ENCLOSURE (B) 9

STUDIES ON THE COMPOSITION OF PYROLIGNEOUS LIQUOR AND ITS USES

by

CHEM. ENG. LIEUT. SAKOTA

Research Period: 1944-1945

Prepared for and Reviewed with Author by
the U. S. Naval Technical Mission to Japan

December 1945

ENCLOSURE (B)9

LIST OF TABLES AND ILLUSTRATIONS

ENCLOSURE (B)9

SUMMARY

The pyroligneous liquor obtained by dry-distillation of pine roots was found to be composed of 4% organic acids, (mostly acetic acid) and mixed ketones. The acidic substances were precipitated as Ca-salts and subjected to dry distillation. The ketone oil thus obtained was 0.06% by vol. of the pine roots and had an octane value of 97 clear, and above 100 with 0.15% lead.

I. INTRODUCTION

A. History of Project

From August 1944 to end of war, research was done on the pyroligneous liquor obtained in yield of 30-50% from the dry distillation of pine roots. This material contains small amounts of acetone and methanol, but insufficient to justify recovery of these compounds. Besides these substances, the pyroligneous liquor contains 4% of organic acids, consisting mostly of acetic acid with some propionic, butyric and other acid homologues. These acid substances were known to be utilized in France for the preparation of "ketone oil".

B. Key Research Personnel Working on Project.

Chem.Eng. Commander H. FUJIMOTO
Chem.Eng. Lieutenant N. SAKOTA

II. DETAILED DESCRIPTION

A. Test Apparatus and Procedure

100 l. of concentrated pyroligneous liquor (d_4^{15} 1.03) were neutralized with Ca-hydroxide, 5% in excess, and filtered. The filtrate was concentrated and evaporated to dryness in open direct-fired steel evaporation dishes, 50 kg of Ca-cake was then heated at 370° for 8 hr. in the apparatus, shown in Figure 1(B)9, which contains 5 iron dishes. The distillate was then rectified several times to separate water from the ketone oil.

B. Experimental Results.

An analysis of the pyroligneous liquor from dry distillation of pine roots is given in Table I(B)9. The crude Ca-salt cake was composed of Ca-acetate 30-40%, tarry matter abbve 10%, and water 5-10%. The tarry matter had a density of 0.780. Properties of the ketone oil obtained by several distillations of this tarry matter are as follows:

Sp. Gr. d_4^{15}	0.7804
Aldehyde(%)	0.01
Acid value	0
Distillation (°C)	
I.B.P.	52
10%	54
20%	55
30%	56
40%	58
50%	68
60%	72
70%	87

ENCLOSURE (B)9

80%	95
90%	107
97%	120
Dry point	135
Octane No. Clear	97
Octane No. 15% Pb	100

A material balance is given in Figure 2(B)9. Preliminary results on analysis of the ketone oil indicated the following approximate composition.

Acetone	50%
Methyl-ethyl-ketone	10%
Higher ketones	20%
Balance	10%
Total	100%

III. CONCLUSIONS

The pyroligneous liquor from pine roots was composed mostly of organic acids of the fatty series, with some ketones and methanol. The organic acids, separated as Ca-salts, were subjected to dry distillation, and the tar, or so-called ketone oil, showed an octane value of 97 clear, and above 100 with lead.

Table I(B)9
DISTILLATION AND PROPERTIES OF PYROLIGNEOUS LIQUOR

Time	D. Distil'n. Temp. °C.		Properties of Pyroligneous Liquor				
	Oven I	Oven II	Sp. Gr. d_{15}^{15}	Org. Acid as Acetic Acid (%)	Acetone (%)	Methanol (%)	Dissolved Tar (%)
07 20							
10 20	70	95	1,010	0.87	0.21	0.48	2.6
11 20	70	95	1,012	1.08	0.21	0.67	4.0
12 20	110	115	1,015	1.97	0.20	0.76	3.8
13 20	185	145	1,017	4.42	0.21	1.03	4.0
14 20	260	200	1,018	4.27	0.21	1.02	7.5
15 20	265	325	1,032	6.27	0.19	1.01	8.0

ENCLOSURE (B)9

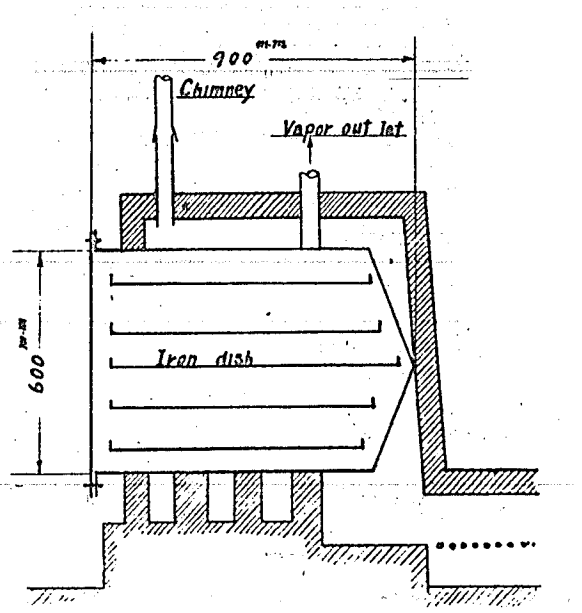

Figure 1(B)9
APPARATUS FOR CARBONIZATION OF CA-ACETATE

ENCLOSURE (B)9

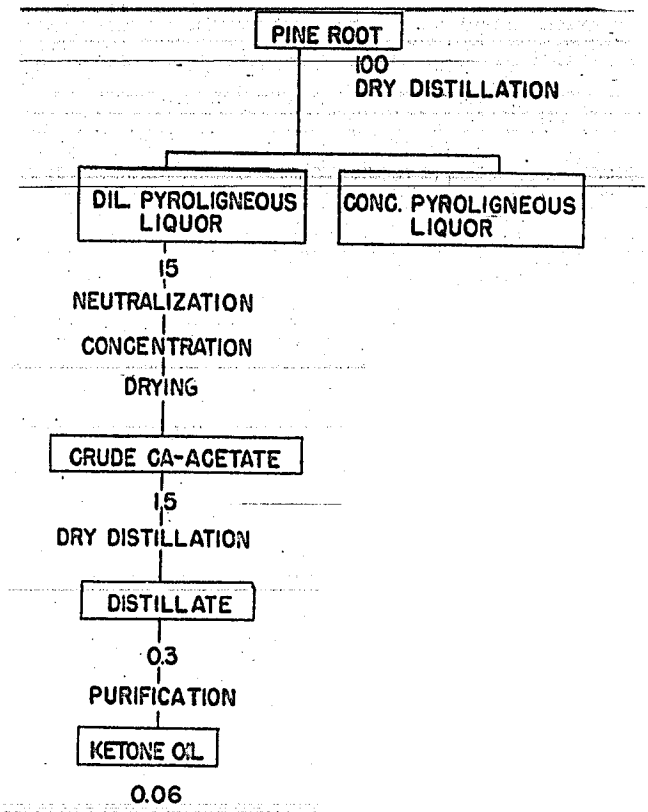

Figure 2(B)9
MATERIAL BALANCE
(yields in wt.%)