improve the consistency of ignition.

The ignition characteristics of fuels based on hydrazine hydrate have been studied in considerable detail and are the subject of a separate report,(1).

Attempts were made to produce self-igniting fuels from common fuels or solvents by adding a self-ignitable fuel and catalyst. Tests on benzene, toluene, kerosene, and gasoline have been in general unsatisfactory because of catalyst insolubility. However, ignitable solutions have been made from o-toluidine-acetone-acid cuprous chloride, and from p-toluidine-ethyl alchol-cobaltous nitrate.

III. EXPERIMENTAL WORK

PART I.

The primary attack on the problem of developing self-igniting fuels for use with hydrogen peroxide has been on the broadest possible basis. Exploratory tests have been made on widely varying types of compounds to determine qualitatively, what types of compounds have potentially good ignition characteristics.

The exploratory tests were very simple. The liquid or solid compound or mixture to be tested was mixed 90 per cent Becco hydrogen peroxide in drop quantities on a stainless steel plate and the results observed. At first, compounds were picked somewhat at random with the object of covering a great deal of ground.

A. Single Compounds

The results of preliminary tests on single components are presented below under four main headings.

Table I

Drop Test Results with 90% Hydrogen Peroxide and Single Compounds

- -

No Immediate Decomposition

acetal
acetic acid
acetone
acetone oxime
acetyl acetone
o-aminophenol hydrochloride
benzaldehyde semicarbazone
benzyl chloride
benzyl peroxide
cinnamaldehyde
cyclohexane
cysteine hydrochloride
decahydronaphthalene (decalin)
dibutylamine
dimethylgloxime
dimethyl mercury
ethyl acetoacetate
formic acid
furfural
furfuryl alcohol
glycine
p-hydroxyphenyl-glycine
isopropyl ether
manganese tartrate
mercuric bromide
mercuric iodide
mercury succinimide
methionine
methyl mercuric chloride
methyl mercuric hydroxide
methyl mercuric iodide
p-nitroaniline
nitrobenzene
nitroguanidine
nitromethane
2-nitropropane
p-nitrosodiphenyl amine
oxalic acid
paraldehyde
paraformaldehyde
phenol
phosphorous
picric acid
silver benzoate
silver citrate
sodium isoamylate
tetramethyl ammonium bromide
tetranitromethane
o-thiocresol
m-thiocresol
thio-β-naphthol
o-toluidine
2,4,6-trichlorophenol
trinitrotoluene
triphenyl guanidine
urea
m-xylene
zinc chloride

[illegible]

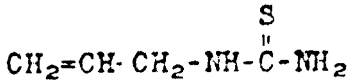
- ammonium vanadate
- aniline
- benzotriazole
- benzylamine
- catechol
- chromic nitrate
- diacetylamino-
  azotoluene
- ethanol diethylamine
- ethyl nitrate
- hexamethylene
  tetramine
- manganous citrate
- manganous dipyridyl vanadate
- manganous oleate
- [illegible]aniline
- molybdic oxide
- nickelous acetate
- o-phenylenediamine
- phenyl hydrazine hydro-
  chloride
- pyridine
- sodium nitroprusside
- sodium p-nitrosophenolate
- thiophenol
- m-toluidine

Vigorous Decomposition

- amyl nitrite
- cobaltous carbonate
- cobaltous formate
- cobaltous oxalate
- ethanol amine
- ethylene diamine
- ethylene diamine cupric
  chloride
- ethylene diammonium
  chlorocuprate
- manganese benzoate
- manganese dioxide
- manganese linoleate
- silver carbonate
- sodium amalgam
- triethylene tetramine
- vanadium pentoxide

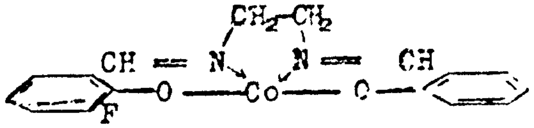
Ignition

allyl thiourea - - - - - - - -



$CH_2{=}CH{-}CH_2{-}NH{-}\overset{\overset{S}{\parallel}}{C}{-}NH_2$

Fluomine - - - - - - - - - - -



hydrazine hydrate - - - - - - - $H_2N{-}NH_2 \cdot H_2O$

ferrous β-naphthalene sulfonate

Salcomine - - - - - - - - - - -

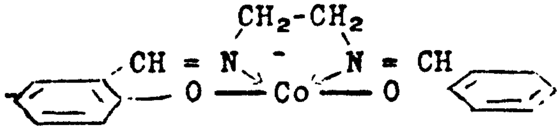


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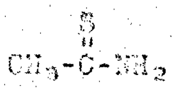
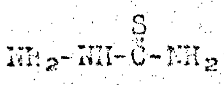
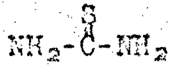
Table I. (Cont'd)

Ignition (Cont'd)

thioacetamide - - - - - - - - - -

$CH_3-\overset{\overset{S}{\|}}{C}-NH_2$

thiosemicarbazide - - - - - - - -

$NH_2-NH-\overset{S}{C}-NH_2$

thiourea - - - - - - - - - - - - - -

$NH_2-\overset{S}{C}-NH_2$

Discussion

Consideration of the compounds causing vigorous decomposition shows first that nine of the fifteen are metallic salts which could best be described as hydrogen peroxide decomposition catalysts. Of the other six, three are amines and two are amine complexes with an inorganic component. The remaining compound, amyl nitrite, is a rather unstable and easily oxidizable compound.

In eight cases ignition was observed. Hydrazine hydrate has been well known for its reactivity with hydrogen peroxide and had been used as the base for several German self-igniting fuels. For consistent ignition in such use it is necessary to add a catalyst, but there is frequent ignition in drop tests even without catalyst. Fluomine and salcomine are complex compounds containing groups similar to amines, which are linked to cobalt. Iron salts of naphthalene sulfonic acids were reported by the Germans to be good fuel catalysts. We found that iron β—naphthalene sulfonate itself was ignitable. The remaining four compounds all contain the thioamide group.

B. Use of Catalysts

There were several indications of the desirability of using a metallic compound in fuels, which would act as a reaction catalyst. Among these was the German use of catalyst with hydrazine fuels and the reactivity of the complex compounds described above. Some catalyst contribution might be expected as a result of hydrogen peroxide decomposition, but it will be shown below that there are other effects to consider as well.

It had been reported that concentrated hydrogen peroxide was decomposed by cobaltous nitrate in an alkaline solution (2). This was confirmed by adding a drop of dilute sodium hydroxide to a drop of peroxide containing cobaltous nitrate. The reaction was violent. This compound would seem to be a reasonable additive to organic bases.

Copper and iron had been used as catalysts in hydrazine based fuels. Tests were planned using salts of these metals as catalysts and also including nickel because of its proximity to the other catalysts in the periodic table. The procedure involved placing a small quantity of the solid catalyst on a stainless steel plate, adding a drop of fuel and then a drop of hydrogen peroxide. Results are shown in Tables II, III and IV.

Table II.

Drop Tests with Various Compounds and 90 % Hydrogen Peroxide -- Catalysts: Cu^{+1}, Cu^{+2}, Ni^{+2}, Co^{+2}, Fe^{+2}, and Fe^{+3} (x indicates ignition)

No Ignition

acetophenone
acetyl o-anisidine
azobenzene
azoxybenzene
m-benzene disulfonic acid
benzoyl chloride
o-bromoanisole
bromobenzene
cyclohexene
n-decane
3,5-dinitrobenzoic acid
1,4-dioxane
ethanol amine
ethyl bromide
ethylene bromide
ethylene glycol
ethyl fumarate
ethylidene chloride
ethyl nitrate
formamide
n-heptane
n-hexane
isoamyl ether
isopropyl alcohol
lactic acid
levulinic acid
β-methyl naphthol
2-methyl-2,4-pentandiol
methyl n-propyl ketone
o-nitroanisole
nitrobarbituric acid
m-nitrobenzaldehyde
nitrobenzene
o-nitro ethyl benzene
nitroguanidine
p-nitroiodobenzene
nitromethane
3-nitrophthalic acid
propylene glycol
quinone
n-valeronitrile

Ignition with Certain Catalysts

Fuel	Catalyst					
	Cu^{+1}	Cu^{+2}	Ni^{+2}	Co^{+2}	Fe^{+2}	Fe^{+3}
2-amino-5-azoanisole				x	x	
p-aminoazobenzene				x	x	
2-aminothiazole	x	x			x	x
benzonitrite	x					
o-bromobenzoic acid	x				x	

Table II. (Cont'd)

Ignition with Certain Catalysts (Cont'd)

	Catalyst					
Fuel	Cu^{+1}	Cu^{+2}	Ni^{+2}	Co^{+2}	Fe^{+2}	Fe^{+3}
diethylene glycol					x	
diethylene glycol diethyl ether					x	
2,5-dimethyl furan					x	x
furfuryl alcohol	x				x	
isoamyl nitrite					x	x
o-methoxybenzoic acid	x				x	
nicotine	x	x			x	x
p-nitrobenzoic acid					x	
nitron	x			x	x	
p-nitrophenol					x	
α-nitroso-β-napthol					x	
1-phenyl-3-methyl-5-pyrazolone					x	
thiodiglycol	x				x	x
thio-β-naphthol					x	

Table III.

Drop Tests with 90% Hydrogen Peroxide and Organic Bases of Varying Base Strength. (x indicates ignition)

			Catalyst					
Fuel	Kb	None	Cu^{+1}	Cu^{+2}	Ni^{+2}	Co^{+2}	Fe^{+2}	Fe^{+3}
diethylamine	10^{-3}							
piperidine			x	x				
urea (solid)	10^{-4}							
di-isobutylamine							x	x

[illegible]

Drop Tests with [illegible] Peroxide and Organic Bases of Varying Ba[illegible]. (x indicates ignition)

		Catalyst						
Fuel	Kb	[illegible]	[illegible]	[illegible]	[illegible]	Co^{+2}	[illegible]	[illegible]
triethylamine								
benzylamine	10[illegible]		x	x			x	x
ethylene diamine								
hydrazine hydrate (85%)	10^{-6}	x	x	x	x	x	x	x
diethyl m-toluidine	10^{-8}						x	
quinoline	10^{-9}						x	x
phenyl hydrazine			x	x	x	x	x	x
p-toluidine (solid)			x			x	x	x
pyridine								
β-naphthylamine (solid)	10^{-10}		x			x	x	x
o-phenylene diamine			x	x	x	x	x	x
m-toluidine			x	x		x	x	x
anthranilic acid (solid)	10^{-12}		x			x	x	
methyl red (solid)			x			x	x	
acetone oxime	10^{-13}		x				x	
acetanilide (solid)	10^{-14}		x				x	
hexamethylenetetramine (solid)	10^{-14}		x		x		x	
thiourea	10^{-15}		x	x	x	x	x	x
acetamide (solid)								

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Table IV.

Drop Tests on Several Aldehydes with 90% Hydrogen Peroxide. (x, indicates ignition)

	Catalyst					
Aldehyde	Cu^{+1}	Cu^{+2}	Ni^{+2}	Co^{+2}	Fe^{+2}	Fe^{+3}
anisaldehyde	x	x			x	x
benzaldehyde	x	x			x	
butyraldehyde					x	
o-chlorobenzaldehyde		x			x	
cinnamaldehyde	x	x			x	
crotonaldehyde	x	x	x	x	x	x
3,4-diethoxybenzaldehyde	x	x			x	
2,4-dihydroxybenzaldehyde	x	x			x	x
o-ethoxybenzaldehyde	x	x			x	
3-ethoxy-4-hydroxybenzaldehyde	x	x	x		x	x
2-ethylbutyraldehyde						
2-ethylhexaldehyde						
furfural	x	x			x	x
heptaldehyde						
hydrocinnamaldehyde						
m-hydroxybenzaldehyde	x	x			x	
p-hydroxybenzaldehyde	x	x			x	x
isobutyraldehyde		x			x	
p-isobutyraldehyde						
isovaleraldehyde						
o-methoxybenzaldehyde	x	x			x	
m-nitrobenzaldehyde	x	x			x	
p-nitrobenzaldehyde	x	x			x	
paraldehyde					x	

Table IV. (Cont'd.)

Aldehyde	Catalyst Cu^{+1}	Cu^{+2}	[illegible]	Co^{+2}	Fe^{+2}	Fe^{+3}
salicylaldehyde	x	x			x	x
m tolualdehyde	x	x			x	
Valeraldehyde						

Discussion of Tables II, III, & IV.

The tests showed in some cases that activity could be ascribed to the presence of certain functional groups. In the case of organic bases and aldehydes, the ignitability of the compounds initially tested was sufficiently consistent with the presence of the functional group so that a larger number of these compounds was tested and reported separately in Tables III and IV.

The ignitability of the bases is apparantly associated with their tendency to form complexes with the catalyst, and with the stability of such complexes. On the basis of the early experiments with these compounds, it was thought that the base strength of the compounds was a sufficient indication of the complex stability to give some correlation with ignitability. (3) The more complete results plotted in order of base strength in Table III do not support this view, and there is no obvious correlation.

The aldehyde test results are given in Table IV. In general, aromatic aldehydes, notably benzaldehyde derivatives, ignite with H_2O_2 in the presence of Cu^{+1}, Cu^{+2}, and Fe^{+2} catalysts. Aliphatic aldehydes usually do not ignite. Exceptions to the above are the ignition of isobutyraldehyde in the presence of Cu^{+2} and the ignition of butyraldehyde, isobutyraldehyde, and paraldehyde in the presence of Fe^{+2}. Hydrocinnamaldehyde does not ignite with H_2O_2 and any of thecatalysts while crotonaldehyde is ignitable with all the catalysts.

The miscellaneous group (Table II) contains nineteen ignitable compounds. Three of these contain amino groups as well as other functional groups. They might be expected ot ignite on the basis of the results with simple amines. It should be noted that there is one amino compound, ethanol amine, which does not ignite with any of the catalysts. There are also three substituted benzoic acids which ignite, although a fourth, 3-5-dinitro benzoic acid, does not ignite.

The remaining compounds are more difficult to classify. There are two sulfur-containing compounds, two compounds containing the furan ring and three heterocyclic nitrogen compounds. The remaining compounds defy classification. One of these, benzonitrile, ignites only in the presence of cuprous copper. It can be seen from the table that the remaining five compounds, diethylene glycol, diethylene glycol diethyl ether, isoamyl nitrite, p-nitrophenol, and α-nitroso β-naphthol, react only with iron, which is also a hydrogen peroxide decomposition catalyst of reasonable activity. At this point, it [illegible] worthwhile to digress sufficiently to discuss the function of the catalyst in relation to these tests.

C. Effect of Catalysts on Ignition.

Although the nature of these reactions is but poorly understood, it is fairly evident that in most cases the mechanism is more complex than a simple decomposition of the hydrogen peroxide followed by oxidation of the fuel by the liberated oxygen. The large differences in the violence of the reaction with different organic compounds support this viewpoint. Further indication of specific reaction of the catalyst with the fuel constituent is derived from experiments with copper. When copper salts are added to hydrogen peroxide little decomposition occurs, even in strongly basic solution. When an amine is added to the mixture, however, there is violent reaction and in many cases ignition. It is evident that a specific reaction involving the amine is taking place even at room temperature. Studies in dilute solution of the hydrogen peroxide-hydrazine reaction in the presence of copper catalyst have further shown that the hydrazine reacts with a catalytic intermediate. (4) There is no contribution to the reaction rate from oxygen produced as a result of catalytic hydrogen peroxide decomposition.

Under certain conditions, however, it is reasonable to expect some contribution from catalytic decomposition. It is possible to decompose hydrogen peroxide with an active decomposition catalyst, producing an oxidizing atmosphere at such a temperature that simple organic fuels, such as alcohols, ignite when they are sprayed into the decomposition chamber. (5) A combination of the two effects is almost certainly present in the case of iron compounds used as self-igniting fuel catalysts. Iron compounds may cause a rather vigorous decomposition of hydrogen peroxide, although they do not compare favorably with very active decomposition catalysts. On the other hand, the variations in ignitability of different compounds with hydrogen peroxide using iron as catalyst indicate that there is still a considerable specific fuel-catalyst-hydrogen peroxide reaction. The variety of fuels which ignite with iron catalysts and not with the other catalysts indicate the effect of a combined action where the decomposition raises the temperature to a point where rapid oxidation of the fuel takes place either by a catalytic mechanism or by combustion with the liberated oxygen. This viewpoint provides a plausible explanation of the fact that the miscellaneous compounds mentioned above ignite in the presence of iron but not with the other catalysts.

D. Tests with Catalyst Dissolved in Peroxide

Similar tests were made on a variety of compounds using cobalt as the catalyst and dissolving the cobalt in the hydrogen peroxide. This, of course, may not be permissible operationally, but the peroxide decomposition before mixing with fuel was small enough to permit the use of the cobalt solution for these tests. These results are shown in Table V.

Table V.

Drop Test Results with Cobaltous Nitrate Dissolved in 90% Hydrogen Peroxide and Various Fuels.

Fuel	Result
aniline	vigorous decomposition - flames
benzylamine	vigorous decomposition - sparks
dibenzylamine	vigorous decomposition
di-n-butylamine	vigorous decomposition
diethylamine	vigorous decomposition
diethyl o-toluidine	vigorous decomposition - flame
diethyl m-toluidine	vigorous decomposition - flame
diethyl p-toluidine	vigorous decomposition - flame
ethanol amine	vigorous decomposition
ethylene diamine	vigorous decomposition
hydrazine hydrate (85%)	vigorous decomposition - flame
mercury succinimide	no reaction
methylaniline	vigorous decomposition - flame and explosion
p-nitroso diphenylamine	no reaction
nitrosomethyl-aniline	slight decomposition
phenyl hydrazine	vigorous decomposition - flame
tetramethyl-ammonium bromide	slight decomposition
o-thiocresol	moderate decomposition
m-thiocresol	no reaction
thiophenol	moderate decomposition
o-tolidine	vigorous decomposition - flame
c-toluidine	vigorous decomposition - flame
m-toluidine	vigorous decomposition - flame
tri-n-butylamine	vigorous decomposition
triethylene tetramine	vigorous decomposition

Discussion

In general, the experiments with dissolved catalyst bear out the conclusions reached from the first drop tests. The activity of amine compounds is again obvious. There are a few results which should be mentioned because they do not conform. In two cases, compounds containing amino groups did not show vigorous action. Both these amines contain nitroso groups. There are also two sulfur containing compounds, o-thiocresol and m-thiocresol, which did not react vigorously.

E. Vanadium Compounds

German workers have claimed that vanadium compounds were better catalysts for hydrazine hydrate ignition than copper and iron compounds. (6) Potassium and sodium vanadate were found to be weak decomposition catalysts for concentrated hydrogen peroxide, while ammonium meta-vanadate, sodium-meta-vanadate, and vanadium pentoxide are good decomposition catalysts. Results of drop tests with the latter three vanadium catalysts and miscellaneous liquids are shown in Table VI.

Table VI.

Drop Tests with 90% Hydrogen Peroxide, Vanadium Catalysts, and Miscellaneous Compounds

(x Indicates Ignition)

Compound	Catalyst		
	V_2O_5	NH_4VO_3	$NaVO_3$
Acids-Acid derivatives			
benzoyl chloride	x	x	
n-butyric anhydride	x		x
ethyl cyanoacetate			
ethyl fumarate			
formamide			
lactic acid		x	
levulinic acid	x	x	x
Alcohols-Alcohol derivatives			
diethylene glycol			
diethylene glycol diethyl ether			
ethylene glycol			
furfuryl alcohol	x	x	x
isopropyl alcohol			

Table VI (Cont'd)

	Catalyst		
Compound	V_2O_5	NH_4VO_3	$NaVO_3$
2-methyl-2,4-pentandiol			
propylene glycol			
thiodiglycol	x	x	x
Aldehydes-Aldehyde derivative			
acetal	x		
anisaldehyde	x	x	x
benzaldehyde	x	x	x
n-butyraldehyde	x		
crotonaldehyde	x	x	x
o-ethoxybenzaldehyde	x		x
furfural	x	x	x
salicylaldehyde	x	x	x
m-tolualdehyde	x	x	x
Amine type			
benzylamine			
o-bromoaniline			
n-butylamine			
cyclohexylamine		x	
dibenzylamine			x
diethylamine			
di-isoamylamine			
di-isobutylamine	x		
dimethylaniline	x		
ethanol amine			x
ethylamine (33%)			
ethylaniline			

Table VI. (Cont'd)

	Catalyst		
Compound	V_2O_5	NH_4VO_3	$NaVO_3$
ethylene diamine	x	x	
hydrazine hydrate (85%)	x	x	x
o-iodotoluidine			x
methylaniline	x		x
nicotine			
phenyl hydrazine	x	x	x
pyridine			
quinoline			
m-toluidine			
tri-n-butylamine			
triethylamine			
triethylene tetramine	x	x	x
tri-n-propylamine			
Ethers			
anisole			
o-bromoanisole			
1,4-dioxane	x		x
isoamyl ether			
isopropyl ether		x	x
o-nitroanisole	x	x	x
Hydrocarbons-Hydrocarbon derivatives			
bromobenzene			
cyclohexene	x	x	x
decahydronaphthalene (decalin)			
n-decane			
2,5-dimethyl furan	x	x	x

Table VI. (Contd) Catalysts

Compound	V_2O_5	NH_4VO_3	$NaVO_3$
ethylene bromide			
n-heptane			
n-hexane			
nitrobenzene	x		
o-nitro ethyl benzene			
nitromethane			
m-xylene			
Nitriles acetonitrile			
benzonitrile	x		x
n-valeronitrile			
Miscellaneous ethyl nitrate			
isoamyl nitrite			
methyl n-propyl ketone			

- -

The aldehydes ignite rather consistently with these catalysts, and there are several amines which ignite. The scattered results using other types of compounds are rather difficult to explain and may be partially due to the decomposition type of mechanism discussed above with respect to iron.

F. Cobalt Complexes

Numerous solid compounds were prepared by precipitation from mixtures of some of the catalysts and organic bases. These have the general formula, $Co(A)_aX_b$, in which A is an amine and X is a halogen. Two of these complexes, which were formed from alcoholic cobaltous chloride and (1) hydrazine hydrate, and (2) o-toluidine, have been analysed. Their formulas have been found to be (1) $Co(N_2H_4)_2Cl_2$ and (2) $Co(C_7H_7NH_2)_2Cl_2$. Some twenty-five similar compounds have been prepared using representatives of different classes of amines as well as hydrazine. These compounds were tested for reactivity with hydrogen peroxide by observing results when a drop of 90 per cent hydrogen peroxide fell on a small sample of each. Ignition occurred in thirteen cases. The primary amines, both aromatic and aliphatic, apparently form more reactive complexes than secondary or tertiary amines, although ignition occurred with the complexes formed from the latter two types. These results are shown in Table VII.

Table VII.

Drop Tests with 90% Hydrogen Peroxide on Cobalt Complexes Formed from the Following Compounds and Alcoholic Cobaltous Chloride

No Ignition

cyclohexylamine
diethylamine
di-isobutylamine
ethylamine (33% in water)
phenyl hydrazine
pyridine
quinoline
tri-n-butylamine
tri-n-propylamine

Poor Ignition

diethyl-m-toluidine
di-isoamylamine
ethylaniline

Good Ignition

n-butylamine
diethylaniline
hydrazine hydrate
methyl aniline
β-naphthylamine
o-toluidine
m-toluidine
p-toluidine
tribenzylamine
triethylamine

II.

Attempts to Form Homogeneous Ignitable Solutions

A usable fuel must be homogeneous and stable as well as ignitable. The tests in the first part of the report were made in order to find what types of compounds and mixtures were ignitable, with no regard to homogeneity. In this part we are concerned with attempts to apply the information on ignitability to the production of usable mixtures.

The work is reported in several sections for convenience in organization. The first is concerned with attempts to dissolve catalysts in the fuels with which they form ignitable mixtures. The second includes the efforts to dissolve single solid ignitable compounds in suitable solvents while retaining the ignitability. The third section reports attempts to make ignitable mixtures using non-self-igniting but cheap fuels as diluents for the more expensive mixtures.

A. Solution of Catalyst in Ignitable Compounds

The first attempt to dissolve catalysts in ignitable compounds consisted of shaking some of the prospective catalyst with a small quantity of fuel in a test tube, allowing the solid to settle and decanting off the liquid. The liquid was then tested for ignitability by the usual drop test procedure. Results are reported in Table VIII.

Table VIII.

Drop Tests with 90% Hydrogen Peroxide on Fuel Substances with Various Catalysts in Solution

Fuel	Catalyst in Solution	Result
aniline	acid cuprous chloride	vigorous decomposition-flame
	ferric benzoate	slight decomposition
benzaldehyde	ferric benzoate	slight decomposition
benzylamine	potassium cuprocyanide	vigorous decomposition-flame
dibenzylamine	potassium cuprocyanide	vigorous decomposition
ethanol amine	potassium cuprocyanide	vigorous decomposition-flame
ethylene diamine	cuprous cyanide	vigorous decomposition
	ethylene diamine cupric chloride	vigorous decomposition
	ethylene diammonium chlorocuprate	vigorous decomposition
	potassium cuprocyanide	vigorous decomposition
	sodium nitroprusside	vigorous decomposition

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Table VIII. (Cont'd)

Fuel	Catalyst in Solution	Result
hexamethylenetetramine	hydrochloric acid (conc.)	very slight decomposition
	sulfuric acid (conc.)	vigorous decomposition-long delayed flame
hydrazine hydrate (85%)	ethylene diamine cupric chloride	vigorous decomposition-flame
	ethylene diammonium chlorocuprate	vigorous decomposition
o-toluidine	cobalt acetate	vigorous decomposition-sparks
	cobalt ammonium chloride	moderate decomposition-sparks
	cobaltous chloride	vigorous decomposition-flame
	cobalt chromate	slow decomposition-flame
	cobalt sulfate	vigorous decomposition-flame
	copper sulfate	moderate decomposition
	cupric cyanide	moderate decomposition
	ethylene diammonium chlorocuprate	vigorous decomposition
	ferric sulfate	moderate decomposition
	ferrous sulfate	vigorous decomposition-flame
	nitric acid (conc)	vigorous decomposition-flame
	sodium nitroprusside	moderate decomposition

Discussion of Table VIII.

Eight of these compounds ignite satisfactorily, giving vigorous decomposition and a flame. The catalyst in four of these cases was a complex copper compound, and in two cases a cobalt salt. One case of ignition was observed with an iron salt and in the remaining case the fuel was a mixture of o-toluidine and nitric acid.

Further work has been carried out on the aniline-acid cuprous chloride combination. It has been found that on standing a precipitate forms which contains copper. The composition has not been satisfactorily determined. The other combinations have not as yet been investigated further.

Miscellaneous tests.

Samples of o-toluidine hydroiodide, hydrazinium iodide, and hydrazinium chloride were prepared to investigate a report that I^- would improve the ignition of hydrazine hydrate. Both hydrazinium iodide and chloride improve the consistency of ignition of 85% hydrazine hydrate. Alone, o-toluidine hydroiodide produces sparks with concentrated peroxide but it only reacts moderately, without ignition, when dissolved in o-toluidine.

The three vanadium catalysts mentioned in Part I were separately mixed with 85 per cent hydrazine hydrate. They all react with the hydrazine to produce blue, brown, or green mixtures. The ammonium and sodium meta-vanadate mixtures are more consistently ignitable than hydrazine alone while the vanadium pentoxide mixture increases the violence of the ignition but not the consistency of ignition.

These catalysts were also mixed with thiodiglycol. None of the catalysts was very soluble. The mixture with sodium meta-vanadate was inconsistently ignitable while neither of the other mixtures ignited at all with hydrogen peroxide. A mixture of thiodiglycol and ethyl alcohol was shaken with hydrated ferrous chloride for 10 minutes. This mixture reacted explosively in a drop test.

B. Attempts to Dissolve Ignitable Solid Compounds in Fuel Substances

An attempt was made to dissolve two active solid compounds, salcomine and thioacetamide, in an inexpensive solvent. The results of these experiments are shown in Tables IX and X. In all cases a saturated solution of the active solid was used.

Table IX.

Drop Tests on Salcomine Solutions with 90% Hydrogen Peroxide

Slow Decomposition

acetal	furfural
decalin	isopropyl ether
ethyl acetoacetate	paraldehyde
ethyl nitrate	toluene

Vigorous Decomposition

amyl nitrite	ethylene diamine
diethylaminoethanol	nitromethane

Table IX. (Cont'd)

No ignitable solutions were produced, although salcomine itself ignites. In most cases the solubility was very low.

Table X.

Drop Tests on Thioacetamide Solutions with 90% Hydrogen Peroxide

Slight Decomposition

acetal
acetic acid
acetone
amyl acetate
amyl alcohol
anisole
o-anisidine
benzaldehyde
n-butyraldehyde
n-butyric anhydride
decalin

3,4-diethoxybenzaldehyde
diethyl m-toluidine
ethanol amine
o-ethoxybenzaldehyde
ethyl alcohol
ethyl ether
formic acid
n-heptaldehyde
nitrobenzene
thiophenol
m-toluidine

Vigorous Decomposition

aziridine
o-bromoanisole
diethylamine
ethylene diamine

furfural
hydrazine hydrate
salicylaldehyde

Ignition

annisaldehyde

cinnamaldehyde

It is seen that ignition was obtained in only two cases. In each case the solvent was an aromatic aldehyde.

Iron β-naphthalene Suphonate

German sources reported that iron salts of substituted and unsubstituted naphthalene sulfonic acids were good catalysts for the ignition of mixtures of methyl alcohol and pyrocatechol. The ferrous salt of β-naphthalene sulfonic acid was prepared and was found to be soluble in methyl alcohol. Both the salt itself and its methyl alcohol solution are ignitable with concentrated peroxide, even in the absence of pyrocatechol.

A methyl alcohol solution of ferrous β naphthalene sulfonate was mixed with pyrocatechol in the correct proportions to give a fuel similar to the German "Ergol 56". This fuel contained by weight:

7.5% ferrous dibutyl naphthalene sulfonate
62.5% methyl alcohol
30.0% pyrocatechol

The ferrous β naphthalene sulfonate mixture gave a fuel comparable to the German fuel.

Ethylene glycol, propylene glycol, resorcinol, hydroquinone, and o-cresol were successively substituted for the pyrocatechol to determine whether similar compounds would produce an ignitable fuel. The ethylene glycol fuel did not ignite and propylene glycol gave a very long ignition delay. The fuels made from resorcinol, o-cresol, and hydroquinone ignited but had a longer delay and were less consistent than pyrocatechol.

Organo-metallic Compounds

Four organo-metallic compounds were dissolved in several solvents and the resulting mixtures drop tested with peroxide. Alone, copper dibutyl dithiocarbamate, cobalt propionyl acetonate, and cobalt linoleate react with 90 per cent hydrogen peroxide giving a shower of sparks or flames. Chromium dibutyl dithiocarbamate does not react with concentrated peroxide. In most cases the compounds were insoluble in the following solvents: ethyl alcohol, acetone, methyl alcohol, benzene, aniline, and ether. When a compound did dissolve, there was little or no decomposition with peroxide.

Cobalt complexes

The complex compound, cobalt di-p-toluidyl chloride ($Co(C_7H_7NH_2)_2Cl_2$), made from an alcoholic cobaltous chloride solution and an alcoholic solution of p-toluidine produced sparks with concentrated hydrogen peroxide. This compound was mixed with several solvents in an endeavor to produce an ignitable fuel mixture. The results are shown in Table XI.

Table XI.

Drop Tests with 90% Hydrogen Peroxide, Cobalt Di-p-toluidyl Chloride, and Several Solvents

Solvent	Solubility of Complex	Result with H_2O_2
acetic acid	slightly soluble	no reaction
acetic anhydride	soluble	slight decomposition
acetone	soluble	no reaction
alcohol, absolute	soluble	vigorous reaction
alcohol, 95%	soluble	vigorous reaction

Table XI. (Cont'd)

Solvent	Solubility of Complex	Result with H_2O_2
amyl acetate	soluble	slight decomposition
amyl alcohol	soluble	slight decomposition
aniline	slightly soluble	delayed decomposition
benzene	insoluble	no reaction
bromobenzene	slightly soluble	delayed decomposition
carbon disulfide	insoluble	no reaction
di-tert-butyl peroxide	insoluble	no reaction
ether	slightly soluble	vigorous decomposition
formic acid	insoluble	no reaction
gasoline, aviation	very slightly soluble	small delayed decomposition
gasoline, white	very slightly soluble	small delayed decomposition
glycerol	slightly soluble	slight decomposition
hydrazine hydrate (85%)	soluble	explosion
kerosene	very slightly soluble	small delayed decomposition
methyl alcohol	soluble	vigorous decomposition
phenyl hydrazine	soluble	large flash
turpentine	slightly soluble	small delayed decomposition
o-toluidine	soluble	delayed decomposition

The results were negative in most cases. There was ignition in the case of hydrazine hydrate and phenyl hydrazine. Although hydrazine hydrate will ignite inconsistently without catalyst, the catalyst did dissolve in this case and aided the consistency of ignition.

C. Use of Inexpensive Compounds as Diluents

The self-igniting fuel systems so far developed have been for the most part either expensive or scarce. It was the German custom to dilute these fuels as much as possible with non-self-ignitable but easily available fuels. The principal example in practice has been the German dilution of hydrazine hydrate with water and methyl alcohol to produce the various C-Stoffs. It was considered that it might be possible to produce self-ignitability in common fuels (such as gasoline), by the addition of rather small quantities of an ignitable fuel and/or catalyst.

Work on the dilution of hydrazine has been carried out on this project and is included in a separate report on the use of hydrazine hydrate(1)Attempts have also been made to dissolve ignitable fuels and catalysts in hydrocarbon compounds in sufficient quantities to make the solution self-igniting. This is a difficult problem because of the expected low solubility of such mixtures.

The procedure has been to shake a mixture of ignitable fuel, hydrocarbon fuel, and catalyst. Usually both phases were then tested for ignition in drop tests. The results of tests on the hydrocarbon layer on a number of such combinations are reported in Table XII. These results are uniformly negative, probably due to low catalyst solubility. This view is supported by the fact that o-toluidine-kerosene mixtures do ignite when the two fuels are mixed as above but the catalyst is dissolved in the peroxide.

There is some possibility that the catalyst might be introduced in the form of an organo-metallic salt although the ones tried have produced little activity. It might be feasible to produce a stable colloidal solution of the catalyst in the fuel.

Table XII.

Drop Tests with 90 % Hydrogen Peroxide and Hydrocarbon Fuel Mixtures

Hydrocarbon	Mixer	Catalyst	Result
benzene		acid cuprous chloride	no reaction
	water	copper sulfate	no reaction
	water	ferrous sulfate	no reaction
gasoline: aviation (130 octane)		cobaltous di-p-toluidyl chloride	slightly soluble-- slight decomposition

Table XII. (Cont'd)

Drop Tests with 90% Hydrogen Peroxide and Hydrogen Fuel Mixtures

Hydrocarbon	Mixer	Catalyst	Result
gasoline (cont'd) (130 octane) aviation		copper propionyl acetonate	slightly soluble--inconsistent, long delayed ignition
		salcomine	insoluble--no reaction
	85% hydrazine hydrate	sodium nitroprusside & potassium cuprocyanide	slightly miscible no reaction
	phenyl hydrazine	sodium, potassium & cuprous cyanides	immiscible-moderate decomposition
	ethyl alcohol and water	phenyl hydrazine & sodium, potassium and cuprous cyanides	immiscible-- no reaction
	o-toluidine	ammonical copper sulfate	moderate decomposition
	o-toluidine	copper sulfate	immiscible--precipitate-- no reaction
	o-toluidine	acid cuprous chloride	immiscible-- no reaction
	o-toluidine	sodium nitroprusside	slightly miscible slight decomposition
gasoline: white	85% hydrazine hydrate	sodium nitroprusside and potassium cuprocyanide	slightly miscible no reaction
Kerosene		manganese sulfate	insoluble-- no reaction
	duponol	copper sulfate	no reaction
	85% hydrazine hydrate	sodium nitroprusside and potassium cuprocyanide	immiscible -- no reaction
toluene		ferric benzoate	no reaction
		ferrous β-naphthalene sulfonate	no reaction

Results are shown in table XIII for a group of miscellaneous fuels which were diluted with rather large amounts of solvent. Ignition with peroxide was obtained when o-toluidine-acid cuprous chloride was diluted with acetone and when p-toluidine-cobaltous nitrate was diluted with ethyl alcohol.

Table XIII.

Drop Tests with 90% Hydrogen Peroxide and Multi-Component Fuel Systems

Fuel	Diluent	Catalyst	Result
acetone oxime	ethyl alcohol	cobaltous nitrate	vigorous decomposition
a-amino pyridine	ethyl alcohol	cobaltous nitrate	vigorous decomposition
benzene	ethyl alcohol	cuprous chloride	no reaction
hexamethylene tetramine	benzylamine and water	cuprous, sodium, & potassium cyanides	vigorous decomposition-inconsistent ignition
o-phenylene diamine	ethyl alcohol	cobaltous nitrate	vigorous decomposition
phenyl hydrazine hydrochloride	ethyl alcohol and water	cuprous, sodium & potassium cyanides	vigorous decomposition
o-toluidine	acetone	acid cuprous chloride	vigorous decomposition -sparks-flame
	water	cuprous, sodium & potassium cyanides	no reaction
p-toluidine	ethyl alcohol	cobaltous nitrate	vigorous decomposition - flame

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