

DEPARTMENT OF THE INTERIOR
UNITED STATES GEOLOGICAL SURVEY

GEORGE OTIS SMITH, DIRECTOR

WATER-SUPPLY PAPER 235

THE PURIFICATION
OF SOME
TEXTILE AND OTHER FACTORY WASTES

BY

HERMAN STABLER AND GILBERT H. PRATT

PREPARED IN COOPERATION WITH RHODE ISLAND
STATE BOARD OF HEALTH



WASHINGTON
GOVERNMENT PRINTING OFFICE
1909

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THE PURIFICATION OF SOME TEXTILE AND OTHER FACTORY WASTES.

By HERMAN STABLER and GILBERT H. PRATT.

INTRODUCTION.

Scope of paper.—This paper gives a brief outline of the processes of scouring wool, bleaching cotton yarn and cloth, dyeing cotton yarn, and manufacturing oleomargarine, glue, and fertilizer. The waste waters of these processes are considered in detail and means of purifying them are discussed at length, special prominence being given to the investigations of the authors. These investigations, which were begun in pursuance of an agreement for eleven months' cooperative work entered into August 1, 1906, between the United States Geological Survey and the Rhode Island state board of health, consisted of the determination of the effects of special industrial wastes on streams, their persistence, their relation to health, their damage to natural water resources, and the best methods of disposing of them, purifying them, or recovering valuable materials contained in them. The effect of the wastes on streams and the resulting damage to water resources has been detailed in another publication.^a

The cooperative work was conducted under the joint supervision of M. O. Leighton, chief hydrographer of the United States Geological Survey, and Dr. Gardner T. Swarts, secretary of the Rhode Island state board of health, and the expense was shared equally by the organizations represented. The investigation of processes, the general field work, and a part of the laboratory experiments and analyses were conducted by Herman Stabler, assistant engineer, United States Geological Survey. The general laboratory work was conducted by Gilbert H. Pratt, chemist, Rhode Island state board of health. After the close of the cooperative work on June 30, 1907, the purification experiments were continued for several months by the state board of health, and the operation of an experimental purification plant that had been established and the installation and operation of other plants fell under Mr. Pratt's direction.

Acknowledgments.—Information as to processes, free access to factories, assistance in installing and maintaining experimental purification plants, and many other courtesies were extended by the manu-

^a Stabler, Herman, Pollution of the Moshassuck * * * rivers: Spec. Rept. Rhode Island State Board of Health, 1908.

facturers affected by these investigations. The hearty cooperation thus received is acknowledged with thanks. A partial list of those who contributed to the research in this manner is as follows: Messrs. A. A. Sack, J. R. MacColl, F. A. Sayles, K. F. Wood, H. F. Schwarz, F. R. Mason, W. J. Burton, J. P. Farnsworth, F. R. Ames, H. G. McKerrow, S. Turner, and William Hartshorne.

Special acknowledgment is due Dr. Gardner T. Swarts for his personal interest in the investigations and for his active assistance in procuring the cooperation of manufacturing interests.

Free use has been made of the technical literature on the subjects treated. Where specific facts have been taken from such sources due credit is given, but the report also contains material gleaned from a perusal of the history of previous investigations, for which entire credit can not rightly be given to any one author.

WOOL SCOURING.

NECESSITY FOR TREATMENT.

Raw wool, as it comes from the sheep, is heavily impregnated with fatty matter, potassium salts, sand, and miscellaneous organic and inorganic materials. These impurities are derived partly from exudations from the skin of the animal and partly from outside sources, such as the soil over which the sheep moves. As it comes to the textile factory, therefore, wool may contain only from 20 to 80 per cent of fiber from which yarn may be produced. The separation of these impurities from the fiber is one of the most important processes of manufacture. The separation must be practically complete or all subsequent processes will suffer, for imperfectly scoured wool resists the action of mordants and takes a streaky color in dyeing, causes the fabric to become stiff and hard, and otherwise interferes with the production of high-class textiles. The scouring processes depend for their success on the use of softening and solvent agents which bring into solution some of the impurities and loosen others from the fiber so that they may readily be removed.

PROCESSES OF SCOURING.

SCOURING WITH SOAP, ALKALI, AND WATER.

The agents most commonly used for scouring wool are soap, alkali, and water. The operation is usually conducted in a series of three long, shallow iron tanks. These are nearly filled with water, to which soap and alkali are added in desired amounts. The wool is then fed continuously into the first tank, through which it is slowly carried by an ingenious system of moving rakes. Having reached the end of the first tank, the wool passes between rolls, which extract the moisture from it, and it falls into the second tank. Passage through the second and third tanks is accomplished in a similar manner.

The wool emerging from the third tank is practically free from impurities and is ready to be dried, oiled, and subjected to further manufacturing processes. As more and more wool passes through the scouring machine, the liquid in the tanks becomes very foul and the operation finally has to be discontinued. The scouring liquor is discharged from the first tank into the sewer, the liquor in the second tank is discharged into the first, the liquor in the third tank is discharged into the second, and the third tank is filled anew with clean water and other cleaning agents. In this way the waste liquors are highly concentrated, and water, soap, and alkali are used in an economical manner. Potassium soaps and potash are usually employed, because the sodium compounds, though less expensive, have an energetic corrosive action on the fiber, destroy its natural mellowness, and impart to it a yellow tinge. The potassium compounds, on the other hand, tend to whiten the wool and to give it a softened, diffusive character. Olive-oil soaps are most frequently used. The water is maintained during scouring at a temperature between 32° C. (90° F.) and 60° C. (140° F.). The higher temperatures are conducive to better cleansing, but they have a tendency to injure the wool.

For each thousand gallons of water, 40 to 45 pounds of soft soap and 5 to 10 pounds of potash are generally used in scouring from 1,000 to 2,000 pounds of raw wool to produce 600 to 900 pounds of clean wool fiber. These figures are, of course, only approximations; some scourers prefer a large amount of soap and no alkali, and others use very little soap and large quantities of alkali. At some mills the wool, after having been scoured, is rinsed in a running stream of fresh water.

STEEPING AND SCOURING.

As the impurities of wool include potash and wool fat, two valuable substances, special methods of cleansing have been adopted in order to facilitate the recovery of one or both of them from the scouring liquor. Steeping in tepid water as a preliminary to scouring is a procedure much followed in Europe, but little used in this country. The steeping, as usually practiced, is in reality an extractive process, for successive portions of water are passed through the wool, softening the impurities and removing the matters soluble in water. The steep water contains, in consequence, a large amount of potash.

After having been steeped, the wool is scoured as previously described. Although the steeping removes from the wool its natural content of the valuable scouring agent, potash, it renders scouring less costly, for it softens the impurities so that they may easily be removed from the fiber. This results in a saving of time and soap in scouring. The recovery of potash from steep water is another advantage derived from this procedure.

SCOURING WITH VOLATILE SOLVENTS.

Methods of scouring with volatile solvents have been devised in great numbers. The solvents proposed are petroleum-naphtha, carbon disulphide, and carbon tetrachloride. Because of the danger in the use of highly inflammable solvents and the expense entailed by loss of solvent, few of the patented processes have proved to be successful. Some of them, however, provide such perfect control of the solvent that its escape is negligible, and excessive cost and danger of fire have been obviated. Unfortunately these processes are jealously guarded for the exclusive use of the owners, so that their benefits are not open to textile interests generally.

In volatile solvent processes the raw wool is placed in suitable containers, the solvent is passed through it again and again until all the grease is extracted, and the solvent is removed from the wool and container by means of an inert gas. The solvent is recovered from the grease for subsequent use by distillation, and a valuable wool-fat product is left. After treatment with the solvent, the wool can be completely cleansed by warm water alone in the usual forms of scouring apparatus. It is claimed for such processes that the recovered wool fat is sufficiently valuable to pay for the extraction process. The gain in their use is measured partly by the saving in soap in the subsequent scouring process, but the greatest economy is said to be a saving in wool. It is claimed that the net yield of clean wool is far in excess of that obtained from the processes of scouring with soap.

WASTE WATERS FROM SCOURING WOOL.

CLASSIFICATION.

The waste waters from scouring wool vary in composition according to the methods of scouring and according to the differences of practice when the same general methods of scouring are employed. The wastes may be classified as follows: Steep water; sewage from scouring with soap and alkali after steeping; sewage from washing after treatment with volatile solvents; sewage from scouring with soap and alkali, and rinse water after scouring with soap and alkali.

STEEP WATER.

Treatment.—Steeping before scouring is practiced generally in France and Belgium and to a less extent in Germany, Russia, and England, but the method is almost unknown in the United States. As its object is the recovery of the potash in the wool, the steep water is used repeatedly until it becomes highly concentrated before the recovery processes are begun. Although there is considerable difference in practice at individual works, the average amount of liquor is approximately 1,000 gallons for 5,000 pounds of wool treated. The

steep water contains about 20 per cent of the weight of the wool treated, or about 40 per cent of the impurities in the wool. The constituents are mainly potassium compounds of organic and mineral acids, a small amount of similar sodium compounds, sand, dirt, and organic matter of various kinds. The residue obtained by evaporating the steep water amounts to about 120,000 parts per million (1,000 pounds per 1,000 gallons). About 40 per cent of this amount is organic or volatile matter and about 60 per cent is mineral matter. The alkalinity of the liquor corresponds to about 4,000 parts per million (33 pounds per 1,000 gallons) of potassium carbonate. In the recovery process the potassium compounds of organic acids are converted into potassium carbonate, thus largely increasing the yield of crude potash.

The process of treatment is simple. Usually the liquor, heated by waste furnace gases, is fed into an evaporator of the Porion type, passing first through an evaporating chamber and, after concentration has progressed to a suitable degree, passing into an incinerating chamber or common reverberatory furnace, from which crude potash is removed. The waste heat from the incinerating chamber accomplishes the evaporation. The liquor contains a considerable amount of inflammable matter which, burning in the incinerating chamber, assists the evaporation and reduces the amount of coal required for the process. Obviously the more concentrated the liquors are the more economically they can be utilized for the production of potassium salts.

Results.—Table 1 gives analyses of the potash obtained from the foregoing process:

TABLE 1.—*Analyses of potash recovered from wool-steep liquor.*

[Per cent of crude potash.]

	1.	2.	3.	4.	5.	6.	7.
Silica (SiO ₂).....		6.2	6.0	1.4	0.1		
Iron oxide (Fe ₂ O ₃).....				Trace.	Trace.		
Calcium (Ca).....				1.7	1.6		
Magnesium (Mg).....				.6	.5		
Sodium (Na).....	2.0	1.4	1.3	2.0	Trace.		2
Potassium (K).....	48.8	48.5	48.4	49.0	52.7	53.6	48
Carbonate radicle (CO ₃).....	36.7	31.5	29.7	35.2	34.5	37.7	35
Sulphate radicle (SO ₄).....	1.7	1.2	1.3	3.8	3.8	1.5	3
Chlorine (Cl).....	2.9	5.9	8.0	4.3	3.8	3.0	3
Phosphate radicle (PO ₄).....				.9	.9		
Insoluble matter.....	4.9	1.5	1.6			4.2	5
Water.....		2.8	2.8				3
Organic matter and undetermined, etc.....	3.0	1.0	.9	2.5	2.1		1
	100.0	100.0	100.0	100.0	100.0	100.0	100

1. Analysis of potash from Doehren establishment. Analyst unknown. Wagner, Rudolf von, *Manual of chemical technology*; transl. by Sir William Crookes from the 13th German ed. of Dr. Ferdinand Fischer, 1904, p. 298.

2, 3. Analyses by Wérotte, Verviers, 1873. Wagner, Rudolf von, *loc. cit.*

4, 5. Analyses by Marcker and Schulz. Naylor, W., *Trades waste; its treatment and utilization*, 1902, p. 83.

6. Analysis by Maumené and Rogelet. Naylor, W., *loc. cit.*

7. Analysis of potash from a plant using the Richard-Lagerie apparatus. Analyst unknown. Naylor, W., *op. cit.*, p. 85.

The content of potassium is nearly uniform in all the analyses, and it is approximately 50 per cent of the crude potash. In Table 1 the analyses have been calculated in ionic form for purposes of comparison. Table 2 contains a statement of the soluble salts as given by the analysts.

TABLE 2.—*Saline composition of potash recovered from wool-steep liquor.*

[Per cent of crude potash.]

	1.	2.	3.	4.	5.	Mean.
Potassium carbonate (K_2CO_3).....	78.5	68.5	64.3	86.8	75	74.6
Potassium sulphate (K_2SO_4).....	2.7	2.1	2.5	2.8	6	3.2
Potassium chloride (KCl).....	5.7	12.5	16.9	6.2	6	9.5
Potassium silicate (K_2SiO_3).....	8.5	8.0	8.2
Sodium carbonate (Na_2CO_3).....	4.6	3.2	3.1	4	3.7

Potash in this crude form is worth from 3 to 4 cents a pound. It may be purified by extraction and crystallization, treatment that yields fairly pure carbonates, sulphates, and chlorides of potassium. A notable application of this procedure is found at the establishment of W. Graff at Lesum, where the pure salts were manufactured from the crude potash from six wool-scouring plants in 1878. According to Flekkel,^a simple extraction and calcination of crude potash obtained from steep water gives a product which is 85 per cent pure potassium carbonate and is the purest commercial form known.

Costs and values.—A rough estimation of costs and values in this process may not be out of place. Naylor^b quotes a statement placing the coal consumption for evaporating and incinerating at 1 pound for each pound of crude potash produced when a steep liquor of specific gravity 1.072 (10° Baumé, or 14.4° Twaddell) is treated. Fischer^c estimates that 1 pound of Westphalian coal (analysis—carbon, 76.11; hydrogen, 4.52; sulphur, 1.19; oxygen and nitrogen, 10.06; ash, 8.12 per cent) is sufficient to evaporate and to calcine 12 pounds of steep liquor of specific gravity 1.069 (9.6° Baumé, or 13.8° Twaddell) and that the product is about 0.9 pound of crude potash. According to these estimates, evaporation and calcination would require about 745 pounds of coal per 1,000 gallons of steep liquor. If the preliminary concentration were conducted in a triple-effect vacuum avaporator, the amount of coal could probably be reduced to 425 pounds, but such a machine is economically adapted only to the concentration of large quantities of liquor. The following is a rough estimate of costs:

^a Extraction of potash from suint of Russian wool: Bull. Soc. chim., 2d ser., vol. 34, 1880, pp. 332-333.

^b Naylor, W., Trades waste; its treatment and utilization, 1902, p. 85.

^c Fischer, F., Utilization of suint from wool: Dinglers polytech. Jour., vol. 229, 1879, pp. 446-449.

Yearly costs for treating 100 gallons an hour of steep liquor, ten hours a day, three hundred days a year.

Interest, depreciation, and repairs on plant, 15 per cent on investment of \$5,000.....	\$750
• Labor.....	750
Coal, 132 tons, at \$4 (this estimate allows additional coal for banked fires fourteen hours daily).....	528
	2,028

Liberal allowance has been made for all costs. Such a plant would utilize the steep liquor from 5,000 pounds of wool daily, and plants for treating larger quantities of liquor and running twenty-four hours a day could be operated with much greater economy. On the assumption that the foregoing process would recover 84 per cent of the potassium in the wool as potassium carbonate worth 5 cents a pound, and that the remainder is valueless, the receipts would amount to 0.075 cent a pound of wool for each 1 per cent of potassium found by analysis, or 0.05 cent a pound of wool for each 1 per cent of recoverable potassium carbonate. A plant like the one just indicated would, therefore, produce an annual revenue of \$1,125 for each 1 per cent of potassium, or \$750 for each 1 per cent of potassium carbonate in the wool. In order to pay costs as estimated, this plant would necessarily have to operate on liquor from wool containing 1.8 per cent of potassium or 2.7 per cent of recoverable potassium carbonate. Each additional 0.044 per cent of potassium, or 0.067 per cent of recoverable potassium carbonate, would return a profit of 1 per cent on the investment of \$5,000. With the average wool, containing over 4 per cent of recoverable potash, a substantial return on the investment is to be expected.

Production of ferrocyanide.—Another method which has been suggested for utilizing steep water is that of evaporating it to dryness and heating it in a closed retort for the production of potassium ferrocyanide, or yellow prussiate of potash. The residue obtained by evaporation to dryness is an intimate mixture of potassium salts and organic matter, and after the addition of iron filings and nitrogenous waste in proper proportions, it is one of the best mixtures for the production of ferrocyanide. This method was proposed by Havrez^a in 1870, and it was successfully tried on an experimental scale at the Peltzer works in Verviers. Although it is improbable that the method will come into use at wool-scouring establishments, it indicates a market in the chemical industries for a potash residue which may easily be obtained at such works. It is stated that the value of the residue of steep water, when used in this way, is more than twice its ordinary commercial value.

^a Havrez, P., Wagner's Jahresbuch, 1870, pp. 210 and 222; Dinglers polytech. Jour., vol. 195, p. 535.

SEWAGE FROM SCOURING AFTER STEEPING.

As 40 per cent of the impurities of the wool are removed by steeping, the sewage from scouring after steeping contains the remaining 60 per cent of the impurities. This waste differs from ordinary scouring liquor chiefly by reason of the absence of the potassium compounds that are in raw wool. It contains large amounts of organic matter, wool fat, soaps used in scouring, and other substances, and its practical purification can be best effected by the means adopted for ordinary scouring liquor (see pp. 14-17) except those in which the recovery of potash is an important element.

SEWAGE FROM WASHING AFTER TREATMENT WITH VOLATILE SOLVENTS.

Sewage from washing wool after treatment with volatile solvents is closely allied in character to the steep water already considered. It differs from steep water in that it contains a greater percentage of the wool impurities, other than potash, and for this reason it is less valuable for potash recovery. On the other hand, its greater content of organic impurities should give it a greater value than steep water in the production of potassium ferrocyanide. The solids of this liquor consist of about 40 per cent mineral and 60 per cent organic matter. The yearly cost for potash recovery from it would be from \$500 to \$800 greater than that given on page 11 for recovery from steep water for the same quantity of raw wool, and the quality of crude potash would be much inferior, averaging probably not more than 50 per cent pure potassium carbonate. As a larger quantity of inferior potash would be produced, the economy of the process would be considerably lessened by cost of marketing the product unless the potash were purified on the premises. Furthermore, the greater proportion of suspended matter in this liquor makes economic evaporation difficult.

It would seem that the dried residue after evaporation of such liquor would make an excellent fertilizer stock. Attempts to market it in this way, however, have been unsuccessful.

If mere purification without recovery of potash is desired, this liquor can be successfully treated by slow filtration through sand. From 65 to 85 per cent removal of total organic matter and entire removal of suspended matter was effected at a rate of 12,300 gallons per acre per day by this method at the Lawrence sewage experiment station.^a Such purification would cost:

Annual cost of daily purification of 2,000 gallons of sewage from washing wool after treatment by volatile solvents.

Interest, repairs, and depreciation, 15 per cent on an investment of \$1,500.....	\$225
Labor, one-half of one man's time, at \$1.50 a day.....	225
	450

^a Thirty-sixth Ann. Rept. Massachusetts State Board Health, 1904, pp. 276-277.

Whether this treatment would be less expensive than potash recovery would depend chiefly on the quantity and the potash content of the wool scoured and on the amount of water used in scouring

SEWAGE FROM RINSING AFTER SCOURING.

If the wool is rinsed after scouring with soap and alkali the resulting waste water is usually very dilute and contains no materials of sufficient value to warrant their recovery. Purification by common sewage methods is usually satisfactory. Experiments at Lawrence, Mass.,^a indicate that if rinse water, which may vary from 5 to 100 gallons for each pound of wool scoured, is mixed with sewage, it can be satisfactorily purified by filtration through sand at high rates. Rates exceeding 500,000 gallons per acre per day were maintained in some of these experiments.

SEWAGE FROM SCOURING WITH SOAP AND ALKALI.

The sewage which results from scouring wool with soap and alkali is the "wool-scouring liquor" commonly encountered in this country. It is an ill-smelling liquor of brownish color, heavily charged with impurities in solution and in suspension. It contains soap, wool fats, and potash in sufficient quantities to form a heavy emulsion, and on this account the liquor is very hard to filter in the laboratory. The analyses in Table 3 show the general composition of the scouring liquor and some of the variations in character likely to be encountered at different mills.

TABLE 3.—Analyses of wool-scouring liquor.

[Milligrams per liter, unless otherwise stated.]

	1.	2.	3.	4.	5.
Specific gravity.....	1.023	1.027	1.019
Turbidity.....	22,000	120,000	55,000
Scum..... per cent by volume.....	8	Slight.	20
Sediment..... do.....	15	8	20	14
Total solids.....	45,090	116,400	68,060	70,800	41,700
Volatile.....	24,670	85,000	50,710	46,600	29,800
Fixed.....	20,420	31,400	17,350	24,200	11,900
Alkalinity.....	8,000	8,000	3,100	3,800	3,420
Fats.....	14,600	59,000	38,200	31,620	20,200
Oxygen consumed.....	5,680	11,500	5,700	5,200
Nitrogen as free ammonia.....	300	300	164	180
Organic nitrogen.....	663	1,550	795	941

1. Liquor from woolen mill of Lymansville Company, Lymansville, R. I., November 13, 1906.
 2. Average of three samples, Lorraine Manufacturing Company, Pawtucket, R. I., March 13, 1907.
 3. Average for week ending March 29, 1907, Lorraine Manufacturing Company, Pawtucket, R. I.
 4. Average for week ending May 23, 1907, Lorraine Manufacturing Company, Pawtucket, R. I.
 Analyses 1 to 4 by G. H. Pratt and H. Stabler.
 5. Crude suds from Field Head Mills, Bradford, England, May, 1900. Analysis by F. W. Richardson, Bradford public analyst.

^a Thirty-second Ann. Rept. Massachusetts State Board Health, 1900, p. 415.

PURIFICATION OF WOOL-SCOURING LIQUOR.

SEWAGE-TREATMENT METHODS.

The methods of treating wool-scouring liquor in which purification is the sole object include the common processes of sewage purification, namely, sedimentation, precipitation, and filtration.

The Massachusetts state board of health has conducted for several years experiments on the purification of wool-washing wastes by sewage methods, and a summary ^a of five years' work by that body may be given as follows:

1. A considerable amount of suspended matter can be removed by sedimentation. The amount of organic matter deposited can not be materially increased by reasonable amounts of common precipitants alone or in combination. Ferric chloride and lime or lime alone will sometimes precipitate considerable quantities of organic and fatty matter.

2. Acids separate fats, but they give an acid liquor that is hard to treat.

3. Filtration through sand is inexpedient, because the liquor does not nitrify by itself and the filters quickly clog.

4. Scouring liquor mixed with sewage can be purified and nitrified in intermittent sand filters.

5. A higher rate of filtration and less clogging of filters result from holding the mixture of scouring liquor and sewage for bacterial action, including putrefaction, before filtration.

6. Mixtures of scouring liquor and sewage that can be purified in sand filters can not be purified in contact filters.

It is evident from this summary that a high degree of purification can not be obtained by sedimentation, chemical precipitation, or filtration under reasonable conditions of practice.

The results obtained by septic treatment of mixed sewage and wool-scouring liquor for 1900 were very good and are indicated in Table 4, adapted from the report ^b previously mentioned.

TABLE 4.—*Analyses of mixed sewage and wool-scouring liquor before and after purification.*

[Milligrams per liter.]

Source of sample.	Color.	Free ammonia.	Albuminoid ammonia.	Nitrogen as nitrates.	Nitrogen as nitrites.	Oxygen consumed.
Mixture of 11 parts sewage and 1 part scouring liquor		36.50	14.30			164.0
Mixture filtered at rate of 47,600 gallons per acre per day, 6 days a week	215	.19	1.68	16.3	.000	28.3
Mixture passed through septic tank and filtered at rate of 46,100 gallons per acre per day, 6 days a week	173	.22	1.44	29.1	.023	22.5

^a Loc. cit.

^b Op. cit., p. 416.

The liquor treated by this method, however, is very different from the wool-scouring liquor represented by the analyses in Table 3. The scouring liquor used in all the Massachusetts experiments was rather dilute, and the successful filtration experiments were made on this dilute liquor still further reduced by eleven times its volume of sewage. It appears that even the best results obtained in these experiments show that the method can not be employed at many wool-scouring establishments. Treatment of wool-scouring liquor by usual sewage methods, therefore, can not be recommended, except in cases where a comparatively small percentage of purification is all that is desired.

The conclusion reached above is corroborated by the results of treatment by sedimentation at Lymanville, R. I. The wastes at the woolen mill of the Lymanville Company are dye water, 3,000 to 4,000 gallons; piece-scouring and rinsing water, 5,000 to 6,000 gallons; and wool-scouring liquor, 5,000 to 6,000 gallons. Piece-scouring suds and dye water, in intermittent flow during the day, are collected in two 9,500-gallon brick cisterns, which are used alternately. At noon and at the end of the day wool-scouring liquor to which has been added slaked lime to the amount of 50 pounds a day, or 1 pound to about 300 gallons of the combined liquors, is admitted. These cisterns serve primarily as receiving and mixing basins, but the mixed liquors are also freed in them from coarse and heavy suspended matters. The cisterns are cleaned once in three weeks, when about 9 cubic yards of sludge are removed. This sludge is dumped near by until excess of water has drained off, and is then carted away. It contains, as shown by analysis of a sample taken from the dump, total solids, 79.7 per cent (volatile solids, 4.5 per cent; fixed solids, 75.2 per cent); fats, 1.4 per cent; and nitrogen, 0.12 per cent. It is chiefly sand, wholly unsuitable for fertilizing purposes, and it probably can not be utilized in any way.

The combined liquors are pumped from the receiving cisterns into the first of a series of four reservoirs. The sizes of these reservoirs and the periods of storage afforded by them are shown in Table 5.

TABLE 5.—Waste liquor reservoirs at Lymanville, R. I.

Reser- voir No.	Dimensions.	Capacity.	Storage period.
	<i>Feet.</i>	<i>Gallons.</i>	<i>Days.</i>
1.....	45 by 60 by 4	81,000	5.4
2.....	35 by 18 by 4	18,900	1.3
3.....	40 by 18 by 4	21,600	1.4
4.....	45 by 18 by 4	24,300	1.6
.....		145,800	9.7

The system is practically one of sedimentation, though the effluent from reservoir No. 4 passes through a bank of coarse cinders. The first of the reservoirs is cleaned about once in six months, when 25 cubic yards of scum and an equal amount of sludge are removed. Only slight

accumulations are found in the other reservoirs. In a year, therefore, 160 cubic yards of material are removed from the wastes in the receiving cisterns, 100 cubic yards in reservoir No. 1, and about 10 cubic yards in the other reservoirs, making a total of 270 cubic yards of material removed from the wool-scouring wastes. The scum from reservoir No. 1 contains 24.1 per cent total solids (15.4 per cent volatile solids, 8.7 per cent fixed solids), 4.8 per cent fat, and 0.4 per cent nitrogen. The sludge contains 29.5 per cent total solids (14.1 per cent volatile solids, 15.4 per cent fixed solids), 4.2 per cent fat, and 0.5 per cent nitrogen. This material undoubtedly has some manurial value, but the great content of moisture (70 to 75 per cent) renders it practically valueless because of cost of transportation or of drying. Correcting for the moisture and assuming an average weight of 80 pounds per cubic foot for the 270 cubic yards of material, it appears that a total removal of 357,000 pounds per annum is accomplished. This is a maximum figure. The waste liquors contain about 1,000,000 pounds of solid matters. A purification of upward of 36 per cent is therefore effected. It should be noted, however, that by far the greater part (about 75 per cent) of this purification takes place in the receiving basins. In Table 6 analyses of the various waste liquors at Lymanville are presented, together with analyses of the combined liquors at different stages in the sedimentation system.

TABLE 6.—*Analyses of waste waters at Lymanville, R. I.^a*

[Milligrams per liter, unless otherwise stated.]

Source of sample.	Turbidity.	Color.	Sediment.	Organic nitrogen.	Nitrogen as free ammonia.	Oxygen consumed.	Fats.
			<i>Per cent.</i>				
Dye water.....	400	Black....	(b)	7	43	364	14
Piece-scouring suds.....	1,200	Slate....	(c)	69	1	560	900
Wool-scouring liquor.....	22,000	Brown....	15	663	300	5,680	14,600
Inflow reservoir No. 1.....	10,300	do.....	7.2	215	183	1,700	5,940
Effluent reservoir No. 1.....	9,700	do.....	3.0	194	169	1,690	5,430
Effluent reservoir No. 2.....	9,700	do.....	2.2	194	143	1,700	4,950
Effluent reservoir No. 3.....	8,000	do.....	2.1	188	145	1,680	4,380
Effluent reservoir No. 4.....	8,500	do.....	1.3	184	134	1,680	5,070
Final effluent.....	9,700	do.....	1.0	190	133	1,700	4,850

Source of sample.	Chlorine.	Alkalinity.	Total solids.		Solids in solution.	
			Total.	Loss on ignition.	Total.	Loss on ignition.
Dye water.....	5	114	790	690	790	690
Piece-scouring suds.....	100	1,990	4,610	2,140	3,960	1,510
Wool-scouring liquor.....	1,100	8,000	45,090	24,670	31,120	13,080
Inflow reservoir No. 1.....	400	3,500	19,600	12,500	11,080	4,670
Effluent reservoir No. 1.....	400	3,250	16,530	9,400	11,770	5,000
Effluent reservoir No. 2.....	400	3,750	16,010	9,140	11,610	5,050
Effluent reservoir No. 3.....	400	3,750	15,520	8,640	11,240	4,670
Effluent reservoir No. 4.....	400	3,750	15,730	8,760	11,580	4,920
Final effluent.....	400	3,750	15,900	8,960	11,360	4,880

^a Analyses by G. H. Pratt and H. Stabler.^b Considerable.^c Heavy.

The mixing of the wool-scouring liquor with the other waste waters assists materially in the precipitation of solids; but the reduction in impurities, even under these favorable conditions, is comparatively small. It appears from the figures presented in Table 6 that the entire system of reservoirs, considered aside from the receiving basins, with nearly ten days' storage accomplishes a reduction of only 6 per cent in turbidity, 86 per cent in sediment, 19 per cent in total solids, 28 per cent in loss on ignition, 2 per cent in fixed solids, 12 per cent in organic nitrogen, 27 per cent in free ammonia, 18 per cent in fats, and practically nothing in oxygen consumed or solids after filtration. In the first reservoir alone there is a reduction of 6 per cent in turbidity, 58 per cent in sediment, 16 per cent in total solids, 25 per cent in loss on ignition, nothing in fixed solids, 10 per cent in organic nitrogen, 8 per cent in free ammonia, and 9 per cent in fats. There appears to be little gain in prolonging the period of sedimentation beyond five days. It is even probable that a carefully constructed sedimentation basin with a twenty-four to forty-eight-hour period would accomplish as much purification as the entire system of reservoirs in use at Lymansville, for it should be understood that these reservoirs are mere excavations in the earth and that no precautions were taken in their construction to produce an even flow throughout their cross sections. It is probable that the liquor moves through them in a comparatively narrow current and hence is subjected to a much shorter period of sedimentation than their capacities indicate.

PURIFICATION BY RECOVERY.

The methods of purification which involve the recovery of valuable products are more successful in reducing the impurities of wool-scouring liquors than the methods applied for the sake of purification only. The valuable substances which have been recovered on a practical scale are fats and potash, both of which are found in large amounts in the fleece and both of which are added in the scouring process. The most prominent of the methods that have been used successfully will be described.

Cracking process.—The earliest and at present the most usual method of recovery treatment for wool-scouring liquor is known as the "cracking process." In the routine of this process, the liquor is first allowed to settle for a short period to remove the greater portion of insoluble mineral matter. It is then drawn into wooden or lead-lined tanks and treated with sulphuric acid. Acid is added until a breaking up of the liquor or "cracking" is noticed. At this point the liquor has a decidedly acid reaction. The result of the addition of acid is that the fatty compounds are broken up with the formation

of free fats. The separation of the fats from the mother liquor is, however, somewhat difficult. Usually, after one or two days sedimentation, a considerable portion of the fatty matter collects on the surface and a nearly equal amount at the bottom of each tank, while the middle portion is comparatively clear and free from suspended matter. This middle portion is drawn off and run to waste, while the fatty matters at top and bottom are removed to sludge filters covered with sawdust, in order that surplus moisture may drain off.

The material usually placed on these filters amounts to about one-third of the total volume of the wool-scouring liquor. The sludge, after draining about four days on the filters, is collected in cloths and is made into thin cakes. These cakes are placed in presses heated by steam, which melts the fats so that the resulting oil can be removed by pressure. The mixed water and oil which flows from the presses is collected in cisterns and separated by sedimentation. The oil is subjected to purification by being boiled with a little acid, after which it is allowed to settle and cool in order that any remaining traces of water, acid, and other impurities may be run off. The oil, when cool, solidifies, and it is sold under the name of "de gras," or Yorkshire grease. It is worth from $1\frac{1}{2}$ to 3 cents a pound. A large proportion of the mineral and organic impurities of the wool-scouring liquor remains in the presses in the form of hard-pressed cakes, which are removed and generally thrown away as a waste product. Some establishments have extracted these cakes with volatile solvents in order to obtain a greater yield of fats, but such procedure has not generally been financially successful because of loss of solvent. In Europe they have been used to some extent as a fertilizer base, but their value for this purpose is at least subject to doubt.

Detailed examinations were made at the de gras plant of the Lorraine Manufacturing Company, Pawtucket, R. I. At this establishment preliminary sedimentation of the liquor is omitted, and, in consequence, an usually large proportion of suspended mineral matter is carried through the entire process. Three sets of samples were collected. The scouring liquor of these sets is represented by analyses 2, 3, and 4 of Table 3. Analyses of the waste waters from the acid-treating tanks, of waste waters from the separating cistern which receives mixed water and oil from the presses, and of waste waters from the purification of the oil by boiling with acid, are listed in Table 7. Several samples of the press cake were found to contain fats amounting to 25.2 per cent, 26.3 per cent, 16.7 per cent, and 22.7 per cent of the weight of the dried cake.

TABLE 7.—Analyses of waste waters from the de gras plant of the Lorraine Manufacturing Company at Pawtucket, R. I.^a

[Milligrams per liter, unless otherwise stated.]

Date, 1907.....	Treating-tank effluent.			Water from separating cistern.		Water from grease purification.
	Mar. 13.	Mar. 22-29.	May 17-23.	Mar. 29.	Mar. 14.	Mar. 14.
Turbidity.....	1,500	8,000	4,000	4,500	160
Sediment.....	1.2 per cent.	Heavy.	3.5 per cent.	Considerable.	Considerable.	Considerable.
Oxygen consumed.....	1,800	2,160	2,800	1,600	1,320	1,025
Nitrogen as free ammonia.....	330	230	289	80	89	98
Organic nitrogen.....	200	258	346	402	238	150
Acidity as CaCO ₃	1,600	2,200	3,000	500	400	34,000
Fats.....	1,360	4,420	7,000	2,352	2,275	600
Total solids.....	19,600	21,010	27,670	10,570	10,150
Loss on ignition.....	6,900	9,960	13,310	7,970	6,200
Fixed solids.....	12,700	11,050	14,360	2,600	3,950
Shrinkage of wool scoured.....	High.	42 percent.	48 percent.

^aAnalyses by G. H. Pratt and H. Stabler.

Table 8 gives in detail the manner in which the scouring liquor at the Lorraine mill is treated, together with estimates of relative volumes at different steps based on 1,000 gallons of scouring liquor. The probable composition of the various materials is also estimated.

TABLE 8.—Disposal of wool-scouring liquor at the de gras plant of Lorraine mill, Pawtucket, R. I.

Source.	Total amount of material.		Water.		Composition.			
					Total solids.	Volatile solids.	Fixed solids.	Fats.
	Galls.	Pounds.	Galls.	Pounds.	Pounds.	Pounds.	Pounds.	Pounds.
Scouring liquor.....	1,000	8,520	948	7,941	579	406	173	291
Acid used.....	5.2	77	1.5	13
Liquor from acid-treating tanks.....	620	5,290	619	5,164	126	60	66	30
Filter effluent.....	225	2,174	254	2,122	52	25	27	12
Material pressed.....	1,093	80	668	425	321	104	249
Liquor from cisterns.....	167	1,405	167	1,391	14	10	4	3
Liquor from purification of grease.....	7	60	7	60	Trace.	Trace.
Grease recovered.....	183	Trace.	183	Trace.	183
Press cake.....	255	3	27	228	128	101	63
Total liquid effluent.....	1,049	8,929	1,047	8,737	192	95	97	45

Comparison of the raw liquor and the total liquid effluent indicates a reduction of 66.7 per cent in total solids, 76.6 per cent in volatile solids, 46 per cent in fixed solids, and 84.5 per cent in fats. This is a much better purification than has been obtained by the usual sewage treatment methods.

Table 9 gives a comparison of the results obtained at the Lorraine mill and at several English establishments, as calculated from analyses of the raw liquor and of the effluent from the acid-treating tanks:

TABLE 9.—Per cent of solids and grease removed from or added to wool-scouring liquor by cracking process.

Factory.	Total solids.	Volatile solids.	Fixed solids.	Grease.
Greenwood & Hanson.....	-66	-78	- 43	-82
Broadbent.....	+39	+ 9	+125	-20
Schofield.....	- 3	-26	+ 80	-83
Millor.....	- 8	-13	+ 13	-50
Shaw.....	+10	-11	+125	-32
Average.....	- 6	-24	+ 60	-53
Lorraine, March 22-29.....	-69	-80	- 36	-88
Lorraine, May 17-23.....	-61	-71	- 41	-78

NOTE.—Plus signs indicate material added to the liquors; minus signs indicate material removed from the liquors.

The data for the English establishments are quoted from Naylor^a as representing the "rule of thumb" treatment of the early days of the process. The comparison is favorable to the operations at the Lorraine mill.

Turner-Akeroyd process.—A modification of the cracking process has been patented in England by Turner and Akeroyd. The usual procedure is most successfully applied to a highly concentrated liquor. The Turner-Akeroyd modification, on the contrary, is applied most successfully to dilute liquors, and can, therefore, be used for the treatment of piece-scouring suds or other comparatively dilute soapy waste waters. The process is intended to effect a more complete separation of fats than can be accomplished in the ordinary method of treatment. The scouring liquor is collected in a precipitation tank, where it is diluted to such specific gravity that the fats which have separated on the addition of acid can all readily sink to the bottom, leaving a clear supernatant liquor. The liquor is agitated for several hours by blowing air through distributors in the bottom of the tank. Acid is then added, and if this alone will not produce the desired separation of fats, a small amount of fine earth is introduced in order to give weight to the precipitate and carry it to the bottom. The treatment of sludge follows the methods of the ordinary cracking process. The system includes filtration of the liquor from the acid-treating tank through gravel overlain with a few inches of ashes and topped with a sprinkling of sawdust. Grease gathers on the surface of the filter and is collected from time to time and pressed for the recovery of fats.

The following table shows the character of the liquors in a Turner-Akeroyd plant at Pittsfield, Mass.:

^a Naylor, W., Trades waste; its treatment and utilization, 1902, p. 75 (quoted from Third Rept. Rivers Pollution Commissioners, vol. 1, p. 32.)

TABLE 10.—Analyses of waste liquors from Turner-Akeroyd plant at Pontusac mill, Pittsfield, Mass.^a

[Milligrams per liter.]

	Scouring liquor.	Liquor from acid-treating tank.	Filter effluent.
Turbidity.....	4,500	370	50
Oxygen consumed.....	410	89	64
Nitrogen as free ammonia.....	2.6	5.5	5.6
Organic nitrogen.....	47.6	9.1	6.1
Alkalinity as CaCO ₃	980	^b 425	^b 170
Sulphate radicle (SO ₄).....	Small amount.	1,970	1,660
Iron (Fe).....	Trace.	5	2.5
Fats.....	2,140	175	38
Total solids.....	4,740	2,460
Volatile solids.....	3,200	100
Fixed solids.....	1,540	2,360

^aAnalyses by G. H. Pratt and H. Stabler.^bAcidity.

The analyses indicate more thorough purification than is obtained by the other methods of treatment that have been discussed. A sample of press cake from this plant contained 2.6 per cent moisture, 78.4 per cent volatile solids, 19.0 per cent fixed solids, 22.4 per cent fats, and 3.8 per cent nitrogen. Effluent liquors from the ordinary cracking or the Turner-Akeroyd process can be further purified by neutralization with lime and subsequent filtration. This procedure is followed in many European plants where a high degree of purification is essential.

Battage process.—The *battage* process of grease recovery is followed successfully in France. The scouring suds are conveyed into a narrow tank divided into numerous compartments and provided with agitators which beat up the liquor, raising froth which carries the fats to the surface. The greasy lather thus produced is removed by means of scrapers. Various modifications of this process provide for agitation by means of beaters, compressed air, superheated steam, and other agencies, the object of all being to produce a froth which can readily be removed by skimming. The grease removed in this way is then recovered by treatment with acid and by pressing, as in the cracking process. Several qualities of grease may be obtained. The lather produced in the first compartments is light colored, and it yields a grease of much better quality than that which is obtained from the lather skimmed from the last compartments of the apparatus.

Smith-Leach process.—Another process results in the recovery of fats by means directly opposed to those employed in the Turner-Akeroyd patent. The first procedure is concentration instead of dilution, and the scouring liquor is thus reduced to one-tenth or a less proportion of its original volume. It then has specific gravity of about 1.15 (20° Baumé, or 30° Twaddell). At this concentration the liquor has a sirupy consistency, and on sedimentation and cooling the grease collects at the top and is removed by skimming. In the Smith-Leach modification of this process the separation of the grease

from the concentrated liquor is accomplished by means of a centrifugal machine similar to the ordinary cream separator. The grease so obtained is nearly pure wool fat (60 to 70 per cent of the entire grease), which commands a price twice as great as that of the grease recovered by the cracking process.

Potash recovery alone has not been applied to ordinary wool-scouring liquor, but it has been used in connection with grease recovery. When the liquor is treated with acid, the value of the potash product is greatly reduced, because the potassium is changed from the carbonate to the sulphate. The electrolytic regeneration of the acid with coincident production of caustic potash has been suggested for the acid liquors, but so far as known it has not been successful on a practical scale because of the high cost of the electric current required. The liquor resulting from washing wool after degreasing with volatile solvents and the waste liquor from the battage grease recovery can be utilized for potash recovery as in the case of steep liquor. Where concentration is used for grease recovery, the utilization of the concentrated waste for potash recovery is a very simple matter, for a little additional concentration, followed by calcination in a reverberatory furnace or in the ordinary rotary incinerator used for soda recovery in soda pulp mills produces a crude potash which has a substantial commercial value. Extraction of this crude product followed by concentration and calcination of the resulting liquor produces a high grade of potash.

Such treatment is a part of the patented Smith-Leach process, which is the highest development of utilization of wool-scouring liquor that has been put into practice. A plant using this complete process has been in practical operation for several years at the Field Head mills, Bradford, England.

The scouring liquor flows through settling tanks to remove the greater part of the sand and mud. From the tanks the liquor passes to a quadruple-effect Yaryan evaporator, which reduces it to one-tenth or one-fifteenth of its original volume. The condensed vapor from this machine provides distilled water of excellent quality for scouring purposes. After the concentrated liquor has been heated nearly to the boiling point, it is passed through a centrifugal separator, which separates it into an outer layer of sand and mud, a middle layer of soapy liquid containing all the potash, and an inner layer consisting almost entirely of wool grease. The sand and mud adhere to the separator and are removed from time to time by hand. The potash liquor and the wool grease are caught up separately and conveyed to separate receptacles. The wool grease is purified by warming it with water and allowing it to separate again by cooling. The potash liquor, which contains a large amount of fat combined as soap, is further concentrated to about one-fourth its volume, and it is then passed through a revolving cylindrical incinerator in which

the remaining water is evaporated and the organic matter is burned, leaving crude potassium carbonate. In this manner the scouring liquor is entirely disposed of and the following products are derived from it: Distilled water, wool grease, crude potassium carbonate, and sand and mud.

The distilled water is not perfectly pure, but contains small amounts of ammonia and grease, which have passed over during evaporation. The patentees claim that 15 to 30 per cent less soap is required in scouring when this distilled water is used. Moreover, the recovered water is at the temperature required for wool scouring. The wool grease is free from mineral and fatty acids, and it can be used without further purification as a lubricant or for any of the purposes for which Yorkshire grease finds a market. The potash salt contains from 50 to 70 per cent of pure potassium carbonate and it can be used in its crude state for wool scouring, or it can be sold for purification. The sand and mud removed by the separator are without market value. The advantages of the process are the entire destruction of the scouring liquor; the treatment of the suds in a fresh state, so that the whole process can be carried on without the production of the noxious odors so common in the ordinary cracking process; the comparatively small space occupied by the whole apparatus, and the profit which can be realized from the recovered products. The prominent disadvantages are the cost of installation for the evaporator and the coal consumption necessary for the vaporization of the water which the liquor contains. Table 11 shows the composition and the amounts of liquor at different steps in the Smith-Leach process as conducted at Field Head mills. The figures are computed from analyses by F. W. Richardson, and 1,000 gallons of liquor are used as a basis.

TABLE 11.—Disposal of wool-scouring liquor at Smith-Leach plant of Field Head mills, England.

Source.	Total amount of material.		Water.		Composition.				
					Total solids.	Grease.	Organic impurities.	Soluble mineral matter.	Insoluble mineral matter.
From evaporator:	<i>Galls.</i>	<i>Lbs.</i>	<i>Galls.</i>	<i>Lbs.</i>	<i>Lbs.</i>	<i>Lbs.</i>	<i>Lbs.</i>	<i>Lbs.</i>	<i>Lbs.</i>
Crude suds.....	1,000	8,452	970	8,100	352	174	78	73	27
Concentrated suds.....	103	959	73	607	352	174	76	63	39
Condensed water.....	897	7,493	897	7,493	.5	.4	0	.1	0
From separator:									
Grease.....	30	125	.6	5	120	118	.6	1
Potash liquor.....	73	832	72	601	231	56	55	64	56
Earthy matter.....	0	2	0	.5	1.5	.2	0	0	1.3
	103	959	73	606	352	174	56	64	58
From evaporating incinerator:									
Crude potash.....		94	0	0	94	2	64	28
Loss.....		738	72	601	137	56	53	0	28
		832	72	601	231	56	55	64	56

The results of one week's operation of the plant at the Field Head mills are given in the following balance sheet. This record is adapted from the unpublished report of H. McLean Wilson, chief inspector of the West Riding rivers board, under whose direction the test of the plant was made. During the week of the test half of the wool washed was scoured wool. Consequently the waste liquor was very dilute and it forms an extremely conservative basis for estimates. Furthermore, the plant has double the capacity required for the amount of liquor treated and the fixed charges are therefore relatively high. The scouring liquor had presumably about the following composition in parts per million: Total solids 18,400 (volatile solids 13 100, fixed solids 5,300), fatty matters 8,900.

Balance sheet of Smith-Leach plant of Field Head mills, England, during week's test.

Cost of plant.....		\$21,900
Amount of wool scoured.....	pounds..	65,256
Amount of scouring liquor.....	gallons..	66,000
VALUE OF PRODUCTS.		
3,300 pounds of wool fat.....		\$146. 00
2,830 pounds of crude potash.....		93. 98
53,100 gallons of distilled water.....		6. 69
Soap saved by using above water— one-seventh total soap used		10. 22
		256. 89
COST OF OPERATION.		
Coal, 19 tons (41,780 pounds).....		50. 54
Labor, 3 men.....		16. 60
Interest and depreciation, 10 per cent.....		42. 10
		109. 24
Profit.....		147. 65
Profit per annum.....		7,677. 80
Per cent of profit.....		35

The foregoing profits could not be realized in this country, owing to the greater cost of labor and the higher price of coal in the textile centers of the United States. The figures are of interest, however, for they show what has actually been done on a practical scale under adverse working conditions.

ECONOMY OF TREATMENT.

Reports of plants in operation in the United States indicate that the cracking and Turner-Akeroyd processes result in the recovery of sufficient grease to pay all costs of operation. In general, with the market value of recovered grease less than 2 cents a pound, the operations are conducted at a loss, but with higher values a profit is obtainable. The market value ranges from 1½ cents to 3½ cents, with a probable average of 2 cents a pound, so that little, if any, profit is to be expected from these processes under American conditions of practice.

A plant equipped for the Smith-Leach process and capable of utilizing 500 gallons of wool-scouring liquor an hour, ten hours a day, three hundred days a year, would cost approximately \$13,000. The fixed charges of operation (interest, depreciation, and repairs) may be safely estimated at 11 per cent of the cost, or \$1,430 a year. The cost of labor (3 men) would not exceed \$1,650 a year. Fuel, 300 tons of coal at \$4.50, allowing 10 per cent for banked fires fourteen hours daily, would amount to \$1,350 a year if a triple-effect vacuum evaporator is used. If \$200 a year is allowed for incidentals, the total cost of operation would amount to \$4,630. In order to make the estimate more conservative, the costs may be placed at \$5,000 a year, or \$1 for each gallon of the daily capacity of the plant. In return for this expenditure, wool fat valued at 4½ cents a pound and crude potash worth 5 cents per pound of potassium carbonate content would be recovered.

The recoverable wool fat may be safely assumed to be 60 per cent of the fats indicated by analysis of the liquor. Numerous tests on the ash of scouring liquors indicate that the recoverable potash is equal to or greater than the potassium carbonate equivalent of the alkalinity of the liquor, and this equivalent may therefore be taken as a conservative estimate of the recoverable potash. The following estimate of costs, values, and profits, based on the foregoing facts and assumptions, has been made for treatment of the liquors represented by the analyses in Table 3 (p. 13). The numbers for the column headings have the same significance in both tables.

TABLE 12.—*Estimate of costs, values, and profits for application of Smith-Leach process to various wool-scouring liquors.*

	1.	2.	3.	4.	5.	Average (omitting 2).
Value of wool fat.....	\$4,920	\$19,920	\$12,890	\$10,680	\$6,820	\$8,830
Value of potash.....	6,920	6,920	2,680	3,300	2,460	3,840
Cost.....	11,840	26,840	15,570	13,980	9,280	12,670
	5,000	5,000	5,000	5,000	5,000	5,000
Profit.....	6,840	21,840	10,570	8,980	4,280	7,670
Per cent profit on investment of \$13,000.....	53	168	81	69	33	59

Column 2 in Table 12 is calculated from an abnormal analysis, and estimates based on it were therefore omitted from the average. Column 5, representing the liquor treated at the Field Head mills in England, gives lower values than any other. The results previously given for a week's test at those mills indicate a 31 per cent profit from grease and potash under the working conditions—comparatively low labor costs and unnecessarily high cost of plant. The check obtained by this figure is an excellent one. It should be noted that in the test at the Field Head mills an additional 4 per cent profit is derived from the distilled water obtained and the soap saved through its use. This is an additional advantage of the process that is by no means negligible, although it has been omitted from Table 12 in order

that the estimates may be more conservative. If, as has been proved by several years' operation, the dilute liquors at the Field Head mills can be made to yield a profit, it is to be expected that the more concentrated liquors examined in the United States would give even better results. The average of 59 per cent profit, or \$7,670 a year on an investment of \$13,000, leaves a wide margin for marketing recovered products, and this is believed to be a conservative estimate. Larger plants could be operated even more economically, but smaller ones would necessarily be placed at a disadvantage because of relatively higher costs of maintenance. The effect of different concentrations of fat and potash in the liquors is shown in Table 12. Expressed in unit values, 385 parts per million of fat or 150 parts per million of alkalinity make a difference of 1 per cent in the estimated profits. The character of the wool scoured, and more especially the amount of water used in scouring it, influence the economy of treatment.

SUMMARY.

1. Wool is impregnated with many impurities which must be removed before it can be used in the manufacture of textiles.

2. The common methods of cleansing wool are: (a) Scouring with soap, alkali, and water; (b) steeping and scouring; and (c) scouring with volatile solvents. Such treatment produces great quantities of foul liquors containing valuable substances, which can usually be recovered profitably with coincident purification of the liquors.

3. The scouring liquor most frequently encountered in the United States is the waste suds from scouring with soap, alkali, and water. Purification of this liquor by the usual methods of sewage treatment is difficult at best and usually impracticable. Purification combined with recovery of grease or potash is possible and can generally be accomplished at little or no expense. The degree of purification and the coincident profit on the capital invested for three prominent systems of treatment are shown in Table 13.

TABLE 13.—*Degree of purification and profit of different methods of treating wool-scouring liquor.*

	Per cent purification.			Per cent profit on capital invested.
	Total solids.	Volatile solids.	Fats.	
Cracking process.....	65	75	83	0
Turner-Akeroyd process (including filtration).....	48	97	98	0
Turner-Akeroyd process (excluding filtration).....	92	0
Smith-Leach process.....	100	100	100	59

4. All the evidence gathered tends to show that wool-scouring liquor can be entirely destroyed by means of the Smith-Leach process, and that sufficient valuable products can be recovered therefrom to pay all costs of treatment and leave a substantial profit on the capital invested.

BLEACHING AND DYEING COTTON YARN.**BLEACHING PROCESSES AND WASTE WATERS.**

The methods of bleaching and dyeing cotton yarn do not differ in essential principles from those of bleaching and dyeing other cotton goods, but the form of the material and its condition with respect to impurities render the processes more or less distinctive.

Gray boil.—The yarn when brought to the bleachery contains dirt that has accumulated during transportation as well as other impurities that must be removed before bleaching. The first process is cleansing in the gray boil. The yarn is placed by hand in closed iron kiers, after which water and steam are admitted. The desired amount of alkali, usually sufficient to make a 0.5 to 1 per cent solution of caustic soda, is then added, and boiling with open steam pipes is continued for ten or twelve hours. The kiers are so designed that the solution circulates through the yarn, and an open steam exhaust keeps the pressure very low. The natural impurities, including cotton wax, margaric acid, pectic acid, parapectic acid, and albuminous matters, and constituting from 3 to 5 per cent of the weight of the yarn, are emulsified and dissolved by boiling in the dilute alkaline solution, which does not appreciably affect the cotton fiber. At the end of the boil the liquor is drawn off and the yarn is thoroughly rinsed with fresh water.

Bleaching.—After it has been rinsed and drained the yarn is removed from the kiers and placed in stone or concrete vats. Bleaching or "chemic" solution, usually chloride of lime of a strength represented by a specific gravity of 1.01 (2° Twaddell, or 1.5° Baumé), is added until the yarn is covered. The material is steeped for a period of two to four hours, and the solution is drained off and returned to storage tanks for further use. The steeped yarn is then rinsed by covering it with water and draining; two such rinsings are usually given in order to remove excess of chemic solution. A weak acid, or "sour" solution, usually sulphuric acid of 1.005 specific gravity (1° Twaddell, or 0.75° Baumé), is then admitted in sufficient quantity to cover the yarn. The chemic solution remaining in the material reacts with the acid, and bleaching by oxidation results. After steeping a few hours, the acid is drawn into a storage tank and held for further use.

Cotton fiber has the property of concentrating within itself acid from a solution; furthermore, it is sensitive to acid and easily injured by it; for these reasons, and also in order to prevent additional and uncontrolled bleaching action, the material must be cleansed from all traces of acid at this stage. This is accomplished by three successive rinsings with fresh water in the masonry vats and by "soaping," which is customarily done by hanging the skeins of yarn on wooden

sticks and moving them by hand through narrow wooden vats which contain dilute soap solution. The soap effectually neutralizes any remaining acid and leaves the fiber in proper condition. A final rinsing in clear water in similar vats removes the excess of soap from the yarn. The soaping and rinsing may be effected by means of revolving wheels on which the sticks bearing skeins of yarn are fastened like the cars on a Ferris wheel. Partial drying in centrifugal machines, drying by heat, and winding on spools are the finishing processes.

Wastes.—The waste waters from the bleaching processes are described in the following paragraphs. The estimates of quantity of the different materials are based on the treatment of 1 ton of yarn.

Waste A, gray-boil liquor: Usually 250 to 1,000 gallons in amount, with an average of 600 gallons; contains 25 to 30 pounds of sodium compounds expressed in terms of caustic soda and 60 to 75 pounds of impurities from the cotton; reddish brown or garnet in color and has a characteristic odor like that of burnt sugar.

Waste B, gray-boil rinse: Amounts to about 3,000 gallons; contains about 10 to 15 pounds of sodium compounds and 25 to 40 pounds of impurities from the cotton; in reality a diluted portion of waste A. As the rinsing is usually accomplished by a continuous stream of water, this waste varies in character from a concentration nearly equal to that of waste A to nearly pure water.

Waste C, first chemic rinse: About 1,000 gallons in amount; contains small quantities of sodium compounds and cotton impurities and comparatively large amounts of calcium chloride, some sulphates, and some free chlorine, giving total solids of 10 to 15 pounds; characterized by a strong chlorine odor.

Waste D, second chemic rinse: Similar to waste C in quantity and in quality except that it is much more dilute, containing only about one-fifth as much matter in solution.

Waste E, first sour rinse: Amounts to about 1,000 gallons, like the other sour rinse waters and the chemic rinses; has a slight odor of chlorine and contains 20 to 25 pounds of free acid and 10 to 15 pounds of calcium sulphate; other impurities are small in amount.

Waste F, second sour rinse: Practically a dilution of waste E; character determined by the quality of water used for rinsing, from which it differs by having a slightly lower alkalinity and a somewhat increased sulphate content.

Waste G, third sour rinse: Similar to waste F.

Waste H, soap liquor: Amounts to about 4,500 to 5,000 gallons; light blue in color by reason of the addition of a small amount of bluing to whiten the goods; has a characteristic soapy appearance, with high turbidity, alkalinity, and fat content, 30 to 40 pounds of soap having been used.

Waste I, soap rinse: Same in quantity as waste H, of which it is a dilution; contains one-fifth to one-tenth as much soap and organic matter as waste H and is too dilute to warrant much consideration.

Waste J, lime sludge: Chloride of lime is agitated with water in cement-lined tanks in order to prepare the bleaching solution. As the chloride of lime is never pure, an insoluble sludge collects at the bottom of the tanks and is removed periodically. The sludge is semi-liquid in consistency and contains chlorides, hypochlorites, hydrates, carbonates, and sulphates of calcium, together with small amounts of other substances. The amount of sludge is 30 to 50 pounds, varying in quantity and in consistency with the quality of bleaching powder and with minor changes in manipulation.

An excellent opportunity was afforded the writers to study the processes and waste waters from the bleaching and dyeing of cotton yarn at the establishment of the R. D. Mason Company, Pawtucket, R. I.

Table 14 shows the character of the wastes from cotton-yarn bleaching at this establishment. Wastes A, B, E, and H are the most objectionable.

TABLE 14.—Character of waste liquors from bleachery of the R. D. Mason Company, Pawtucket, R. I.^a

[Milligrams per liter, unless otherwise stated.]

	Waste A (gray-boil liquor).	Waste C (first chemie rinse).	Waste D (second chemie rinse).	Waste E (first sour rinse).	Waste F (second sour rinse).	Waste G (third sour rinse).	Waste H (soap liquor).	Waste I (soap rinse).
Date, 1907.....	Mar. 7	Feb. 21	Feb. 21	Feb. 21	Feb. 21	Feb. 21	Feb. 21	Feb. 21
Amount in gallons.....	2, 400	3, 200	3, 200	3, 200	3, 200	3, 200	14, 000	15, 000
Color.....	Orange.	21	28	12	21	21	Light blue.	Slight soapy.
Odor.....	Burnt sugar.	Chlorine.	Slight chlorine.	Slight chlorine.	None.	None.	Soap and chlorine.	Soap.
Turbidity.....	650	10	5	5	5	5	400	50
Sediment.....	Considerable.	Considerable.	Very slight.	Slight.	Slight.	Slight.	Slight.	Very slight.
Oxygen consumed.....	4, 400	17.5	3.5	9.2	1.7	1.5	84	12
Nitrogen as free ammonia.....	6.5	.17	.05	.02	.05	.04	.25	.15
Organic nitrogen.....	234	1.00	.50	1.00	.35	.30	2.00	.90
Chlorine.....	260	635	72	70	24	24	34	20
Alkalinity as CaCO ₃	440	Slight.	Slight.	^b 2, 790	30	30	345	53
Sulphate radicle (SO ₄).....	360	408	78	3, 800	47	44	60	54
Fats.....	3, 520						380	46
Total solids.....	17, 920							
Loss on ignition.....	9, 320							

^a Analyses by Pratt and Stabler.

^b Acid.

DYEING PROCESSES AND WASTE WATERS.

Wet-out process.—Though cotton yarn must be cleansed before it can be satisfactorily dyed, dyeing does not always require so thorough a preliminary treatment of the yarn as bleaching does. The cleansing process, or “wet out,” is conducted in kiers like those used for the gray boil before bleaching. The yarn is piled in these kiers and boiled

with water and steam but without chemicals for about six hours. After the boiling has been completed, the material is rinsed in the kiers and then allowed to drain for several hours, after which it is ready for dyeing. For some colors, the entire bleaching process may be substituted for the wet-out boil.

Dyeing.—Dyeing operations are many and varied, and they are usually conducted in the form of apparatus described for the soaping process in bleaching. Machines are generally employed for heavy colors, and the dye baths are saved for further use. The lighter colors are more often applied by the hand process, and the baths are discharged after having been used but once. The following abstracts from Thorp's text-book ^a give some idea of the varied character of the liquors discharged from a dyehouse for cotton yarn.

Direct dyes, or those which yield full colors without the assistance of mordants, are applied in a boiling bath usually with 10 to 15 per cent of common salt or Glauber's salt. Ingrain colors require in addition "diazotizing" in a cold bath of sodium nitrite acidulated with hydrochloric acid, and "developing" by treatment with phenols, naphthols, or amines. Basic dyes, or those which form insoluble tannates and require mordants on vegetable fibers, are the salts of colorless bases. The cotton is first mordanted in a bath of tannin, turkey-red oil, or soap, and it is then treated in a bath of the dye-stuff. The base of the dyestuff combines with the acid of the mordant to form the color, and the goods are not washed after having been dyed. Acid dyes are applied in a very concentrated bath to which common salt, acetic acid, and alum are added, or the yarn may be first mordanted in a bath of alum and soda or of stannic chloride followed by alum and soda. Tannin treatment may precede mordanting with alum and soda. Mordant dyes for cotton are chiefly turkey reds, logwood blacks, and alizarin colors, and they always require a metallic mordant, such as the salts of aluminum and less frequently those of chromium, iron, and tin. Oil, sodium carbonate, powdered chalk, sodium phosphate, sodium arsenate, and calcium acetate are some of the accessory chemicals that may be used in the processes. After having been dyed, the yarn may be subjected to soap baths. Special dyes include those colors which are prepared or developed directly on the fiber by peculiar processes and include indigo, aniline black, and certain azo colors. The baths may contain sodium hyposulphite, copperas, zinc compounds, milk of lime, potassium bichromate, copper salts, potassium ferrocyanide, and many other chemicals.

Wastes.—Any or all of the classes of dyes mentioned above may be applied in a dyehouse during a single day, or one class may be used

^a Thorp, F. H., *Outlines of industrial chemistry*, 1901, pp. 482-494.

one day and another the next. It is impossible, therefore, to predict the nature or the color of the liquor in a dyehouse sewer. Average results for a day's run offer the best opportunity for comparisons, but even these may vary widely from day to day. The quantity of liquor discharged also varies considerably, as one bath may suffice for fixing some colors, while seven or eight baths may be required for fixing others. The waste liquors from a cotton-yarn dyehouse may be classed under three heads, as follows:

Waste K, wet-out liquor: Orange colored; has a peculiar odor like that of scorched vegetable matter, and contains a large amount (about 3 per cent by weight of the yarn) of the impurities, chiefly organic, removed from the fiber; in quantity it averages about 600 gallons per ton of yarn. Similar in many respects to waste A. (See p. 28.)

Waste L, wet-out rinse: A dilution of waste K, concentrated at first, but becoming more nearly pure water as the rinsing progresses; usually amounts to from 2,500 to 3,000 gallons and contains 10 to 20 pounds of impurities per ton of yarn.

Waste M, sewage from dyeing processes: Of extremely varied character; changes color at frequent intervals and varies in reaction from strongly acid to excessively alkaline. The quantity may be roughly estimated at 20,000 to 30,000 gallons per ton of yarn, and the content of impurities at from 250 to 400 pounds of dyes, and other chemicals per ton of yarn.

The following table shows the general characteristics of waste K and the nature of waste M for one day at a yarn dyehouse:

TABLE 15.—Character of waste liquors at cotton dyehouse of the R. D. Mason Company, Pawtucket, R. I.^a

[Milligrams per liter.]

	Waste K ("wet-out" liquor).	Waste M (dye-house sewage).
Date, 1907.....	Mar. 7.	Mar. 6. ^b
Amount..... gallons.....	2,400	90,000
Color.....	Orange.	Red to purple.
Odor.....	Scorched vegetable matter.	Dyes.
Turbidity.....	70	300
Sediment.....	Considerable.	Heavy.
Oxygen consumed.....	1,140	240
Nitrogen as free ammonia.....	7.2	1.8
Organic nitrogen.....	106	5.25
Chlorine.....	115	250
Alkalinity as CaCO ₃	400	40
Sulphate radicle (SO ₄).....	120	530
Fats.....	429	27
Starch.....	Present.
Total solids.....	4,430	1,470
Loss on ignition.....	2,860	450

^a Analyses by Pratt and Stabler.

^b Composite.

PURIFICATION OF WASTE WATERS.

PREVIOUS INVESTIGATIONS.

Experimental treatment by the Massachusetts state board of health^a of a composite liquor composed of six of the most objectionable wastes from the processes of dyeing, bleaching, and mercerizing cotton yarn, mixed in proportion to the quantity of each waste, showed that about 87 to 90 per cent removal of organic matter was effected by sand filtration, and that about 92 to 93 per cent removal was obtained by the use of chemical precipitants followed by sand filtration. Though the waste was a very turbid brown liquor, the effluents were straw colored. A rate of 50,000 gallons per acre per day was used in the work.

Experimental work on waste dye liquors from a woolen mill was also conducted.^b While this waste is not derived from the industry under discussion, it seems proper to mention here the general conclusions reached, as follows: The waste dye liquor from a woolen mill can be successfully treated by sand filtration at sewage rates; a large part of the color will be removed, but more surface raking is required than is necessary with sewage. The addition of copperas followed by lime in amounts averaging about one-half ton each for 1,000,000 gallons will effect good color removal when the liquor is allowed to settle about one hour; the resulting supernatant liquor can be filtered through sand at rates approximating 2,000,000 gallons per acre per day with good results. The sludge will be about 1.5 per cent of the volume of the liquor treated and can be satisfactorily filtered at a rate of 40,000 to 50,000 gallons per acre.

Though the wastes from a cloth dyeing and finishing works at Ravenna, Ohio, studied by Stabler,^c were liquors from dyeing wool, they resemble those under discussion sufficiently to justify mention. His work indicated that for chemical precipitation of such liquors lime and copperas together gave better purification at less cost than either alone. Separate treatment of the concentrated dye wastes was more economical than treatment with the rinse water, "crab" waste, and a waste from washing and dyeing wool. Straining these four combined through sifted coke breeze at a rate of 520,000 gallons per acre per day removed from one-third to one-half the organic matter present. Results from the operation of an experimental plant at Ravenna confirmed the conclusion drawn from laboratory tests on the use of lime and copperas as precipitants, and indicated

^a Thirty-eighth Ann. Rept. Massachusetts State Board Health, 1906, p. 301.

^b Thirty-fifth Ann. Rept. Massachusetts State Board Health, 1903, p. 271.

^c Stabler, Herman, Disposal of waste water from cloth dyeing and finishing works at Ravenna, Ohio. Ohio Sanitary Bull., vol. 10, 1906, p. 189.

that the combined wastes were fairly well purified when treated with 12 pounds of copperas and from 8 to 10 pounds of lime per 1,000 gallons, at a cost of about 7½ cents. The concentrated dye waste, taken alone, showed about 70 per cent removal of organic matter when treated with 42 pounds of copperas and 15.8 pounds of lime per 1,000 gallons, at a cost of about 23 cents.

LABORATORY EXPERIMENTS.

Several experiments, which were not refined in detail, were made in the laboratory by the writers on liquors from the Mason establishment. Waste A, the gray-boil liquor, was treated with different amounts of sulphuric acid. Complete coagulation was most nearly obtained by using 58 pounds of sulphuric acid per 1,000 gallons. The liquor became clear in about two minutes and the coagulum, probably a very impure pectic acid, was reduced in half an hour to 5.6 per cent, by volume, of the original liquor. Analyses before and after treatment showed the following results:

TABLE 16.—Analyses of waste A before and after treatment with sulphuric acid.^a

[Milligrams per liter.]

	Before treatment.	After treatment.
Oxygen consumed.....	4,400	3,320
Organic nitrogen and nitrogen as free ammonia.....	240	156
Fats (ether-soluble matter).....	3,520	2,050
Alkalinity as Ca CO ₃	440	—3,500
Sulphate radicle (SO ₄).....	360	7,900
Totalsolids.....	17,920	20,280
Loss on ignition.....	9,320	9,580

^a Analyses by Pratt and Stabler.

These analyses do not show great improvement in the quality of the liquor, though there is a decided reduction in oxygen consumed, nitrogen, and fatty matters. The dried coagulum was largely volatile, 96.6 per cent being lost on ignition. Less visible purification was procured by the use of greater or less amounts of acid and by the substitution of hydrochloric for sulphuric acid. The substance removed by this coagulation contained much coloring matter, and was readily soluble in alkaline solutions, especially solutions of the caustic alkalies.

Equal parts of waste A, the gray-boil liquor, and waste K, the "wet-out" liquor, were treated with sulphuric acid in the ratio of 58 pounds of the acid to 1,000 gallons of waste. The coagulum gathered and the liquor cleared as in the preceding experiment. The following

analyses show, as would be anticipated, that the relative purification is similar to that of the former experiment; 98.1 per cent of the dried coagulum was lost on ignition:

TABLE 17.—*Analyses of wastes A and K (mixed) before and after treatment with sulphuric acid.*^a

[Milligrams per liter.]

	Before treatment.	After treatment.
Oxygen consumed	2,770	1,720
Organic nitrogen and nitrogen as free ammonia.....	177	113
Fats (ether-soluble matter).....	1,975	1,370
Alkalinity as CaCO ₃	420	-3,750
Sulphate radicle (SO ₄).....	240	7,200
Total solids.....	11,180	13,400
Loss on ignition.....	6,090	6,590

^a Analyses by Pratt and Stabler.

Waste E, the first sour rinse, and waste H, the soap liquor, were combined in the proportion in which they occur in the bleachery, 1 part of the former to 4.38 parts of the latter. The result was a coagulation of the soap. The following analyses of the combined liquors and the filtrate after coagulation disclose very decided purification of both wastes:

TABLE 18.—*Results of mixing wastes E and H.*^a

[Milligrams per liter.]

	Combined liquors.	Filtrate.
Color.....	Light blue.	15
Turbidity.....	330	0
Oxygen consumed.....	70	23.2
Organic nitrogen and nitrogen as free ammonia.....	2.02	1.75
Fats.....	310	12
Acidity as CaCO ₃	240	200

^a Analyses by Pratt and Stabler.

Waste M, the dyehouse sewage, was treated with milk of lime at the rate of 2½ pounds per 1,000 gallons (17.5 grains per United States gallon) and 3½ pounds per 1,000 gallons (23.3 grains per United States gallon). The former mixture was filtered after standing sixteen hours, and the latter after standing five hours, and the filtrates were both subjected to analysis, with results as shown in Table 19.

TABLE 19.—*Results of treating waste M with lime.*^a

[Milligrams per liter.]

	Waste M (dye-house sewage).	After treatment with 2½ pounds of lime per 1,000 gallons, and after 16 hours' sedimentation.	After treatment with 3½ pounds of lime per 1,000 gallons, and after 5 hours' sedimentation.
Color	Reddish purple.	Light straw.	Very light straw.
Turbidity	300	0	0
Sulphate radicle (SO ₄).....	530	350	350
Alkalinity as CaCO ₃	40	124	80
Oxygen consumed.....	240	102	99
Organic nitrogen and nitrogen as free ammonia	7.05	3.25	3.45

^a Analyses by Pratt and Stabler.

The color of the filtrates faded on standing. Both parts of the experiment show very good decolorization and removal of more than 50 per cent of the organic matter. The lower percentage of lime, with longer time for reaction and sedimentation, seems to give fully as good results as the second treatment.

No experiments were made with the lime sludge, waste J. It is a precipitant of great value and could doubtless be used in place of lime in a precipitation plant, thus reducing the cost of chemicals and disposing of an additional waste.

EXPERIMENTS AT PAWTUCKET, R. I.

Waste M, the liquor causing most serious pollution from the R. D. Mason plant, except the bleach-house boils, is discharged through the dyehouse drain. Filtration experiments having been started at another plant with kier liquors (see p. 47), the work at this plant was confined to the wastes from the dyeing processes, the wet-out liquor, however, not being included in samples taken or applied to the filters, as it was discharged very early in the morning.

Character of wastes.—The liquors which are discharged from the dyeing processes at this establishment are typical. They are disagreeable to look at, on account of the coloring matter in them, and they exert an influence on the stream into which they are discharged which is objectionable not only because of the unsightly appearance but also because of the organic matter and chemicals which they contain in solution. Samples of the liquor taken at different times have shown, as would be expected, great variations in its quality, the colors varying between the widest extremes and the organic and mineral contents showing similar fluctuations. A fairly accurate conception of the average character of the liquor with which experiments were made can be obtained from the analysis of the composite sample, given in line 1 of Table 20. The analyses of nine samples of the dye water,

collected on different days between October 10, 1907, and January 18, 1908, are also given in this table, and they show great variation in results; but an average of them, as shown by line 11, represents a liquor of nearly the same strength as the composite sample.

TABLE 20.—Analyses of liquor from dye drain of R. D. Mason Company.^a

[Milligrams per liter, unless otherwise sated.]

Sample No.	Date.	Oxygen consumed.	Nitrogen as free ammonia.	Organic nitrogen.		Solids.		Loss on ignition.		Number of bacteria per cubic centimeter.
				Total.	Dis-solved.	Total.	Dis-solved.	Total.	Dis-solved.	
1.....	1907. Mar. 6	240	1.8	5.2	1,470	450	Very high.
2.....	Oct. 10	102	1.3	2.9	2.4	1,140	1,092	206	168	
3.....	Oct. 17	144	.3	1.3	790	612	356	182	20,000
4.....	Oct. 31	257	1.4	6.4	4.8	906	782	426	340	100,000
5.....	Nov. 8	85	1.0	2.7	1.5	1,576	1,482	262	248	40,000
6.....	Nov. 21	155	1.0	1.0	570	290	
7.....	Dec. 20	354	1.0	3.0	1,174	764	Less than 10,000.
8.....	1908. Jan. 3	194	.7	1.8	740,000
9.....	Jan. 10	660	4.9	2.5	5,000
10.....	Jan. 18	218	.7	15.5	Less than 10,000.
11.....	241	1.4	4.1

^a Analyses by G. H. Pratt.

1. Composite of samples taken every half hour from 9 a. m. to 5.30 p. m.

2 to 10, inclusive. Single samples taken between 10 a. m. and 11.45 a. m., at time when filters were dosed.

11. Average of 2 to 10, inclusive.

Sand filters.—It was decided to study the results which could be obtained by applying this waste dye liquor to sand filters. The applied liquor was not selected with special reference to its appearance or composition, but was generally drawn directly from the dyehouse effluent when the attendant was ready to dose the filters. In case the effluent appeared very dilute, however, the dosing was postponed a few minutes until a normal sample could be taken. Two galvanized iron filters were installed, each 6 feet high and 20 inches in diameter, with slightly inclined bottoms to permit thorough draining through faucets at the lowest level. Underdrainage was provided by a foot of coarse cinders overlain by 10 inches of crushed stone approximately the size of pea coal. The filtering material was 3½ to 4 feet of sand, having an effective size of 0.24 millimeter and a uniformity coefficient of 3.4. The filters were set up in the yard and were subject to all effects of the weather.

Effluents.—Filter No. 1 was started on October 1, 1907, with a dose of 5 gallons a day, or at the rate of 100,000 gallons per acre per day, six days in the week, and that size of dose was continued till November 22. During this time the filter delivered an effluent of extremely good appearance, free from color due to dyes (average reading = 48), and low in organic content as shown by analysis. (See Table 21.)

TABLE 21.—*Analyses of effluent from filter No. 1 treating dye liquor.^a*

[Milligrams per liter, unless otherwise stated.]

Sample.	Date.	Color.	Oxygen consumed.	Nitrogen as free ammonia.	Organic nitrogen.	Nitrogen as nitrates.	Nitrogen as nitrites.	Chlorine.	Alkalinity as CaCO ₃ .	Total solids.	Loss on ignition.	Number of bacteria per cubic centimeter.	Rate of filtration (gallons per acre per day)
1	Oct. 10		75	9.6	1.65	0.47	0.008	796	211	2,250	100,000
2	Oct. 17		85	7.2	.65	.64	0.50	.176	1,001	87	800	100,000
3	Oct. 31		18	5.0	.80	.20032	82	493	61	73,000	100,000
4	Nov. 8		32	4.6	.80	.29016	56	696	82	100,000
5	Nov. 21		30	5.5	.16	.23011	106	524	64	36,700	100,000
6	Dec. 13	Wine.....	20.0	.16	.64	Present.	Present.	501	74	60,000	400,000
7	Dec. 20	Brownish wine...	140.0	1.36	1.54	Present.100	1,143	348	50,000	400,000
8	Jan. 3	Dark brown.....	99.2	.44	1.50100	50,000	400,000
9	Jan. 10	Dark reddish brown.....	84.0	.49	1.85	Present.	4.000	10,000	100,000
10	Jan. 18	Light brown.....	22.8	.18	1.38	1.75700	10,000	100,000
11	Feb. 21	Violet.....	42.0	.26	1.20	.70020	100,000
12			6.4	.81	.37

^a Analyses by G. H. Pratt.

1 to 11, inclusive, represent single samples taken between 10 a. m. and 12 m.

12. Average of 1 to 5, inclusive.

From November 22 to January 4 the filter was dosed at the rate of approximately 400,000 gallons per acre per day, and the combined effect of cold weather and the increased rate caused the effluent to show color; from December 13 until the last dosing on February 21 the effluent was not of good appearance, nor did it give a satisfactory analysis, though from January 4 until the filter was stopped the dose was the same as originally—100,000 gallons per acre daily. The exposure of the cans to extremely cold weather probably had an appreciable influence on the work of the filter during the latter part of the run. The fact that the experimental filters were entirely above ground allowed much greater chilling effects on their contents than would occur in a plant where only the tops are exposed. The surface of neither filter was raked during the run, but the sand was leveled two or three times. The detailed analyses of the effluent from filter No. 1 are given in Table 21.

Filter No. 2 was a duplicate of No. 1 as to construction and filtering material. It was started at the same time, October 1, 1907, at the rate of approximately 200,000 gallons per acre per day, and this rate was continued throughout the run of the filter. The same deterioration in quality and appearance of the effluent during extremely cold weather was noticed, and it is probable that to the excessive exposure of the cylinders, to which reference has been made, were due largely the comparatively poor results obtained during the latter part of the run. For the purpose of comparison, therefore, it seems best to select the results obtained during the same period used for figuring on filter No. 1—that is, from October 1 through November 21.

During this period the filter gave an effluent of fairly good appearance, at times even lower in color than that from No. 1, but a little color from the dyes seemed to pass through it always, and sometimes the shade was marked. The figures for organic nitrogen and oxygen consumed were higher on this filter, as would be expected at the higher rate.

TABLE 22.—*Analyses showing organic purification of dye liquor by filtration.*^a

Source.	Milligrams per liter.			Per cent removal.		
	Nitrogen as free ammonia.	Organic nitrogen.	Oxygen consumed.	Nitrogen as free ammonia.	Organic nitrogen.	Oxygen consumed.
Waste from dye drain.....	1.80	5.21	240
Average of 5 analyses of effluent from filter No. 1, when run at 100,000-gallon rate....	.81	.37	6.4	55	93	97
Average of 5 analyses of effluent from filter No. 2, when run at 200,000-gallon rate....	.89	.67	11.8	51	87	95

^a Analyses by G. H. Pratt.

The effluents from both filters always contained nitrites, but the nitrates were never high, although they were always present to some extent. The percentage of removal effected by filter No. 1 at the 100,000-gallon rate and by filter No. 2 at the 200,000-gallon rate on nitrogen as free ammonia, organic nitrogen, and oxygen consumed is shown in Table 22, for which the analysis of the composite sample of March 6 (line 1, Table 20) has been used as a basis for calculating percentage removal.

Detailed analyses of the effluent from filter No. 2 are shown in Table 23.

TABLE 23.—*Analyses of effluent from filter No. 2 treating dye liquor.*^a

[Milligrams per liter, unless otherwise stated.]

Sample.	Date.	Color.	Oxygen consumed.	Nitrogen as free ammonia.	Organic nitrogen.	Nitrogen as nitrates.	Nitrogen as nitrites.	Chlorine.	Alkalinity as CaCO ₃ .	Total solids.	Loss on ignition.	Number of bacteria per cubic centimeter.	Rate of filtration (gallons per acre per day).
1	1907.												
2	Oct. 10		47	3.2	2.45	0.43	(?)0.05	712	133	15,000	200,000
3	Oct. 17		35	6.2	.89	.48	.40	1,030	68	5,000	200,000
4	Oct. 31	L. purple.	28.5	.56	1.53	(?)	.14	597	92	158,000	200,000
5	Nov. 8		45	8.9	.50	.46	.14	82	31	764	110	200,000
6	Nov. 21		40	12.2	.07	.47	.026	208	30	676	81	69,000	200,000
7	Dec. 13	Wine.	27.2	.13	.56	Present.	Present.	743	74	100,000	200,000
8	Dec. 20	Brownish.	83.0	.61	1.67	Present.	1.000	22	700	223	25,000	200,000
9	1908.												
8	Jan. 3	D. brown.	68.8	.21	.93	Present.	.140	20	10,000	200,000
9	Jan. 10	D. y. brown.	85.0	.64	1.95	Present.	4.000	25,000	200,000
10	Jan. 18	D. p. brown.	28.0	.28	1.62	1.85	5,000	200,000
11	Feb. 21	Violet.	46.0	.20	1.25	1.00	200,000
12	11.8	.89	.67	200,000

^a Analyses by G. H. Pratt.

1 to 11, inclusive, represent single samples taken between 10 a. m. and 12 m.
12. Average of 1 to 5, inclusive.

Effect on sand in filters.—When the plant was dismantled on March 5, 1908, the sand in the filters was sampled at the top inch, 6 inches down, 2 feet down, and just above the crushed stone. The sand was not at all clogged, nor had it become so at any time during the run, but it was badly discolored for about 6 inches down, and discolored channels ran through the rest of the bed, though the larger part of the main body had apparently not been affected in color. The results of determining albuminoid ammonia and loss on ignition on the samples of sand, given in Table 24, show that even the surface sand was not extremely high in stored organic matter, and that it would have been suitable for a much longer run without being replaced.

TABLE 24.—Analyses showing albuminoid ammonia and loss on ignition of filter sands.^a

Sand from filter No. 1.			Sand from filter No. 2.		
Source.	Albumi- noid am- monia.	Loss on ignition.	Source.	Albumi- noid am- monia.	Loss on ignition.
	<i>Parts per million.</i>	<i>Per cent.</i>		<i>Parts per million.</i>	<i>Per cent.</i>
Top inch.....	71	1.31	Top inch.....	65	1.10
6 inches down.....	19	.42	6 inches down.....	19	.44
2 feet down.....	15	.37	2 feet down.....	14	.37
Bottom.....	12	.38	Bottom.....	8	.32

^aAnalyses by G. H. Pratt.

COST OF TREATMENT.

The following estimate, indicating the approximate cost of treating the dyehouse waste by sand filtration at the rate of 100,000 gallons per acre per day, is based on a flow of 100,000 gallons a day:

Estimated cost of sand filtration of dyehouse waste.

Cost of plant (flushing tank with automatic regulator and 1 acre of filtration beds).....	\$5,000
Annual cost of maintenance:	
Interest on \$5,000 at 5 per cent.....	250
Depreciation and repairs at 8 per cent.....	400
Labor.....	160
	810

Another estimate has been prepared on the assumption that the laboratory experiments with lime precipitation are a fair index of practical treatment of the waste in a plant similar to the apparatus used for water softening or for mechanical filtration of water.

Estimated cost of purification of dyehouse waste by lime precipitation.

Cost of plant (apparatus for chemical treatment, sedimentation, and filtration; pump; and sludge beds, with total area of about 400 square feet).....	<u>\$4,000</u>
Annual cost of maintenance:	
Interest on \$4,000, at 5 per cent.....	200
Depreciation and repairs, at 10 per cent.....	400
Pumping, at 0.25 cent per 1,000 gallons.....	75
Labor.....	75
Lime, at \$6 a ton (treatment at rate of 3 pounds per 1,000 gallons).....	270
	<u>1,020</u>

These figures indicate that the choice between the two methods of treatment, so far as cost is concerned, is determined by the area available. Mechanical treatment can be conducted on one-tenth the area necessary for sand filtration, and this is a distinct advantage in an urban community, because it appreciably reduces the cost of installation and interest. The effluent from the sand filter, however, is decidedly better in quality, showing a removal of over 90 per cent of the organic matter, as against a removal of little more than 50 per cent by the mechanical plant. The effluent from a plant of either style is satisfactory in color, which is the first requisite in purification of a dye liquor. Sand filtration at the rate of 200,000 gallons per acre per day would reduce the cost of the plant by 50 per cent and would make the annual cost of maintenance less than \$500. The combined factors of reduced area and lower cost of maintenance under such conditions indicate that sand filtration is usually the most desirable method of treatment. Local conditions as to area obtainable and final disposal of effluent, however, necessarily determine whether the waste liquor from a yarn dyehouse can be purified most advantageously in a mechanical plant, by means of sand filters, or by a combination of the methods.

The conclusions drawn from these investigations are in accord with those of a previous less detailed study by the Rhode Island state board of health of the dyehouse waste from the R. D. Mason mill, possibly mixed at times with bleach-house waste. They are also consistent with the work of other investigators, though the proposed rates of filtration are somewhat higher.

SUMMARY.

1. Waste waters from dyeing cotton yarn can be satisfactorily purified by filtration through sand. If the rate of filtration is 100,000 gallons per acre per day, six days a week, 90 to 97 per cent of the organic matter and practically all color can be removed from the liquor with an investment of \$50 (not including cost of land) and at an

annual cost (including interest, depreciation, repairs, and labor) of \$8.10 per 1,000 gallons per day. If the rate of filtration is doubled, 85 to 95 per cent of the organic matter can be removed and color can usually be reduced to a satisfactory extent, though this item will vary somewhat with changes of temperature in the filtering medium and changes of intensity of color in the applied waste. The investment of the annual cost for such purification would be, respectively, \$25 and \$4.50 per 1,000 gallons per day.

2. Laboratory experiments indicate that 50 to 60 per cent of the organic matter and all the objectionable color can be removed from the waste waters by precipitation with about 3 pounds of lime per 1,000 gallons (21 grains per gallon). A mechanical plant operating by this method would cost \$40, and would require an annual outlay (including interest, depreciation, repairs, pumping, chemicals, and labor) of about \$10.20 per 1,000 gallons per day.

3. Laboratory experiments indicate that yarn-bleaching liquors may be purified. Gray-boil liquor treated with sulphuric acid and filtered loses about 35 per cent of its organic matter, but the cost of treatment is relatively very high for the benefits derived. Similar results are indicated for a mixture of gray-boil and wet-out liquors. Filtration or sedimentation of mixed soap liquors and sour-rinse water produces a highly purified effluent.

BLEACHING COTTON PIECE GOODS.

BLEACHING PROCESSES AND WASTE WATERS.

The routine processes in a cotton-goods bleachery are described below. These processes, however, are followed only in a general way at Saylesville, where the writers conducted investigations, for there, as at other progressive bleacheries, experience has suggested many modifications of detail and of apparatus.

The pieces of goods, having first been marked for identification, are sewed together into one continuous piece or web, and this web, spread out to the full width of the goods or gathered into a rope, as the nature of the treatment may determine, is drawn from one process to another until the treatment is completed. It first usually passes close to a red-hot plate or cylinder in order to singe loose fiber and lint. After singeing it may be given a preliminary washing in water in order to soften the impurities, which are chiefly sizing materials, such as starch or china clay, but it is more frequently subjected at once to the kier boil.

For this process the cloth is packed in large iron vats or kiers and boiled for several hours with caustic soda or with lime and steam. The kiers have false bottoms, and circulating devices arranged so that the boiling liquor flows on the goods and then passes downward

through them, softening and removing the impurities. For the finer grades the caustic boil is used, consisting of 40 to 50 pounds of caustic soda and 1,000 gallons of water with each ton of material. A large amount of the organic matter is dissolved by this liquor, which becomes very dark colored and loses part of its causticity. For the coarser materials the lime boil is used. It consists of 50 to 60 pounds of lime, as milk of lime, with 1,000 gallons of water for each ton of goods treated. Much of the organic matter forms insoluble compounds with the lime in this boil and remains on the cloth, but the greater part passes out with the waste liquor. After the boil has been completed the liquor is drawn into the sewer and the goods are drained.

When the goods are removed from the kier, they carry with them more than one-third of the kier-boil liquor, together with insoluble matters that have been loosened from or deposited on the fabric. This matter is removed by washing machines, in which the rope of cloth passes over rollers immersed in a stream of running water. As 8,000 to 10,000 gallons of water are used in this process for each ton of material, the waste water is very dilute, containing only 4 to 5 per cent as much foreign matter as the kier-boil liquor, though its portion of suspended matter is relatively greater. The amount of impurities removed from the cloth in the kier boils and in the subsequent washing is generally from 8 to 15 per cent of the weight of the cloth.

After the cloth has been passed through the washing machines the bleaching is continued. The fabric is passed through chloride of lime solution, is rinsed, and is allowed to stand for a few hours in stone or masonry vats. Souring by weak sulphuric or hydrochloric acid, rinsing, treatment in baths of sulphurous acid and soda ash follow in succession. The chemic, sour, and soda-ash solutions are used over and over. The rinse waters are run to waste, and they carry with them small amounts of cloth impurities and mineral matter from the baths. The quantity of water used in these processes is large, and the waste waters are in consequence very dilute. They were not included in the investigations made by the writers at Saylesville.

A piece of cloth in some of the later bleaching or finishing processes is often found to be stained or dirty. If it does not seem necessary to subject this a second time to the entire bleaching procedure, it may be washed in "dolly" washers preparatory to finishing, but only a small percentage of the goods in the bleachery reaches these washers, in which the cleansing process consists merely in soaping the goods and washing them in water. About 350 gallons of water and $1\frac{1}{2}$ pounds of soap are used for each ton of material bleached. The waste waters are somewhat soapy in appearance and they contain but little refuse matter.

The chemic solution is prepared by agitating commercial chloride of lime with water. The lime compound is not chemically pure, and a semiliquid sludge, which settles out, is discharged as a waste product. It amounts to about 5½ gallons (45 to 50 pounds) per ton of material bleached. It is similar to milk of lime in appearance and contains chiefly calcium compounds. A partial analysis of it gave the following results: Total solids, 18.6 per cent; loss on ignition, 0.34 per cent; insoluble in hydrochloric acid, 0.89 per cent; reaction, alkaline.

The analyses of wastes from the Saylesville bleachery in Table 25 show the great concentration of the kier liquors and the comparative weakness of the other waste waters. The same fact is brought out even more clearly by the estimates in Table 26.

TABLE 25.—*Character of liquors at Saylesville, R. I.*^a

[Milligrams per liter.]

	Waste from the lime boil.	Waste from the caustic soda boil.	Rinse water from the lime boil.	Rinse water from the caustic soda boil.	Effluent from the dolly washers.
Color.....	Orange.	Reddish brown.	Gray.	Light orange.	Soapy.
Odor.....	Lime.	Alkali.	Lime.	Alkali.	Soapy.
Turbidity.....	120	600	260	220	350
Sediment.....	Heavy.	Slight.	Heavy.	Considerable.	Heavy.
Oxygen consumed.....	5,200	5,000	154	168	136
Nitrogen as free ammonia.....	5	5	0.3	0.2	0.03
Organic nitrogen.....	190	233	6.4	8.0	1.15
Alkalinity as CaCO ₃	1,840	3,600	84	180	52
Fats.....	848	974	20	130	84
Total solids.....	17,230	18,280	808	850	848
Loss on ignition.....	11,970	10,860	532	520	612

^aAnalyses by Pratt and Stabler.TABLE 26.—*Quantity of waste liquors and solids in same, Saylesville, R. I.*

Waste.	Gallons per diem.	Tons per diem.		
		Total.	Inorganic.	Organic.
Caustic and lime boils.....	60,000	6.70	2.30	4.40
Rinse waters from same.....	825,000	3.40	1.20	2.20
Dolly washer wastes.....	24,000	.08	.02	.06
Other waste liquors.....	5,091,000	2.32	2.23	.09
	6,000,000	12.50	5.75	6.75

PURIFICATION OF WASTE BLEACHING LIQUORS.

So far as known to the authors, the purification of the concentrated liquors from the kiers has not heretofore been attempted, though treatment of the combined waste liquors of bleacheries has been effected with varying degrees of success.

Purification in England.—The bleaching processes in England, according to Naylor,^a are somewhat different from those herein outlined, and the liquors, though similar in quality of impurities, are widely different in concentration from those which were studied at Saylesville. The waste liquors in England contain principally organic matter, such as resins, fats, starches, waxes, and soaps, both in suspension and in solution, lime and calcium salts in solution, china clay in suspension, and soluble salts of sodium and potassium.

The sodium and potassium salts, principally chlorides, are not at present susceptible to practical precipitation nor to any other mode of elimination, except the commercially impossible method of recovery by evaporation of the water. Most of the lime is precipitated as sulphate by the sulphuric acid of the sour liquors, if the wastes are allowed to mix and settle. The precipitate, however, is usually not sufficiently dense, and carries with it the buoyant soaps, starches, or fats, the result being a turbid liquor resembling an emulsion, though eventually clarification nearly always takes place. Such purification is too tedious and requires far too much settling space. Up to the present time the most successful method of clarification is by means of precipitation tanks, successful installations of which are described by Naylor. At Leyland, Lancashire, 500,000 gallons daily of mixed liquors from dyeing and bleaching cotton are treated by precipitation with lime and alumina ferric. The supernatant liquor is usually discharged into the river, but it is sometimes filtered through 5 feet of sand and furnace clinkers, after which it is again used in the bleachery. The amount of waste water is estimated at 50,000 gallons per ton of cloth. The following figures from Naylor's account show the concentration of the liquor that is treated and the purification that takes place.

TABLE 27.—*Purification of bleaching liquors at Leyland, Lancashire, England.*

Source.	Solids in parts per million.		Per cent removal.		
	Total.	Volatile.	Total.	Volatile.	Remarks.
Raw liquor.....	1,660	680	Highly discolored.
Supernatant liquor.....	1,320	400	20	41	Clear.
Effluent from filter.....	660	100	60	85	Clear and sparkling.

Precipitation appears to be satisfactory only as a preliminary process, the greater part of the purification being accomplished by sand filtration. The sludge removed by precipitation contains about 95 per cent water and is, so far as known, valueless. Accord-

^a Naylor, W., *Trades waste; its treatment and utilization*, 1902, pp. 164-180.

ing to the foregoing figures, the sludge amounts to about 2.68 pounds of dry material or 53.6 pounds of wet material per 1,000 gallons; 0.93 pound of alumina ferric per 1,000 gallons is used; the amount of lime used is not stated. Although no estimates of cost are given, a plant of this character would cost about \$15,000 in the United States, excluding cost of land; and the total annual cost of operation and maintenance would amount to \$6,000, or about 4 cents per 1,000 gallons.

Naylor states that a similar purification plant at the works of Grafton & Co. is unable to accomplish results as satisfactory as those at Leyland. The admixture of soapy liquors at this plant renders the deposition of a precipitate difficult, and it was concluded that satisfactory treatment by precipitation could not be accomplished, unless the soapy liquors were segregated and treated with very large quantities of precipitants. Naylor states also that application of bleachery wastes to land is unsatisfactory because they tend to undergo acid fermentation, and that artificial filters soon became choked and failed to work. He recommends bacterial treatment in septic tanks, followed by filtration through sprinkling filters. Such installation proved successful in treating mixed bleaching and dyeing liquors at the works of Peel, Tootal & Co., after precipitation tanks and ordinary continuous-flow filters had been found to be unsatisfactory. The septic tank of this company is of sufficient size to provide three days' storage and it was seeded with sewage sludge. Putrefaction is maintained by allowing the sewage of employees to enter the waste waters and by occasional additions of sewage sludge. The filter material is cinders graded from not more than $2\frac{1}{2}$ inches diameter at the bottom to not less than one-fourth inch diameter at the surface. A similar plant was installed at Leyland after a septic tank and contact filters had proved to be unsatisfactory. Septic tanks and filters can not be maintained unless the chloride of lime solution is separated from the wastes.

Purification in Massachusetts.—Experiments in the United States on the purification of bleachery wastes have been reported by the Massachusetts state board of health.^a The liquor treated was much more dilute than the kier liquors used in the experiments by the authors. It was "a mixture of the stronger" bleachery waste waters. The fairly clear supernatant liquor, which was left after the soap in the mixture had been precipitated, was applied at the rate of 50,000 gallons per acre per day to a filter of 3 feet of sand having an effective size of 0.25 millimeter. Table 28 shows the extent of the reduction in solids accomplished by sedimentation and filtration.

^aThirty-eighth Ann. Rept. Massachusetts State Board Health, 1906, p. 300.

TABLE 28.—*Purification of stronger bleaching liquors in Massachusetts.*

Source	Solids in milligrams per liter.		Per cent removal.		Remarks.
	Total.	Volatile.	Total.	Volatile.	
Raw liquor.....	2,926	696	—	—	Fairly clear. Clear, colorless, non-putrescible.
Supernatant liquor.....	2,494	548	15	21	
Effluent from filter.....	1,821	148	38	79	

A removal of about 90 per cent of the organic matter was effected, as measured by determinations of both albuminoid ammonia and oxygen consumed. The raw liquor contains much more mineral matter than that treated by precipitation and filtration at the plant at Leyland, England, but the content of organic matter is practically the same. Precipitation appears to give more satisfactory results than sedimentation, though the filtration reported by Naylor^a is less efficient, as may be expected from comparison of the filtering media that were used.

Sedimentation, precipitation, and septic treatment of bleachery wastes appear to be satisfactory only as preliminary processes; and the degree of purification increases in the order in which the processes are named. The best secondary treatment seems to be bacterial filtration, sprinkling filters and sand filters having proved to be satisfactory and contact filters unsatisfactory.

TREATMENT OF LIME-BOIL AND CAUSTIC-BOIL LIQUORS.

LABORATORY EXPERIMENTS.

The authors' experiments were confined to the most concentrated of all bleachery wastes, the lime-boil and the caustic-boil liquors. Sulphuric acid or the sludge from chloride of lime solution produces a heavy precipitate when mixed with the kier liquors, and precipitation with these substances was tried in the laboratory. The precipitate with acid, although apparently of great bulk and composed almost entirely of organic matter, contains so small a percentage of the impurities that this method of treatment was rejected. The best result obtained was a 17 per cent removal of volatile solids from the caustic-boil liquor with acid in the proportion of 52 pounds of sulphuric acid to 1,000 gallons of waste.

The use of lime sludge on the caustic-boil liquor also failed to give satisfactory results, but the sludge is an effective precipitant for the lime-boil liquor. The mixing of 3.2 volumes of sludge with 100 volumes of liquor gave a 32 per cent reduction in volatile solids, but smaller amounts of sludge gave less efficient purification. The use of

^a Naylor, W., *Trades waste; its treatment and utilization*, 1902, pp. 164-180.

such sludge on lime-boil liquors to aid other coagulants in purification by precipitation would probably be economical. The amount of sludge produced at Saylesville is sufficient to precipitate only about a third of the lime-boil liquors. If subsequent treatment of all the liquor by bacterial methods is contemplated, the use of lime sludge is undesirable because of the inhibiting effect of its hypochlorites on bacterial growth.

EXPERIMENTS AT SAYLESVILLE, R. I.

Description of purification plant.—A small plant, consisting of storage tank, septic tank, and filters for the treatment of mixed lime-boil and caustic-boil liquors was installed at the Saylesville bleachery. The storage tank, an old boiler shell of 750-gallon capacity, was so placed that 460 gallons of liquid could be discharged from it into the septic tank by gravity, and a drain plug was provided to facilitate the discharge of all the remaining liquor if desirable. The septic tank, an open iron vat about 8 feet long with a capacity of 350 gallons under the conditions of operation, had sloping sides and a rounded bottom. It varied in width from $2\frac{1}{2}$ feet at the top to $1\frac{1}{2}$ feet near the bottom, and it had a maximum depth of about $2\frac{1}{2}$ feet. An inflow pipe connected with the storage tank and regulated by a valve discharged downward at mid depth of the septic tank. The effluent was taken from mid depth by a similar pipe, and no baffles were used to direct the flow. A drainpipe provided for sludge removal and for cleaning.

Each of the 7 filters was an open galvanized-iron cylinder 6 feet deep and 20 inches in diameter, with a filtering area of 2.18 square feet, or one twenty-thousandth of an acre. The filters were exposed to all weather conditions. The filtering media were found on the bleaching premises. An immense bank of good quality filter sand was available, which without treatment had the following characteristics: Effective size, 0.27 millimeter; uniformity coefficient, 4.8; smaller than 0.10 millimeter, 1 per cent; lime by hydrochloric acid test, none. Coke, the only other filtering medium that was used, was obtained from a water-gas plant on the premises. It was broken in order to obtain pieces varying from one-half inch to $2\frac{1}{2}$ inches in diameter.

Operation of storage tank.—A pipe was laid from the storage tank to the kier house and branches were connected to the kiers used for the lime boil and the caustic boil. A steam jet forced the liquors from the kiers to the storage tank every morning when the kiers were emptied. Usually the liquors from both lime and caustic boils were available, in which case the mixed liquors were used to fill the tank, but sometimes only one class of kier liquor could be obtained and occasionally no liquor could be procured. Generally, however,

the storage tank was filled five or six times a week with scalding-hot krier liquors. The valve on the pipe connecting the storage and the septic tank was so regulated that 460 gallons of liquor were discharged from the storage tank in about fifteen hours. On July 16 there was a slight evidence of septic action in the storage tank, which was confirmed by the bacterial counts on samples collected July 12 and July 23; accordingly the tank was flushed weekly after July. Table 29 gives the results of testing 13 samples of the untreated liquor which was used in the work on experimental purification. The examinations cover a period of about four months and the samples were dipped from the storage tank. These analyses represent a very strong alkaline waste.

TABLE 29.—Character of untreated waste liquor at Saylesville, R. I.^a

[Milligrams per liter.]

Date.	Residue on evaporation.			Nitrogen as free ammonia.	Organic nitrogen.	Oxygen consumed.	Number of bacteria per cubic centimeter.
	Total.	Loss on ignition.	Fixed.				
1907.							
June 7.....	17,412	11,962	5,450	2.5	191	5,200	550
June 14.....	18,086	10,964	7,122	4.0	222	5,160	1,000
June 21.....	20,544	12,952	7,592	4.0	226	5,280	1,000
June 26.....	17,240	11,412	5,828	3.0	228	5,220	10
July 12.....	16,676	9,264	7,412	1.5	229	3,540	^b 1,900,000
July 23.....	14,980	8,652	6,328	1.5	196	3,260	^b 11,800,000
July 31.....	5.5	210	5,080	425
August 8.....	18,576	12,380	6,196	3.0	201	3,760	90,000
August 20.....	5.7	210	4,960	10,000
August 28.....	9.8	160	5,340	Low.
September 11.....	6.5—	203	5,060	600
September 18.....	6.6	148	3,740	1,500
September 27.....	4.0	125	3,780	400
Average.....	4.4	196	4,570

^aAnalyses by G. H. Pratt.^bSome evidence of septic action in storage tank, July 16, 1907.

Operation of septic tank.—The septic tank was dosed five or six times a week with 460 gallons of waste liquor, and, since the capacity of the tank was only 350 gallons, a complete change of its contents was effected each time. The septic period, with regular dosing, varied from twelve to twenty-one hours, with a probable average of sixteen hours. The tank was filled on May 22, 1907, and a keg full of sewage sludge was distributed through the contents to start bacterial action. Active gasification accompanied by a characteristic pigpen odor was apparent in a few days. On May 30 regular dosing was begun. On August 7 the septic tank contained a little over a foot of heavy black sludge, which was beginning to escape with the effluent from the tank. This deposit was therefore drawn off, leaving only about 1 to 2 inches in the bottom, and the operation of the tank was resumed. The plant was dismantled on November 26 on account of the piping that would have been exposed to very severe weather.

The results in Table 30 were obtained on 13 samples of liquor taken as it flowed from the septic tank to the filters; they cover a period of about four months.

TABLE 30.—*Character of septic tank effluent, Saylesville, R. I.*^a

[Milligrams per liter.]

Date.	Residue on evaporation.			Nitrogen as free ammonia.	Organic nitrogen.	Oxygen consumed.	Number of bacteria per cubic centimeter.
	Total.	Loss on ignition.	Fixed.				
1907.							
June 7.....	13,776	9,182	4,594	5.0—	181	4,040	14,000,000
June 14.....	15,606	10,264	5,342	6.5—	231	5,320	21,000,000
June 21.....	14,692	8,132	6,560	1.5—	176	3,620	11,500,000
June 26.....	15,780	8,964	6,816	1.5—	196	2,660	12,000,000
July 12.....	15,160	7,312	7,848	2.5—	229	2,320	10,710,000
July 23.....	14,020	7,532	6,488	4.0	203	2,560	21,200,000
August 8.....	12,800	7,200	5,600	14.8	198	2,820	3,600,000
August 20.....				14.8	136	3,880	1,000,000
August 28.....				18.0	155	3,880	70,000
September 11.....				7.5—	232	4,000	190,000
September 18.....				15.6	158	2,740	120,000
September 27.....				5.0	146	3,020	160,000
October 11.....				7.4	139	2,440	143,000
Average.....				8.0	183	3,330

^a Analyses by G. H. Pratt.

Operation of filters.—The effluent from the septic tank was applied to all the filters except No. 6, which was dosed for a short time with liquor from the storage tank, but later with the septic-tank effluent. Filters Nos. 1 and 2 each had 4½ feet of coke for the filtering medium; they were operated first as continuous trickling filters and later as intermittent trickling filters dosed by siphons. Filters Nos. 3 to 7, inclusive, were operated as sand filters, each containing as the filtering medium 4½ feet of sand underlain by 1 foot of coke; the filters were dosed at different intervals and the rates of filtration were varied. Determinations of albuminoid ammonia in samples of sand collected from these five filters on November 26 are given in Table 38. Analyses by G. H. Pratt of samples of the effluents from all the filters are detailed in Tables 31 to 37, inclusive.

TABLE 31.—*Character of effluent from filter No. 1, Saylesville, R. I.*

[Milligrams per liter.]

Date.	Turbidity.	Sediment.	Color.	Odor.	Residue on evaporation.			Nitrogen as free ammonia.	Organic nitrogen.	Oxygen consumed.	Number of bacteria per cubic centimeter.
					Total.	Less on ignition.	Fixed.				
1907.											
June 7.....	300	Very heavy...	Dark beer...	Burnt sugar and alkali.....	11,230	6,956	4,274	4.0—	85	3,000	5,250,000
June 14.....	380	Heavy.....	Dark brown.....	Decidedly disagreeable and musty.....	12,742	8,762	4,580	3.5—	131	3,140	11,500,000
June 21.....	600	Considerable.....	Black coffee.....	Hogpen and moldy.....	10,580	6,592	3,888	3.5—	128	2,250	8,500,000
June 26.....	1,200do.....do.....do.....	9,244	4,560	4,684	8.0—	147	1,520	1,200,000

TABLE 32.—*Character of effluent from filter No. 2, Saylesville, R. I.*

[Milligrams per liter.]

Date.	Turbidity.	Sediment.	Color.	Odor.	Residue on evaporation.			Nitrogen as free ammonia.	Organic nitrogen.	Oxygen consumed.	Number of bacteria per cubic centimeter.
					Total.	Less on ignition.	Fixed.				
1907.											
June 7.....	a 500	Very heavy...	Very dark beer...	Burnt sugar and alkali.....	12,146	7,638	4,508	5.5—	120	3,600	3,720,000
June 14.....	a 800do.....	Light in ruddy brown.	Decidedly disagreeable and moldy.....	13,266	7,874	5,392	4.5—	201	4,340	35,000,000
June 21.....	a 1,000do.....	Muddy coffee.....	Hogpen.....	15,612	8,728	6,884	3.5—	196	3,840	15,700,000
June 26.....	a 800	Considerable.....	Coffee.....do.....	15,620	8,304	7,256	2.3—	191	2,580	1,000,000

a Very great.

TABLE 33.—Character of effluent from filter No. 3, Saylesville, R. I.
[Milligrams per liter.]

Date.	Tur- bid- ity.	Sediment.	Color.	Odor.	Residue on evaporation.		Nitrogen as free ammonia.	Organic nitrogen.	Nitrogen as—		Oxygen con- sumed.	Number of bacteria per cubic centimeter.
					Total.	Loss on ignition.			Fixed.	Nitrates.		
1907.												
June 7	a 220	Considerable.	Whitish.	Distinctly moldy.	2,472	772	3.0—	5.0	Trace (?)	0.002	145	Low.
June 14	b 350	Slight.	Pale straw, darker on standing.	do.	3,454	1,040	.0—	7.0	Trace.	Trace.	288	2,500,000
June 21	b 180	Very slight.	Lemon.	Decidedly moldy.	6,408	3,056	1.5—	34.1	Trace.	Trace (?)	640	1,500,000
June 26	c 120	Slight.	Lemon juice.	do.	7,040	3,304	1.5—	28.0	0.00 (?)	.000	538	40,500
July 12	(c)	do.	Weak tea.	Distinctly moldy.	8,656	3,528	12.5—	42.0 (?)	.00 (?)	.000 (?)	406	90,000
July 23	(c)	do.	Muddy beer.	Distinctly unpleasant and moldy.	9,492	3,504	32.8	76.4	Trace (?)	.000	562	30,000
July 31	(c)	Very slight.	Dark tea.	Faintly unpleasant.			33.6	82.8	Trace (?)	.000	664	1,300,000
August 8	(b)	None.	Muddy beer.	do.			44.3	73.0	Trace (?)	.000	615	270,000
August 20	(c)	Considerable.	Muddy brown.	do.			48.8	48.8	.00 (?)	.000	460	160,000
August 28	(c)	Very slight.	Muddy beer.	Distinctly unpleasant			42.6	38.1	.00 (?)	.000	400	50,000
September 11	(c)	do.	Yellowish.	Faintly unpleasant.			22.1	16.0	.00 (?)	.000	148	65,000
September 18	(c)	Slight.	Yellowish brown.	Distinct of celery.			23.8	18.0	.00 (?)	.000	150	11,000
September 27	(d)	do.	do.	Very faintly unpleas- ant.			38.1	38.1	.00 (?)	.000 (?)	246	13,000
October 11	(a)	Very slight.	Reddish brown.	do.			14.8	25.8	.00 (?)	.020 (?)	180	11,200
October 23	(d)	None.	Dark ale.	Faintly unpleasant.			6.2	41.0	.00 (?)	.050 (?)	530	216,000
November 1	(c)	Very slight.	Dark beer.	Distinctly sweetish.			11.1	84.5	Trace (?)	.000	1,200
November 13	(c)	Slight.	Brown.	do.			10.7	45.1	Trace (?)	.000	1,510	28,200

a Very slight. b Great. c Decided. d Slight. e Very decided.

TABLE 34.—Character of effluent from filter No. 4, Saylesville, R. I.

[Milligrams per liter.]

Date.	Tur- bid- ity.	Sediment.	Color.	Odor.	Residue on evaporation.			Nitrogen as free ammonia.	Organic nitrogen.	Nitrogen as—		Oxygen con- sumed.	Number of bacteria per cubic centimeter.
					Total.	Loss on igni- tion.	Fixed.			Nitrates.	Nitrites.		
1907.													
June 7.....	a 170	Considerable..	Dark straw.....	Decidedly moldy.....	5,546	2,746	2,800	3.0—	34	0.00	0.000	1,270	8,750,000
June 14.....	a 105	Slight.....	Pale straw.....	do.....	7,040	3,520	3,520	1.0—	37	.00	.000	1,290	8,500,000
June 21.....	a 140	do.....	Weak tea.....	do.....	9,948	4,940	5,008	2.5—	65.2	.00	.000	1,400	800,000
June 26.....	b 120	do.....	do.....	do.....	10,828	5,460	5,368	9.5—	74.0 (?)	.00 (?)	.000	1,160	40,000
July 12.....	(b)	Very slight.....	Muddy dark beer..	Decidedly moldy and alkali.	11,136	4,000	6,536	36.0—	91.0 (?)	.00 (?)	.000 (?)	710	85,000
July 23.....	(b)	Slight.....	Muddy beer.....	Distinctly unpleasant and moldy.	11,104	4,320	6,784	55.8	98.4	Trace (?)	.000	805	80,000
July 31.....	(c)	Very slight.....	Dark tea.....	do.....				53.3	94.8	Trace (?)	.000	700	50,000
August 8.....	(a)	None.....	do.....	Faintly unpleasant..				55.0	86.9	.00 (?)	.000	675	60,000
August 20.....	(b)	Very slight.....	Muddy ale.....	do.....				46.7	63.6	.00	.000	625	40,000
August 28.....	(a)	do.....	Dark beer.....	Distinctly unpleasant				48.4	54.1	Trace.	.000	555	10,000
September 11.....	(b)	Slight.....	Brown.....	Faintly unpleasant..				28.3	28.3	Trace.	.000	232	2,000
September 18.....	(b)	Very slight.....	do.....	do.....				28.3	28.8	.00 (?)	.000	276	2,000
September 27.....	(b)	Slight.....	Yellowish brown..	Barnyard.....				50.4	56.6	.00 (?)	.000	362	500
October 11.....	(c)	Very slight.....	Turbid brown.....	Faintly moldy.....				30.8	44.7	.00 (?)	.000	320	8,400
October 23.....	(a)	Considerable.....	Turbid brown.....	Faintly hogpen.....				30.3	45.5	.00 (?)	.000	572	18,400
November 1.....	(c)	Slight.....	Turbid dark beer..	Distinctly sweetish and unpleasant.				23.0	89.4	Trace (?)	.000	1,100	9,000
November 13.....	(b)	do.....	Dark beer.....	do.....				17.6	52.9	Trace (?)	.100 (?)	790	9,000

a Great.

b Decided.

c Very slight.

d Slight.

e Very great.

f Very decided.

TABLE 35.—Character of effluent from filter No. 5, Saylesville, R. I.

[Milligrams per liter.]

Date.	Turbidity.	Sediment.	Color.	Odor.	Residue on evaporation.		Nitrogen as free ammonia.	Organic nitrogen.	Nitrogen as—		Oxygen consumed.	Number of bacteria per cubic centimeter.
					Total.	Fixed.			Nitrates.	Nitrites.		
1907.												
June 7.....	a 220	Considerable..	Dark straw....	Decidedly moldy....	7,032	3,332	2.5—	52.5	0.00	0.000	1,580	12,000,000
June 14.....	a 150	Slight.....	do.....	do.....	11,090	5,190	1.0—	81.0	.00	.000	1,800	5,200,000
June 21.....	a 100	Very slight..	Weak tea.....	do.....	13,052	6,252	5.5—	88.8	.00	.000	1,690	900,000
June 26.....	b 100	Slight.....	Tea.....	do.....	13,136	6,700	17.0—	99.0(?)	.00(?)	.000	1,450	40,000
July 12.....	(b)	Very slight..	Dark beer.....	Decidedly moldy and alkali.	12,356	5,512	46.5—	100.5(?)	.00(?)	.000(?)	1,850	60,000
July 23.....	(b)	do.....	Very dark ale....	Decidedly unpleasant and moldy.	12,312	5,040	65.6	109.0	Trace(?)	.000	1,060	50,000
July 31.....	(c)	Slight.....	Turbid light beer	Decidedly moldy....			46.3	80.8	Trace(?)	.000	650	380,000
August 8.....	(c)	Very slight..	Dark beer.....	Distinctly moldy....			47.6	97.2	Trace(?)	.000	760	100,000
August 20.....	(b)	do.....	Muddy ale.....	Faintly unpleasant..			41.8	76.3	.00(?)	.000	665	110,000
August 28.....	(a)	Considerable.	Brownish black.	Hoppen.....			44.3	85.3	.000	.000	730	5,000
September 11.....	(a)	do.....	Muddy brown....	do.....			38.4	59.0	.000	.000	520	24,000
September 18.....	(a)	Slight.....	Dark muddy brown.	Barnyard.....			44.3	58.6	.00(?)	.000	450	330,000
September 27.....	(a)	do.....	Dark brown.....	do.....			46.3	60.7	.00(?)	.000	358	25,000
October 11.....	(d)	do.....	Reddish brown....	Hoppen.....			22.6	56.6	.00(?)	.060(?)	310	12,800
October 23.....	(a)	Considerable.	Turbid brown....	do.....			15.6	52.1	.00(?)	.050(?)	382	77,000
November 1.....	(a)	do.....	Chocolate.....	Distinctly sweetish..			12.7	76.7	Trace(?)	Trace(?)	588	510
November 13.....	(a)	Very slight..	Turbid dark beer	Distinctly sweetish and unpleasant.			9.0	40.2	Trace(?)	Trace(?)	510	6,800

a Great.

b Decided.

c Slight.

d Very great.

TABLE 36.—Character of effluent from filter No. 6, Saylesville, R. I.

[Milligrams per liter.]

Date.	Turbidity.	Sediment.	Color.	Odor.	Residue on evaporation.		Nitrogen as free ammonia.	Organic nitrogen.	Nitrogen as—		Oxygen consumed.	Number of bacteria per cubic centimeter.
					Total.	Loss on ignition.			Nitrates.	Nitrites.		
1907.												
July 23.....	None.....	None.....	Strong clear tea.	Sweetish of sugar.	10,580	4,400	3.0—	28.7	Trace (?)	0.600	1,250	20,000
July 31.....	do.....	Very slight.....	Dark wine.	Distinctly sweetish and moldy.			a 4.5	57.4			940	
August 8.....	Very slight.....	do.....	Dark tea.	Distinctly unpleasant			4.5	64.4	Trace (?)	.000	640	20,000
August 20.....	None.....	Considerable.....	Dark vinegar.	do.			16.0	54.1	0.00 (?)	.000	655	80,000
August 28.....	Very great.....	do.....	Muddy coffee.	Distinctly moldy			48.1	46.3	0.00 (?)	.000	490	10,000
September 11.....	do.....	Heavy red.....	Reddish brown.	Distinctly sweetish and earthy.			17.2	38.5	(?)	Trace.	390	32,000
September 18.....	Very great (red).....	do.....	Very turbid red brown.	Distinctly sweetish.....			16.4	19.2	.00 (?)	Trace.	285	3,500
September 27.....	Very great.....	do.....	Red brown.....	Sweetish and earthy.			27.1	46.3	Trace (?)	Trace (?)	380	200
October 11.....	do.....	do.....	Reddish brown.	do.....			26.7	37.3	.00 (?)	Trace (?)	280	5,400
October 23.....	do.....	do.....	do.....	do.....			20.5	54.5	.00 (?)	.050 (?)	380	8,400
November 1.....	do.....	do.....	Brown.....	Earthy and sweetish.			13.5	60.3	Trace (?)	Trace (?)	680	800
November 13.....	Very slight.....	Slight.....	Dark porter.....	Distinctly sweetish.....			10.3	57.0	Trace (?)	Trace (?)	810	11,800

a High (?).

TABLE 37.—Character of effluent from filter No. 7, Saylesville, R. I.

[Milligrams per liter.]

Date.	Turbidity.	Sediment.	Color.	Odor.	Residue on evaporation.		Nitrogen as free ammonia.	Organic nitrogen.	Nitrogen as—		Oxygen consumed.	Number of bacteria per cubic centimeter.
					Loss on ignition.	Fixed.			Nitrates.	Nitrites.		
1907.												
July 23.....	Great.....	Considerable (red).	Orange brown...	Sweetish of sugar.....	8,576	3,072	5,504	20.0	Trace (?)	0.200	374	20,000
July 31.....	Slight.....	Very slight.....	Dark ale.....	Distinctly sweetish and moldy.				102.0			1,420	600,000
August 8.....	Great.....	do.....	Very dark porter	Distinctly unpleasant				117.8		.000	1,620	2,600,000
August 20.....	Decided.....	Considerable.....	Very dark brown	Very faintly unpleasant				56.6	0.00 (?)	.000	850	120,000
August 28.....	Slight.....	Very slight.....	Dark beer.....	ant.				41.0		.000	490	50,000
September 11.....	Decided.....	Slight.....	Brownish yellow	Faintly unpleasant				28.3		Trace.	186	4,000
September 18.....	do.....	do.....	Yellowish brown	do.....				23.3		.00 (?)	234	6,500
September 27.....	Great.....	Considerable (red).	Reddish brown	do.....				51.0	Trace (?)	.000 (?)	380	11,000
October 11.....	Very great.....	do.....	do.....	Very faintly unpleasant				41.8	.00 (?)	.100 (?)	300	10,600
October 23.....	Great.....	Heavy (red).	do.....	ant.				44.3	.00 (?)	.100 (?)	320	15,000
November 1.....	Very decided.....	None.....	Dark beer.....	Very faintly unpleasant				61.1	Trace (?)	.000 (?)	344
November 13.....	Very great.....	Heavy (red).	Light coffee	Distinctly sweetish.....				45.5	Trace (?)	.160 (?)	390	4,400

^a High (?).

TABLE 38.—*Albuminoid ammonia in sand from filters, Saylesville, R. I.*^a

[Milligrams per liter.]

Source of sand.	Filter No.				
	3.	4.	5.	6.	7.
Surface.....	586	558	129	248	555
6 inches down.....	232	246	155	210	161
2 feet down.....	75	56	^b 65	101	121
4 feet down.....	45	41	56	83	67

^a Analyses by G. H. Pratt.^b Sample taken a little more than 2 feet down.

Filter No. 1 was operated as a continuous trickling filter four days a week from May 30 to June 7, but the distribution of the liquor over the surface was poor. The filter was dosed at the rate of 70,000 gallons per acre, a net rate of 40,000 gallons per acre per day. It was operated as an intermittent trickling filter dosed by a siphon with fair distribution over the surface from June 8 to June 26, the dose being applied five days a week at the rate of 70,000 gallons per acre, a net rate of 50,000 gallons per acre per day.

Filter No. 2 was operated as a continuous trickling filter four days a week from May 30 to June 7. The distribution of liquor over the surface was poor. The filter was dosed at the rate of 120,000 gallons per acre, a net rate of 70,000 gallons per acre per day. It was operated as an intermittent trickling filter dosed by a siphon with fair distribution over the surface from June 8 to June 14, during which period the dose was applied five days a week at the rate of 120,000 gallons per acre, a net rate of 85,000 gallons per acre per day. It was operated as a contact filter from June 15 to June 26, with one treatment a day six days in a week. It was filled in an hour, and was emptied in one-half hour, the contact lasting eight hours. It was dosed at the rate of 650,000 gallons per acre per day, a net rate of 560,000 gallons per acre per day.

Filter No. 3 was dosed once daily six times a week from May 30 to June 14, at the rate of 20,000 gallons per acre, a net rate of 17,000 gallons per acre per day. It was dosed once daily three times a week from June 15 to June 26, at the rate of 40,000 gallons per acre, a net rate of 17,000 gallons per acre per day; once daily six times a week from June 27 to October 15, at the rate of 20,000 gallons per acre, a net rate of 17,000 gallons per acre per day; and once daily six times a week from October 16 to November 26, at the rate of 40,000 gallons per acre, a net rate of 34,000 gallons per acre per day. On September 11 the surface of the sand was leveled on account of a slight depression in the middle. On October 11 the sand was apparently in as good condition as when operation of the filter began. No clogging and only slight discoloration of the surface was evident. On

November 26 the sand in the center for about $2\frac{1}{2}$ feet down was considerably darker in color than that on the sides.

Filter No. 4 was dosed once daily six times a week from May 30 to June 14, at the rate of 40,000 gallons per acre, a net rate of 34,000 gallons per acre per day, and once daily three times a week from June 15 to June 26, at the rate of 60,000 gallons per acre, a net rate of 26,000 gallons per acre per day.

During the rest of the time that it was operated, except the period from September 27 to October 7, the filter was dosed once daily at the rate of 30,000 gallons per acre, a net rate of 26,000 gallons per acre per day. On September 11 the surface was leveled on account of a slight depression in the sand. On September 27 the surface of the sand was black and the filter had hardly become dry from the dose of the day before. Wet weather prevailed at that time, and the dosing was delayed until the sand had become dry. By October 11 the condition of the surface had become much improved and the filter was apparently disposing of the dose satisfactorily, but there was some question as to whether the filter would remain efficient without being scraped. During the first part of November the dose just disappeared from day to day, and on November 13 a small amount of liquor was still on the surface of the sand, which was black with a slimy deposit. On November 26 the surface of the sand was dark-colored for about 8 inches down from the surface, but it was fairly clean below that point.

Filter No. 5 was dosed once daily six times a week from May 30 to June 14 with a dose equivalent to 80,000 gallons per acre, a net rate of 69,000 gallons per acre per day. From June 15 to June 26 it was dosed once daily three times a week, at a rate of 80,000 gallons per acre, a net rate of 34,000 gallons per acre per day. From June 27 to July 24 the dose was equivalent to 40,000 gallons per acre six times a week, a net rate of 34,000 gallons per acre per day. From July 25 to August 8 the dose was equivalent to 80,000 gallons per acre six times a week, a net rate of 69,000 gallons per acre per day. From August 8 to September 1 the dose was applied three times a week at the rate of 80,000 gallons per acre, a net rate of 34,000 gallons per acre per day. From September 12 to November 26 the dose was at the rate of 60,000 gallons three times a week, a net rate of 26,000 gallons per acre per day. Surface clogging was apparent on July 31, and on August 5 about one-quarter inch of the surface sand was removed. During the latter part of August the filter was not disposing of the dose satisfactorily, and though it had not received a dose for ten days the filter was still wet on September 11. A considerable depth of very black sand with a slimy coating on top at that time necessitated removal of about 3 inches of sand near the perimeter and about 8 inches at the center, after which the filter was

leveled and resurfaced with 6 inches of new sand. By November 26 the sand had become nearly black for a depth of 8 inches, except at the center, where it was dark-colored for a depth of $2\frac{1}{2}$ feet. Otherwise the filter was in good condition.

Filter No. 6 was dosed with the raw liquor twice a day six times a week from July 13 to August 21, with an amount equivalent to 10,000 gallons per acre, a net rate of 17,000 gallons per acre per day; and once daily six times a week from August 22 to September 27 and from October 7 to October 11, at the rate of 30,000 gallons per acre, a net rate of 26,000 gallons per acre per day. From October 12 to November 26 septic liquor was applied at the rate of 60,000 gallons per acre three times a week, a net rate of 26,000 gallons per acre per day. On September 11 a slimy deposit on the sand was found to retard the operation of the filter, but the film was not disturbed. On September 27 the deposit was found to be so much thicker that dosing was discontinued till October 7, when the surface had become thoroughly dry. On October 11 practically all the previous dose was still on the filter; after the surface seal had been broken in order to allow the liquid to drain into the filter, 3 inches of dark-colored, slimy sand was removed. The considerable amount of discolored sand that was left was mixed with a trowel, and 3 inches of new sand was put on the surface. Though septic liquor was applied instead of raw liquor after that experience, the rate of filtration had become materially decreased at the end of October and the sand did not become dry between doses. On November 13 the surface of the sand was discolored and slimy and filter was not dry. On November 26 the sand was very dark-colored at the surface, and nearly black between 6 and 12 inches in depth, but the rest of the sand was in good condition.

Filter No. 7 was dosed twice a day six times a week, from July 13 to August 21, at the rate of 10,000 gallons per acre, a net rate of 17,000 gallons per acre per day; and from August 22 throughout the operation of the filter it was dosed twice a week at the rate of 90,000 gallons per acre, a net rate of 26,000 gallons per acre per day. The surface of the filter received no attention except on September 11, when the sand was leveled on account of a depression in the middle. On November 26 the sand was in good condition and clean for about 2 feet below the surface, then for about 1 foot it was slightly discolored. Below that layer the sand was in good condition.

Results of experimental work.—Table 39 presents a summary of the analytical results of work at Saylesville, together with the percentage removals of organic matter effected by the different filters; the averages of the analyses of raw and septic liquor are used as the bases of the computations. The rates of filtration and some other essential data are also included.

TABLE 39.—Summary of results of purification experiments, Saylesville, R. I.

Source of sample; effluent from—	Number of samples analyzed.	Source of applied liquor.	Number of doses weekly.	Dose per acre.	Net rate per acre per day.	Organic nitrogen.	Nitrogen as free ammonia.	Organic nitrogen and free ammonia.	Oxygen consumed.	Percentage removal based on organic nitrogen.	Percentage removal based on organic nitrogen and ammonia.	Percentage removal based on oxygen consumed.
			Gallons.	Gallons.	Gallons.	Milligrams per liter.	Milligrams per liter.	Milligrams per liter.	Milligrams per liter.	Percentage	Percentage	Percentage
Storage tank.....	13	Lime and caustic				196	4	200	4,570	7	5	27
Septic tank.....	13	Storage tank				183	8	191	3,330	26	26	31
Filter No. 1 a.....	3	Septic tank	70,000	70,000	50,000	135	6	141	2,900	b 10	b 8	b 30
Filter No. 2 a.....	1	do	120,000	120,000	85,000	201	5	206	3,240	b 16	b 8	b 30
Do. c.....	2	do	650,000	650,000	560,000	194	3	197	3,210	83	83	82
Filter No. 3 d.....	3	do	40,000	40,000	17,000	31	1	32	3,482	79	79	80
Do. d.....	12	do	20,000	20,000	17,000	39	26	65	747	69	66	66
Do. d.....	2	do	40,000	40,000	34,000	57	9	66	1,280	80	80	80
Filter No. 4 d.....	3	do	40,000	40,000	34,000	36	2	38	1,310	62	60	61
Do. d.....	6	do	60,000	60,000	26,000	70	6	76	1,594	65	62	61
Do. d.....	13	do	30,000	30,000	26,000	64	38	102	1,198	57	47	47
Filter No. 5 d.....	4	do	80,000	80,000	69,000	78	24	102	1,184	52	40	64
Do. d.....	3	do	34,000	34,000	34,000	87	27	114	1,955	43	16	67
Do. d.....	4	do	40,000	40,000	34,000	105	25	161	433	68	57	71
Do. e.....	6	do	60,000	60,000	26,000	58	56	83	385	74	71	81
Do. e.....	6	do	10,000	10,000	17,000	51	7	63	365	81	69	92
Filter No. 6 d.....	4	Storage tank				37	26	72	1,066	69	62	81
Do. e.....	5	do	30,000	30,000	26,000	57	15	9	1,066	60	57	68
Do. e.....	3	Septic tank				74	9	83	330	77	67	80
Filter No. 7 d.....	4	do	10,000	10,000	17,000	42	22	64				
Do. d.....	8	do	90,000	90,000	26,000							

a Operated as a trickling coke filter.

b Increase.

c Operated as a contact coke filter.

d Operated as an intermittent sand filter.

e After resurfacing.

The unsatisfactory work done by filters Nos. 1 and 2, operating as trickling coke and as contact filters, warrants their exclusion from any further consideration; the best purification accomplished by filter No. 1, operated as a trickling filter at the low rate of 50,000 gallons per acre per day and dosed with septic liquor, showed the removal of only about 30 per cent of the organic matter. The work done by the intermittent sand filters showed variations which were dependent on the changes in the rates and on the general condition of the filters in respect to clogging. The following conclusions seem warranted, after a careful consideration of all phases of the question:

1. Filtration of the raw liquor from the storage tank gives good removal for a time, but the bed soon becomes clogged. The use of the septic tank is desirable, for it insures a better general condition of the filters although the quality of the effluent may be somewhat inferior.

2. The septic period used in this work—about sixteen hours—seems to be satisfactory. Occasional seeding of the tank with fresh sewage sludge would probably improve the action.

3. The use of rates exceeding 26,000 gallons per acre per day appears to reduce the efficiency of the filters and renders their effluents unsatisfactory. Clogging of the surface results in a comparatively short time.

4. It is better to apply the liquor to the filters in large doses with long intermediate intervals rather than in smaller doses more frequently. This is emphasized by the satisfactory work and condition of filter No. 7 during a four months' run. The occasional removal of a few inches of sand and resurfacing maintains a satisfactory condition of filter and effluent.

5. No oxidation to nitrites and nitrates can be expected in filtration of this waste at the rates used.

6. The liquor can be filtered after septic treatment with good results (80 to 85 per cent removal of organic matter), at rates approximating 25,000 gallons per acre per day by dosing the beds with 90,000 gallons twice a week. If the net rate of filtration is maintained at 20,000 gallons per acre per day, over 85 per cent removal of organic matter is effected.

ESTIMATES OF COST.

The approximate cost of a purification plant to treat lime-boil and caustic-boil liquors, and the cost of its maintenance, are shown in the following tables. The estimates apply to a plant including a storage or equalizing tank, a septic tank, and intermittent sand filters capable of treating 60,000 gallons a day. They are based on the costs of several sewage-treatment plants in New England.

TABLE 40.—*Estimates of cost of installing kier-boil liquor treatment plant.*

Rate of filtration in gallons per acre per day.	Filtration area.	Total cost.	Cost per 1,000 gallons of daily capacity.
	<i>Acres.</i>		
15,000	4.0	\$20,000	\$330
20,000	3.0	16,000	270
25,000	2.4	13,600	230
30,000	2.0	12,000	200

TABLE 41.—*Estimates of annual maintenance costs for kier-boil liquor treatment plant.*

Rate of filtration in gallons per acre per day.....	15,000	20,000	25,000	30,000
Interest on investment.....	\$1,000	\$800	\$680	\$600
Depreciation and repairs.....	\$840	\$820	\$800	\$780
Labor.....	\$640	\$480	\$280	\$320
	\$2,480	\$2,100	\$1,860	\$1,700
Cost per 1,000 gallons.....cents.....	13.8	11.7	10.3	9.5

The foregoing estimates are believed to cover all usual cases and they can probably be reduced considerably for most installations.

SUMMARY.

Bleaching processes give rise to the discharge of great quantities of waste waters which are undesirable additions to streams.

The dilute mixed waste waters have been partly purified in practice in England and experimentally in Massachusetts. These installations indicate that sedimentation, precipitation, and septic treatment are satisfactory only as preliminary processes; that the degree of purification increases in the order in which the processes are named; and that the best secondary treatment is bacterial filtration, chlorine solutions being excluded. Sprinkling filters and sand filters have done satisfactory work, but contact filters have proved unsatisfactory.

The investigations of the authors show that 1 per cent of the entire volume of waste waters contains about 40 per cent of the mineral pollution and 65 per cent of the organic pollution from a bleachery. This small portion of the waste consists of the waste kier liquors produced by boiling the unbleached cloth with caustic soda or caustic lime. If the waters derived from rinsing the cloth after boiling are included, 15 per cent of the total volume of waste waters contains 61 per cent of the mineral and 98 per cent of the organic pollution from a bleachery.

Laboratory experiments indicate that neutralization of the concentrated kier liquors by acid produces a voluminous precipitate but results in little actual purification. The lime sludge from the chloride of lime solution tanks precipitates lime-boil liquors and removes about

one-third of the volatile solids, but such treatment is ineffective for caustic-boil liquors.

Field experiments on mixed kier liquors covering a period of six months indicate that septic treatment followed by sand filtration at rates not exceeding 30,000 gallons per acre per day removes 80 to 85 per cent of the organic matter that the waste contains. This is accompanied by still greater reduction of the polluting qualities and by marked improvement in the appearance of the liquor, probably rendering it incapable of causing a nuisance even in a small stream. The cost of this treatment amounts to about 10 cents per 1,000 gallons. In this way 50 to 55 per cent of the organic matter in all wastes from the bleachery may be removed at a cost of about 0.1 cent per 1,000 gallons.

THE MANUFACTURE OF OLEOMARGARINE, FERTILIZER, AND GLUE.

PROCESSES AND WASTE WATERS.

Oleomargarine and other butter substitutes are made from mixtures of animal and vegetable oils. Tallow and lard, the most important sources of stock, are contained in membranous cells which soon putrefy and cause the fats to become rancid. Therefore the stock is washed and cooled with water and rendered to obtain an oil free from animal tissue. The rendering is usually accomplished by digestion with steam under about 50 pounds pressure in iron containers. The cell walls are broken down and the melted fat, which rises above the tissue or "cracklings" and the water, can be decanted. The impure water is discharged as sewage or utilized for recovery of valuable materials.

A large amount of fertilizer is made from slaughterhouse wastes, such as blood, waste meat, bones, hoofs, hair, tainted meat, diseased animals, and tankage or residue from rendering and glue-making processes. In converting these substances into commercial fertilizer, blood is dried at a moderate heat and crushed to powder; bones may be ground without preliminary treatment, or they may be treated with a volatile solvent or boiled with steam to remove fats and gelatin and then ground for fertilizer; and tankage is dried, powdered, and mixed with other fertilizing material. The valuable ingredients sought for in all stock are nitrogen, potassium, and phosphorus.

Glue is prepared from animal tissues, which on heating with water lose their structure, swell up, and finally go into solution. When they become cool, such solutions form a jelly which dries into the hard translucent mass known as glue. The raw material includes hide trimmings, "skivings," "fleshings," and other untanned refuse of the tannery; earlaps, heads, feet, tails, and other slaughterhouse

offal; and "green" or fresh bones. Green bones are used in manufacturing "bone glue;" the other materials are used for the production of "hide glue."

The stock for hide glue is salted, washed, and then mixed with milk of lime and allowed to stand for several weeks. The liming softens and swells the tissues, converts the fats into lime soaps, and dissolves a large part of the blood, flesh, coriin and other matters which will not form glue. The material is then washed in water and acid. The sewage from liming and washing is largely used for fertilizer.

Bone glue is not essentially different from hide glue. The fatty matters are removed from the fresh bones by boiling with water and skimming or by extracting with a volatile solvent. The bones are then crushed and treated with dilute acids to remove calcium phosphate and other salts, which are valuable as fertilizers. Washing with lime water removes traces of acid.

The stock for bone or hide glue, having been cleansed by the treatment just described, is boiled with water or steamed in a digester until practically all the glue-forming material has been dissolved. Several successive boilings may be used. The glue solution is filtered, evaporated to the desired consistency in a vacuum pan, and run into trays in which it cools and forms a jelly, which is cut into plates and dried for market.

It may be seen from the foregoing description that the industries of making oleomargarine, fertilizer, and glue are closely allied. The raw materials are the wastes from slaughterhouses and markets. The processes, however, vary greatly in different establishments. In one plant utilizing these raw materials attention may be given to the manufacture of a single product. In another plant all by-products may be taken into account and the sewage or waste waters from one process may be utilized to the fullest extent as raw materials for the manufacture of a secondary product. A detailed description of processes would not, therefore, be of general application, and variation in concentration and in difficulty of purification of waste waters is to be expected with variation in processes and products of manufacture. The L. B. Darling Fertilizer Company, at whose establishment experimental work was conducted, attempts to recover the greatest possible amount of valuable material from the stock that is used; consequently, the waste waters are small in quantity and low in concentration as compared with the sewage from plants that are less economically operated.

Practically the only waste from this plant, and the waste that was used in the experimental work, was the discharge over a weir in the last of a series of boxes, or tanks, through which a waste liquor was passed for the purpose of increasing the recovery of fat and animal

tissue by gathering the scum and the sediment. This liquor is a waste from "tankage," and the greater part of the fatty matter has been taken from it before it is run into the boxes. The writers have found no account of experimental filtration of a waste similar to that from the series of skimming tanks. It is a putrescible, ill-smelling liquor, very high in organic matter in solution and in suspension, both carbonaceous and nitrogenous. The nitrogen content is enormous, because of the animal composition of the materials used at the plant. It is extremely high in bacterial life, the number of organisms per cubic centimeter being invariably in the millions. The average for ten samples is 13,800,000 bacteria per cubic centimeter. Other characteristics are high turbidity, very great sediment, and a dirty-brown color.

Analyses of samples of this waste show that it is extremely variable in strength. Twelve samples give an average figure for organic nitrogen of 979 parts per million, and for oxygen consumed of 988 parts per million. The detailed analyses are given in Table 42.

TABLE 42.—Analyses of waste water from factory of L. B. Darling Fertilizer Company.

[Milligrams per liter.]

Date.	Time.	Residue on evaporation.			Nitrogen as free ammonia.	Total organic nitrogen.	Nitrogen as—		Oxygen consumed.	Fats.	Number of bacteria per cubic centimeter.
		Total.	Less on ignition.	Fixed.			Nitrates.	Nitrites.			
1907.	April 24.....	5,744	4,836	908	79	500			500	575	
	May 28.....	7,606	6,604	992	123	726			475	1,240	25,000,000
	July 22.....	3,508	3,041	467	105	356			470		13,300,000
	August 8.....	11,329	11,468	1,864	254	259	0.00 (?)	2.000	c 340	1,996	18,000,000
	August 10.....	11,824	10,148	1,676	170	1,916		.000	1,150		13,500,000
	August 20.....				209	1,310	Trace (?)	.000	990		4,400,000
	August 28.....				248	1,036	Trace (?)	2.000	1,380		13,800,000
	September 11.....				108	603	.00 (?)	2.400	440		7,000,000
	September 18.....				117	660			745		16,500,000
	October 10.....				166	962	.00 (?)	2.400	990		18,800,000
	October 17.....				172	1,332			1,910		18,800,000
	November 1.....	9.45 a. m.			127	1,434			1,600		7,900,000
November 13.....	10.30 a. m.				886	.50 (?)	2.000	1,200			

a Composite of samples taken hourly from 8 a. m. to 6 p. m., inclusive.

b Unfiltered.

c Filtered.

EXPERIMENTAL PURIFICATION OF WASTES.

OPERATION OF PURIFICATION PLANT.

Sand filtration without preliminary treatment was conducted from July 26 to November 14, 1907. Two galvanized-iron filters, 20 inches in diameter, were kept in operation, out of doors and uncovered, throughout the period. Both filters contained $4\frac{1}{2}$ feet of sand, having an effective size of 0.23 millimeters and a uniformity coefficient of 3.4. Three or four inches of fine cinders, not screened and containing a little dust, supported the sand, and these cinders in turn rested on 8 or 10 inches of larger cinders.

Filter No. 1 was started at the rate of approximately 20,000 gallons per acre per day. This dose, applied once a day for six days in the week, was continued till August 21, when it was increased to 100,000 gallons a day. The dose was made 40,000 gallons a day on September 12, and it was decreased to the original dose of 20,000 gallons a day on October 22. The surface of the filter was not raked during the run, except when removals of sand were made, at which time the surface was leveled before dosing the filter was resumed. The behavior of this filter demonstrated that surface clogging occurs at any of the rates that were tried and that the interval between cleanings is dependent on the size of the dose, being necessarily more frequent with the higher rates. While the rate of dosing was 100,000 gallons a day the filters clogged so badly that it was difficult to apply the entire dose from day to day, because so much of the previous dose was left on the filter that there was not room above the sand for more liquor.

The condition of the surface finally became so bad, the sand being discolored and covered with a slimy deposit, that it became necessary, on September 11, to remove about $1\frac{1}{2}$ inches of the sand on top. No new sand was put on, but the surface was leveled. Dosing was resumed, but at the rate of 40,000 gallons. The same condition of slow drying soon developed, and conditions became such that the surface would not dry from day to day. Consequently a slimy layer about one-eighth inch thick, which sealed the surface, together with half an inch of the top sand, was removed on October 21, and the surface was leveled after the top layer of 3 to 4 inches had been mixed. No new sand was put on, and dosing was resumed at the 20,000-gallon rate. By the middle of November surface clogging was again apparent, and the filter was gradually approaching the condition noted on previous occasions. When the filter was dismantled on December 13 dosing had been discontinued about ten days. The sand was clean in appearance, there being little discoloration. The clogging was caused by a surface deposit, grayish in color, and not more than one-sixteenth to one-eighth inch thick after it had dried into the top sand.

Filter No. 2 was started at a rate of 60,000 gallons an acre. Dosing once daily for six days a week was continued until October 22. From that date until the dismantling of the filter the dose was applied every other day at the rate of 40,000 gallons per acre per day, making the net rate for the latter period 20,000 gallons per acre per day. An inch of sand was removed from the surface of the filter on September 11, after which dosing was resumed without putting on new sand. Sand was again removed on October 21, as a slimy layer about one-eighth inch thick was sealing the surface at this time. This top layer and about one-half inch of sand was removed; the surface was then leveled, and after the top layer of 3 to 4 inches of sand had been mixed dosing was resumed without putting on new sand. The conditions after that date were similar to those existing in filter No. 1, and the appearance of the sand when the filter was dismantled on December 13 was much the same as that of the sand in the other filter.

When the experiments were discontinued samples of sand were collected from both filters at the top inch, at 6 inches down, and at the bottom. The results of tests for albuminoid ammonia and loss on ignition on these samples are given in Table 43. They confirm what was noticed regarding the satisfactory condition of the sand below the surface at the time of dismantling. The analyses of the samples from the top inch indicate the presence of a large amount of nitrogenous organic matter in the clogging film.

TABLE 43.—*Partial analyses of sands after use in filtration.*

Source of sample.	Albuminoid ammonia.	Loss on ignition.
	<i>Milligrams per liter.</i>	<i>Per cent.</i>
Filter No. 1:	^a 3,156	12.1
Top inch.....	145	.67
6 inches down.....	87	.47
Bottom.....		
Filter No. 2:	2,262	7.3
Top inch.....	147	.57
6 inches down.....	124	.51
Bottom.....		

^a Probably too low.

The quality of the effluents from the filters, as determined by analyses of samples taken at about 10 a. m., is shown in Table 44.

TABLE 44.—Analyses of effluents from filters Nos. 1 and 2 at the establishment of the L. B. Darling Fertilizer Company.

Date.	Appearance.			Odor.	Nitrogen as free ammonia.	Total organic nitrogen.	Nitrogen as—		Oxygen consumed.	Number of bacteria per cubic centimeter.	Net rate of filtration (gallons per acre per day).
	Turbidity.	Sediment.	Color.				Nitrates.	Nitrites.			
FROM FILTER NO. 1. [Milligrams per liter.]											
1907.											
August 1.....	Decided.	Very heavy (iron).	Red.....	Distinctly unpleasant.	119	67.2	1.00(?)	Trace.	68.0	50,000	20,000
August 8a.....	Slight.	Heavy (iron).....	Iron.....	do.....	320	35.7	Trace(?)	0.50 (?)	62.0	50,000	20,000
August 20.....	Decided.	Slight (iron).....	Very light brown.	do.....	502	11.5	Trace(?)	5.50	36.0	140,000	20,000
August 28.....	Great (iron).	do.....	Red (iron).	Decidedly unpleasant.	420	14.8	Trace(?)	.000	50.0	38,000	100,000
September 11.....	Very great (iron).	Heavy (iron).....	Red.....	Distinctly unpleasant.	895	41.5	(?)	.000	87.0	20,000	100,000
September 18.....	do.....	do.....	Red (iron).	do.....	699	17.9	.00(?)	.000	109.0	500,000	40,000
October 10.....	do.....	do.....	Red.....	Faintly unpleasant.	701	52.8	.00(?)	.000	135.0	310,000	40,000
October 17.....	Decided (iron).	Considerable (iron).	do.....	Distinctly unpleasant.	527	24.8	.00(?)	.000	118.0	400,000	40,000
November 1.....	Very decided.	None.....	Reddish brown.	Decidedly unpleasant.	661	43.5	1.00(?)	.000	150.0	190,000	20,000
November 13.....	do.....	do.....	do.....	Distinctly unpleasant.	624	25.9	.50(?)	.000(?)	130.0	190,000	20,000
FROM FILTER NO. 2.											
1907.											
August 1.....	Decided.	Heavy.....	Turbid.....	Distinctly unpleasant.	271	46.7	0.50(?)	Trace.	54.0	40,000	60,000
August 6 b.....	Slight.	Heavy (iron).	Iron.....	Distinctly unpleasant and muddy.	509	12.7	Trace(?)	0.050(?)	46.0	40,000	60,000
August 20.....	do.....	do.....	Red (iron).	Distinctly unpleasant.	626	10.7	Trace(?)	.000	65.0	40,000	60,000
August 28.....	Great (iron).	Slight (iron).....	do.....	Decidedly unpleasant.	831	26.9	Trace(?)	.000	86.0	1,400,000	60,000
September 11.....	Decided (iron).	Considerable (iron).	Light brown.	Distinctly unpleasant.	530	14.4	(?)	.000	70.0	4,200,000	60,000
September 18.....	Decided.	Very slight.	do.....	do.....	710	17.7	.00(?)	.000	110.0	450,000	60,000
October 10.....	Very great (iron).	Heavy (iron).	Red.....	Faintly unpleasant.	500	12.8	.00(?)	.040	72.0	1,000,000	60,000
October 17.....	Decided (iron).	Considerable (iron).	do.....	Distinctly unpleasant.	740	19.7	.00(?)	.000	118.0	4,900,000	60,000
November 1.....	Very decided.	None.....	Reddish brown.	Decidedly unpleasant.	822	29.2	1.00(?)	.020(?)	150.0	400,000	20,000
November 13.....	Decided.	do.....	Yellowish brown.	Distinctly unpleasant.	658	22.0	.70(?)	.000(?)	115.0	400,000	20,000

^a Residue on evaporation: Total, 1.486; fixed, 1.064; loss on ignition, 422.

^b Residue on evaporation: Total, 1.908; fixed, 1.268; loss on ignition, 640.

Experiments were made with the object of coagulating the waste with chemicals in order to obtain a decided removal of organic matter before filtration, but the results were not satisfactory. The addition of aluminum sulphate resulted in excellent clarification on several occasions, but at other times the coagulum had no effect whatever, and the addition of soda ash did not aid coagulation. Copperas and lime also were tried, but the results were not encouraging.

RESULTS OF FILTRATION.

A summary of the determinations of organic nitrogen, organic nitrogen plus free ammonia, and oxygen consumed for the filter effluents, together with the percentage of removal by filtration represented by these estimates, is given in Table 45.

TABLE 45.—*Summary of results of filtration at the establishment of the L. B. Darling Fertilizer Company.*

Source of sample.	Number of samples analyzed.	Number of doses weekly.	Net rate of filtration (gallons per acre per day, approximately).	Milligrams per liter.			Percentage of removal. ^a							
				Organic nitrogen.	Organic nitrogen + free ammonia.	Oxygen consumed.	Organic nitrogen.		Organic nitrogen + free ammonia.		Oxygen consumed.			
							A.	B.	A.	B.	A.	B.		
Crude waste.....	12	979	1,136	988
Effluent, filter 1....	3	6	20,000	38	352	55	96	97	69	74	94	94	94	94
Do.....	2	6	100,000	28	686	69	97	97	40	34	93	92	92	92
Do.....	3	6	40,000	32	674	121	97	97	41	40	88	88	88	88
Do.....	2	6	20,000	35	678	140	96	97	40	48	86	90	90	90
Effluent, filter 2....	8	6	60,000	20	618	78	98	98	46	49	92	92	92	92
Do.....	2	3	20,000	26	766	133	98	98	33	42	86	90	90	90

^a Columns A are calculated from the average of 12 samples of crude waste as a basis, but columns B are calculated from the samples of crude waste that were collected when the corresponding samples of filter effluent were taken.

The figures for percentage of removal, based on the estimates of organic nitrogen and oxygen consumed under widely different rates of filtration, are almost uniformly high. The decomposition of organic nitrogen is nearly the same in all cases, but the completeness of the oxidation, indicated by free ammonia, varies, showing some advantages in favor of the lower rates of filtration. The operation and the condition of the filters themselves are the proper sources of information regarding the best rates to use.

A high percentage of removal of organic matter can be effected by filters, but constant attention to the surface of the sand, with frequent scraping and ultimate addition of new sand, would be necessary. It is doubtful whether, with the rate as low as 20,000 gallons, filters would do satisfactory work longer than five or six weeks unless attention were given to the surface. It is possible that a rate of

60,000 gallons could be used, but conditions requiring slight sand removal in less time than a month would probably result. If attention were not given to the surface when needed, a nuisance would probably result from the odors given off by the putrid waste, but if the surface were kept fairly open, thus allowing the liquor to get quickly below the surface, such trouble might be avoided. Preliminary filtration at a rate of about 60,000 gallons, followed by a second filtration at the same or even a little higher rate, would give excellent results.

It would be advisable to construct near the filters a storage or dosing tank capable of holding one day's flow, in order to avoid the necessity of continual discharge on the surface of the sand. Considerable sediment and scum would, of course, collect in the storage tank, and the supernatant liquor would not clog the sand nearly so rapidly as the waste itself. The interval between scrapings would also be longer, and the accumulated scum and sediment could be treated like the material now recovered in the series of tanks. If filtration were interrupted by severe winter weather, such conditions would prevail only for a comparatively short period and that at a time when the crude waste would do least damage to a stream. The waste liquor, even then, would be considerably reduced in strength by sedimentation in the storage tank, which would increase the organic matters returned for utilization.

COSTS OF FILTRATION.

Table 46 gives estimates of the costs of installation, operation, and maintenance of a plant for purifying tank waste from establishments similar to that of the L. B. Darling Fertilizer Company. The estimates are based on the results of experiments with the waste from the factory just mentioned. The installation provides for sand filtration and for a tank for storing one day's supply of liquor, 25,000 gallons.

TABLE 46.—*Estimates of cost of plant for filtering tank waste.*

Net rate of filtration (gallons per acre per day).	Filtration area.	Cost of plant.	Annual cost of maintenance and operation.	Cost per 1,000 gallons.
	<i>Acres.</i>			<i>Cents.</i>
10,000	2.50	\$11,000	\$2,200	29.3
20,000	1.25	6,000	1,200	16.0
40,000	.625	3,500	700	9.3
60,000	.417	2,700	540	7.2
100,000	.250	2,000	400	5.3

The cost of operation and maintenance is based on fixed charges of 15 per cent of the cost of the plant for interest, repairs, and depreciation, and 5 per cent for labor. This makes ample allowance for the high concentration of the waste, for usually the charges are about 11 per cent and 3 per cent, respectively, for sewage-treatment plants of this type. The tank capacity that is provided allows for considerable sedimentation and for a dosing tank.

EVAPORATION—RECOVERY TREATMENT OF WASTES.

VALUE OF RECOVERED PRODUCT.

Table 47 gives estimates of the fertilizing value of the products obtained by evaporating and drying 25,000 gallons of tank waste daily. The unit values for nitrogen, phosphoric acid, and potash are those proposed by the Massachusetts agricultural experiment station.^a

TABLE 47.—*Estimated value of products obtained by evaporating and drying tank waste.*

VALUES BASED ON A COMPOSITE SAMPLE COLLECTED JUNE 16, 1908.

	Milligrams per liter.	Pounds per 1,000 gal- lons.	Value per pound.	Value per 1,000 gal- lons.	Value per day.
Organic nitrogen (N).....	595	4.96	\$0.185	\$0.918	\$22.94
Phosphoric acid (P ₂ O ₅).....	106	.88	.05	.044	1.10
Potash as K ₂ O.....	126	1.05	.05	.052	1.31
Total solids.....	5,300	44.2	.023	1.014	25.35
Volatile solids.....	4,190	34.9	.026	.918	22.94
Fixed solids.....	1,110	9.3	.010	.096	2.41

VALUES BASED ON AN AVERAGE OF TWO COMPOSITE SAMPLES AND A MEAN OF 11 SNAP SAMPLES.

Organic nitrogen (N).....	774	6.45	\$0.185	\$1.195	\$29.88
Phosphoric acid (P ₂ O ₅).....	^a 138	1.15	.050	.058	1.44
Potash as K ₂ O.....	^a 164	1.37	.050	.068	1.71
Total solids.....	^a 6,900	57.5	.023	1.321	33.03
Volatile solids.....	^a 5,450	45.4	.026	1.195	29.88
Fixed solids.....	^a 1,450	12.1	.010	.126	3.15

^a Estimated from ratio to nitrogen in sample of June 16, 1908.

This shows the value of the waste based on the average to be \$1.32 per 1,000 gallons.

COST OF TREATMENT.

The next natural question is the cost of installation of a complete independent plant operating ten hours a day and a similar estimate for the plant probably required as an addition to a factory. These figures are as follows:

^a Bull. No. 51, March, 1894.

Estimated cost of plant for evaporating and drying 25,000 gallons of tank waste daily.

Complete independent plant:

Boilers and setting, flues, stack, feed pumps, coal pocket, piping, and heaters	\$8,000
Evaporator (triple-effect)	10,000
Dryer	500
Building	3,500
Incidentals	2,000
	<hr/>
	24,000
	<hr/>

Plant probably required as an addition to a factory:

Boilers and setting, flues, feed pumps, piping, and heaters..	5,400
Evaporator (triple-effect)	10,000
Building	1,000
Incidentals	1,500
	<hr/>
	17,900

The cost of operation of both of the above-described plants, the complete plant and the probable addition to the existing plant, is of course the figure to determine the practicability of the recovery process as applied to the waste. These estimates are as follows:

Estimated cost of operation of plant for evaporating and drying 25,000 gallons of tank waste daily.

Complete independent plant:

Fixed charges (11 per cent)	\$2,607.00
Attendance (\$33 per week)	1,716.00
Coal (1,500 tons, at \$4.50)	6,750.00
Supplies	227.00
	<hr/>
Total annual cost	11,300.00
Cost per day	37.67
Cost per 1,000 gallons	1.51
	<hr/>

Plant probably required as an addition to a factory:

Fixed charges (11 per cent)	1,969.00
Attendance (\$17 per week)	884.00
Coal (1,500 tons, at \$4.50)	6,750.00
Supplies	227.00
	<hr/>
Total annual cost	9,830.00
Cost per day	32.77
Cost per 1,000 gallons	1.31

CONCLUSIONS.

As the value of the waste per 1,000 gallons and the estimated cost of operating the plant which would be required as an addition are practically identical, evaporation would just about pay for itself at this plant were the strength of the waste maintained at about total solids—6,900 parts per million and organic nitrogen 774 parts per million.

Reference to Table 47 shows that an increase of 52 parts per million in total solids adds 1 per cent per 1,000 gallons to the value of the waste. If, therefore, the more dilute portions could be diverted and only the more concentrated liquor used in the evaporation plant, a small profit would undoubtedly be obtained. The figures for the complete independent plant indicate that a waste containing 7,900 parts per million total solids would be valued at \$1.51 per 1,000 gallons, or the cost of operation of this plant. Thus a waste of greater strength than the one just mentioned and of the same character as that with which the experiments were made could be handled with a margin of profit in an independent plant.

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