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QUALITATIVE IDENTIFICATION AND QUANTITATIVE ESTIMATION  
OF NYLON IN THE PRESENCE OF SILK, WOOL AND COTTON

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QUALITATIVE IDENTIFICATION AND QUANTITATIVE ESTIMATION  
OF NYLON IN THE PRESENCE OF SILK, WOOL AND COTTON

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## INTRODUCTION

Nylon is the new textile fiber announced by Du Pont in the fall of 1938. It is the first organic textile fiber of an entirely mineral origin to be known to the world, being derived from coal, air and water. Scientifically it can be defined as a synthetic fiber having a protein-like structure built up by the polymerization of amides. Nylon is a generic name which covers all fibers or filaments capable of being drawn (spun) from polymeric amides.

Nylon has extraordinary properties of tensile strength, elastic recovery from elongation, and insensitivity to moisture which make it a superior textile fiber. It imparts increased strength to yarns and fabrics composed of nylon in mixture with other fibers. Although at present only small quantities of nylon have reached the textile market, an eight million dollar plant intended for commercial production is under construction by the Du Pont Company at Seaford, Delaware. Since it seems apparent that the cost of nylon will be relatively high because of the expensive processing necessary to its production, it may be expected that a great use will be made of it in mixture with other fibers to give added strength. For this reason it is important to have available methods for the qualitative identification and quantitative estimation of nylon in admixture with other fibers. This problem was undertaken for the purpose of discovering, if possible, workable procedures for such identification and estimation.

## HISTORICAL

According to United States Patent 2,130,948, the superpolyamide fibers called by the generic name nylon are hydrolyzed by hydrochloric acid to the dicarboxylic acid and the hydrochloride of the diamine from which the polyamide was formed. The patentee claims that fibers spun from poly-pentamethylene sebacamide (formed from pentamethylene diamine and sebacic acid) are completely resistant to the common organic solvents and can be immersed in boiling toluene for one week without noticeable effect. On heating with strong mineral acid, such as hydrochloric, hydrobromic, sulfuric, or phosphoric the fibers disintegrate and are hydrolyzed to sebacic acid and the mineral salt of pentamethylene diamine.

Von Bergen (10) has described the behavior of nylon in the burning test, on dry distillation and in reaction with certain chemicals. He states that the fiber is insoluble in boiling 5% caustic soda solution. He also found nylon to resist the carbonizing process, but to be somewhat weakened by it.

He points out the value of the microscope for identifying nylon in fiber mixtures. Nylon resembles cuprammonium rayon most closely in microscopical appearance, but can be distinguished from it by means of the swelling test. When measured in glycerine and water nylon shows no increase in width, while cuprammonium rayon swells 50% or more.

With the standard sulfuric acid test for the separation of wool and vegetable fibers, Von Bergen discovered that nylon

disintegrates to a white brittle mass, and that the method has to be modified as it takes more than fifteen minutes to disintegrate the nylon fibers completely.

## EXPERIMENTAL

## A. Materials

I. Chemicals.

Acetic acid. Glacial (99.5%) from Mallinckrodt Chemical Works.

Calcium thiocyanate. Purified liquid from J. T. Baker Chemical Co. Sp. gr. at 28°C 1.39 (1.36 at 70°C).

Carbon tetrachloride. Analytical reagent from Mallinckrodt Chemical Works. Boiling range 76°-78°C.

Diethyl ether. Absolute ether, reagent grade, from Merck and Co. Sp. gr. not above 0.710 at 25°/25°.

Ethyl alcohol. 95%, not denatured, from American Alcohol Corp. B.p. 78.5°-79°.

Malt diastase. U. S. P. 9 from the Wilson Laboratories.

Potassium Hydroxide. Reagent grade from Merck and Co.

Sodium Carbonate. Anhydrous powder 'C. P. Baker's Analyzed' from J. T. Baker Chemical Co.

II. Yarns.

Cotton. 2/40's mercerized yarn 'Durene' from Aberfoyle Manufacturing Co.

Nylon. 86 denier bright yarn from Dupont. 45 denier delustered yarn from Dupont.

Silk. Pure dye crochet silk, shade 3 (white) from Belding Heminway.

Wool. 2/20's yarn from W. J. Steel.



## B. Apparatus

The apparatus used for extraction was a slight modification of the Wasitzky micro-extractor. (Morton<sup>1\*</sup> gives a diagram and discussion of this apparatus.) This equipment is claimed<sup>2\*</sup> to be as accurate for samples of 1.2 grams as the larger Soxhlet type extractor is for samples of 25 grams.

A hot plate with approximately 1/4 in. sheets of asbestos for varying the temperature was used to maintain constant temperatures.

Fifty milliliter beakers were used to contain solutions for all treatments of samples.

For weighings a Seeder-Kohlbusch chainomatic balance was used.

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<sup>1\*</sup> Morton, Avery Adrian. 'Laboratory Technique in Organic Chemistry'; p. 202. McGraw-Hill Book Company, New York, 1938.

<sup>2\*</sup> Morton, loc. cit.

### C. Preparation of Yarns

For Qualitative Tests. Four meter lengths of nylon were scoured for fifteen minutes in 0.5% olive-oil soap solution, rinsed in warm distilled water until apparently free from soap, and air-dried.

For Quantitative Tests. Approximately 0.5 gram skeins of yarns were used for quantitative work. Samples of mixed fibers were prepared by braiding together two 0.5 gram skeins (one 0.5 gram skein of each fiber) to make an approximately one gram sample. Four strands of each kind of fiber were used for braiding in order to make a broader braid and to give a more intimate mixture of the yarns, which would be more nearly like a woven fabric.

The nylon, wool and cotton yarns were scoured in 20 ml. of a 0.5% olive-oil soap solution at constant volume for fifteen minutes (the nylon and cotton being maintained at the boiling point; the wool at 70°-80°C). Yarns were rinsed in distilled water until apparently free from soap, extracted for six hours with ethyl alcohol, and then for six hours with diethyl ether. After the ether extraction the yarns were air-dried, rinsed three times with squeezing in 25-30 ml. portions of warm distilled water, and air-dried.

The skeins of wool and cotton were then extracted with carbon tetrachloride for two hours, air-dried, washed thoroughly with squeezing in hot distilled water. Each sample was then immersed in 20 ml. of a 2% solution of malt diastase at 50°C, squeezed while immersed, removed and squeezed again. This

procedure was repeated with the same solution for three times. The sample was then allowed to remain immersed in the same solution at 50°-60°C for one hour, removed and rinsed twelve times with squeezing in fresh 25-30 ml. portions of hot distilled water, air-dried, and dried to constant weight at 105°-110°C.

The silk skeins were scoured in 20 ml. of a boiling 0.2% olive-oil soap solution at constant volume for thirty minutes, immersed for twenty minutes in a boiling 0.1% solution of sodium carbonate, rinsed, air-dried, extracted for six hours with ethyl alcohol, and then for six hours with ether, air-dried, washed three times with squeezing in warm distilled water and dried. Samples were then extracted with carbon tetrachloride and desized in the same manner as for the wool and cotton yarns.

#### D. Qualitative Tests

The following qualitative tests were made with the idea of discovering some peculiarity in behavior which would enable the qualitative identification and the separation on a quantitative basis of nylon from the other common textile fibers.

Ignition Test. When placed on a platinum foil and held in a non-luminous flame the yarns show a sharp melting point, and will not burn until after the fibers have melted. The ash left when the yarn is held directly in the flame is an extremely hard round globule, pale yellowish in color if the yarn is held in a non-luminous flame, but a darker brown to black color if held in a luminous flame (such as that of a match). The ash more closely resembles that of cellulose acetate, but is very much harder, being extremely difficult to crush.

The odor of the burning nylon fiber is very faintly like that of burning wool. It is more suggestive of the odor from cooking celery or green beans, being quite distinctive and hence of value in identifying the fiber when not in mixture with other fibers.

COLOR TESTS: Millon's test. Nylon did not give the characteristic brick red color with Millon's reagent. Instead the fibers took on a faint yellowish tinge as the solution was boiled.

Xanthoproteic test. With nitric acid the yellow color of xanthoproteic acid is not produced; instead, the yarns are completely disintegrated, producing a clear solution with

the acid.

Diphenylamine in H<sub>2</sub>SO<sub>4</sub> solution. There is no color change with the nylon yarns.

Iodine solution. Nylon stains a very dark brown, almost black, color which is not removed by prolonged washing.

Neocarmine. With this dye nylon gives a green color most like that given by wool, but distinguishable from it by being a pure green color, whereas wool gives a yellowish-green color.

SOLUBILITY TESTS: Glacial acetic acid. Nylon is very readily soluble in hot glacial acetic acid. On cooling the solution sets to a thick, white, jell-like mass. On drying in thin layers this substance leaves a tough, white, cellophane-like film. In cold glacial acetic acid nylon does not dissolve even after standing for two weeks.

Acetone. Nylon is not soluble in the hot or cold reagent.

Lead acetate solution (conc.) There is no apparent disintegration of the fibers in a boiling solution at constant volume for five hours.

Calcium thiocyanate (sp. gr., 1.36 at 70°C). Nylon fibers are apparently not soluble.

Loew's Reagent. (Copper-glycerol solution) Nylon is not soluble and there is no visible change in the fiber.

Basic zinc chloride. In boiling solution the yarns are soluble, but leave a white opalescent film along the walls of the test tube which will not dissolve although subjected to prolonged boiling.

Ammonical nickelic hydroxide solution. There is no

apparent deterioration of the fibers.

Schweitzer's reagent. Nylon is not soluble. After standing in the solution for seven days there was still no apparent sign of solubility.

Aluminium chloride solution. This reagent seemingly had no effect on the fibers, either when cold or boiling.

Sulfuric acid (conc.) Fibers are soluble in the cold reagent without difficulty.

Hydrochloric acid (conc.) Nylon is very readily soluble in the cold.

Potassium hydroxide (5%). Nylon yarns are insoluble with no apparent weakening of the fiber in the boiling, as well as the cold, solution.

## E. Methods of Quantitative Analysis

### I. Preparation of yarns for analytical work.

Purification method for nylon. (Data given in Tables I--VI.) The effect of extraction with carbon tetrachloride for two hours was first tried on the yarns as received. In order to determine whether or not any soluble material was completely removed by the length of time allowed for extraction, and also to compare the effectiveness of the type of extractor used here with that of the larger Soxhlet type, four samples were extracted for a second two hour period. Results (Table II) showed the first two hour extraction to have been effective in removing all material soluble in carbon tetrachloride.

Since it was thought that carbon tetrachloride might possibly be an ineffective solvent for removing all spinning fluids, etc., one group of the samples which had been extracted was given the scouring treatment as used for wool and cotton fibers. An additional loss in weight, even greater than that for the carbon tetrachloride extraction, was found. A second group of samples made up of the yarn as received were given the scouring treatment only. In this case the loss in weight was found to agree with the total loss on the first group as caused by both the carbon tetrachloride extraction and scouring treatment, thereby indicating that all soluble material is effectively removed by scouring alone.

The standard treatment of carbon tetrachloride extraction and enzyme desizing given to union fabrics for removal of fill-

ing material was used on the scoured nylon samples. For analytical work with yarns this treatment is not necessary, but in analysis of a fabric of mixed fibers such a treatment would always be given. To make conditions as analogous as possible to actual analysis of a mixed fabric, it was judged better to subject the yarns to this treatment.

Samples were extracted with carbon tetrachloride for two hours, rinsed thoroughly in hot water and dried. Each sample was immersed and squeezed while immersed in about 20 ml. of 2% diastase solution at 50°-60°C, removed and squeezed. This alternate immersion and squeezing was repeated three times with the same solution. The samples were then allowed to remain immersed in the same solution at 50°-60°C for one hour, removed and rinsed with squeezing twelve times in fresh portions of warm distilled water, air-dried, and dried to constant weight at 105°-110°C.

Results show that with nylon yarns this treatment gives erratic results, although it always produces an increase in weight of the yarn. It was thought better to omit this treatment with nylon samples used in the following analytical work.

Purification of yarns other than nylon. (Data given in Tables VII, VIII and IX.) It was desired to have some check on the results obtained with 0.5 gram samples as compared with those for larger samples. Also, as a check on the reliability of the extractor used in this work, the loss in weight of the yarns due to the scouring treatment was determined. Since this loss was consistent within each group of



samples (wool, silk and cotton) it was considered that the method used here gives reliable purification.

The slight variation in weights of silk, wool and cotton samples after carbon tetrachloride extraction and diastase treatment was negligible, as would be expected for unsized yarns, and gave an even smaller average change in weight than that reported by investigators using larger samples. (6)

## II. Analysis of mixed fibers.

As nearly as practical, the method of analysis was desired to fit into and follow the already existing and accepted methods of analysis for union fabrics. The tests for which data are given were made with the thought of using the following scheme in the analysis of a fabric containing the common textile fibers, silk, wool, and cotton in addition to nylon.

a. Removal of silk by the calcium thiocyanate method. ( $\text{Ca}(\text{SCN})_2$  of sp. gr. 1.20-1.21 at  $70^\circ\text{C}.$ )

b. Removal of wool with boiling 5% potassium hydroxide solution.

c. Analysis of the residue of nylon and cotton by removing nylon with acetic acid and weighing the residue as cotton.

For the silk-nylon analysis. (Data given in Tables X and XI.) The method as used by Mease and Jessup (6) was tried on one sample of a silk-nylon mixture. Silk was found to be completely dissolved out in much less than the length of treatment (sixty minutes) given by them.

The method given in A. S. T. M. (1) was tried and found to give more satisfactory results, but the silk was completely dissolved out in less than the fifteen minutes treatment as recommended there.

It was finally decided to use a modification of the two above methods. Using approximately 0.5 gram samples of nylon and one gram samples of silk-nylon mixed yarns, the following treatment was given each sample.

The weighed sample was agitated vigorously for five minutes in 40 ml. of a clear aqueous solution of calcium thiocyanate (sp. gr., 1.20-1.21 at 70°) made just acid to litmus with acetic acid and maintained at 70°C  $\pm$ 2°. The residue was removed by filtration through a weighed Gooch crucible (no asbestos mat), removed from the crucible and agitated vigorously for one minute in a fresh 40 ml. portion of calcium thiocyanate at 70°C. The residue was again removed by filtration through the Gooch crucible and thoroughly washed with hot distilled water until free of calcium thiocyanate as shown by testing the wash water with ferric chloride solution. The residue was dried to constant weight at 105°-110°C.

For the wool-nylon analysis. (Data given in Tables XII and XIII.) The standard method for determining cotton in the presence of wool as used by Mease and Jessup (6) and as given in specifications of the United States government (3,5) was used to determine nylon in the presence of wool.

Approximately 0.5 gram skeins of nylon and one gram skeins of a mixture of nylon and wool were treated in the

following manner:

Each sample was immersed for ten minutes in a boiling 5% aqueous solution of potassium hydroxide (previously boiled to remove air). Sufficient solution was used to keep fibers covered during treatment (about 30 ml.). The solution was filtered by suction through a weighed Gooch crucible (no asbestos mat), washed once with hot distilled water, once with a 5% aqueous solution of acetic acid, and then with distilled water until neutral to litmus. The residual fibers were dried to constant weight in an oven at 105°-110°C.

For the cotton-nylon analysis. (Data given in Tables XIV and XV.) It was desired to find the concentration of acetic acid, the temperature and the length of treatment which would most effectively remove the nylon and yet least affect the cotton.

Acetic acid solutions were carefully prepared to contain the following percentages by weight of acetic acid: 95%, 90%, 85%, 80%, 75%, 70%, 65%, 60%, 55%, and 50%. Previous tests had showed that at concentrations of less than 50% no disintegration of the fibers apparently took place, even when boiled at constant volume over a period of as many as eight hours. Working with small scoured test samples (four meter lengths of yarn in 5 ml. of solution) it was found that in boiling solutions of 95%, 90%, 85%, and 80% acetic acid, nylon yarns were completely dissolved in two to four seconds. At 75% and 70% the time for solution at boiling nearly doubled and the solution was less clear than for the higher concentrations. Below these concentrations solution became

increasingly more difficult and the resulting solution less and less clear.

By varying the temperature with concentrations between 99.5% (glacial) and 80%, fibers were found to be very slowly and difficultly soluble below a temperature of 80°C.

Acetic acid solutions of 76%, 77%, 78%, 79%, 81%, 82%, 83%, and 84% were prepared and used as above. Such slight change in concentration was found to be negligible in affecting the solution of the fibers.

Since 80% acetic acid appeared to be the lowest effective concentration for solution of nylon, it was decided to experiment with this concentration. At the boiling temperature (about 105°C) solution took place in three to four seconds; at 90°-95°C in about fifteen seconds; at 85°-90°C in about sixty seconds; at 80°-85°C in about five minutes, with a less clear solution.

Small test samples of cotton-nylon yarns braided together were immersed in solutions of 80% acetic acid at 85°-90°C. The nylon was found to dissolve five to six times more slowly in the presence of cotton than by itself. When the temperature was increased to boiling, the nylon yarn was rapidly and apparently completely dissolved from the cotton which appeared to be unaffected by the treatment.

From these preliminary experiments, the following analytical procedure was worked out and used for treating the cotton and cotton-nylon samples.

Each sample was agitated in 30-35 ml. of 80% by weight acetic acid for five minutes, filtered hot through a weighed

Gooch crucible (no asbestos mat), washed twice, filling the crucible each time, with boiling 80% acetic acid, then with hot distilled water until the wash water was neutral to litmus, and dried to constant weight at 105°-110°C.

## F. Data

TABLE I

Effect of Carbon Tetrachloride Extraction  
on Nylon Yarn as Received

	Reagent Time Temperature	CCl <sub>4</sub> Two hours Boiling
: Sample :	Weight in grams	Weight in grams
: Number :	before Extraction	after Extraction
:	:	Percent
:	:	Change
:	:	:
: 1 :	0.4769	0.4744
:	:	-0.52
: 2 :	0.4851	0.4833
:	:	-0.37
: 3 :	0.4594	0.4569
:	:	-0.54
: 4 :	0.4754	0.4727
:	:	-0.57
: 5 :	0.4974	0.4946
:	:	-0.56
: 6 :	0.5173	0.5148
:	:	-0.48
: 7 :	0.5004	0.4976
:	:	-0.56
: 8 :	0.4949	0.4931
:	:	-0.36
: 9 :	0.4490	0.4460
:	:	-0.67
: 10 :	0.4994	0.4978
:	:	-0.32
: 11 :	0.4980	0.4974
:	:	-0.12
: 12 :	0.4956	0.4939
:	:	-0.34
: Average :		-0.49

TABLE II

Effect of a Second Carbon Tetrachloride  
Extraction on Nylon Yarn

	Reagent	CCl <sub>4</sub>	
	Time	Two hours	
	Temperature	Boiling	
: Sample:	Weight in grams	Weight in grams	Percent
: Number:	before Extraction	after Extraction	Change
: 3	0.4569	0.4569	±0.00
: 4	0.4727	0.4727	±0.00
: 5	0.4945	0.4945	±0.00
: 7	0.4976	0.4974	-0.04
: Average:			-0.01

TABLE III

Effect of Scouring Treatment on Nylon Yarn  
Previously Extracted with Carbon Tetrachloride

	Reagent	0.5% soap sol'n.			
	Time	15 minutes			
	Temperature	Boiling			
: No.:	Wt. (gm.) before	Wt. (gm.) before	Wt. (gm.) after	Percent Change On	Percent Change Total
: :	CCl <sub>4</sub> Ext'n	Scouring	Scouring	Scouring	
: 1	0.4769	0.4744	0.4694	-1.05	-1.60
: 2	0.4851	0.4833	0.4791	-0.87	-1.24
: 3	0.4594	0.4569	0.4529	-0.88	-1.42
: 4	0.4754	0.4727	0.4697	-0.64	-1.12
: 5	0.4974	0.4945	0.4905	-0.81	-1.39
: 6	0.5173	0.5148	0.5113	-0.68	-1.16
: 7	0.5004	0.4976	0.4928	-0.97	-1.52
: Average:				-0.84	-1.35

TABLE IV

Effect of Scouring Treatment on Nylon Yarn  
as Received

	Reagent		0.5% soap sol'n.	
	Time		15 minutes	
	Temperature		Boiling	
:Sample:	Weight in grams	::	Weight in grams	::
:Number:	before Treatment	::	after Treatment	::
:		:		:
:	1	:	0.4976	:
:		:	0.4907	:
:		:		:
:	2	:	0.5001	:
:		:	0.4930	:
:		:		:
:	3	:	0.4982	:
:		:	0.4909	:
:		:		:
:	4	:	0.4980	:
:		:	0.4913	:
:		:		:
:	5	:	0.4952	:
:		:	0.4882	:
:		:		:
:	6	:	0.4996	:
:		:	0.4924	:
:		:		:
:	Average:	:		:

TABLE V

## Effect of Diastase Treatment on Nylon Yarn

	Reagent		2% diastase sol'n.	
	Time		60 minutes	
	Temperature		50°-60°C	
:Sample:	Weight in grams	::	Weight in grams	::
:Number:	before Treatment	::	after Treatment	::
:		:		:
:	1	:	0.4907	:
:		:	0.4945	:
:		:		:
:	2	:	0.4930	:
:		:	0.4966	:
:		:		:
:	3	:	0.4909	:
:		:	0.4936	:
:		:		:
:	4	:	0.4913	:
:		:	0.4929	:
:		:		:
:	5	:	0.4882	:
:		:	0.4896	:
:		:		:
:	6	:	0.4924	:
:		:	0.4953	:
:		:		:
:	7	:	0.4905	:
:		:	0.4924	:
:		:		:
:	Average:	:		:



TABLE VI

Effect of Scouring Treatment on Nylon Yarn  
Previously Treated with Diastase

	Reagent		0.5% soap sol'n.
	Time		15 minutes
	Temperature		Boiling
: No. :	Wt. (gm.) after Scouring :	Wt. (gm.) before Scouring :	Wt. (gm.) before Desizing :
:	Percent Change :	Percent Change :	Percent Change :
: 1 :	0.4921 :	0.4945 :	0.4907 :
:	-0.49 :		+0.29 :
: 2 :	0.4926 :	0.4966 :	0.4930 :
:	-0.81 :		-0.08 :
: 3 :	0.4900 :	0.4936 :	0.4909 :
:	-0.73 :		-0.18 :
: 4 :	0.4892 :	0.4896 :	0.4882 :
:	-0.08 :		+0.21 :
: 5 :	0.4912 :	0.4929 :	0.4913 :
:	-0.35 :		-0.02 :
: 6 :	0.4953 :	0.4957 :	0.4924 :
:	+0.08 :		+0.59 :

TABLE VII

Effect of Scouring Treatment on Silk Yarn  
Followed by Diastase Treatment

Reagent	(1) 0.2% soap sol'n. (2) 2% diastase sol'n.
Time	(1) 30 minutes (2) 60 minutes
Temperature	(1) Boiling (2) 50°-60°C

No.	Scouring Treatment			Desizing	
	Wt. (gm.) before	Wt. (gm.) after	Percent Change	Wt. (gm.) after	Percent Change
1	0.4402	0.4321	-1.85	0.4302	-0.44
2	0.4412	0.4333	-1.80	0.4333	±0.00
3	0.4449	0.4369	-1.80	0.4366	-0.07
4	0.4381	0.4304	-1.76	0.4306	+0.05
5	0.5362	0.5256	-1.98	0.5272	+0.30
6	0.5166	0.5080	-1.67	0.5078	-0.04
	Average:		-1.81		-0.03

TABLE VIII

Effect of Scouring Treatment on Wool Yarn  
Followed by Diastase Treatment

Reagent	(1) 0.5% soap sol'n.
	(2) 2% diastase sol'n.
Time	(1) 15 minutes
	(2) 60 minutes
Temperature	(1) 70°-80°C
	(2) 50°-60°C

No.	Scouring Treatment			Desizing	
	Wt. (gm.) before	Wt. (gm.) after	Percent Change	Wt. (gm.) after	Percent Change
1	0.5958	0.5657	-5.05	0.5652	-0.08
2	0.6269	0.5956	-5.00	0.5949	-0.12
3	0.6134	0.5826	-5.02	0.5823	-0.05
4	0.6664	0.6341	-4.85	0.6342	+0.02
5	0.6551	0.6236	-4.81	0.6245	+0.14
6	0.7077	0.6731	-4.89	0.6736	+0.07
	Average:		-4.94		-0.003

TABLE IX

Effect of Scouring Treatment on Cotton Yarn  
Followed by Diastase Treatment

Reagent	(1) 0.5% soap sol'n.
	(2) 2% diastase sol'n.
Time	(1) 15 minutes
	(2) 60 minutes
Temperature	(1) Boiling
	(2) 50°-60° C

No.	Scouring Treatment			Desizing	
	Wt. (gm.) before Scouring	Wt. (gm.) after Scouring	Percent Change	Wt. (gm.) after Desizing	Percent Change
1	0.5080	0.4956	-1.94	0.4971	+0.30
2	0.4843	0.4718	-2.58	0.4735	+0.34
3	0.4932	0.4829	-2.04	0.4820	-0.19
4	0.5006	0.4914	-1.84	0.4907	-0.14
5	0.5007	0.4915	-1.84	0.4910	-0.10
6	0.4951	0.4844	-2.16	0.4853	+0.19
	Average:		-2.07		+0.07

TABLE X

## Effect of Calcium Thiocyanate Treatment on Nylon

	Reagent			Ca(SCN) <sub>2</sub> , sp. gr. 1.20- 1.21 at 70°		
	Time			5 minutes		
	Temperature			70°C		
No.	Wt. (gm.) before Treatment	Wt. (gm.) after Treatment	Percent Change	Percent Recovery	% Devia: from Average	Correc- tion Factor
1	0.4687	0.4729	+0.89	100.90		0.9911
2	0.4884	0.4897	+0.27	100.26	-0.02	0.9974
3	0.4692	0.4711	+0.41	100.40	+0.10	0.9960
4	0.4957	0.4957	±0.00	100.00	-0.27	1.0000
5	0.4912	0.4940	+0.57	100.57	+0.27	0.9943
6	0.4708	0.4717	+0.19	100.19	-0.09	0.9981
	Average:		+0.29	100.28		0.9972

<sup>1</sup> 15 minute treatment

TABLE XI

Effect of Calcium Thiocyanate Treatment on Nylon  
In Presence of Silk

		Reagent	Ca(SCN) <sub>2</sub> , sp. gr. 1.20- 1.21 at 70°			
		Time	5 minutes			
		Temperature	70°C			
Original Sample						
No.	Wt. (gm.)	Wt. (gm.)	Wt. (gm.)	Percent	Percent	
	Sample	Silk	Nylon	Silk	Nylon	
1	0.9415	0.4302	0.5113	45.69	54.31	
2	0.9988	0.5272	0.4716	52.78	47.22	
3	0.9060	0.4366	0.4694	48.19	51.81	
4	0.9193	0.4306	0.4887	46.84	53.16	
5	0.9242	0.4333	0.4909	46.88	53.12	
6	0.9787	0.5078	0.4709	51.89	48.09	
Analysis Data						
No.	Wt. (gm.)	Percent	Percent	Percent	% Devia:	
	Residue	Silk	Nylon	Recovery	Average	Correction Factor
1	0.5149	45.31	54.69	100.70		0.9930
2	0.4790	52.04	47.96	101.57		0.9845
3	0.4739	47.69	52.31	100.96	+0.36	0.9905
4	0.4918	46.50	53.50	100.63	+0.03	0.9937
5	0.4935	46.60	53.40	100.53	-0.07	0.9947
6	0.4722	51.75	48.25	100.28	-0.32	0.9972
Average:				100.60		0.9940

<sup>1</sup> 15 minute treatment

<sup>2</sup> 60 minute treatment

TABLE XII

Effect of 5% Potassium Hydroxide Treatment on Nylon

	Reagent	5% KOH sol'n.				
	Time	10 minutes				
	Temperature	Boiling				
No.:	Wt. (gm.):	Wt. (gm.):	Percent:	Percent:	% Devia:	Correction:
:	before	after	Change	Recovery	from	Factor
:	Treatment:	Treatment:	:	:	Average:	:
1	0.4907	0.4894	-0.27	99.75	-0.30	1.0025
2	0.4382	0.4369	-0.30	99.70	-0.34	1.0033
3	0.4791	0.4799	+0.17	100.17	+0.11	0.9984
4	0.4938	0.4929	-0.19	99.82	-0.23	1.0018
5	0.4906	0.4916	+0.20	100.20	+0.15	0.9980
6	0.4695	0.4727	+0.68	100.68	+0.63	0.9953
	Average:		+0.05	100.05		0.9999

TABLE XIII

Effect of 5% Potassium Hydroxide Treatment on Nylon  
In Presence of Wool

		Reagent	5% KOH sol'n.			
		Time	10 minutes			
		Temperature	Boiling			
		Original Sample				
No.	Wt. (gm.)	Wt. (gm.)	Wt. (gm.)	Percent	Percent	
	Sample	Wool	Nylon	Wool	Nylon	
1	1.0400	0.5652	0.4748	54.36	45.65	
2	1.0861	0.5949	0.4912	54.77	45.22	
3	1.0517	0.5823	0.4694	55.35	44.64	
4	1.1265	0.6342	0.4923	56.30	43.70	
5	1.1176	0.6245	0.4931	55.88	44.12	
6	1.1150	0.6736	0.4414	60.42	39.58	
		Analysis Data		Percent	% Devia:	
No.	Wt. (gm.)	Percent	Percent	Nylon	from	Correction:
	Residue	Wool	Nylon	Recovery	Average	Factor
1	0.4762	54.21	45.79	100.32	+0.10	0.9968
2	0.4900	54.88	45.12	99.76	-0.46	1.0024
3	0.4710	55.21	44.78	100.34	+0.12	0.9966
4	0.4931	56.23	43.77	100.16	-0.06	0.9984
5	0.4950	55.71	44.29	100.40	+0.18	0.9959
6	0.4429	60.28	39.72	100.34	+0.12	0.9966
Average:				100.22		0.9978



TABLE XIV

Effect of 80% Acetic Acid Treatment on Cotton

	Reagent			80% CH <sub>3</sub> COOH sol'n.		
	Time			5 minutes		
	Temperature			Boiling		
: No.:	:Wt. (gm.):	:Wt. (gm.):	: Percent:	: Percent:	: % Devia:	: Correction:
:	: Treatment:	: Treatment:	: Change:	: Recovery:	: Average:	: Factor:
: 1 :	0.4735 :	0.4756 :	+0.44 :	100.44 :	-0.23 :	0.9956 :
: 2 :	0.4989 :	0.5018 :	+0.58 :	100.57 :	-0.19 :	0.9942 :
: 3 :	0.4853 :	0.4887 :	+0.70 :	100.70 :	+0.02 :	0.9930 :
: 4 :	0.5007 :	0.5040 :	+0.66 :	100.66 :	-0.02 :	0.9935 :
: 5 :	0.4814 :	0.4862 :	+0.99 :	101.00 :	+0.31 :	0.9901 :
: 6 :	-0.4820 :	0.4856 :	+0.75 :	100.75 :	+0.07 :	0.9926 :
:	Average:	:	+0.69 :	100.68 :	:	0.9932 :

TABLE XV

Effect of 80% Acetic Acid Treatment on Cotton  
In Presence of Nylon

		Reagent	80% CH <sub>3</sub> COOH sol'n.				
		Time	5 minutes				
		Temperature	Boiling				
		Original Sample					
No.:	Wt. (gm.)	Wt. (gm.)	Wt. (gm.)	Percent	Percent		
:	Sample	Cotton	Nylon	Cotton	Nylon		
1	0.9803	0.4866	0.4937	49.64	50.36		
2	0.9863	0.4971	0.4892	50.40	49.60		
3	0.9807	0.4907	0.4900	50.04	49.96		
4	0.9831	0.4910	0.4921	49.94	50.06		
5	0.9565	0.4841	0.4724	50.61	49.39		
6	0.9694	0.4884	0.4810	50.38	49.62		
		Analysis Data			Percent	% Devia:	
No.:	Wt. (gm.)	Percent	Percent	Percent	Cotton	from	Correction:
:	Residue	Cotton	Nylon	Recovery	Average	Factor	
1	0.4933	50.32	49.68	101.37	-0.34	0.9864	
2	0.5052	51.22	48.78	101.64	-0.08	0.9840	
3	0.4998	50.96	49.04	101.85	+0.12	0.9818	
4	0.4991	50.77	49.23	101.65	-0.07	0.9838	
5	0.4929	51.53	48.47	101.82	+0.09	0.9822	
6	0.4981	51.38	48.62	101.98	+0.25	0.9805	
Average:				101.72		0.9832	

## DISCUSSION OF RESULTS

From the results of qualitative tests the following plan can be set up for the qualitative identification of nylon in mixtures with the other common textile fibers of cotton, silk, wool, cellulose acetate and regenerated celluloses.

For rapid determination in an undyed fabric neocarmine gives reliable results. Nylon is dyed a pure, medium to pale green, most like the color given by wool, but distinguishable from it by not having a yellowish tint and by being less intense.

In a dyed fabric a microscopical examination would be of first value. In this investigation no microscopical work was done. Von Bergen (10) gives a quite complete microscopical description and presents microphotographs of longitudinal and cross-sectional views of nylon fibers. Cotton, wool and silk are easily distinguishable from nylon by microscopical examination; cellulose acetate and the regenerated celluloses are less readily distinguishable.

For chemical verification of the microscopical examination the following tests are of value:

To distinguish nylon from cotton: Nylon is quickly soluble in concentrated (above 80%) boiling acetic acid; cotton is not.

To distinguish nylon from wool: Nylon does not give a positive test with Millon's reagent; wool does.

Nylon does not give a positive xanthoproteic test; wool does. Instead, nylon rapidly disintegrates in the cold con-

centrated nitric acid.

Nylon is not soluble in boiling 5% potassium hydroxide; wool is.

To distinguish nylon from silk: Nylon does not give a positive test with Millon's reagent; silk does.

Nylon is not soluble in calcium thiocyanate solution of sp. gr. 1.20-1.21 at 70°C; silk is.

To distinguish nylon from cellulose acetate: Nylon is not soluble in acetone; cellulose acetate is.

To distinguish nylon from regenerated celluloses: Nylon is not soluble in calcium thiocyanate of sp. gr. 1.36 at 70°C; regenerated celluloses are.

Results of the work done on purification of nylon yarns show that scouring in 0.5% olive-oil soap solution, followed by a six hour extraction with ethyl alcohol and a six hour extraction with ether, gives satisfactory purification.

The use of diastase as a desizing enzyme for nylon is not satisfactory. Scouring of desized samples, although it did produce a loss in weight, did not cause them to return to their weight before diastase treatment. This indicates that the gain is not chiefly due to mechanical causes, as incomplete rinsing of the diastase, because in that case scouring would cause the yarns to return to their weight before desizing. It is suggested that some other more suitable enzyme could be found for union fabrics containing nylon.

The calcium thiocyanate method for analysis of silk-nylon mixtures gives acceptable results, without the necessity of applying a correction factor.

Results of the 5% potassium hydroxide method of wool from wool-nylon mixtures show it to be a very satisfactory method for analysis of this union. SEP 25 1939

The use of acetic acid for quantitatively estimating cotton in the presence of nylon is not quite as satisfactory a method as the two preceding ones proved. It may be that the increase in weight of the cotton yarns when treated alone is due to a slight acetylation of the cellulose molecule. However, it is believed that the greater increase in weight of the cotton residue from the cotton-nylon mixture is not due chiefly to acetylation, but rather to difficulty in washing out the product formed with nylon and acetic acid, which rapidly solidifies as the solution cools. It is suggested that in order to work out an entirely satisfactory procedure with acetic acid, data should be obtained for other concentrations of acetic acid at various temperatures and various periods of time. A better method for rinsing the cotton residue would be of especial value.

However, when it is considered that an analysis by this method is correct to within one per cent or less, and that consistent results are given, it is clear that this procedure can well be used, particularly when the correction factor is applied.

The carrying out of the analyses on a semi-micro scale proved very convenient, and, in view of the results given, very satisfactory. The saving in reagents and yarns required for the analysis and the greater ease of manipulation is considered an improvement over the use of larger quantities.

## SUMMARY

1. In undyed fiber mixtures nylon is most readily distinguished by dyeing with neocarmine.
2. Chemically nylon is best distinguishable from
  - a. Cotton, by the solubility of nylon in hot concentrated acetic acid.
  - b. Silk, by the insolubility of nylon in calcium thiocyanate, sp. gr. 1.20-1.21 at 70°C.
  - c. Wool, by the insolubility of nylon in boiling 5% potassium hydroxide.
  - d. Cellulose acetate, by the insolubility of nylon in acetone.
  - e. Regenerated celluloses, by the insolubility of nylon in calcium thiocyanate of sp. gr. 1.36 at 70°C.
3. The purification of nylon yarns for experimental purposes is adequately accomplished by scouring in 0.5% olive-oil soap solution followed by extraction with alcohol and with ether.
4. Malt diastase is not a suitable enzyme for use in the desizing of nylon.
5. The use of calcium thiocyanate, sp. gr. 1.20-1.21 at 70°C, to remove silk in the analysis of a silk-nylon mixture is satisfactory.
6. The use of boiling 5% potassium hydroxide to remove wool in the analysis of a wool-nylon mixture gives accurate results.
7. The analysis for nylon in cotton-nylon mixtures by

the solution of nylon in boiling 80% acetic acid gives satisfactory results when a correction factor for cotton of 0.98 is applied.

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## AUTOBIOGRAPHY

I, Katherine Austin Lathrop, was born June 16, 1915 at Lawton, Oklahoma. I attended the public schools of Lawton, graduating from the highschool of that system in May, 1932. From September, 1932 to May, 1934, I attended Cameron State School of Agriculture at Lawton. In May, 1936, I received the Bachelor of Science degree in Household Arts from Oklahoma Agricultural and Mechanical College.

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