

NRL REPORT NO. C-3299

**FILTER PAPER STUDIES IV  
EFFECT OF REPLACING ESPARTO  
WITH VISCOSE RAYON FIBER**

H. W. Knudson and S. J. Pasternak

Hollingsworth & Vose Company  
East Walpole, Massachusetts

June 10, 1948

Approved by:

Dr. P. Borgstrom, Superintendent, Chemistry Division



**NAVAL RESEARCH LABORATORY**

CAPTAIN H. A. SCHADE, USN, DIRECTOR  
**WASHINGTON, D.C.**

APPROVED FOR PUBLIC  
~~RELEASE - DISTRIBUTION~~  
UNLIMITED

**DISTRIBUTION**

<b>BuShips</b> Attn: Code 688	1-7
<b>BuMed</b>	8-9
<b>ONR</b> Attn: Code N482	10-11
<b>Dir., USNEL</b>	12-13
<b>Med. OinC, NMRI, Bethesda</b>	14-15
<b>Atomic Energy Commission</b> Attn: Technical Library	16-18
<b>OCSigO</b> Attn: SIGTM-S	19
<b>CO, SCEL</b> Attn: Dir. of Engineering	20-21
<b>Chemical Corps, USA</b> Attn: Office of the Chief	22
Attn: Technical Command, Army Chemical Center	23-24
Attn: Med. Div., Army Chemical Center	25
Attn: CO, Naval Unit, Army Chemical Center	26
Attn: CO, Camp Detrick, Frederick, Md.	27-28
<b>Hollingsworth and Vose Company (Via CO, ONR, Boston)</b>	29-30
<b>RDB</b> Attn: Technical Library	31
Attn: Navy Secretary	32
<b>Science and Technology Project</b> Attn: Mr. J. H. Heald	33-34
<b>NRL Secret File Room</b>	35-40

## PREFACE

This Naval Research Laboratory report consists of two Research and Development reports written by the Hollingsworth and Vose Company, East Walpole, Massachusetts, on Navy Contract N7-ONR-430.

These reports are identified as follows:

“Research and Mill Trial on the Development of a Domestic Substitute for Esparto Fiber in the Navy Type H-60 Filter Paper”, First Quarter of Contract N7-ONR-430, referred to as the N-7 Trial.

“Research and Mill Trial on the Development of a Domestic Substitute for Esparto Fiber in the Navy Type H-60 Filter Paper”, Second Quarter of Contract N7-ONR-430, referred to as the N-8 Trial.

This report concludes the work on the first half of the contract. Filter paper studies are being continued by the Hollingsworth and Vose Company and additional reports will be published when received.

**This page intentionally blank.**

## CONTENTS

Abstract	vi
----------	----

### N-7 TRIALS

INTRODUCTION	1
LABORATORY WORK	1
THE MILL RUN	5
RECOMMENDATIONS	8

### N-8 TRIALS

INTRODUCTION	9
LABORATORY WORK	9
THE MILL RUN	12
RECOMMENDATIONS	16

## ABSTRACT

This is an interim report describing in detail two mill runs and the associated laboratory research work on the effect of replacing esparto with viscose rayon fiber in the Navy H-60 filter paper. It is shown that an exceedingly superior filter paper can be manufactured using causticized viscose in place of esparto fiber. In general, the viscose-asbestos filter paper exhibits no deleterious "break" or "relaxation" effects. Further study is required to determine optimum conditions of the causticizing treatment, washing, viscose fiber size and length, and general mill procedure.

**RESEARCH AND MILL TRIAL ON THE DEVELOPMENT OF  
A DOMESTIC SUBSTITUTE FOR ESPARTO FIBER  
IN THE NAVY TYPE H-60 FILTER PAPER  
(N-7)**

**INTRODUCTION**

This report is a summary of the work done for the first quarter of Contract N7-ONR-430 (July 1, 1947 to October 13, 1947). A statement of the general objectives and an outline of previous work accomplished are contained in past reports of this series.\*

The mill trial reported here is designated as the N-7 trial, H & V Lot No. 167, dated October 13, 1947.

On the basis of laboratory handsheet data, it was decided to work with a furnish of causticized viscose rayon and asbestos, or causticized viscose rayon, rope, and asbestos. Handsheets made from this furnish showed excellent smoke filtration efficiency and impressive tensile strength. Viscose rayon will be referred to as viscose.

**LABORATORY WORK**

It has been found that with proper causticizing, 1/8 in. rayon flock of 1.5 denier would curl considerably. This curled fiber could accommodate a considerable amount of asbestos and when felted on conventional paper-making equipment produced a sheet possessing a surprising amount of strength.

Following is a summary of handsheet data obtained from furnishes of 75% causticized viscose (1/8 in. flock, 1.5 denier) and 25% Blue Bolivian asbestos.

**Effect of Caustic Concentration**

A 5:1 weight of causticizing liquid to weight of viscose was used in all cases for a causticizing period of two hours.

---

\* H. W. Knudson, "Filter Paper Studies I. Effect of Replacing Esparto with Yucca Fiber," NRL Report C-3172, September 1947; H. W. Knudson and S. J. Pasternak, "Filter Paper Studies II. Effect of Replacing Esparto with Wood Pulp Fiber," NRL Report C-3225, January 1948, and "Filter Paper Studies III. Effect of pH and Added Electrolytes," NRL Report C-3226, January 1948.

TABLE I

Effect of Caustic Concentration on Performance of Handsheets

Caustic Conc. (Wt. % NaOH)	Tensile (#/in.)	Resistance (mm H <sub>2</sub> O)	DOP Penetration (%)	% Efficiency
10.0	4/16	87	.035	3.98
12.5	5/16	98	.008	4.18
15.0	10/16	102	.006	4.14
17.5	11/16	107	.005	4.02
20.0	12/16	118	.003	3.90
22.5	15/16	111	.005	3.87
25.0	1	118	.003	3.83
27.5	1 1/16	114	.004	3.86

As can be seen from Table I, the tensile strengths of the sheets increase with concentration of caustic. The variations in resistance are caused somewhat by the variations in the weight of the sheet; however, resistance seems to increase slightly with concentration of caustic. Though there are variations in the efficiencies, these variations are not in excess of variations encountered in data from any single type furnish. However, it is reasonable to assume that there is probably a slight decrease in efficiency at the higher caustic concentrations due to the increase in resistance.

#### Effect of Causticizing Time on Tensile Strength

A 5:1 weight of causticizing liquid to weight of viscose at the various caustic concentrations was used.

TABLE II

Effect of Causticizing Time on Tensile Strength of Handsheets

Caustic Conc. (Wt. % NaOH)	Time (Min.)	Tensile (#/in.)	Time (Min.)	Tensile (#/in.)	Time (Min.)	Tensile (#/in.)
17.5	5	8/16	60	9/16	120	11/16
20.0	5	12/16	60	12/16	120	12/16
22.5	-	---	60	14/16	120	15/16
25.0	5	1	60	1	120	1

Apparently at caustic concentrations above 17.5% and below 25%, time of causticizing has no appreciable effect on the tensile strength of the sheet.

#### Effect of the Ratio of Causticizing Liquor to Fiber on Tensile Strength

A 20% caustic solution was used to causticize viscose for a period of 15 minutes.

TABLE III

## Effect of Caustic/Fiber Ratio on Tensile Strength of Handsheets

Causticizing Liquor to Fiber Ratio (Wt.)	Tensile (#/in.)
5.0:1	10/16
7.0:1	1 9/16
7.5:1	2 3/16
10.0:1	2 4/16
12.5:1	2 5/16
15.0:1	2 10/16

Table III shows that tensile increases with increased caustic liquor ratio over the ranges tested. A sharp increase in tensile is evident between ratios of 5:1 and 7.5:1, and beyond this ratio, tensile increases slowly but steadily with increases in ratio to 15:1.

## Effect of Temperature on Causticizing

All the above data were obtained by causticizing at room temperature. Laboratory experiments indicated that moderate temperature changes did not affect causticizing noticeably. Though complete data were not obtained it was ascertained that causticizing would proceed normally over the temperature range that might be encountered in the mill run.

## Discussion of Causticizing

In summary, the tensile strength of a 75% causticized viscose and 25% asbestos hand-sheet increases with the concentration of caustic and the ratio of causticizing liquor used to treat the viscose. Evidently temperature during treatment and length of treatment do not affect the causticizing in the ranges considered.

At the higher caustic concentrations and causticizing ratios, the viscose tends to form a gelled mass which may be difficult or impossible to wash. The tendency to form a gel is enhanced by excessive agitation and slow dilution. If the concentration of caustic be permitted to increase beyond a certain maximum concentration, the viscose fibers tend to agglomerate during washing, plug the washing screen, and prevent removal of the residual caustic. If the concentration of caustic be high enough during dilution, mild agitation will cause complete gellation of the mass, and if possible to wash, the viscose becomes hard and brittle and a major portion of it loses its fibrous structure. Also if the caustic is not removed completely the resulting sheet is too harsh and too high in resistance.

Evidently the choice of causticizing conditions becomes a balance between desired strength and a minimum of operational difficulties plus a safety margin. It was decided to set tentatively the operating conditions at 20% caustic (NaOH) at a 10:1 causticizing-liquor-to-viscose ratio on a weight basis.

## Summary of Handsheet Data

Following is a summary of data from handsheets made of 25% asbestos and 75% causticized viscose. The viscose was causticized with 20% caustic at 10:1 causticizing liquor ratio. After two hours the mass was added to approximately 20 times its weight of water and washed free of caustic.

Table IV is a summary of the physical properties of the paper.

TABLE IV

## Physical Properties

Caliper	0.039 in.
Ream Weight	135 lb.
Tensile	3.1 lb.

The above values are average for several handsheets made. The tensile is well above specifications, and the sheet appeared to have good handling and folding characteristics.

Typical smoke filtration performance of the sheet is recorded in Table V.

TABLE V

## Performance of Handsheets

Sample	DOP Penetration (%)	Resistance (mm H <sub>2</sub> O)	Efficiency (%)
1	.005	115	3.74
2	.015	101	3.79
3	.003	120	3.77
4	.003	116	3.90

The initial efficiencies recorded are excellent, probably as high as any ever encountered. Efficiencies as high as the above have been approached in the past, but never before was it possible to reproduce such results at will. The above results are easily reproducible.

Table VI is a record of effect of DOP exposure on smoke penetration of a sheet.

As can be seen, no "break" occurred with exposure to DOP over the test period. The efficiency of the sheet during the test did drop slightly, but this was due entirely to increase in resistance with exposure, since penetration actually dropped with exposure. This paper had the best "non-break" performance ever recorded at this laboratory.

Upon checking the asbestos distribution and sheet formation by means of DOP penetration vs flow rate, it was found that very good asbestos distribution and sheet formation were realized.

As a further possibility, rope fiber was added to this type of furnish. Table VII is the performance record of typical sheets containing 20% rope.

TABLE VI

## Effect of DOP Exposure on Performance

Time of Exposure (Min.)	DOP Penetration (%)	Resistance (mm H <sub>2</sub> O)
0	.003	120
2	.003	120½
4	.003	121
6	.003	122
8	.003-	122½
10	.003-	123
45	.002	130
90	.001	141

TABLE VII

## Performance of Handsheets Containing 20% Rope

Sample	DOP Penetration (%)	Resistance (mm H <sub>2</sub> O)	Efficiency (%)
1	.028	102	3.48
2	.020	105	3.52
3	.020	106	3.49

As can be seen there is a slight decrease in efficiency with the addition of 20% rope, but this decrease is not exceptional, and since in practice 10% rope would be considered the maximum rope addition, it was felt that, if greater strength were required, small amounts of rope could be added to the furnish without markedly decreasing the filtering efficiency.

On the basis of the preceding data it was decided to make a machine run with a furnish of 25% asbestos and 75% viscose, causticized as outlined previously.

## THE MILL RUN

## Preparation of Stock for Mill Trial

The predetermined amount (10 times the weight of the fiber) of 20% caustic solution was made up in a rotary boiler and 650 pounds of viscose 1/8 inch flock of 1.5 denier were added and the rotary was closed. The rotary was turned over for 10 minutes, and the fiber was dumped into the beater as washing permitted.

## Manufacturing Data

One beater was furnished with 90 pounds of asbestos. The asbestos was given a hard beat for approximately 20 minutes after which it was fairly well defibered. The slurry was dropped to the beater chest, and the beater was refilled with water.

The causticized viscose was added slowly to the beater just before the rotating roll, and the stock was continually washed. The stock did not plug the washing screen and no other difficulties were encountered. After three hours of continuous washing, the viscose was free of caustic and was dropped into the beater chest with the asbestos. It was estimated that about 150 pounds of viscose were lost by solution and through the washer screen during washing, and that approximately 500 pounds were dropped into the beater chest.

The resulting furnish consisted of:

500 lb. Causticized viscose	85%
90 lb. Asbestos	15%
<u>590 lb.</u>	<u>100%</u>

The stock was "backed-up" in the Jordan (sent through under pressure with the Jordan plug backed off) to insure thorough mixing and was sent to the machine chest to be felted into paper. Approximately 10% more asbestos was added to the stock from the auxiliary asbestos feed line as the stock was fed to the Fourdrinier.

The last half of the stock had approximately 10% rope added to it in an attempt to increase the tensile strength.

In an effort to minimize calendering, all but one calender roll were jacked up permitting the use of one light roll to soften up the sheet.

No great difficulty was experienced in formation and handling of the paper over the machine once steps were taken to compensate for the high shrinkage of the sheet during drying.

#### Performance of the Paper

Average values of physical tests of this paper are recorded in Table VIII.

TABLE VIII

Physical Properties of Mill Run Sheets

Caliper (in.)	Ream Weight (lb.)	Tensile (Length) (lb.)	Moisture (%)	Comment
.045	156	2	5.3	First half of run
.039		2.75		Second half with rope

Allowances should be made for possible inaccuracy in the low ranges of these tests. As can be seen, the addition of 10% rope fiber increased the tensile from 2.0 to 2.75 pounds.

Table IX is a record of performance characteristics of the paper as it was sampled directly from the machine over a period of about three hours.

From Table IX, based on initial efficiencies, this is undoubtedly the best filter paper ever made on a machine at this company. Further, as seen from the data, the addition of 10% rope did not abnormally decrease the efficiency of the filter but did increase the tensile

strength. Evidently small amounts of rope may be added to the furnish without an appreciable loss in efficiency but with a material increase in strength.

TABLE IX

## Performance Data of Samples Taken Directly from Paper Machine

Sample	DOP Penetration (%)	Resistance (mm H <sub>2</sub> O)	Efficiency (%)	Comment
1	.008	111	3.69	Start of rope addition
2	.008	111	3.69	
3	.010	108	3.71	
4	.010	110	3.64	
5	.005	118	3.64	
6	.010	105	3.81	
7	.010	105	3.81	
8	.011	103	3.84	
9	.008	114	3.59	
10	.009	116	3.49	

## Effect of DOP Exposure

Table X shows the effect of DOP exposure on smoke penetration.

TABLE X

## Effect of DOP Exposure on Performance of N-7 Trial Sample

Time of Exposure (Min.)	DOP Penetration (%)	Resistance (mm H <sub>2</sub> O)
0	.005	118
1	.005	118
2	.005	119
5	.005	119
10	.005	120

This paper showed little or no "break" with exposure to DOP smoke during the test.

## Performance vs Flow Rate

Through a study of DOP penetration vs flow rate, imperfections in sheet formation, such as pinholes, become apparent. Penetration will decrease normally with decreased flow rate if good sheet formation and asbestos distribution are realized. Table XI is a record of such a study.

It is evident that good asbestos distribution and sheet formation were realized.

## Relaxation with Aging

The possibility of relaxation upon aging -- increased penetration accompanied by small decreases in resistance -- was checked, and typical results are recorded in Table XII.

TABLE XI

## Performance vs Flow Rate of N-7 Trial Sample

Flow Rate (1/m)	DOP Penetration (%)	Resistance (mm H <sub>2</sub> O)
85	.012	110
42 $\frac{1}{2}$	.008	55
21 $\frac{1}{4}$	.004	28

TABLE XII

## Effect of Aging on Performance of N-7 Trial Sample

Interval after Manufacture	DOP Penetration (%)	Resistance (mm H <sub>2</sub> O)
Direct from machine	.012	112
7 days	.012	110
11 days	.013	109

From the above data there is no doubt that relaxation did not take place in this paper. The variations in both penetration and resistance shown above are caused by sampling at different machine positions and different machine times.

## RECOMMENDATIONS

In view of the fact that the N-7 papers made from causticized viscose and asbestos and causticized viscose, rope, and asbestos exhibited such remarkable filtration properties, impressive strength, little or no "break", and no relaxation with aging, it is recommended that work be continued on this type of furnish. Attempts should be made to find the best causticizing conditions to give the desired strength and filtering efficiency with the minimum difficulty in mill production. Further attempts should be made to investigate the optimum length of flock and diameter of viscose fiber for this application.

If all the production difficulties can be brought under control, there is little doubt that the causticized rayon fiber can be substituted for the esparto fiber in the Navy H-60 type filter paper and at the same time produce a much improved filter material.

\* \* \*

RESEARCH AND MILL TRIAL ON THE DEVELOPMENT OF  
A DOMESTIC SUBSTITUTE FOR ESPARTO FIBER  
IN THE NAVY TYPE H-60 FILTER PAPER  
(N-8)

INTRODUCTION

This report is a summary of the work done for the second quarter of Contract N7-ONR-430 (October 13, 1947 to December 31, 1947). A statement of the general objectives and an outline of previous work accomplished are contained in past reports of this series.\*

The mill trial reported here is designated as the N-8 trial, H & V Lot No. 248, dated December 18, 1947.

From the N-7 mill trial it was found that causticized viscose served as an excellent substitute for esparto in type H-60 Filter Paper. Since the N-7 method of causticizing in the rotary boiler was considered awkward as well as hazardous for the operators, it was decided to attempt a simplification of the causticizing procedure. Further, since the 1.5 denier, 1/8 in. viscose flock was the only size of flock considered at that time, the subsequent investigation was planned to find the optimum fiber dimensions of diameter, and length.

LABORATORY WORK

From laboratory work it was found that viscose flock could be causticized at caustic concentration less than 20% if the consistency of the pulp were reduced from the 9% used in the N-7 run. The amount of actual caustic used by either process was approximately the same. Successful preliminary causticizing was done in the laboratory beater, and subsequent work revolved about perfection of beater causticizing.

By experimentation it was found that 1.5 denier, 1/8 in. flock could be causticized in the beater by an 8% caustic solution if the stock were at about a 4% consistency. It was found that the degree of causticizing was influenced by caustic concentration, pulp consistency, and degree and length of agitation.

The following procedure was used successfully in the laboratory beater. An 8% solution of caustic was made up in the beater. With the beater roll raised, enough viscose flock was added to give a consistency of about 4%. Circulation was continued until the desired degree of causticizing was reached as ascertained by sampling. After 20 minutes the causticizing was usually completed and was stopped by discontinuing circulation. The

---

\* NRL Reports C-3172, C-3225, and C-3226.

causticized mass was then diluted in three times its volume of water and was washed. The resulting product was the equivalent of the causticized fiber obtained in the manner described in the N-7 report.

Samples of viscose flock, 1.5 denier - 1/8 in., 1.5 denier - 1/4 in., 1.5 denier - 3/8 in., and 3 denier - 1/8 in., were obtained. Each type and combinations of the types were causticized and tested. The results of the tests are recorded below. Henceforth, the following designations for the viscose flock will be used:

Type "A" --- 1.5 denier - 1/8 in.  
 Type "B" --- 1.5 denier - 1/4 in.  
 Type "C" --- 1.5 denier - 3/8 in.  
 Type "D" --- 3 denier - 1/8 in.

#### Physical Tests

All of the following handsheets, except where designated otherwise, were made from furnishes of 25% Blue Bolivian asbestos and 75% causticized viscose flock (by weight) of varied dimensions in proportions as noted. No sheets of Type "C" fiber were made since this causticized flock would snarl so completely that it was impossible to form handsheets. Typical tensile strengths are recorded below.

TABLE I  
 Tensile Strengths of Various Handsheets

B.B. Asbestos (Wt. %)	Viscose (Wt. %)	Viscose Furnish (Parts by weight)	Tensile (Unpressed) (#/in. @ 130# R. W.)
25	75	100 "A"	1.5
25	75	100 "B"	1.4 ?
25	75	100 "D"	.6
25	75	50 "A", 50 "D"	1.0
25	75	80 "A", 20 "B"	1.3
25	75	80 "D", 20 "B"	.7
0	100	100 "A"	2.0
0	100	100 "B"	3.7
0	100	100 "D"	1.1

From the above tests it would appear that the Type "A" flock when felted with asbestos formed the strongest asbestos bearing sheet. It should be noted that these tensiles are for unpressed sheets; that is, during formation they were not wet pressed because it was found that more uniform handsheets were obtained in this manner. Though tensiles obtained by this method are not directly comparable to machine tensiles (usually machine tensiles are 1.5 to 2.5 times greater), this method does serve as a means to compare tensiles of a particular series of sheets. A question mark was inserted in Table I at the tensile of the sheet made from 25% asbestos and 75% Type "B" viscose flock as it was felt that this tensile was too low since on the basis of the tensiles of non-asbestos bearing sheets (Table I), the decrease in tensile of this type upon addition of asbestos was disproportionate with the decreases in tensile of the other types studied. This contention finds further support

in that handsheets of Type "B" asbestos bearing paper made in the past have had tensiles higher than that recorded in Table I.

**Filtration Tests**

Following is a summary of typical smoke filtration performance of the asbestos bearing sheets.

**TABLE II**  
Performance Data of Various Asbestos-Viscose Handsheets

B.B. Asbestos (Wt. %)	Viscose (Wt. %)	Viscose Furnish (Parts by Weight)	DOP Penetration (%)	Resistance (mm H <sub>2</sub> O)	Efficiency (%)
25	75	100 "A"	.007	108	3.84
25	75	100 "B"	.007	102	4.07
25	75	100 "D"	.003	105	4.30
25	75	50 "A", 50 "D"	.006	103	4.10
25	75	80 "A", 20 "B"	.006	108	3.91
25	75	80 "D", 20 "B"	.007	100	4.15

From Table II it appears that the asbestos bearing sheets having the highest efficiency were made with 75% Type "D" viscose and 25% Blue Bolivian asbestos. There is a slight decrease in efficiency with the substitution of 50 parts Type "A" or 20 parts Type "B" for an equivalent amount of Type "D" flock. This behavior is contrary to past experience with other furnishes where efficiency increased with decreased fiber diameter. The asbestos bearing sheets made with Type "B" flock also had very good efficiency in spite of the fact that fiber formation was very poor in these samples. This type furnish should be investigated further.

**"Break" Tests**

Following is a summary of "break" data on these samples.

**TABLE III**  
Effect of DOP Exposure on Performance of Asbestos-Viscose Handsheets

Time (Min.)	25% Asbestos 75% Viscose (100 "A")* DOP		25% Asbestos 75% Viscose (100 "B")* DOP		25% Asbestos 75% Viscose (100 "D")* DOP	
	Penetration (%)	Resistance (mm H <sub>2</sub> O)	Penetration (%)	Resistance (mm H <sub>2</sub> O)	Penetration (%)	Resistance (mm H <sub>2</sub> O)
0	.008	105	.008	104	.005	101
1	.008	105	.008	105	.004	101
2	.009	105	.009	105	.006	102
3	.009	106	.009	106	-	-
4	-	-	.008	106	-	-
5	.009	106	.008	107	.006	103
	75% Viscose (50 "A", 50 "D")*		75% Viscose (80 "A", 20 "B")*		75% Viscose (80 "D", 20 "B")*	
0	.008	101	.010	106	.013	93
1	.008	101	.010	106	.013	93
2	.009	102	.010	107	.014	93
3	.008	102	.012	107	.014	94
4	.009	102	.012	108	.014	94
5	.009	103	.012	108	.014	94

\* Parts by weight of various viscose flocks.

From Table III apparently all the samples exhibited remarkable resistance to "break." An attempt to differentiate between the sheets based on the above data would be impossible.

#### Performance vs Flow Rate

In an attempt to check mechanical perfection and asbestos distribution, DOP penetration vs flow rate were made. Desirable formation and asbestos distribution are normally indicated by a regular decrease in penetration with decreased flow rate. Table IV lists such data for these samples.

TABLE IV  
Performance vs Flow Rate

B.B Asbestos (%)	Viscose (%)	Viscose Furnish (Parts by Weight)	85 l/min.		42½ l/min.		21¼ l/min.	
			Res. (mm H <sub>2</sub> O)	DOP Pen. (%)	Res. (mm H <sub>2</sub> O)	DOP Pen. (%)	Res. (mm H <sub>2</sub> O)	DOP Pen. (%)
25	75	100 "A"	105	.008	53	.005	26	.003
25	75	100 "B"	101	.008	50	.005	25	.002
25	75	100 "D"	99	.010	50	.007	25	.003
25	75	50 "A", 50 "D"	99	.006	50	.004	26	.001
25	75	80 "A", 20 "B"	105	.006	53	.004	26	.001
25	75	80 "D", 20 "B"	98	.010	49	.006	25	.002

From this study it is evident that in all cases good asbestos distribution and mechanical perfection were realized.

#### Summary of Laboratory Work

In the series reported where the strongest asbestos bearing sheets were those made with Type "A" flock. This sheet also possessed a rather high filtering efficiency. It should be noted that on occasion sheets made with Type "B" viscose have had higher tensiles than those made with Type "A."

The highest filtering efficiency was obtained with the sheets made from Type "D" flock, but sheets formed from this material were so weak that such a furnish probably could not be handled on the machine. However, an asbestos-bearing furnish composed of 50 parts Type "A" and 50 parts Type "D" causticized viscose had a high filtering efficiency as well as enough strength to be handled on the machine.

On the basis of these experimental data it was decided to make a machine run with a furnish of:

- 25% Blue Bolivian asbestos
- 75% Causticized viscose
  - 50 parts "A" (1.5 denier 1/8 in.)
  - 50 parts "D" (3 denier 1/8 in.)

#### THE MILL RUN

##### Preparation of Stock for Mill Trial

A predetermined amount of 8% caustic solution (NaOH) was made up in the beater and 300 pounds of 1.5 denier - 1/8 in. flock and 300 pounds of 3 denier - 1/8 in. flock were added

to the beater just in front of the rotating roll, which was in the raised position throughout the entire operation. The resulting consistency was approximately 4%. The stock was circulated and samples were taken periodically. After 30 minutes the fiber was causticized to the proper degree. The caustic mass was dropped into three times its volume of water in the beater chest with the beater chest agitator running.

For the purpose of removing caustic, it was decided to run the stock over the Four-drinier wire removing the stock as wet broke, since previous conventional beater washing had led to considerable operating difficulty. At this point the fiber was returned to the beater for final washing to remove the last traces of caustic. The yield of causticized fiber was approximately 350 pounds (about 58%), but this yield is not considered representative of regular production running. There is a certain minimum loss of fiber for any normal quantity of fiber treated in this manner, and naturally this percentage loss would be smaller the larger the starting batch.

#### Manufacturing Data

When the causticized viscose was washed free of caustic the batch was dropped to the beater chest. The beater was furnished with 90 pounds of Blue Bolivian asbestos. The asbestos was given a hard beat for approximately 30 minutes after which it was well defibered. The asbestos was dropped into the beater chest and thoroughly mixed with the viscose.

The resulting furnish consisted of:

350 lb	Causticized viscose	80%
90 lb	Blue Bolivian asbestos	20%
<u>440 lb</u>		<u>100%</u>

The stock was "backed-up" in the Jordan (sent through under pressure with the Jordan plug backed-off) to insure thorough mixing and was sent to the machine to be felted into paper. Approximately 5% more asbestos was added to the stock from the auxiliary asbestos feedline as the stock was fed to the Fourdrinier.

To minimize calendering all but one calender roll were jacked up permitting the use of one roll to softenup the sheet.

The major difficulty in handling this paper was setting up the machine to allow for the considerable shrinkage of the sheet during drying. To insure better handling of this type furnish on the paper machine, more experience in handling causticized viscose on the machine would be necessary.

#### Performance of the Paper

Average values of physical tests of this paper are recorded in Table V.

Allowances should be made for testing inaccuracies, quite possible in these low tensile ranges. The tensile of 2 lb recorded above is not exceptionally high; however, probably no difficulty would be encountered in fabricating this material on the Navy gas mask canister.

Table VI is a record of the smoke penetration and air resistance characteristics of the paper as it was sampled directly from the machine over a period of about two hours.

TABLE V

## Physical Properties of Mill Run Sheets

Caliper	0.036 in
Ream Weight	128 lb
Tensile Length	2 lb
Cross	1 lb

TABLE VI

## Performance Data of Samples Taken Directly from Paper Machine

Sample	DOP Penetration (%)	Resistance (mm H <sub>2</sub> O)	Efficiency (%)
1	.010	100	4.00
2	.012	98	4.00
3	.020	97	3.82
4	.016	101	3.76
5	.011	112	3.54
6	.012	110	3.56
7	.014	108	3.56
8	.004	119	3.70
9	.004	120	3.67

The efficiencies recorded are somewhat higher than those for the N-7 Trial run. The large variations in efficiency (though a more or less normal occurrence) make it rather difficult to compare this furnish to the N-7 furnish (75% causticized 1.5 denier, 1/8 in. viscose flock and 25% Blue Bolivian asbestos). The lowest efficiency recorded above is not much greater than the lowest recorded for the N-7 run, but the highest efficiency recorded above is somewhat higher than the corresponding value for the N-7 run. This latter fact probably serves best to indicate the greater possibilities of the N-8 type furnish.

Based on the above data the new method of beater causticizing of viscose flock is a satisfactory substitute for the previous rotary boiler method.

## Effect of DOP Exposure

The rate of change in penetration and resistance of a sample exposed to DOP smoke is normally checked and reported as "break." The "break" data are recorded in Table VII.

The rate of "break" was exceedingly slow and is definitely satisfactory.

## Performance vs Flow Rate

A standard practice is to test the paper for pin holes and for poor asbestos distribution by measuring the smoke penetration at various flow rates through the sample. Table VIII presents these data on the N-8 paper.

TABLE VII

Effect of DOP Exposure on Performance of N-8 Trial Sample

Time of Exposure (Min.)	DOP Penetration (%)	Resistance (mm H <sub>2</sub> O)
0	.004	118
1	.005	119
2	.005	119
3	.005	119
4	.006	119
5	.005	119

TABLE VIII

Performance vs Flow Rate of N-8 Trial Sample

Flow Rate (l/m)	DOP Penetration (%)	Resistance (mm H <sub>2</sub> O)
85	.004	120
42½	.002	60
21¼	.001	30
85	.004	120

This paper showed a normal decrease in penetration with flow rate; hence it may be assumed that there were no pin holes and that the distribution of asbestos was good.

#### Relaxation with Aging

The possibility of relaxation upon aging -- increased penetration accompanied by small decreases in resistance -- was checked and typical results are recorded in Table IX.

TABLE IX

Effect of Aging on Performance of N-8 Trial Sample

Interval After Manufacture	Penetration (%)	Resistance (mm H <sub>2</sub> O)
Direct from machine	.004	119
5 hours	.004	118

From the above it is seen that the N-8 paper did not relax with aging for the time interval indicated. Referring to the N-4 Trial\*, in which a detailed study of aging was recorded, it was shown that if any relaxation takes place it is definitely apparent three hours after manufacture and probably reaches a maximum in from three to six hours after manufacture.

\* NRL Report C-3225.

## RECOMMENDATIONS

Based on the filtration results recorded in this run, the N-8 furnish probably made the best machine-made paper tested at this mill. However, the tensile strength of this paper was not exceptional, and work should be continued to improve it.

Evidently, beater causticizing of viscose flock is possible. This production procedure is simpler and permits better control than rotary boiler causticizing. From all evidence the resulting product from beater causticizing is equivalent to that from the rotary boiler. Work should be continued to increase the available information concerning beater causticizing.

Since this type of paper presents a difficult problem of handling on the paper machine, in view of the abnormal shrinkage, it is recommended that more experience in machine handling be planned for the future.

In view of the fact that only a relatively small number of available sizes of viscose flock have been evaluated, work should continue in obtaining and evaluating other possible sizes.

\* \* \*