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The Amount and Identification of Amyl Alcohols in a Commercial Mixture of Pentanols

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T1162

THE AMOUNT AND IDENTIFICATION OF AMYL ALCOHOLS
IN A
COMMERCIAL MIXTURE OF PENTANOLS

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MILWAUKEE, WISCONSIN
May 1930

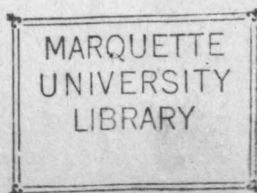


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The writer wishes to express the most sincere gratitude to Dr. J. E. Koch and Dr. H. Hejralich, for their patient and constant assistance in directing the work on this problem.

INTRODUCTION

For many years the pentanols were regarded, largely, as scientific curiosities. The scarcity of their sources and the expense of production caused little attention to be given them. The discovery of natural gas and its products furnished a cheap and plentiful source for larger production.

The history of these alcohols is interesting.

PREFACE

The Amount and Identification of Amyl Alcohols in a Commercial Mixture of Pentanols.

Casing head gasoline obtained from natural gas yields five commercial products. One of these is a mixture of amyl alcohols. As treated by the Sharples Company, West Virginia, pentane is separated and treated with chlorine. Three chlorine derivatives are produced. These are treated with caustic soda and may form eight possible amyl alcohols.¹ The products of the normal pentane, only, are the subject matter of this thesis.

The writer wishes to express the most sincere gratitude to Dr. J. R. Koch and Dr. H. Heinrich, for their patient and constant assistance in directing the work on this problem.

¹Eugene E. Ayers, Amyl Alcohols from the Pentanes; 21:899-904

INTRODUCTION

For many years the pentanols were regarded, largely, as scientific curiosities. The scarcity of their sources and the expense of production caused little attention to be given them. The discovery of natural gas and its products furnished a cheap and plentiful source for laquer production.

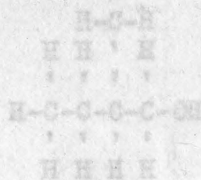
The history of these alcohols is interesting.

Substances of Amyl or Pentyl formation are derived from the three pentanes.

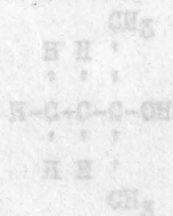
Normal pentane can yield one primary and two secondary alcohols. Isopentane can produce two primary one secondary and one tertiary alcohol. Tetramethyl methane can yield but one single primary alcohol.

The following table gives the emperical formulas for all possible monohydroxy amyl alcohol all of which are known.

IV. 2 Methyl Butanol (1)
Secondary Butyl Carbinol



V. 2 Methyl Butanol (3)
Di-methyl Ethyl Carbinol
Tert. Amyl Alcohol



VI. 2 Methyl Butanol (30)
Methyl Iso Propyl Carbinol

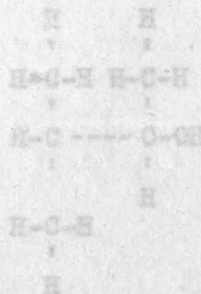


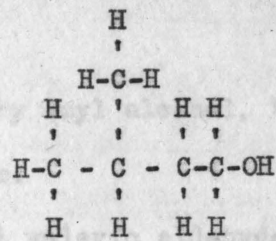
TABLE I

Empirical Formulas for Amyl Alcohols

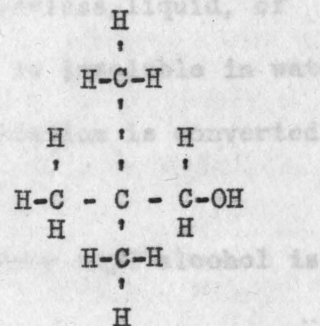
<p>I. Amyl Alcohol: N. Amyl Alcohol:</p>	<pre> H H H H H H-C-C-C-C-C-OH H H H H H </pre>
<p>II. Pentanol (2) Methyl Propyl Carbinol</p>	<pre> H H-C-H H H H H-C-C-C - C-OH H H H H </pre>
<p>III. Pentanol (3) Di-ethyl Carbinol</p>	<pre> OH H H H H H-C-C-C-C-C-H H H H H H </pre>
<p>IV. 2 Methyl Butanol (1) Secondary Butyl Carbinol</p>	<pre> H H-C-H H H H H-C-C-C-C-OH H H H H </pre>
<p>V. 2 Methyl Butanol (2) Di-methyl Ethyl Carbinol Tert. Amyl Alcohol</p>	<pre> CH₃ H H H-C-C-C-OH H H CH₃ </pre>
<p>VI. 2 Methyl Butanol (30) Methyl Iso Propyl Carbinol</p>	<pre> H H H-C-H H-C-H H-C ---- C-OH H H-C-H H </pre>

TABLE I (Continued)

VII. 2 Methyl Butanol (4)
 Iso Butyl Carbinol
 Primary Iso Amyl Alcohol



VIII. 2,2 Dimethyl Propanol
 Tert. Butyl Carbinol



Any information on the various properties and preparations of amyl alcohols is either scattered or only available in foreign books. Therefore, it was thought to be worth while to summarize below the more important properties and preparations.

I. Normal butyl carbinol or normal primary amyl alcohol, has been prepared in either of the two following ways:

1. By addition of hydrogen to normal valeric aldehyde.
2. From normal pentane. It is a colorless liquid, of fusel odor, boiling at 137° . It is insoluble in water, has sp. gr. .8296 at 0° and on oxidation is converted into normal valeric acid.

II. Methyl-propyl carbinol or first secondary amyl alcohol is prepared:

1. By addition of hydrogen to propyl-methyl ketone.
2. Together with normal butyl carbinol from the chlor-amyliodide. This is converted into the alcohol by moist silver oxide. substitution products of normal pentane.
3. From ethyl-allyl and hydriodic acid followed by replacement of iodine and the oxy radical. It is a colorless liquid, boils at 120° - 122° and has sp.gr. .825. It is converted into propyl-methyl ketone by oxidation.

III. Di-ethyl carbinol or B secondary amyl alcohol is prepared from ethyl formate and zinc ethyl by heating.

It is a colorless liquid, has fusel-like odor. The specific gravity is .832 at 0° C and 819 at 16° C, it boils at 116° - 117° C. It yields a ketone or propionic and acetic acids upon oxidation.

IV. Isobutyl carbinol, or first primary isoamyl alcohol is the one known for the longest time as an amyl alcohol obtained by fermentation. It is found invariably in fusel oils, and is separated from them

by fractional distillation as an unpleasant smelling liquid of fusel-like odor. Its specific gravity is .825 at 0° C. Its boiling point is 129°-130° C. It is soluble in all proportions in alcohol and ether, but it is insoluble in water. Its vapor causes coughing when inhaled, and acts as a poison. When quickly fractionated from fusel oil it gives a rotation to the left in the plane of polarization. This optical activity is lost when the liquid is heated for a long time or when it is distilled from caustic alkali. There is no apparent chemical change. This gives evidence that the optical activity belongs directly to an isomer mixed with it. Oxidation of the isoamyl alcohol produces isovaleric aldehyde and acid.

V. Methyl-isopropyl carbinol, secondary isoamyl alcohol or amylene hydrate is prepared by heating isobutyl carbinol (IV) with zinc chloride, by uniting it with hydriodic acid, to obtain secondary isoamyl iodide. This is converted into the alcohol by moist silver oxide. It has a boiling point at 106°-108° C. The vapor, heated to 200° C splits into isoamylene and water. Sulphuric acid mixed with it produces the same result without heat. Oxidation produces carbonic and acetic acids.

VI. Ethyl-dimethyl carbinol or tertiary amyl alcohol, is obtained from zinc methyl and propionic chloride, and the subsequent decomposition of this product by water. It boils at about 100° C. Apparently it solidifies at -10°. Oxidation yields carbonic and acetic acids.

VII. Second primary isoamyl alcohol is thought to be the active admixture in fermentation amyl alcohol (IV). Its boiling point (125°-129°) though nearly the same, but there is a difference from the "active" in the properties of the respective acid sulphates.²

²Dr. J. Wislicemus: "Adolph Strecker's Organic Chemistry." 126-128.

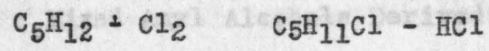
VIII. Tertiary Butyl carbinol is the last amyl alcohol to be known. It has a boiling point of K-113-114° C. Its vapor tensions is 16 m.m. at 20° C and 533.5 m.m. at 100° C.³

Modern research has found an abundant and cheap material in natural gas. The identity of the amyl alcohols was fixed by chlorinating them to form the pentanes. The synthetic processes use hydrolysis, with caustic soda, on the chlorinated derivatives of the pentanes to produce the amyl alcohols.

The importance of the amyl alcohols is of great concern in industry because of its being an ingredient in the expensive lacquers and varnishes.

The method followed by the Sharples Co. is of interest:

The reactions simply expressed take the forms:



The company has been able to produce six of the possible seven amyl chlorides, and the corresponding six amyl alcohols.⁴

The three pentanes from natural gas are, normal pentane, B.P. 36° C; Isopentane, B.P. 28° C; and Tetramethylmethane, B.P. 9.5° C.

Tetramethylmethane is the best known. It is easily obtained as a two percent constituent of Texas and West Virginia gasolines. By chlorination and hydrolysis tertiary butylcarbinol, boiling point 114° C is produced. But this alcohol is not regarded as suitable for commercial synthesis.

³Bellstein: 1, 383. Ergänzungsband, 1, 193.

⁴Ayres, E. E., Industrial and Engineering Chemistry 21, 899-900 (1929)

In the synthetic process sodium oleate is used for two purposes:

(1) "To put the water into colloidal contact with the amyl chloride which permits the most effective hydrolysis of tertiary and secondary chlorides. *It attacks the carbon atom with the least number of hydrogens.*

(2) It is, itself, in colloidal contact with the amyl chloride, so that primary and secondary chlorides can react to form the amyl oleates, which can be easily hydrolyzed with caustic soda to produce amyl alcohols.⁵

It is claimed that special treatments produce amychlorine derivative desired. According to one result of the Sharples Company, the yield at any temperature, from secondary chlorides is about 48 per cent in the absence of water (or alcohol) while in the presence of water it varies from 65 per cent at 180° C to 100 per cent at 100° C.⁶

The latest results obtained by the above company are given below:

1. to Mixed Amyl Alcohols Derived from Pentanes ^a *obtained by fractional distillation.*

	B.P. °C	Percentage of Mixture
CH ₃ CH ₂ CH ₂ CH ₂ CHOH	138	26
CH ₃ CH ₂ CH ₂ CHOH CH ₃	119	8
CH ₃ CH ₂ CHOH CH ₂ CH ₃	115.7	8
CH ₃ CH ₃ CHCH ₂ CH ₂ OH	130.5	16
CH ₂ OH CH ₃ CHCH ₂ CH ₃	128	32

^aTertiary amyl alc. prod. during the process is taken off in a separate fraction.⁷

⁵Ibid. 21, 900.

⁶Ibid. 21, 902.

⁷Clark, Lee H.: Ind. & Eng. Chem. 22, 440 (1930)

Interest in pentyl alcohols has greatly increased with their commercial use. Scientific interest is based, largely, upon the experimental proof of the Markownikoff Rule.⁸ According to this rule the chlorine atom seeks the carbon atom with the least number of hydrogens. When caustic soda is added the hydroxyl group replaces the chlorine. Since a secondary carbon holds two hydrogens and a tertiary carbon has none, the chlorine should go to the secondary or tertiary carbon atoms. The succeeding hydroxyl group then forms a secondary or a tertiary alcohol.

Fractional distillation seemed the most logical method for attacking this problem. This method will separate each fraction. The alcohols can then be identified by their boiling points, their indices of refraction and their density.

The object of the following investigation is,

1. to separate the alcohol mixture into its constituents by fractional distillation.
2. to identify the various alcohols thus obtained by means of their boiling points, refractive indices and specific gravities.

⁸Cohen, Julius B., Ph.D., D.Sc., F.R.S.

Plate I.

Fractional distillation is one of the most widely used operations in the chemical industry. Its operation is simple, cheap and efficient.

It is always practical if the boiling points of the ingredients are not too close together, or if the material is not a constant boiling mixture.

The fractionating column is of great importance. The still heads may be of different types. These are common. I, The Vigreux column

consists of glass tubing with bulbous sections. II, The Hempel type is a glass column filled with glass beads. III. The Allihn condenser consists

of glass jacket containing a series of glass tubes. The efficiency of a fractionating column is increased by increasing the number of theoretical plates.

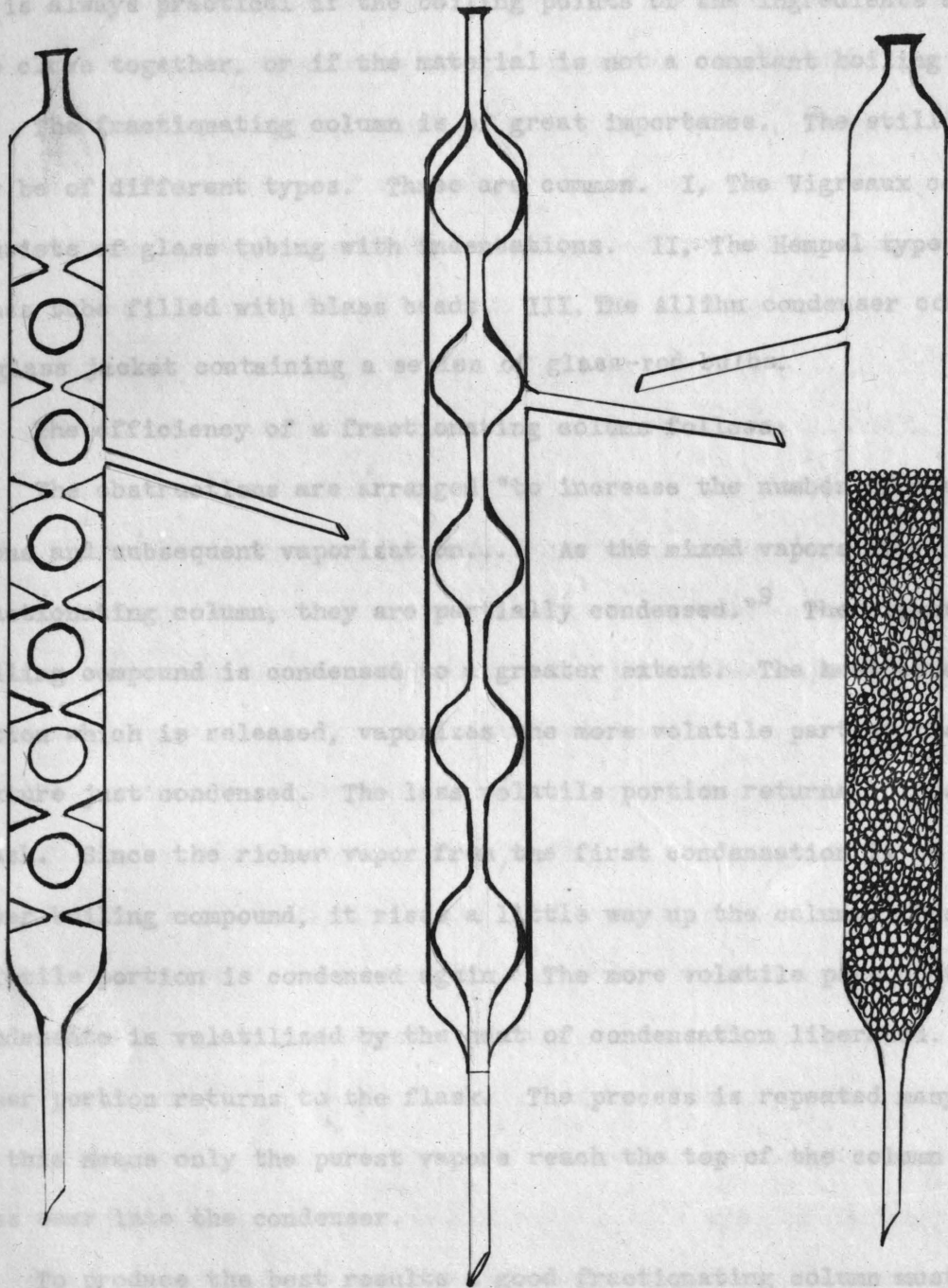
The plates are arranged to increase the number of theoretical plates. As the mixed vapors rise up the fractionating column, they are repeatedly condensed.

The less volatile compound is condensed to a greater extent. The more volatile portion which is released, vaporizes and the more volatile portion of the mixture is condensed.

The less volatile portion returns to the flask. The richer vapor from the first condensation rises to the next condensation stage. The less volatile compound, it rises a little way up the column. The less volatile portion is condensed again. The more volatile portion of this condensation is volatilized by the loss of condensation liquid. The other portion returns to the flask. The process is repeated many times.

By this means only the purest vapors reach the top of the column and pass over into the condenser.

To produce the best results a good fractionating column must



I.

III.

II.

EXPERIMENTAL RESULTS

Fractional distillation is one of the most widely used operations in the chemical industry. Its operation is simple, cheap and efficient. It is always practical if the boiling points of the ingredients are not too close together, or if the material is not a constant boiling mixture.

The fractionating column is of great importance. The still heads may be of different types. Three are common. I, The Vigreux column consists of glass tubing with indentations. II, The Hempel type is a glass tube filled with glass beads. III, The Allihn condenser consists of glass jacket containing a series of glass-rod bulbs.

The efficiency of a fractionating column follows: The obstructions are arranged "to increase the number of condensations and subsequent vaporization.... As the mixed vapors start up the fractionating column, they are partially condensed."⁹ The higher boiling compound is condensed to a greater extent. The heat of condensation which is released, vaporizes the more volatile part of the mixture just condensed. The less volatile portion returns to the flask. Since the richer vapor from the first condensation is of the lower boiling compound, it rises a little way up the column. The less volatile portion is condensed again. The more volatile part of this condensate is volatilized by the heat of condensation liberated. The other portion returns to the flask. The process is repeated many times. By this means only the purest vapors reach the top of the column and pass over into the condenser.

To produce the best results a good fractionating column must

⁹ Adkins and McElvain: "Practice of Organic Chemistry" 163-164.

comply with the following points:

1. It must be high.
2. It must reflex well.
3. It must be shielded against loss of heat due to radiation.
4. It must provide intimate contact of the liquid and vapor, therefore must not slant.
5. The temperature of the column should be so regulated as to maintain a temperature just below the boiling point of the high boiling constituent; and just at the boiling point of the lower one.

The Methods of heating the column may be by steam, an oil bath, or an electrical heating spiral.¹⁰

The apparatus arranged for this experiment was arranged as described below. In view of the boiling points of the amyl alcohols being close together a special device was worked out. This idea helped to prevent superheating especially for the lower fractions.

The apparatus consisted of a large round bottom flask. (A three liter flask for the first two distillations, a one liter flask for the others). The still-head fit^{ted} into a rubber stopper in the flask. This still-head consisted of an inner column with a regular series of bulbs. Around this column a shell-jacket-like those on ordinary condensers was sealed. Two side arms permitted the constant flow of steam to bathe the column. Above the large column a smaller still head (length) was secured. This was of the indentation type and with a side arm for delivery, Into the rubber stopper of the small still head was placed the thermometer. The mercury bulb was just below the side arm opening. The side arm led

¹⁰Adkins and McElvain, Practical of Organic Chemistry - 166

into an ordinary cold water condenser. The receivers were 100 cc graduates. These were of importance since they showed immediately the number of cc for each fraction. For the index of refraction an Abbe Refractometer was used. Specific gravity weighings were made in an Abbe Pyknometer. The heat was supplied by a Bunsen burner and steam heat. The entire apparatus was wrapped heavily in asbestos and was surrounded by a tall asbestos-lined pasteboard chimney which extended upward to the side arm. Over the top of the chimney was a flat cover.

One gallon of the commercial mixture of pentols was used. The fractions of the first two distillations were taken for every two degrees. The latter ones, for each degree.

It was necessary to make the first distillation of the one gallon (3,750^{cc}) in three portions, 1500^{cc}, 1500^{cc}, and 750^{cc} parts. For the first two distillations the fractions of like boiling points were put together.

After the second distillation of the one gallon the index of refraction was taken for each fraction, and recorded. These indices were used to make new groups. All readings of like index were placed in a group. These groups were again distilled. This process was repeated several times.

In all fractions up to one hundred degrees two layers separated out. The layer of unknown substance fell to the bottom of the receiver, being heavier than the alcohols.

To force this portion of the mixture over the steam heat was used on the fractionating column. No alcohol distilled over above one hundred degrees centigrade (steam heat). This steam heating was used, not to heat the vapors but to cool them. The action of steam cooling of vapors

is analogous to cold water on liquids of ordinary boiling temperature.

When all distillation ceased, steam heat was removed and the Bunsen burner only was used. This furnished heat as high as 137°C.

The problem at this point was:

1. The identification of the alcohols.
2. The percentage of each.
3. The boiling points of the ingredients.
4. The index of refraction of the ingredients.
5. The specific gravity of the ingredients.
6. The identification of the second layer.

The data obtained is recorded in the following tables.

Table II shows the boiling point range of the various fractions together with the refractive indices. The succession is generally uniform. The high refractive index for the first fraction is evidently due to the presence of unidentified impurities which boil over at a low temperature. The refractive indices were used to combine smaller fractions into larger groups. Table III shows these groups.

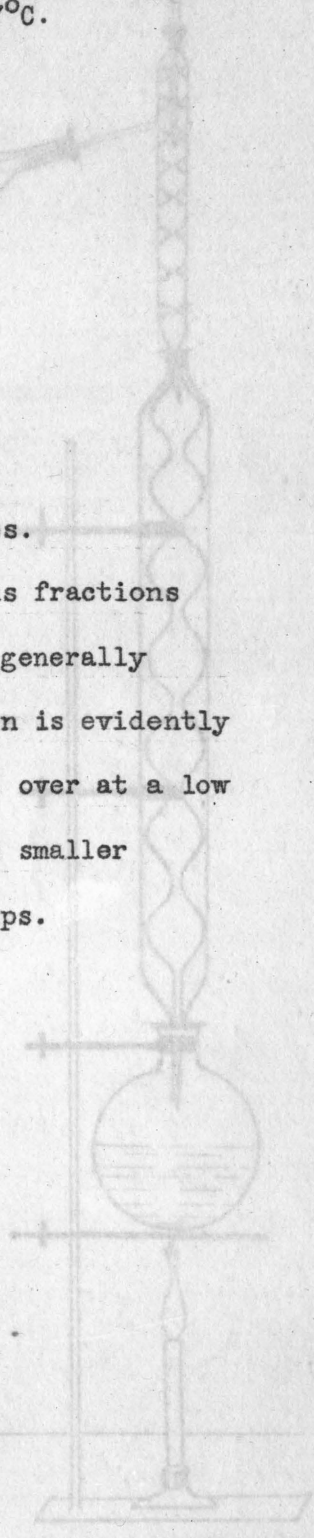
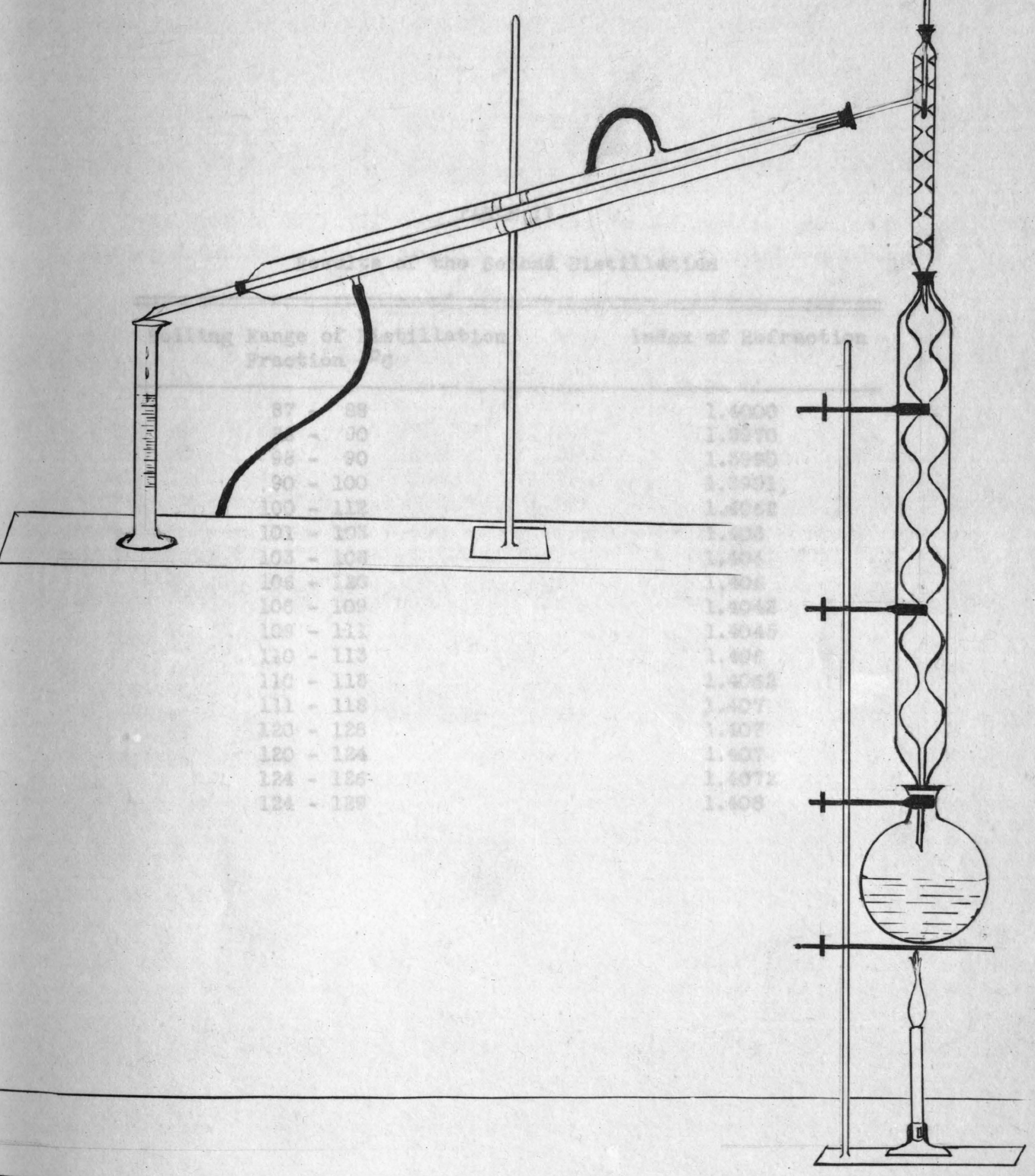


Plate I.



Boiling Range of Fraction	Boiling Point of Fraction	Index of Refraction
87 - 88	88	1.4000
88 - 90	90	1.4070
90 - 90	90	1.4080
90 - 100	100	1.4001
100 - 112	112	1.4052
101 - 104	104	1.400
103 - 104	104	1.404
106 - 106	106	1.406
106 - 109	109	1.4042
109 - 111	111	1.4045
110 - 113	113	1.404
110 - 118	118	1.4052
111 - 118	118	1.407
120 - 128	128	1.407
120 - 124	124	1.407
124 - 126	126	1.4072
124 - 129	129	1.408

TABLE II

Results of the Second Distillation

Boiling Range of Distillation Fraction -°C		Index of Refraction
87 - 88		1.4000
88 - 90		1.3970
98 - 90		1.3990
90 - 100		1.3991
100 - 112		1.4062
101 - 103		1.403
103 - 106		1.404
106 - 120		1.406
106 - 109		1.4042
109 - 111		1.4045
110 - 113		1.406
110 - 118		1.4062
111 - 118		1.407
120 - 128		1.407
120 - 124		1.407
124 - 126		1.4072
124 - 129		1.408

Table IV records the boiling points and the refractive indices after the first groups which were separated according to their refractive indices, were distilled. The new groups were again separated and labelled M., N., O., P., and Q. as shown in Table V. This procedure shows the extensive range of fractions which are obtained with water. It will be noted that the fractions, beginning with the second group, are much higher than the original

TABLE III

Fractions of Table II Grouped According to Refractive Indices

Group A		Group B		Group E	
I.R. - 1.4040-1.4052		I.R. - 1.4060-1.4062		I.R. - 1.403	
B.P. °C	I.R.	B.P. °C	I.R.	B.P. °C	I.R.
103-106	1.4040	106-120	1.406	101-103	1.403
106-109	1.4042	110-113	1.4062		
109-111	1.4045	110-118	1.4062 (2)		
100-112	1.4052	116-118	1.406		
Group C		Group D		Group F	
I.R. - 1.407-1.4072		I.R. -1.4077-1.408		I.R. - 1.3961-1.3991	
B.P. °C	I.R.	B.P. °C	I.R.	B.P. °C	I.R.
118-120	1.407	122-128	1.4077	88-90	1.3961
120-124	1.407	124-129	1.408	90-100	1.3991
122-124	1.4073 (3)				1.399
124-126	1.4072				

Table IV records the boiling point and the refractive indices after the first groups which were arranged according to like refractive indices, were distilled. The new groups were again gathered and labelled M., N., O., P., and Q. as shown in Table V. The E column shows the extensive range of fractions still combined with water. It will be noted that the fractions begin at lower temperatures and rise to ones higher than the original liquids.

TABLE IV
Results of the Third Distillation

B.P. -°C	R.I. --B	R.I. --A	R.I. --C	R.I. --D	R.I. --E-H ₂ O
87-100	1.3980	1.402			1.398
100-102	1.403	1.404			1.3982
102-104	1.4035	1.4045			1.398
104-106	1.404	1.405			
106-108		1.4052			1.403
108-110	1.405	1.4055			
110-112	1.4055	1.4055	1.405		
112-114	1.406	1.4065	1.4065		
114-116	1.4062	1.4068	1.4069		
116-118	1.4062	1.407	1.407		
118-120	1.4074	1.407	1.407	1.4068	
120-122	1.4075	1.4075	1.407		
122-124	1.4075		1.407	1.4075	
124-126	1.4075	1.403	1.408	1.407	
126-128			1.408	1.4071	
128-130			1.408	1.4075	1.407

Groups Classified from Results of Distillation in the
 District of Columbia

TABLE IV

Results of the Third Distillation

B.P. -°C	R.I.--B	R.I.--A	R.I.--C	R.I.--D	R.I.--E-H ₂ O
87-100	1.3980	1.402			1.398
100-102	1.403	1.404			1.3962
102-104	1.4035	1.4045			1.398
104-106	1.404	1.405			
106-108		1.4052			1.403
108-110	1.405	1.4055			
110-112	1.4055	1.4055	1.405		
112-114	1.406	1.4065	1.4065		
114-116	1.4062	1.4068	1.4068		
116-118	1.4062	1.407	1.407		
118-120	1.4074	1.407	1.407	1.4068	
120-122	1.4075	1.4075	1.407		
122-124	1.4075		1.407	1.4075	
124-126	1.4075	1.408	1.408	1.407	
126-128			1.408	1.4071	
128-130			1.408	1.4078	1.407

Contributing
 Group

A

Y

S

D

B

TABLE V

Groups Compiled from Table IV According to Like
Indices of Refraction

Group M

Contributing Group	B.P. °C	I. R.	No. cc.
A	104-106	1.405	27
	106-108	1.4052	41
	108-110	1.4055	30
B	110-112	1.4055	62
B	108-110	1.405	44.5
	110-112	1.4055	310
C	110-112	1.405	32
			<u>546.5</u>

Group N

Contributing Group	B.P. °C	I. R.	No. cc.
A	112-114	1.4065	55
	114-116	1.4068	51
B	112-114	1.406	114
	114-116	1.4062	30
	116-118	1.4062	51
C	112-114	1.4065	72
	114-116	1.4068	115
			<u>488</u>

Group O

Contributing Group	B.P. °C	I. R.	No. cc.
A	116-118	1.407	110
	118-120	1.407	27.5
X	114-117	1.407	22
C	116-118	1.407	
	118-120	1.407	41
	120-122	1.407	82.5
	122-124	1.407	51
D	118-120	1.4068	42
	124-126	1.407	78
	126-128	1.4071	89
B	124-126	1.407	20
			<u>563.0</u>

TABLE V (continued)

Group P

Contributing Group	B.P. °C	I. R.	No. cc.
X	117-118	1.4078	22
	118-120	1.4078	36
B	118-120	1.4074	15
	120-122	1.4075	30.5
	122-124	1.4075	18.8
A	120-122	1.4075	18
D	122-124	1.4075	69
			209.3

Group Q

Contributing Group	B.P. °C	I. R.	No. cc.
A	124-126	1.408	61
C	124-126	1.408	55
	126-128	1.408	52
	128-130	1.408	35
D	128-130	1.408	21
			224

Table VI shows the boiling points and refractive indices after

the fourth distillation. These indices were taken to place the fractions into the last groups collected. The object was to group for the specific gravity test.

B.P.	yield	yield							
100-101									
101-102									
102-103	1.4023	23.5							
103-104	1.4023	52							
104-105	1.4023	85							
105-106	1.4023	40							
106-107	1.4023	31							
107-108	1.404	10							
108-109	1.404	42							
109-110	1.404	71	1.404	28.5					
110-111	1.405	75	1.405	30					
111-112	1.4055	27.5	1.405	48					
112-113	1.405	30	1.405	20.5	1.405	22			
113-114	1.405	12	1.405		1.405	23			
114-115	1.405	20	1.405		1.405	24.5			
115-116	1.405	12.5	1.405		1.405	25.5			
116-117			1.405		1.405	26.5			
117-118	1.405	12	1.405		1.405	28.5			
118-119	1.405	22.5	1.405		1.405	41			
-119							1.4055	19	
119-120			1.4055	25	1.4055	25	1.405	10	
120-121			1.405	2	1.4055	24.5	1.405	13.8	
121-122			1.405	24	1.4055	1.4055	25	10	
122-123					1.405	5	1.405	21	
123-124					1.405	21.5	1.4055	20.5	
					1.4055	21.5	1.4055	20	
		Residue II	c. c.						
125-126				1.405	21	1.405	21	1.4055	20
126-127						1.405	23	1.405	21
127-128	1.405		20.5			1.405	22.5	1.405	20
128-129	1.4055		27.5			1.405	2		
129-130	1.4055		53						
		Residue I							
130-131	1.410		14.5						
131-132	1.410		79						
132-133	1.410		63						
133-134	1.4105		34						
134-135	1.411		8						
135-136	1.411		8						

TABLE VI

Results of Fourth Distillation of Each Group of Table V

B.P. °C	R.I.-M yield	c.c.	R.I.-N yield	c.c.	R.I.-O yield	c.c.	R.I.-P yield	c.c.	R.I.-Q yield	c.c.
100-101	1.4015	12								
101-102	1.402	14.5								
102-103	1.4023	13.5								
103-104	1.4023	32								
104-105	1.4023	35								
105-106	1.4025	40								
106-107	1.4025	31								
107-108	1.404	10								
108-109	1.404	42								
109-110	1.404	71	1.404	38.5						
110-111	1.405	75	1.405	30						
111-112	1.4055	27.5	1.405	49						
112-113	1.406	30	1.405	50.2	1.405	52				
113-114	1.406	12	1.405		1.405	23				
114-115	1.406	20	1.405		1.405	66.5				
115-116	1.406	12.5	1.405		1.405	82.5				
116-117			1.405		1.405	74.5				
117-118	1.406	12	1.406		1.406	95.2				
118-119	1.406	22.5	1.406		1.406	41				
-119									1.4052	19
119-120					1.4062	62	1.4062	23	1.406	10
120-121					1.407	8	1.4068	28.5	1.406	19.5
121-122					1.407	22	1.4068	31.2	1.406	10
122-123							1.407	34	1.406	21
123-124					1.407	62.5	1.407	20	1.4065	30.2
					1.4072	31.8		22.2	1.4068	30
		Residue II		c.c.						
125-126					1.407	21	1.407	11	1.407	30
126-127							1.407	11	1.407	20
127-128		1.409		25.5			1.407	23.5	1.407	19
128-129		1.4095		27.5			1.407	8		
129-130		1.4095		55						
		Residue I								
130-131		1.410		86.2						
131-132		1.410		79						
132-133		1.410		63						
133-134		1.4105		34						
134-135		1.411		8						
135-136		1.411		8						

TABLE VII

Groups Formed from Table VI According
to Like Refractive Index

Contributing Group	B.P. °C	I. R.	c.c.	Sp. Gr.
Group I				
Contributing Group	B.P. °C	I. R.	c.c.	Sp. Gr.
1. Gr. "M"	101-102	1.402	14.5	
	102-103	1.4023	13.5	
	103-104	1.4023	32	
	104-105	1.4023	35	
			95.0 cc.	
Group VI				
Contributing Group	B.P. °C	I. R.	c.c.	Sp. Gr.
1. Gr. "M"	107-108	1.404	10	
	108-109	1.404	42.2	
	109-110	1.404	71	
2. Gr. "N"	109-110	1.404	38.5	
			161.5 cc.	
Group VII				
Contributing Group	B.P. °C	I. R.	c.c.	Sp. Gr.
1. Gr. "M"	111-112	1.4055	27.5	
	112-113	1.406	30	
	113-114	1.406	12	
2. Gr. "P"	114-115	1.406	20	
	115-116	1.406	12.5	
			102.0 cc.	
Group III				
Contributing Group	B.P. °C	I. R.	c.c.	Sp. Gr.
1. Gr. "M"	110-111	1.405	75	
2. Gr. "N"	110-111	1.405	30	
	111-112	1.405	49	
3. Gr. "N"- "O"	112-113	1.405	50.2	
	112-113	1.405	52	
	113-114	1.405	23	
	114-115	1.405	66.5	
	115-116	1.405	82.5	
	116-117	1.405	74.5	
			427.7 cc.	
Group IV				
Contributing Group	B.P. °C	I. R.	c.c.	Sp. Gr.

TABLE VII (continued)

Group V

Contributing Group	B.P. °C	I. R.	c. c.	Sp. Gr.
1. Gr. "M"	117-118	1.406	12	
	118-119	1.406	22.5	
2. Gr. "N"- "O"	117-118	1.406	95.2	.81137
	118-119	1.406	41	
3. Gr. "O"	118-119	1.406	61.5	
			<u>232.2</u> cc.	

Group VI

Contributing Group	B.P. °C	I. R.	c. c.	Sp. Gr.
1. Gr. "Q"	119-120	1.406	10	.8097
	120-121	1.406	19.5	
	121-122	1.406	10	
	122-123	1.406	21.3	
	123-124	1.4065	30.2	
			<u>91.0</u> cc.	

Group VII

Contributing Group	B.P. °C	I. R.	c. c.	Sp. Gr.
1. Gr. "O"	120-121	1.407	8	.81022
	121-122	1.407	22	
	123-124	1.407	62.5	
	124-125	1.4072	31.8	
	125-126	1.407	21	
2. Gr. "P"	120-121	1.4068	28.5	
	121-122	1.4068	31.2	
	122-123	1.407	34	
	123-124	1.407	20	
	124-125	1.407	22.2	
	125-126	1.407	11	
	126-127	1.407	11	
	127-128	1.407	23.5	
	128-129	1.407	8	
3. Gr. "Q"	124-125	1.4068	30	
	125-126	1.407	30	
	126-127	1.407	20	
	127-128	1.407	19	
			<u>433.7</u>	

The results in Table VIII are formed from the preceeding table with some groups re-arranged. Fraction III and fraction IV were combined into fraction III, and all numbers of fraction adjusted accordingly, that is, fraction V becomes fraction IV, fraction X becomes fraction IX, etc. The fractions 101°-105°C and 101°-103°C were redistilled. From this distillation 10° below 100° were placed with the water fraction. The fractions 102°-4°C, 104°-8°C and 100°-02°C were placed in Table VIII. The new groups were tested for primary, secondary, and tertiary carbon atoms by the zinc-chloride-hydrochloric-acid reagent test. Only the larger fractions were used.

The reagent is made by dissolving 136 g. (1 mole) of anhydrous zinc chloride in 105 g. (1 mole) of concentrated hydrochloric acid with cooling.

Results are produced with 2 ml. of the alcohol in a test tube, to which 12 ml. of the reagent at 26-270 is quickly added. The mixture is shaken and the tube closed with a stopper. "Alcohols lower than hexyl are soluble, but tertiary alcohols react so fast and the separation of the tertiary chloride proceeds so rapidly that two phases are observed from the time of mixing. On standing, within five minutes or less the clear solution becomes cloudy in the case of the secondary alcohols and undergoes no change other than darkening in the case of the primary. After one hour a distinct upper layer is visible in the case of all the secondary alcohols except isopropyl.

To distinguish with certainty between tertiary and secondary alcohols, another sample of the alcohol is mixed with concentrated hydrochloric acid alone. Tertiary butyl and amyl alcohols immediately react to form the insoluble chloride which rises to the surface. Solutions of the secondary alcohols should remain clear." 11

11Howard J. Lucas, Journal of the American Chemical Society, 52: 803, (1930)

VERIFICATION OF RESULTS

1. A large portion of the original mixture, 1225.2^g, according to 56 per cent was lost in the process of the experiment. The greater loss was caused by volatilization. Smaller portions were taken as samples for the refractive indices, densities, transference of heat etc. and the optical densities etc.

TABLE VIII

Final Results from the Groups of Table VII

Fraction	Boiling Point °C	c.c.	Refractive Index	Temperature	Specific Gravity	Class of Alcohol
I	102-8	43	1.4055	23	.8077	Tertiary
II	107-10	161.5	1.404 (3)	23	.8077	Tertiary
III	110-17	529.7	1.4055 (19)	24	.8116	Secondary
IV	117-19	232.2	1.406 (7)	23	.8094	Secondary
V	119-24	91	1.406 (5)	24	.8074	Secondary
VI	120-25	433.7	1.4068(3)-1.407(5)	23	.8078	Primary
VII	128-30	82.5	1.409(1)-1.4095(2)	23	.8120	Primary
VIII	130-33	228.2	1.410 (3)	22.5	.8081	Primary
IX	133-36	67.5	1.4105(1)-1.411(2)	23	.8108	Primary
Intermediate						
X	100-01	12	1.4015 (1)	23	.8157	Tertiary
XI	100-02	20	1.403	23	.814	Tertiary
XII	105-7	71	1.4025 (2)	22.5	.8071	Tertiary
XIII	119-20	85	1.4062	23	.8081	Secondary
XIV	119	19	1.4052	22.5	.796	Secondary
XV	127-8	25.5	1.409	23	.8129	Primary

- a. Primary alcohols are present in fractions V, VI, VII, VIII, IX, IV to the extent of 845.5^g, which formed 57.25 per cent of the total amount.
- b. Secondary alcohols are present in fractions III, IV, XIII to the extent of 845.5^g which formed 57.25 per cent of the total amount.
- c. Tertiary alcohols are present in fractions I, II, XII to the extent of 295.5^g which formed 1.95 per cent of the total amount.

Using the Reilstein and Sharpless boiling temperatures to identify the individual alcohols the following results were obtained:

VERIFICATION OF RESULTS

1. A large portion of the original mixture, 1226.2^{cc}, amounting to 34 per cent was lost in the processes of the experiment. The greater loss was caused by volatilization. Smaller portions were taken as samples for the refractive indices, densities, transference of containers, and the special identity tests.

2. The fractions still containing water were eliminated for the present.

3. The time limit does not permit a final redistillation. The products obtained possess properties which approach the best results given by standard authorities. Any discrepancies prove that the products are not yet pure enough. The variations occur in:

1. The large range in boiling temperature.
2. In refractive indices.
3. Several results in specific gravity.

4. The test for primary, secondary and tertiary alcohols shows that,

- a. Primary alcohols are present in fractions V, VI, VII, VIII, IX, XV to the extent of 927.9^{cc}; which formed 37.12 per cent of the total amount.
- b. Secondary alcohols are present in fractions III, IV, XIII to the extent of 846.9^{cc} which formed 33.91 per cent of the total amount.
- c. Tertiary alcohols are present in fractions I, II, XII to the extent of 295.5^{cc} which formed 1.83 per cent of the total amount.

Using the Beilstein and Sharples boiling temperatures to identify the individual alcohols the following results were obtained:

1. N. Amyl Alcohol 67.5^{cc}; 2.07 per cent
2. 2 Methyl Butanol (1) 111.0^{cc}; 4.044 per cent
3. 2 Methyl Butanol (4) 228.2^{cc}; 9.138 per cent
4. Pentanol (2)
5. 2 Methyl Butanol (3) 529.7^{cc}; 21.21 per cent

It will be noted that only the fractions closest to the correct temperatures were taken. Without doubt the redistillation of the intermediate fractions would separate the mixtures into the various pure alcohols thus increasing the percentage of identified alcohols. It is safe to conclude that the results are correct as approximations since the properties of the identified alcohols agree closely with those of the Sharples Corporation.

Table IX compares the boiling point of the alcohols obtained in the present investigation with the boiling point recorded in Beilstein and by the Sharples Solvent Corporation.

2 Methyl Butanol Act. (5)	113	110-117, III	129.7
Tertiary	102	102-106, I	
		107-110, II	104.8

INGREDIENTS PRESENT IN ADDITION TO THE

AMT. ANALYSIS

TABLE IX

Identification of Alcohols

	Beilstein	Sharples	Present Results	
	Boiling Temperature °C	Boiling Temperature °C	Boiling Temperature °C	No. c.c.
Primary:				
N. Amyl Alcohol	137	138	136, IX	67.5
2 Methyl Butanol (1) Act.	129	128	127-8, XV	111
2 Methyl Butanol (4) Act.	132	130.5	128-30, VII	
2 2 di Methyl Propanol (1)	113		130-133, VIII	228.2
Secondary				
Pentanol (2) Act.	119	119	117-119, IV	232.2
Pentanol (3)	115	115.7		
2 Methyl Butanol Act (3)	113		110-117, III	529.7
Tertiary				
	102		102-108, I	
			107-110, II	204.5

- The density was .807 against water at 20°C.
- This .001 difference readily accounted for, since one alcohol of which the liquid had the spec. grav. was present.
- The index of refraction was 1.462 vs. 1.455 for water.
- The boiling point of the pure liquid was 99°.

From these properties, the presence of water was verified.

2. The Chlorine Content.

A second substance which caused the higher fractions to vary somewhat in boiling temperatures and refractive indices was chlorine.

To identify this chlorine the halogen test or sodium test was used. The expected precipitate appeared. That the precipitate was chlorine is clear, since chlorine is the only halogen used in producing the amyl alcohols.

INGREDIENTS PRESENT IN ADDITION TO THE

AMYL ALCOHOLS

1. The Water Fraction.

Two substances appeared in the process of distillation. These were water and chlorine.

The water appeared in every distillation which had a boiling temperature near 100°C. The entire portion of water containing alcohol was separated from the mixture proper. Because of the limited space of time the last fractions were set aside for future analysis. The water appeared as a second layer below the alcohols. All the large fractions were extracted and tested for identity. The proofs that this layer is water are:

1. It was heavier than alcohol.
2. It boiled over with fractions of 100° and below when the steam heat was used.
3. Its presence shows that it is somewhat soluble in alcohol. This is in accordance with fact.
4. The density was .997 against water at .996 at 23°C.
5. This .001 difference readily accounted for, since some alcohol of which the liquid had the odor, still was present.

The index of refraction was 1.362 vs. 1.333 for water.

The boiling point of the pure liquid was 99°.

From these properties the presence of water was verified.

2. The Chlorine Content.

A second substance which caused the higher fractions to vary somewhat in boiling temperatures and refractive indices was chlorine.

To identify this chlorine the hologen test on sodium fusion was used. The expected precipitate appeared. That the precipitate was chlorine is clear, since chlorine is the only hologen used in producing the amyl alcohols.

TESTS AND PROPERTIES

I. Amyl Alcohol.

Absorption of light in TABLE X

Volume change The Portion Containing Water

	Amt. of Alc. (c.c.)	Amt. of Water (c.c.)
1st Distillation	3,750	25
2nd Distillation		95
3rd Distillation		30
		<u>150</u>

Temperature °C	Index of Refraction	No. of c. c.
87- 90	1.397	13
90- 91	1.397	16
91- 92	1.3973	10
92- 93	1.3975	18
93- 94	1.3984	14
94- 95	1.3984	16
95- 96	1.399	15.5
96- 97	1.400	15
97- 98	1.4005	19.5
100-101	1.402	27.5
101-102	1.402	12
103-104	1.4026	23
105-106	1.404	18
106-107	1.404	12.5
108-109	1.4045	21
110-111	1.4052	27
111-112	1.4055	19
112-114	1.406	18.5
114-115	1.4065	12.5
115-118	1.407	17
118-120	1.4075	12.5
		<u>357.5</u>

Soluble in 8 parts of H₂O.

Tryscopic behavior in alcohol-water mixture. Refractive surface coated of various organic substances.

The alcohol is strongly soluble in water. Absorption of light in ultraviolet light in the presence of SO₂, into carbon and acetaldehyde.

VI. 2 Methyl Butanol (3).

B. Dextro-rotatory - soluble in alcohol.

TESTS AND PROPERTIES

I. Amyl Alcohol.

Absorption of light in the ultraviolet.

Volume changes when mixed with CHCl_3 .

Thermal changes when mixed with CH_3 .

Insoluble in water.

Ternary mixture - possible amyl alcohol; H_2O

MiOH
OH
propy OH

II. Pentanol (2)

A. Inactive form:-

a. Solubility in H_2O - one part in 6 of H_2O .

B. Dextro Rotatory:

a. Prepared by forming phthalic acid meno ester followed by
1 racime, a strychnine.

b. Soluble in beiszene.

c. Soluble in absolute alcohol.

III. Pentanol (3)

IV. 2 Methyl Butanol (1)

B. Laevo-rotatory - absorption of light in ultraviolet.

V. 2 Methyl Butanol (2)

Properties very well investigated.

Soluble in 8 parts of H_2O .

Kryoscopic behavior in nitrobenzene; in aniline; and aniline-water mixture. Influence on surface tension of various organic substances.

The alcohol in aqueous solution decomposes in the ultraviolet light in the presence of FeCl_3 into acetone and acetaldehyde.

VI. 2 Methyl Butanol (3).

B. Dextro-Rotatory - soluble in alcohol.

VII. 2 Methyl Butanol (4)

TABLE XI

The Amyl Alcohols

The commercial product is the best investigated alcohol but most data are given for the impure product. The properties

in the preceding table are for the pure synthetic inactive alcohol.

Name	Boiling Point	D ₂₀	n _D ²⁰	A _D ²⁰
Amyl Alcohol				
Pentanol (1)	Soluble in 50 parts of H ₂ O - Becomes turbid at 50 °C.			
Primary N Amyl Alcohol	K 740m.m. 137.5-138 °C	D ₂₀ ²⁰ = 0.8162		
N. Amyl Alcohol	K 740m.m. 137 °C	D ₂₀ ²⁰ = 0.8168		
	K 13m.m. 49-50 °C	D ₂₀ ²⁰ = 0.89918		
Pentanol (2)	K 760m.m. 115.9 °C	D ₂₀ ²⁰ = 0.811	n _D ²⁰ = 1.407	A _D ²⁰ = -0.139
Methyl Propyl Carbinol		D ₂₀ ²⁰ = 1.3605	n _D ²⁰ = 1.4127	
Sec. Amyl Alcohol				
A - Inactive Form				
B - Optically Rotatory	K 118.5-119.5 °C	D ₂₀ ²⁰ = 0.8189	n _D ²⁰ = 1.4053	(Optical Rot.) A _D ²⁰ = -15.30
C - Laevo Rotatory	No properties known			
Pentanol (3)	K 750m.m. 115.1-1.5 °C	D ₂₀ ²⁰ = 0.8157	n _D ²⁰ = 1.4036	
Diethyl Carbinol	K 750m.m. 115.53 °C		n _D ²⁰ = 1.41243	
2 Methyl Butanol (1)				
Sec. Butyl Carbinol				
A - Inactive Form	K 740m.m. 128.5-129.5 °C	D ₂₀ ²⁰ = 0.8341		
		D ₂₅ ²⁵ = 0.8178		
B - Laevo Rotatory or				
c. Amyl alcohol	K 128 °C	D ₂₀ ²⁰ = 0.816		
C - Laevo Rotatory or				
e. Amyl alcohol	No constant known			

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TABLE XI

The Amyl Alcohols

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Name	Boiling Point	Density	Refractive Index	Viscosity
I. Amyl Alcohol				
Pentantol (1)	K 758m.m. 137-137.5 °C	D-0/0--0.8296	N-20/D = 1.4101	
Primary N Amyl Alcohol	K 749m.m. 137.5-138 °C	D- $\frac{20}{20}$ --0.8162		
N. Amyl Alcohol	K 740m.m. 137 °C	D- $\frac{20}{20}$ --0.8168		
	K 13m.m. 49-50 °C	D- $\frac{25}{25}$ --0.80915		
II. Pentanol (2)				
Methyl Propyl Carbinol	K 760m.m. 118.9 °C	D- $\frac{19}{4}$ --0.811	N- $\frac{19}{D}$ = 1.407	A 25C--0.039 gm cm/sec
		D- $\frac{20}{D}$ --1.8303	N- $\frac{20}{D}$ = 1.4127	
Sec. Amyl Alcohol				
A - Inactive				
Form:				
B - Dextro-Rotatory	K 118.5-119.5 °C	D- $\frac{12.3}{4}$ --0.8169	N- $\frac{20}{D}$ = 1.4053	(Optical Rot.) A- $\frac{19}{D}$ -- 13.86°
C - Laevo Rotatory	No properties known			
III. Pentanol (3)				
Diethyl Carbinol	K 750m.m. 116.1-.3 °C	D- $\frac{20}{4}$ --0.8157	N- $\frac{20}{D}$ = 1.40565	
	K 760m.m. 115.53 °C		N- $\frac{14.7}{D}$ = 1.41243	
IV. 2 Methyl Butanol (1)				
Sec. Butyl Carbinol	No properties given			
A - Inactive				
Form	K 749m.m. 128.5-129.5/ °C	D- $\frac{0}{0}$ = 0.8341		
		D- $\frac{25}{25}$ = 0.8179		
B - Laevo-Rotatory or				(Optical Rot.)
d. Amyl alcohol	K 128 °C	D- $\frac{20}{4}$ --0.816		A- $\frac{20}{D}$ --5.90°
C - Dextro-Rotatory or				
e. Amyl alcohol	No constant known			

TABLE XI

Name	Boiling Point	Density	Melting Point	Viscosity
V. 2 Methyl Butanol (2)	K 760m.m. 102-103 °C	$D_{\frac{25}{4}} = -0.8066$		at 25°C -- 0.036997 gm/cm/sec.
Dimethyl Ethyl Carbinol	K 766m.m. 102.3-103 °C	$D_{\frac{50}{4}} = -0.7824$	M.P. -8.4°C	at 50°C -- 0.01401 gm/cm/sec.
Tertiary Amyl Alcohol		$D_{\frac{85}{4}} = -0.7441$	M.P. -12°C	at 85°C -- 0.00573 gm/cm/sec.
Amylene Hydrate				
VI. 2 Methyl Butanol (3)				
Methyl Iso Propyl Carbinol				
Sec. Amyl Alcohol	K 760m.m. 112.9-113.9 °C			at 25°C -- 0.0351 gm/cm/sec.
A - Inactive Form	K 768m.m. 111.3-111.5 °C			
B - Dextro- Rotatory	K 110-112 °C	$D_{\frac{15.8}{4}} = -0.8225$ $D_{\frac{51}{4}} = -0.7916$ $D_{\frac{71}{4}} = -0.7722$	(Refractive Index) $N_{\frac{20}{D}} = 1.3973$	(Optical Rot.) $A_{\frac{19}{D}} = 4.020$
b C - Leava Rotatory:	No properties given			
VII. 2 Methyl Butanol (4)				(Viscosity)
Iso-Butyl Carbinol	K 760m.m. 132.00 °C	$D_{\frac{0}{0}} = -0.82390$		at 15°C -- 0.04810 gm/cm/sec.
Prim. Iso Amyl Alcohol	K 762m.m. 130-130.5 °C	$D_{\frac{15}{4}} = -0.81289$ $D_{\frac{30}{4}} = -0.80175$		at 30°C -- 0.02960 gm/cm/sec.

TABLE XI

Name	Boiling Point	Vapor Tension
VIII. 2,2-Dimethyl Propanol (1)	K 113-114 °C	16m.m. - 20 °C 50.5m.m. - 46 °C
Tert. Butyl Carbinol		533.5m.m. - 100 °C
Primary		
N. Amyl Alcohol	137 °C	
2 Methyl Butanol (1) (Act)	129 °C	
2 Methyl Butanol (4) (Act)	132 °C	
2,2-di Methyl Propanol (1)	113 °C	
Secondary		
Pentanol (2) (Act)	119 °C	
Pentanol (3)	115 °C	
2 Methyl Butanol (3) (Act)	113 °C	
Tertiary		
2 Methyl Butanol (2)	102 °C	

SUMMARY

The subject matter of this paper is the analysis of a commercial mixture of pentanols.

1. A history of the preparation and properties of the amyl alcohols is given.
2. A discussion of fractional distillation and the special apparatus devised for this experiment, is made.
3. Special properties, such as, specific gravity and refractive indices are compared with those in a standard table.
4. Special tests to verify the existence of primary, secondary and tertiary alcohols, and the specific alcohols present as such, are given.
5. The presence of foreign substances are identified.

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