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> PUBLIC HEALTH BULLETIN No. 97 OCTOBER, 1918

# STUDIES ON THE TREATMENT AND DISPOSAL OF INDUSTRIAL WASTES

(Made under the supervision of Earle B. Phelps)

1. THE TREATMENT AND DISPOSAL OF STRAWBOARD WASTE By HARRY B. HOMMON

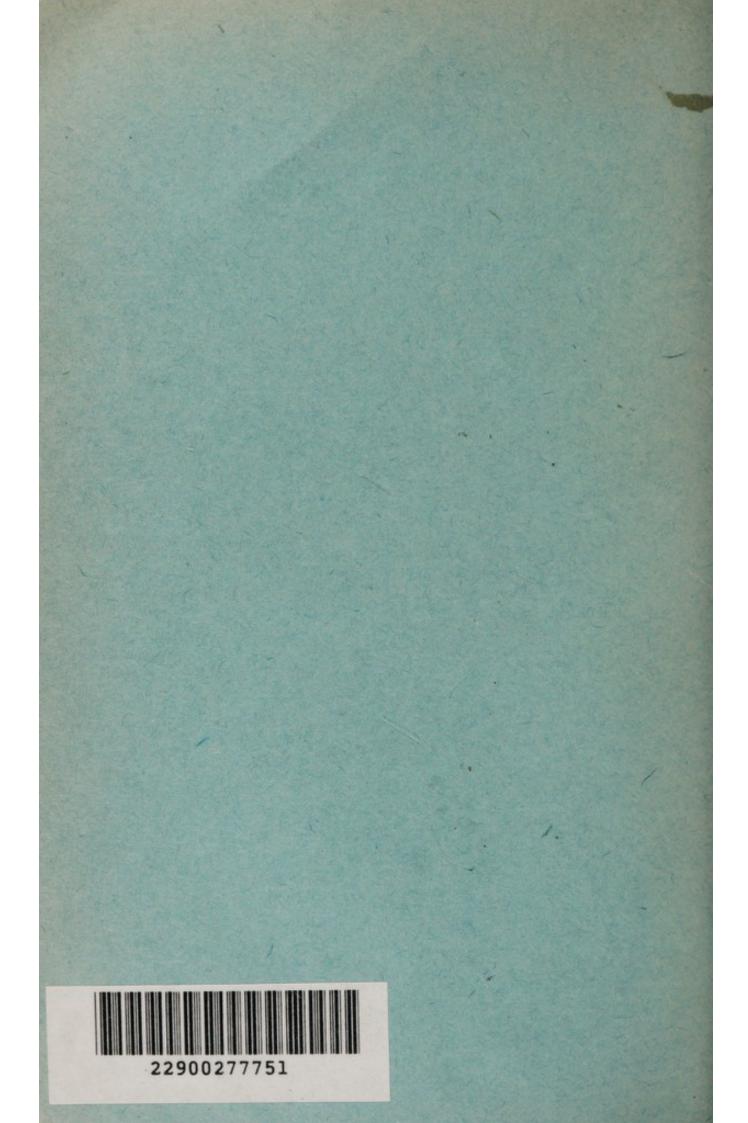
2. THE DETERMINATION OF BIOCHEMICAL OXYGEN DEMAND OF INDUSTRIAL WASTES AND SEWAGE

By EMERY J. THERIAULT and HARRY B. HOMMON

PREPARED BY DIRECTION OF THE SURGEON GENERAL



WASHINGTON GOVERNMENT PRINTING OFFICE 1918



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# STUDIES ON THE TREATMENT AND DISPOSAL OF INDUSTRIAL WASTES.

#### EARLE B. PHELPS,

Professor of Chemistry, Hygienic Laboratory, U. S. Public Health Service.

Any general program for the prevention of stream pollution must be based upon a complete economic study of the many phases of the problem. Stream pollution affects the public health directly when it becomes the means of transmission of the organisms of infectious, water-borne disease from one person to another. It affects the public health indirectly when it becomes a contributory agent to that final result. The admission of such infectious material as city sewage into a water supply is a direct menace, but its ill effects may be largely offset by sewage treatment, dilution, and natural purification in the stream and purification of the water supply. Economically it is best to utilize these three lines of defense to varying degrees, relying more or less upon the one or the other of them, according to the requirements of each situation.

The presence of industrial wastes in such a stream constitutes an indirect menace to the public health in so far as they may draw upon the stream's natural purifying power, thereby delaying or preventing the ultimate disposal of directly infectious matter. These wastes, furthermore, may add to the burden of water-treatment works and decrease both their efficiencies and their margins of safety. Finally, stream pollution by industrial wastes dulls the esthetic sense of a community, and, by presenting apparently insuperable barriers to any real progress toward a clean stream, may delay or permanently prevent the proper treatment of the more dangerous sewage pollution.

Economically, the complication introduced by the industrial-waste problem is still greater. The relation of sewage treatment to water supply is a social or community problem, to be dealt with in terms of public welfare and at public cost. The utilization of a stream for the disposal of an industrial waste, however great the resulting evil, may be a vital necessity for the well-being of an industry upon which the prosperity of the community itself largely depends.

In approaching this problem, therefore, it becomes necessary to determine whether any alternative method of disposal, or any remedial method of treatment, be available or can be devised. Such a method must, first of all, be reasonable in cost, consideration of reasonableness being based upon the relative additional charge the treatment in question will impose upon the cost of the final products of the industry. An industry making a high-grade bond paper, for example, could in general sustain a much heavier purification charge than one making the same quantity of strawboard.

Even this general rule, however, is not a safe one in every case, for the particular bond paper might conceivably be in such close competition with papers made by other processes that a slight additional tax would overthrow the balance and drive it from the market, whereas the cheaper paper might conceivably have less active competition and be in position to withstand a considerable readjustment of manufacturing costs.

While these economic considerations form no part of the present series of investigations, they must underlie any general and enduring program for the prevention of stream pollution and the reclamation of stream resources now being wasted through lack of such a program. In particular they impress upon the investigator the importance of economical procedures. For while it may be true that some industries are in position to increase their manufacturing costs by the installation of expensive treatment works, it is certainly true that most of them are not and that the more expensive the recommended remedy the less the likelihood of its adoption.

Utilization of waste products is naturally the ideal solution. In many cases in the past this has been found possible, and investigations which have been begun to relieve a nuisance have succeeded in developing recoveries of great value. The development of the market for hydrochloric acid, at one time a waste and a nuisance, is a case in point. Opportunities for such striking results are not common in this age of conservation, but partial recoveries of by-products, and some reduction in the net cost of treatment, is always a possibility.

In the series of investigations of various industrial waste problems, which have been carried on by the United States Public Health Service during the past three years, it has been the controlling aim to develop methods of treatment and disposal which would serve the fundamental purpose of conservation of stream resources in the most inexpensive manner. In recognition of the fact that partial treatments of varying degrees of completeness are desirable to meet the varying requirements of practice, alternative methods and supplementary methods have frequently been worked out.

It has been the further aim to perform all the experimental work upon as large a scale as practicable and at the works themselves rather than in the laboratory. The experiments are therefore large scale-testing station experiments, carried over a sufficient length of time to justify definite conclusions as to their practicability.

In recognition, however, of the essential value of laboratory research in many of these problems there has been carried on simultaneously with the field studies many more refined laboratory experiments, the results of which have been of the most practical value. The analytical control of the testing station operation has also been of a laboratory nature.

The present report deals with the treatment and disposal of the waste arising from the manufacture of strawboard. The strawboard industry is strongly developed throughout the middle Western States, especially along the Ohio Valley. From the point of view of waste disposal, it is characterized by the production of a large volume of a particularly harmful waste in the course of manufacture of a very cheap product. Economically the margin allowable for purification treatment is extremely small, while the possibilities of harm are great. The alkaline and highly turbid character of the waste makes it especially harmful to fish life. Its high content of very stable coloring matter produces an undesirable, although quite harmless, discoloration in the stream, while its tendency to precipitate sewage and other pollutional matter, along with its own great bulk of sediment, brings about conditions of physical nuisance in the stream which have for 20 years or more led to litigation, to makeshift remedies, and even to the closing of the mills.

At the suggestion of the Indiana State Board of Health, it was decided to undertake a new investigation of this problem, although it had already received considerable study, especially by the water resources branch of the United States Geological Survey. This work has been done in cooperation with the State board, whose representatives have been especially helpful in securing a suitable location for the work and have kept in close touch with its progress throughout.

Sanitary Engineer H. B. Hommon has planned the investigation and has been in actual charge throughout. In the design and construction of the testing station plant he was ably assisted by Sanitary Engineer Harry R. Crohurst, while the actual operation of the plant has been under the immediate care of Sanitary Bacteriologist Walter E. Brown, in resident charge.

Sanitary Engineer H. H. Wagenhals gave very valuable assistance in assembling the data for this report and Sanitary Bacteriologists Emery J. Theriault and Hugh M. Campbell have been responsible for the analytical work. The studies of the plankton in the activated sludge tests were contributed by Plankton Expert W. C. Purdy.

The officials of the American Strawboard Co. have been most courteously helpful throughout, and Mr. Charles Macy, superintendent of the Noblesville Mill, through his personal interest, has greatly assisted this work.

# THE TREATMENT AND DISPOSAL OF STRAWBOARD WASTE.

HARRY B. HOMMON, Sanitary Engineer, U. S. Public Health Service.

### INTRODUCTION.

The disposal of the waste arising from the manufacture of paper from straw is a problem that has been before the manufacturer since the early days of the industry. Riparian landowners whose streams have been seriously polluted by the waste have had little difficulty in establishing their claim of injury in the courts, and, as a decision often granted a permanent injunction against the discharge of the waste into the stream, the strawboard companies were frequently obliged to undertake extensive remedial measures. As a result there grew up a custom of storing all liquid wastes in large reservoirs, depending upon evaporation and percolation to dispose of the water. (See Figs. 1 and 2.) There are in use at the present time many of these settling ponds, some of which are almost filled up with the solid matters from the wastes. As the ponds become filled new land is purchased and made into ponds, until, in many instances, the area covered by the water in the ponds has become so great that the companies are finding it difficult to provide sufficient storage capacity.

This method of disposal is unsatisfactory in many respects. The storage of such large volumes of the waste, which may at any time find its way into the river through the washing out of the dikes, constitutes a continuing danger. There is also, in certain seasons, some nuisance from odors, and the ponds may serve as a breeding place for mosquitoes. The sludge that fills up the ponds makes the land worthless for farming on account of the large amount of lime, so that the companies are not only losing the rental of the land, but also practically the whole value. At some mills 75 acres are either under water or filled up with sludge. It is apparent at the outset, therefore, that this method is uneconomical and, in time, will be more expensive than the construction and operation of a disposal plant.

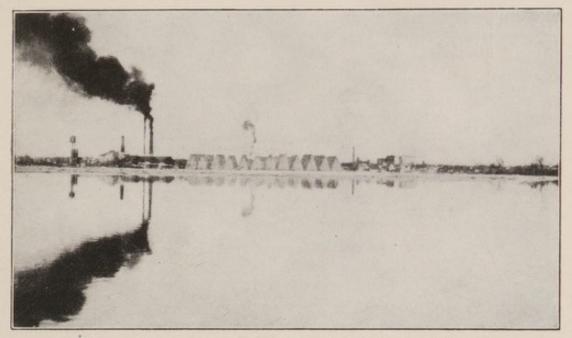


Fig. 1.-Settling pond. Strawboard mill and stacks of straw in background.



Fig. 2 .--- Effluent ditch and settling ponds.



State boards of health, particularly those of Indiana, Ohio, and Illinois, where the majority of strawboard plants are located, have long appreciated the seriousness of the problem, and it was through a request and promise of cooperation from the Indiana State Board of Health that the studies described in this report were made.

#### PREVIOUS INVESTIGATIONS.

Sackett,<sup>1</sup> in 1905, undertook on behalf of the United States Geological Survey an investigation of methods of treatment. He concluded that the addition of carbon dioxide and milk of lime is effective when properly applied and that under favorable conditions the process could be economically maintained. It is stated, however, that the manufacturers regard the expense of the process as prohibitive. Sackett also found that plain sedimentation, chemical precipitation by sulphate of alumina or sulphate of iron, filtration through sand, and treatment with lime alone were either not effective or so expensive as to be commercially impracticable.

The studies of the Geological Survey were continued by Phelps,<sup>2</sup> who recommended plain sedimentation for one hour, followed by mechanical filtration through sand at a rate of 100,000,000 gallons per acre per day. Under these conditions, experimental results gave removals of 98 per cent of the suspended solids, 77 per cent of the total organic matter, as measured by the oxygen consumed test, and 93 per cent of the suspended organic matter similarly measured. Wash water amounted to 9 per cent in the experimental plant, but it was estimated that this could be reduced to 5 per cent in a filter of suitable depth. Although it was shown that the first cost of such a plant is the principal element of cost in the process and would compare favorably with the costs of disposal by impounding basins, the process recommended has not thus far been adopted.

SUMMARY OF THE RESULTS OF THE PRESENT INVESTIGATION.

The present studies were undertaken to develop, if possible, a method of purification and sludge disposal which would more satisfactorily meet the requirements of the various State boards of health and fish and game commissioners, and at the same time be practical and within the economic resources of the strawboard companies. After conference with the officials of the Indiana State Board of Health the matter of location was taken up with the officers of the American Strawboard Co. It was decided to build a testing station

<sup>&</sup>lt;sup>1</sup>The Disposal of Strawboard and Oil-Well Wastes, R. L. Sackett and Isalah Bowman, U. S. Geol. Survey, W. S. Paper 113, 1905.

<sup>&</sup>lt;sup>2</sup> The Prevention of Stream Pollution by Strawboard Waste, Earle B. Phelps, U. S. Geol. Survey, W. S. Paper 189, 1906.

at that company's plant at Noblesville, Ind. This was done and the station was operated from January 1, 1915, to July 1, 1916. During the progress of these tests it was found that sedimentation of the waste in tanks for a period of two to three hours, followed by filtration through cinders, would produce a final effluent suitable for discharge into a stream without danger of creating a nuisance or killing fish. It was also learned that the sludge accumulating in the tanks could be dried over beds of cinders within a period of 30 days, and that the dried material could be hauled away in wagons and dumped either on land as a fertilizer or on lowland as a fill. Fertilizing tests carried out on the strawboard farm on a large scale proved that this material has valuable fertilizing ingredients which are worth, to the land, about as much as ordinary barnyard manure.

It was demonstrated that fish could live in the effluent from the filters and that the sanitary quality was such that samples were not putrescible when stored in stoppered bottles at room temperature for a period of about four days.

The plant as operated during the latter part of the tests appeared practical to the officials of the American Strawboard Co., and at the close of the tests the Public Health Service was requested to design a permanent unit, for installation by the company, for the purpose of demonstrating on a large scale the possibility of duplicating the results obtained in the smaller plant.

#### THE MANUFACTURE OF STRAWBOARD.

The process of making strawboard is the same to-day as it was in 1905, when Prof. Sackett made the first report on methods of purifying the wastes, and as a description of the various operations in a strawboard mill is essential to an understanding of the problem, the following is quoted from that report:

#### STEAMING IN ROTARIES.

"The process employed in the United States is as follows:

"The straw is first subjected to a cooking process by steam and lime. A large ellipsoidal rotating steel boiler, called a 'rotary,' shown in \* \* \*, figure 3, is filled with straw, which is then cooked down with steam, then again filled and cooked down until the rotary boiler has been completely filled. The process of filling a rotary occupies from 6 to 12 hours. The final charge consists of about 6 tons of straw and 30 bushels, or 2,100 pounds, of lime in the form of milk of lime. This mixture is then rotated and cooked under 40 pounds of steam pressure for 12 hours. Figure 3 shows the steam line extending through one of the trunnions and the worm gear which rotates

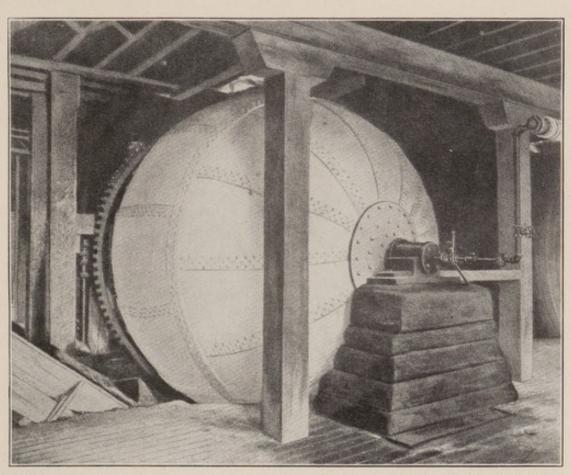


Fig. 3.-Rotary digestor. (From United States Geological Survey Water Supply Paper No. 113.)

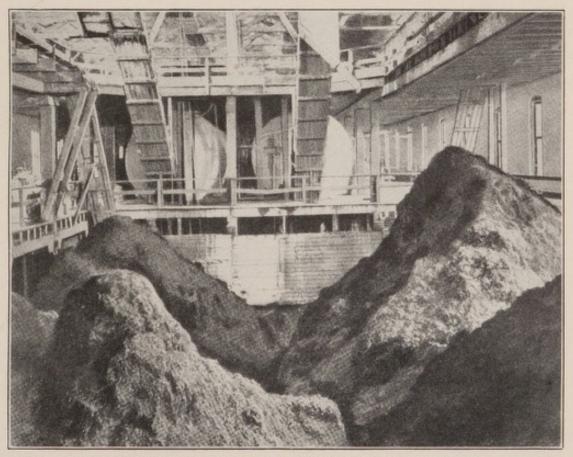


Fig. 4.—Rotaries and stock piles. (From United States Geological Survey Water-Supply Paper No. 113.)

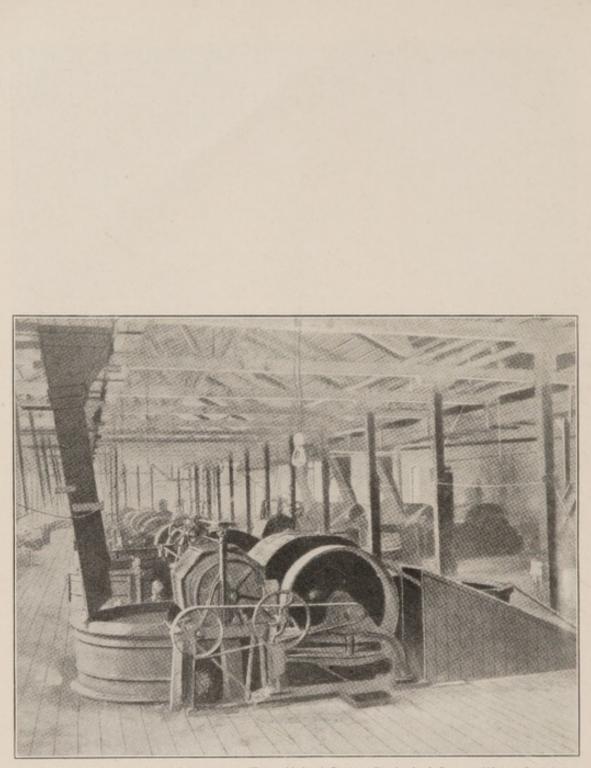


Fig. 5.—Pulp washers and beaters. (From United States Geological Survey Water-Supply Paper No. 113.)

the cylinder. This apparently severe chemical and mechanical action results in a rapid softening of the woody fiber and in the reduction of the straw to a dark-yellow pulpy mass. This 'stock,' as it is called, is stacked in piles 10 to 15 feet high to drain. Concerning the action which takes place in the rotaries the Journal of the Society of Chemical Industry says:<sup>1</sup>

The chemical action of the milk of lime on the encrusting materials surrounding the straw fiber is not a vigorous one. These encrusting materials are not completely, nor, indeed, to a great extent, separated from the cellulose. The mineral matter remains in the product practically untouched, and if any less quantity than that corresponding to the percentage in the original straw operated upon exists in the prepared pulp it is due rather to the washing after digestion than to any solvent action of the milk of lime. Milk of lime under certain conditions has a bleaching action upon the straw. It neutralizes the organic acids usually found when fibrous plants are heated for any length of time in the presence of water.

"The yield of pulp at this point will be from 75 to 80 per cent of the weight of the original material.

"Figure 4 shows the rotaries, charged from the floor above, and the elevators that carry the stock from the rotaries and dump it in the piles shown in the foreground. At the extreme left is an endless chain elevator, which carries the stock to the beater room.

"The material is allowed to stand in these piles for 24 hours or more to drain. After it is thus drained it contains about 50 per cent of water and from 6 to 7 per cent of lime. This is equivalent to from 12 to 14 per cent of lime in the dry straw. Since the original charge of lime and straw was in the proportion of 2,100 pounds of lime to 14,100 pounds of straw and lime, or about 14 per cent lime, practically none of the latter has drained out with the condensed steam. This drainage from the stock piles forms but a small part of the waste sewage. It is straw-colored and very turbid, carrying a small quantity of fiber broken fine in the rotaries.

#### WASHING.

"This process is much more drastic, and it is here that the great volume of waste is produced.

"The stock is run through washing machines for the purpose of removing the lime. A row of washers in operation is shown in figure 5. At the left is the chute from the conveyor above, down which the stock is fed. To the right of the chute are the water pipes. The washing machine consists of an oval channel about 3 feet wide, around which the stock travels, being supplied with copious volumes of water. Across this channel is placed a cylinder, 42 inches in diameter and 42 inches long, having longitudinal ribs or flanges about

<sup>1</sup> For Feb. 28, 1894, p. 101.

three-fourths of an inch square in section and three-fourths of an inch apart. Meshing with this, like the teeth of geared wheels, is an idler below, of similar size and form. These wheels, revolving, lift the water and straw to a level several inches higher than that in the oval channel, whence it flows by gravity half way around its course to a point where it meets a revolving brass screen of fine mesh, through which a part of the water escapes, carrying with it the finer particles of fiber and free lime. The remaining straw, with additional volumes of fresh water, now passes many times through the rolls which further mash and break the fiber, and around to the screen, where more straw, lime, and water escape. The total waste is enormous. It now takes about 40,000 gallons of water to wash 1 ton of straw. About 3,200 pounds of straw and 560 pounds of lime are required to make 2,000 pounds of board. A small amount of lime remains in the board; hence 1,200 pounds of straw and about 500 pounds of lime are washed out by the 40,000 gallons of water. An idea of the volume of waste may be conveyed by giving the capacity of an average mill. Such a plant uses 50 tons of straw and nearly 10 tons of lime during every 24 hours. From 1,000,000 to 2,000,000 gallons of water are employed in the rotaries, washers, and vats. This volume of water carries away with it about 19 tons of the straw and practically all of the 10 tons of lime each 24 hours.

#### DRYING.

"After the washing process the straw, with a considerable volume of water, is led to a train of rolls consisting of three parts-first, the wet end; second, the hot rolls; third, the trimming and cutting machine. As it comes from the washers the material is run into vats, where it is mixed with large quantities of water and passed over hollow cylinders having fine wire-cloth faces, which allow the water to escape, leaving the fiber on the surface of the cylinder. The fiber is then taken by woolen felts, which are pressed down on the surface of the cylinder. This makes a web of paper on the felt. The pulp, which is now about one-third straw and two-thirds water, travels up and down, over and under a double train of hot rolls, heated by steam that is carried in through hollow bearings. As the pulp passes on through the train it is constantly pressed and dried, until finally it is separated from its cloth support and goes to the trimming machine, where it is cut into sheets of proper size. It now contains about 10 per cent of water and a small quantity of lime. The board is manufactured in many thicknesses and weights. Just before it is trimmed it may be coated on one or both sides with a thin paper facing or finish."

### STRAWBOARD WASTES.

The volume and character of the wastes from the strawboard mill at Noblesville were studied throughout the entire period of the tests. It was felt, however, that these might not be representative of the wastes from the industry as a whole, and following the completion of the tests, surveys were made of seven other mills.

#### SURVEY OF STRAWBOARD MILLS.

During these surveys weirs were installed to measure the volume of wastes and several sets of representative samples taken for analvsis. The data obtained are summarized in tabular form for comparison. The completeness of this data is an evidence of the interest the officials of these different plants are taking in this work.

Raw material and water used.-In Table No. 1 are tabulated the raw materials used and the volume of waste from the various wasteproducing processes employed in the manufacture of strawboard.

Mill.	Product.	Tons ra	w materi	Volume waste per ton of product (1,000 gallons).					
		Straw.	Paper stock.	Straw clip- pings.	Lime.	Acid 18° B.	Beater.	Ma- chine.	Total.
Noblesville, Ind. <sup>1</sup> Do Noblesville, Ind. <sup>5</sup> Circleville, Ohio <sup>3</sup> Eaton, Ind. <sup>4</sup> Yorktown, Ind. <sup>5</sup> Lafayette, Ind. <sup>5</sup> Vincennes, Ind Rockport, Ind. <sup>5</sup> Urbana, Ohio Do	Manila-lined straw- board. Strawboard	1.84 1.60 1.70 1.48 1.60 .86 1.71 1.62 1.85 .28	0.37	0.28	0.16 .17 .18 .22 .17 .12 .15 .11 .23 .03	0.0025 .0014 .0053 .0020 .0028 .0006	18.7 17.9 21.3 22.2 14.5 7.3 27.2 14.5 9.5 1.9	19.6 18.5 25.2 16.1 4.1 5.0 16.0 11.9 26.4 25.1	42.2 38.3 38.0 46.5 40.0 18.6 12.3 43.2 26.4 35.9 35.5

TABLE No. 1.—Raw materials used and wastes produced in strawboard industry.

Period covered, August, 1915, to February, 1916.
Period covered, March, 1917. Data for other mills obtained January, February, and March, 1917.
2,000 gallons of machine waste are unpolluted.
1,700 gallons of total waste are unpolluted.
Part of machine waste used in beaters.

The amount of straw used per ton of board varies from 1.48 to 1.84 tons. Considering that only three days were covered in the survey of each mill, except the Noblesville plant, where the tests were made, these figures agree remarkably well. It is a well-known fact among strawboard men that the quality of straw varies not only from one season to another but for different periods in the same season and for different localities tributary to each mill, so that the differences found were, in a measure at least, due to the particular grade of straw used at the strawboard plants at the time the survey was made. The first figures given for Noblesville are the averages for the period, August, 1915, to February, 1916, while the survey of the other mills was made in January and February, 1917. During the time which the Noblesville results cover, the straw was of very poor quality for paper making, and this accounts for the relatively high ratio of raw material to finished strawboard. Later results obtained in March, 1917, showed a ratio of 1.6 tons straw to 1 ton of product.

The amount of lime used per ton of board varied from 0.23 ton at one mill to 0.11 ton at another. The figures for all the mills are weekly averages and therefore do not represent the actual amount used on the days the survey was made, but they should be comparable, and the difference in the amount of lime used is probably due to the particular quality of straw used during the week the data were obtained. The amount used at Noblesville in the first period was based on weekly averages for the seven months from August, 1915, to February, 1916. The second period in March, 1917, corresponds with the data obtained on the survey.

No accurate amount of the hydrochloric acid used was available except at three mills, and these figures were more in the nature of an average than the actual amount used on the days the mills were visited. The acid is used for removing the lime from the screens in the beaters and on the paper machines and from the felts, so that the actual amount used at any one time will depend on the condition of the screens and felts and would vary at different cleanings. The average amount used per ton of board made at all the mills where data was available, except at Urbana when using only 15 per cent straw, is 5.6 pounds, and this figure is probably correct within reasonable limits for long periods. This amount of acid is not enough to neutralize the lime in the waste during the time the screens are washed and has no bearing on the disposal problem.

The volume of waste produced per ton of board manufactured varies with the kind of stock used and also with the same kind of stock at the different mills. The waste was measured in every case over a weir set in the discharge line and read at half-hour intervals over a period of three days. For Noblesville the results given are the average for longer periods and were obtained from a weir set in the waste flume and are therefore more representative than those taken over the three-day period at the other mills.

The beater wastes at the different mills show wide variations. At one mill where 7.300 gallons are reported, the figure would be increased to 10,900 if the stock were all straw instead of one-third paper stock, from which there is no beater waste. Even this figure is lower than those from any other mill and it is accounted for by the rigid economy practiced in the water used while washing the lime and dirt from the straw. At the other extreme, where 27,000 gallons are reported for the beater waste, the mill was only recently placed in operation and an economical routine had not been established. Leaving out the mills using only part straw stock, the average beater waste is 18,200 gallons, which is probably a fair average figure. Local conditions, such as the quality of straw, the water supply, etc., no doubt have some effect on this waste. However, it seems reasonable to assume that in case of necessity, such as would arise where purification becomes necessary, the mills now producing the larger volumes of waste could cut them down considerably with a probable saving of product.

The machine wastes show an even greater variation. And here again local conditions have considerable bearing on the total volume. In the two cases where 4,100 and 5,000 gallons are recorded, most of the machine waste is used in the beaters to wash out the lime and dirt from the straw. The average volume of machine wastes for all the mills, except those using a part of these wastes in the beaters, is 19,900 gallons, and this is, no doubt, a fair average. It will be shown in a succeeding table that the suspended matter in the beater wastes is not higher where the machine wastes are used in the beaters; in fact, these wastes contain less suspended matter. It is apparent that more stock can be recovered by using the machine waste in the beaters.

The total wastes from all the strawboard plants visited show about the same variation from one another that existed in the individual wastes. Omitting the two mills where the machine wastes were used over to a large extent, the average is 38,500 gallons. This corresponds very closely with the average figures obtained at Noblesville covering a long period, and is no doubt a correct measure of the total waste of strawboard mills where machine waste is not used in the beaters and when there is no particular economy in the use of water. There is no doubt that this volume could be cut down to about one-half if the necessity should arise, and the reduced volume, as judged by the analyses where the volume of the total waste is less than one-half the average, would not present a more difficult purification problem, gallon for gallon, than the waste as discharged at the present time.

Composition of wastes.—In Table No. 2 are given the analyses of the different kinds of wastes from the different sources corresponding to the product made.

#### BEATER WASTES.

at our reolite		Results in parts per million.												
Loaving dist	Leal Dillion services	Suspended solid:			lids. Nitro-		Alkalinity as CaCO3.		ed, 30 C.	Oxygen de- mand 20° C				
Mill location.	Product.	Settlea c.c.p 100 c.		er c.		Supernatant after 24 hours.	c.	Ammoniacal.	ate.	onate.	Oxygen consumed, minutes-96° C.			
-Maginine anar	e estis bistor	1 hr.	2 hrs.	24 hrs.	Total.	S u p e after	Organic.	Ammo	Carbonate.	Bicarbonate.	Oxygei	1 day.	5 days.	10 days
Vincennes, Ind Rockport, Ind	Strawboarddo. do. do. Strawboard (1 paper) Strawboarddo. do. do. Chip board (15 per cent straw stock).	3.3 2.8 5.3 6.7	2.6 5.2 6.5	3.5 7.0 6.0	3,480 3,450 3,830 6,903	1,230 1,460 970 1,020 1,360	72 49 61 89	4.0 2.5 2.0 2.3 3.0 1.0 2.6	1,130 640 210 570 0 1,610	1,640 430 320 2,630 770 280 1,100 1,700 1,130	3,400 2,400 1,480 2,830 2,220 1,650 2,120 2,680	480 70 60 480 410 250 430 338	2,400 2,090 1,440 2,380 1,970 1,250 1,620 2,112	3, 30 2, 65 2, 05 3, 20 2, 50 1, 85 2, 25 3, 15
He for a bear	МА	СН	IN	EV	VAST	ES.						1		
Circleville, Ohio Eaton, Ind Yorktown, Ind Lafayette, Ind Vincennes, Ind Rockport, Ind	Manila-lined straw- board. Buckskin and straw- board. Strawboard. do. Strawboard (‡ paper) Strawboard do. Chip board( 15 per cent straw stock).	1.8 2.1 2.0 2.2 3.5 2.5 2.2	1.8 2.1 2.0 2.2 3.2 2.2 2.2	1.5 2.6 1.5 2.2 3.8 2.0 2.2	920 590 820 630 970 1,440 475 545	170 370 60 310 460 230 160 147	11.6 16.0 15.0 20.0 43.0 12.5 19.0 20.8 10.4	.6 2.0 1.0 .9 .8 .4 1.5	0 140 0 0 0 0 35	460 385 200 760 550 405 385 595	250 320 320 490 970 220 375 533	20 60 38 80 190 37 70 96 32	200 215 280 640 120 230 247	
Patrice and the	М	IXF	ED	w.	ISTE	S.2	10.00							101
Yorktown, Ind	per board. Strawboard and ma- nila lined. Strawboard and buckskin. Miscellaneous Chip board. Strawboard. Strawboard. do. do.	···· 5.8 1.5 2.7	···· ···· 5.1 1.5 2.8	 6.3 1.5 2.5	2,435 2,080 1,080 2,500 3,000 1,030 4,090	 350 80 1,340	34.0  34.4 50.0 34.0 62.0	···· 2.0 .8 2.0	200 150 90 215 260 140 450	740 640 280 740 550 285 1,830	1,040 980 1,800 1,290 400 2,320	35 40  55 80 310		1,44
Vincennes, Ind	Strawboard (1 paper) Strawboarddo. do. Chip board (15 per cent straw stock).	3.8	$3.8 \\ 5.0$	3.7	1,950 2,230	720	72.0 38.0 42.0 37.8 14.8	.5 1.0 1.7	0 0 425	635 735 870	1,640 1,050 1,570 1,062 355	$220 \\ 350 \\ 156$	850 1,000 707	1,2 1,4

<sup>1</sup> Data for Noblesville covers period from August, 1915, to February, 1916. Data for other mills is the average from three separate sets of samples, from each mill, taken in January, February, and March, 1917. <sup>2</sup> Analyses are for samples collected at main outlet of mills.

Under beater wastes the only product that is considered is strawboard, as only straw is required to go through this process previous to making it into paper. The settleable solids in the first three columns were determined by pouring a measured volume of the waste into a conical-shaped glass graduated at the point in cubic centimeters and reading the volume of solids that settled out at the end of 1, 2, and 24 hours. The results show the volume of suspended matter that will settle from 100 volumes of waste water in the periods of time designated at the top of the columns. The amount of suspended matter that will settle out in 1 hour is practically the same as that in 2 hours, and in 24 hours the increase is very small. These figures check the data obtained in the operation of the testing stations, allowance being made for the fact that in the tanks the waste was in motion, while in the settling glasses it was quiescent. A two-hour detention period in the settling tanks removed 95 per cent of all the settleable solids.

The results agree as closely as could be expected, except for two of the mills. One of these uses a large part of the machine waste in the beaters, which would tend to increase the suspended matter, and at the other mill the high results are probably due to abnormal operation at the time the samples were taken or to a low grade of straw. The suspended matter in the supernatant in the settling glasses agrees in a general way with that found in the effluent of the settling tanks at the testing station.

The organic nitrogen is high in the Noblesville samples, but fairly uniform for the other samples, the variations being readily accounted for by the different quality of straw used. The free ammonia is low in all the samples and has no particular significance.

The high alkalinity results are due to the lime washed out of the stock after coming from the rotaries. For the most part the variations in the values as tabulated for the different mills find natural explanation in the amount of lime used, as given in Table No. 1. There are, however, other factors, such as the concentration of the waste due to the volume of water used, the quality of the lime, and the hardness of the wash water, which readily account for such unexpected results as were found at Eaton.

The oxygen consumed is high in all the samples and the results check very closely the oxygen demand for the 10-day incubation period at 20° C. given in the last column. The comparison of the 1-day oxygen demand with the oxygen consumed and the 10-day oxygen demand shows very clearly that these wastes do not undergo decomposition to any extent during the first 24 hours, but after that time the rate increases until practically complete oxidation has taken place at the end of 10 days.

The analyses of machine wastes show that most of the material in the wastes is cellulose in the form of short fibers. With suspended matter ranging from 475 to 1,440 p. p. m. the organic nitrogen for

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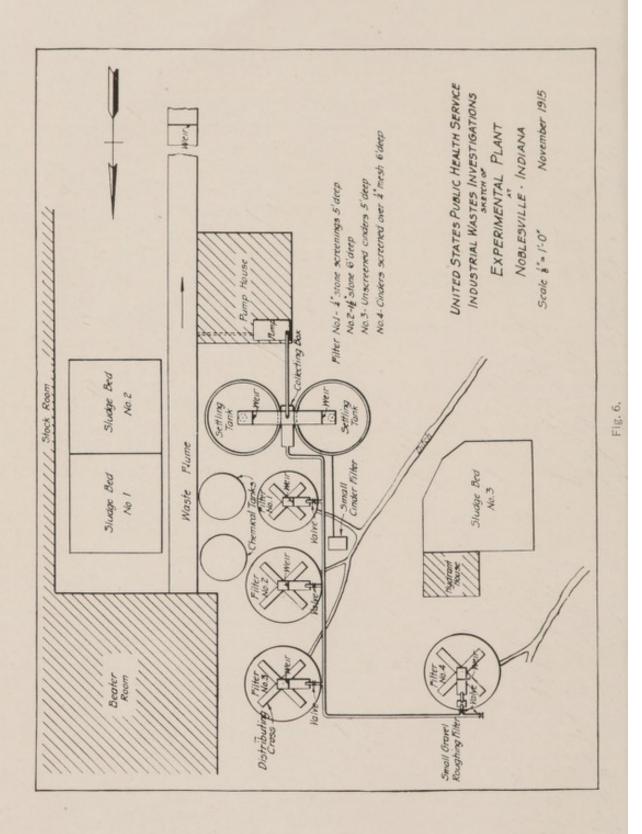
the extremes was only 10.4 and 43 p. p. m., respectively, the oxygen consumed 191 and 970 p. p. m., and the 10-day oxygen demand 111 and 890 p. p. m. Attention is again called to the close agreement between the oxygen-consumed results and the 10-day oxygen demand. The settleable solids show that practically all the material that can be removed by quiescent settling has been removed at the end of one hour.

In the samples collected from the mixed wastes or the composite from the entire plant practically all the settleable solids were removed by 1-hour quiescent settling. The suspended matter varies considerably, but the average is not far from the results obtained at Noblesville for the long period covered by the tests. The suspended matter in the supernatant, except at the Yorktown and Lafayette mills, was lower than was obtained in the effluent of the settling tanks. The organic nitrogen is high in the wastes from the mills where the machine wastes are used in the beaters, but it does not fluctuate with the volume of water used as would be expected. The average free ammonia found in the mixed wastes for all the plants is 1.3 p. p. m. and the average for the beater and machine wastes, which make up the total, are 2.4 and 0.9 p. p. m., respectively, so that the amount found bears a very close relation to that computed from the ratio of the volume of beater and machine wastes to each other.

The alkalinity of the mixed wastes is about the same as in the beater and machine wastes when mixed together in proportion to the volume of flow, and in the wastes from the other stock it is about the same as the alkalinity of the water of the mill supply. The oxygen consumed and the oxygen demand results likewise correspond in a general way to the figures that would be obtained if computed from the analyses of the machine and beater wastes according to the ratio of the volumes of these wastes to each other.

On the basis of the figures given in the table for the average total volume of waste per ton of strawboard manufactured, and on the rated capacity of the strawboard mills in this country in 1918 (Lockwood's Directory of the Paper and Stationery Trades, 1918) there will be approximately 20,000,000,000 gallons of waste discharged from the various plants during the year. Computed on the basis of the average suspended matter in the mixed wastes from all the plants visited (Table No. 2) there will be approximately 161,000 tons of dry suspended solids 'discharged from all the plants. There certainly is a large field for experimental work along the lines of recovering a large part of this material, most of which is short fiber and some of it washed and ready to be made into paper if the proper machinery can be invented to handle it.





### DESCRIPTION OF TESTING STATION.

The original plans for the testing station were based on the earlier studies made by Sackett and by Phelps, with provisions for changing the arrangement and type of the devices to conform with the possible development of the studies from chemical precipitation and mechanical filtration to other well-known methods of purifying sewage. (See Fig. 6.)

The station was located in the rear of the American Strawboard plant and adjacent to the flume carrying all the waste from the mill.

#### PUMPING EQUIPMENT.

A 2-inch horizontal centrifugal pump direct-connected to a  $5\frac{1}{2}$ horsepower marine gas engine was used for raising the waste from the flume to the settling tanks. The pump was set so that the waste flowed through the pipe connecting it with the waste flume to the discharge outlet and the discharge lift was 15 feet. This arrangement increased the discharge lift somewhat but had the advantage of having the pump always primed. The waste was discharged from the pump to the settling tanks through a 2-inch pipe provided with a by-pass running back of the waste flume. The by-pass was located just above the pump and the amount pumped to the tanks was controlled by a valve in this line. To increase or decrease the amount of waste treated in the tests the valve in the by-pass line was either opened or closed.

#### SETTLING TANKS.

Tank No. 1.—This was a round wooden tank 8 feet in diameter and 10 feet deep. This tank was operated as a Dortmund tank from January 1 to October 6, 1915; as an Imhoff tank from November 2, 1915, to March 6, 1916; and as a Dortmund tank from March 6 to July 1, 1916. The total capacity was 3,770 gallons. During the first period the working capacity was 3,580 gallons, with a settling chamber of 2,262 gallons, and sludge storage capacity of 176 cubic feet. As an Imhoff or Dortmund tank after November 2 the flow section had a volume of 3,640 gallons and the sludge section 201 cubic feet. The difference in the available volume during the two periods was effected by raising the overflow level. The waste discharged from the pump was received in a weir box set directly over tanks Nos. 1 and 2, where it was divided equally between them by weirs set in the ends of the weir box over each tank.

The waste was applied to the Dortmund tank through a 6-inch No. 22 galvanized iron tube fastened to the weir box and extending down into the tank  $5\frac{1}{2}$  feet and the effluent was taken off through four side outlet ells and four tees spaced equally apart in a pipe forming a square and set in the tank so that the corners were 1 foot from the side of the tank.

Sludge was removed from the tanks through a 4-inch pipe with valves outside the tank and an elbow turned down in the center of the tank 4 inches from the bottom.

When this tank was converted to an Imhoff tank, the dosing tube and outlet collecting pipes were removed and a galvanized-iron funnel (see fig. 7) was installed to separate the flow section from the sludge-digestion chamber. The waste was discharged into a gutter, 6 by 6 inches, built around the gas vent. Overflowing this, it passed down under a circular baffle wall 15 inches from the side of the tank and 2 feet deep, and was collected in a gutter 4 inches wide, extending around the inside of the tank. The sludge pipe remained the same as originally installed.

In the final period of operation of this tank, March 6 to July 1, 1916, the waste entered through a dosing tube extending down to within 6 inches of the bottom, passed up through the sludge, and between the separating baffles of the Imhoff design and out over the collecting trough around the inside of the tank.

Tank No. 2.—This was operated as a Dortmund tank from January 1 to October 6, 1915, and as an Imhoff tank from November 2, 1915, to July 1, 1916. The capacities of the tank during the two periods and the method of applying and removing the waste were the same in this tank as for No. 1 during the corresponding periods.

Tank No. 3—Chemical reaction tank.—This was a round wooden tank 6 feet in diameter and 10 feet deep. Its capacity was 2,015 gallons below the flow line, which was 6 inches below the top of the tank. Allowing 6 feet for settling capacity the sludge chamber had a capacity of 103 cubic feet.

This tank received the combined waste from tanks 1 and 2, and also the chemical solutions, through a weir box set directly over the tank. The dosing tube and collecting system used throughout the entire period of operation were similar to those in tanks 1 and 2 during the first period. There were no baffles.

Tank No. 4.—Chemical settling tank.—This was a round wooden tank 6 feet in diameter and 10 feet deep with a total capacity of 3,770 gallons and a working capacity of 3,580 gallons below the flow line, 6 inches from the top. Allowing 6 feet for flow section there was a sludge storage capacity of 176 cubic feet. There were no baffles.

This tank received the entire effluent from tank 3, and during the entire period of operation, January 1 to September 14, 1915, the same kind of dosing tube and collecting system for removing the waste was

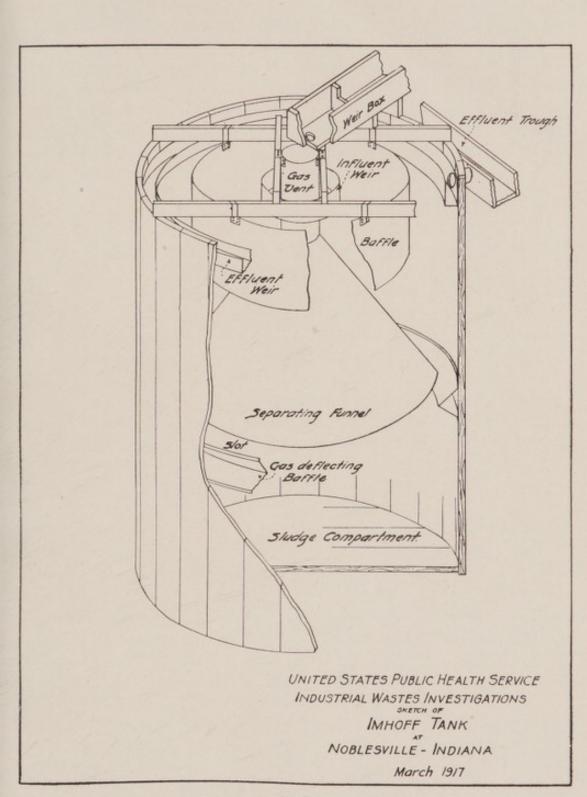


Fig. 7.

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used in this tank as in tanks 1 and 2 during the first period in service of these tanks.

Chemical solutions tanks.—These were two round wooden tanks, each 5 feet in diameter and 6 feet deep, with a capacity of 884 gallons when filled to within 6 inches of the top. They were used for dissolving the chemicals and contained no baffles or stirring devices. Outlet pipes at the bottom of each tank were connected with orifice boxes which controlled the rate of application to the waste entering the chemical reaction tank. The elevation of these tanks was such that the solutions flowed by gravity to the waste treated.

#### FILTERS.

Mechanical filter.—This filter was constructed in a round wooden tank 8 feet in diameter and 6 feet deep. A 4-inch water line connected with the mill storage tank passed through the side of the tank at the bottom and ended in a tee at the center. From this tee another 4-inch line was taken through the side of the tank and carried to a weir box 6 feet below the bottom of the tank, where it was submerged to maintain a negative head. Over the bottom there was constructed a framework consisting of two squares made of 2 by 4 inch studding, resting on posts and placed concentric with the tank. The smaller square had 2 feet on a side and the larger  $5\frac{1}{2}$  feet. On these were . placed 2 by 2 inch slats, radiating from the center, and so set that they were 15 inches apart at the outside of the tank and 2 inches at the center. A strainer plate of one-sixteenth-inch iron, cut in three sections, was placed on the slats. Holes one-sixteenth inch in diameter were drilled on 1-inch centers over the entire area of the plate.

A layer of gravel, 6 inches deep, averaging in size from one-fourth to one-half inch, was placed over the strainer plate and over the gravel a one-fourth-inch screen. About the screen there were 6 inches of gravel averaging one-eighth to one-fourth inch in diameter and another screen with one-eighth-inch mesh. Two feet of sand, with an effective size of 0.36 millimeters, and a uniformity coefficient of 1.33, were placed over the one-eighth-inch screen.

Settled waste was applied to the filter by means of a cross of galvanized-iron troughs, 6 by 6 inches, which also served to remove the wash water through an overflow connected to the end of one arm. This arrangement was not satisfactory and the rectangular troughs were replaced by a large V-shaped trough extending across and 18 inches above the surface of the sand. The rates at which the filter was operated were controlled by a valve on the effluent line from the tank and the length of run was regulated by the time required to use up the total head (12.2 feet) while maintaining a specified rate. The wash water was removed through this trough, but there was also provided an outlet 8 inches above the surface of the sand to drain off part of the water standing on the surface after washing.

The filter was operated from January 1 to July 27, 1915, without any changes in construction except in the troughs as noted above.

Small cinder filter.—This filter was 4.45 square feet in area,  $4\frac{1}{2}$  feet deep and filled with unscreened cinders but thoroughly washed with water after being placed in the bed. The bottom was sloped toward the outlet pipe screwed into the side of the frame of the filter at the base. The influent was applied through a rubber tube working as a siphon and regulated by means of a screw cock. Distribution was obtained by moving the jet from the rubber tubing over the surface.

The influent to this filter was the settled waste from the secondary settling tank No. 4 from July 30, 1915, to September 10, and from primary settling tanks Nos. 1 and 2 from September 20, 1915, to March, 1916, after which from tank No. 1 alone. There were no changes or modifications in the structure of the filter during the entire period of operation except that the filter was washed on May 13, 1916, to see how far it was clogged.

Slow sand filter.—The area of this filter was 2.4 square feet and the depth 4 feet 3 inches. The sand rested on a bed of stone screenings 1 inch thick, and under the screenings there were 3 inches of  $1\frac{1}{2}$ -inch limestone. The bottom sloped toward an outlet pipe screwed into the bed flush with the base of the filter. The influent to the filter was applied through rubber tubing acting as a siphon and the rate controlled by a screw cock. The amount of waste treated on this filter, as well as on the small cinder filter, was measured every hour in a graduated cylinder. The influent was from the secondary settling tank No. 4 during the entire period of operation, July 29 to September 11, 1915.

Filter No. 1.—In October, 1915, the chemical-reaction tank was dismantled and reconstructed as filter No. 1. The area was 28.27 square feet, or 0.000649 acre, and the depth of the tank 10 feet. Concrete was placed over the bottom and sloped from  $1\frac{1}{2}$  inches to one-fourth inch at the outlet. Over the concrete there were placed 4 inches of round stone varying in size from  $2\frac{1}{2}$  to 4 inches, and above this layer there were 2 inches of  $1\frac{1}{2}$ -inch limestone, and on top of this stone 5 feet of limestone screenings, averaging about one-fourth inch in diameter. The amount of waste treated in this filter was measured every hour in a graduated cylinder and the rate controlled by a valve located over the distributing troughs. These troughs were onehalf inch deep and 2 inches wide and made in the form of a cross, the arms extending from the center to within 1 inch of the side of the tank, where they were supported by blocks of 2 by 4 inch studding nailed to the tank. The troughs were reinforced on the bottom to keep them from sagging in the middle, and once a week the cross was moved to a second set of blocks set at 45° to the first set to obtain a better distribution of the waste over the surface of the filter. Notches were cut in each side of the troughs and staggered to assure an even flow to each section of the filter.

No changes in the construction of the filter were made from the time it was placed in operation, November 2, 1915, till the close of the tests, July 1, 1917. The method of operating was changed to learn the highest rates that could be maintained, and on May 13 the filter was washed with water to determine the extent of clogging. Up to March, 1917, the influent was the effluent from tanks Nos. 1 and 2, and after that date it was from tank 2 alone.

Filter No. 2.—This filter was constructed in the chemical settling tank following the dismantling of the mechanical filter. It was 50.27 square feet or 0.00115 acre in area. Concrete was placed over the bottom, sloping from 2 inches to one-fourth inch at the outlet, and a layer of round stones, 3 to 4 inches in diameter, placed on the concrete, and above this a layer of crushed limestone  $5\frac{1}{2}$  feet deep, with an average size of  $1\frac{1}{2}$  inches. The same arrangements for measuring and controlling the rate on this filter were used as in No. 1. The movable cross for distributing the influent was always kept level and the notches free of clogging material.

The filter was operated throughout the period of the test, November 2, 1915, to July 1, 1916, without changes in the construction, but the operation was varied to include different rates and it was washed on May 13, 1916. The influent was the settled waste from tanks Nos. 1 and 2 till March, 1916, and after that from tank No. 2.

Filter No. 3.—The mechanical filter was dismantled and this filter constructed in its place. The area was 50.27 square feet or 0.00115 acre. The underdrainage for this filter was the same as for the others already described, and the filtering material was 5 feet of unscreened cinders produced at the plant. The same method of applying the waste and controlling the rates was used as already described for the other filters.

This filter was operated as originally constructed from November 2, 1915, to July 1, 1916, and the operation changed only as regards the rates. It was washed May 13, 1916, the same as all the other filters by applying water to the surface through a 1-inch hose.

Small gravel roughing filter.—This filter was constructed of galvanized iron, had an area of 0.444 square foot at was 2 feet 4 inches deep. It consisted of three parts, the main body of the filter 8 inches square in plan and 2 feet deep, a section 8 inches wide by 12 inches long by 2 inches deep soldered into the bottom of the filter, and a pipe 2 inches in diameter soldered into the bottom section and extending up to the level of the filter surface with an elbow turned away from the filter. Two feet of fine gravel, averaging about one-eighth inch in diameter were placed in the main body of the filter, and below this, in the section 8 inches by 12 inches, there were 4 inches of graded coarse gravel to hold up the fine gravel. The 2-inch pipe leading from the lower section to the top of the filter served to carry the filtered waste from the bottom to the top and permitted the filter to be flooded all the time for the purpose of coagulating the suspended matter, as will be explained in more detail under the operation of the filters. Water for washing the filter was applied through a 1-inch hose connection soldered into the elbow at the top of the 2-inch pipe and an overflow provided at the top of the filter removed the water at the surface of the filter.

This filter, operating as a roughing filter, was designed to treat all of the waste going to filter No. 4, and was operated from November 21, 1915, to May 13, 1916, receiving as influent the settled waste from tanks Nos. 1 and 2 till March, 1916, and after that date the effluent from No. 2.

Filter No. 4.—The tank in which this filter was constructed had not been used up to the time the plant was remodeled, in October, 1915. It was a round wooden tank 8 feet in diameter and 10 feet deep, and as a filter it consisted of a concrete sloping bottom, overlaid with 5 inches of coarse and fine stone graded to prevent the filtering material, consisting of 5 feet of cinders, screened over a onefourth-inch screen, from matting together over the bed of the filter.

The control of the operation was the same for this filter as for No. 1, already described, and the influent was the effluent from the small roughing filter from November 2, 1915, to May 13, 1916, and from the latter date to the close of the tests the settled effluent from tank No. 2. There were no changes in construction during the entire period, but the rates varied, and on May 13 the filter was washed by applying water to the surface through a 1-inch hose.

#### SLUDGE-DRYING BEDS.

Three sludge beds were constructed, two in the rear and between the testing station and the stock room of the strawboard mill and the other one in front of the tanks. The bottom of the beds sloped to a central drain. Beds Nos. 1 and 2 were each 10 feet square and inclosed by a water-tight wooden frame, and No. 3 was 138 square feet in area and was inclosed by dikes of earth. Straw, free of chaff and compacted, was placed to a depth of 4 inches in bed No. 1 and 5 inches of unscreened cinders were placed in bed No. 2. There were  $7\frac{1}{2}$  inches of screened cinders placed in bed No. 3 and tile were laid over the bottom to facilitate the drainage. Wooden troughs were used to remove the sludge from the tanks to the drying beds.



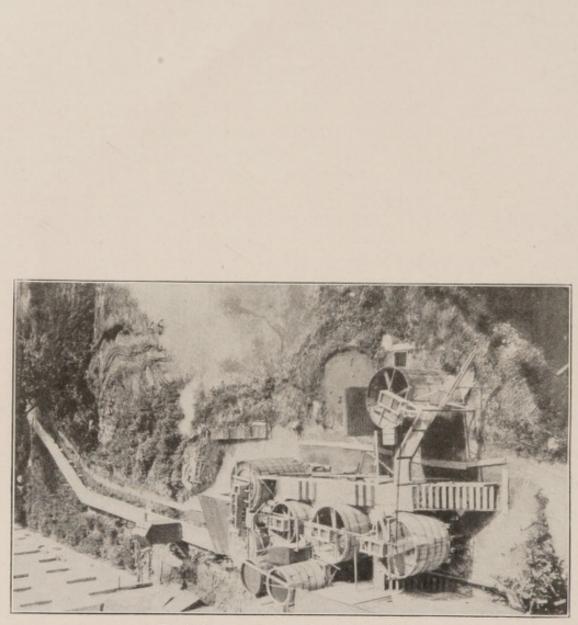


Fig. 8.-Bird's-eye view of the testing station.

In the following table a summary is given showing the design and operation of the various units throughout the tests:

and the second	-				1	
and barre	Dimen	sions.	Filtering	Period of	Influent.	Type of units.
Units.	Diam- eter.	Depth.	material	operation.	innuent.	Type of diffes.
Settling tank	Feet. 8	<i>Feet</i> . 10	·····	Jan. 1, 1915, to Oct. 6, 1915.	Raw waste	Dortmund tank.
NO. 1.	1000	append in	In Decide 1	Nov. 2, 1915, to Mar. 6, 1916.	do	Imhoff tank.
		13.81	and manufactor	Mar. 6, 1916, to	do	Dortmund tank.
Settling tank	8	10		July 1, 1916. Jan. 1, 1915, to	do	Do.
No. 2.				Oct. 6, 1915. Nov. 2, 1915, to	do	Imhoff tank.
Chemical reac- tion tank.	6	10		July 1, 1916. Jan. 1, 1915, to Sept. 14, 1915.	Combined efflu- ents, tanks Nos.	Chemical precip- itation tank.
Chemicalsettling	8	10		do	1 and 2. Effluent chemical reaction tank.	Settling tank.
tank. Chemical solu-	5	6		do	reaction tank.	Solution tanks.
.tion tanks (2). Mechanical filter.	8	6	3 feet of sand	Jan. 1, 1915, to	Effluent chemical settling tank.	Filter.
Small cinder fil-	1000	44	and gravel. Screened cin-	July 27, 1915. July 30, 1915, to	do	Do.
ter.			ders.	Sept. 10, 1915. Sept. 20, 1915, to July 1, 1916.	Effluent settling tanks Nos. 1 and 2.	Do.
Slow sand filter	12.4	41	Sand	July 29, 1915, to	Chemicalsettling	Do.
Filter No. 1	. 6	10	5 feet of lime- stone screen-	Sept. 11, 1915. Nov. 2, 1915, to July 1, 1916.	tank. Effluent from set- tling tanks Nos. 1 and 2.	Do.
Filter No. 2	. 8	3 10	5.5 feet of 11-	do	Contraction of the second s	Do.
Filter No. 3	. 8	6 6	5 feet of un- screened cin-		do	Do.
Roughing filter	. 10. 444	1 23	ders. 2 feet fine gravel, 4 inch-		do	Do.
Filter No. 4	. 8	8 10	es coarse. 5feet of screened		Effluentroughing	Do.
			cinders.	May 13, 1916. May 13, 1916, to July 1, 1916.	filter. Effluent settling tank No. 2.	Do.

TABLE No. 3.—Table showing design and operation of testing units.

<sup>1</sup> Area in square feet.

Approaching the testing station from the steps shown in the photograph (see fig. 8) the screened cinder filter is on the right, and the unscreened cinder filter to the left. Proceeding down the platform the next tank in order is the 2-inch limestone filter, and next to this is the filter containing limestone screenings. The two large round tanks farthest from the steps are the settling tanks. The two small tanks to the left of the picture were used for dissolving the chemicals in connection with the operation of the mechanical filter. Just beyond the two settling tanks is the pump house in which were located the gas engine and pump. The suction of the pump extended out into the flume shown leading away at the far end of the picture. The boxes in front of the pump house were used for fertilizer tests. A fish box is shown to the right and beyond the screened cinder filter bed to the right of the steps, and just beyond the fish box is shown one of the sludge beds. The sludge beds indicated on the plan at the left of the flume were not constructed when the picture was taken. The waste discharged from the pump was measured in a weir box located on top of the settling tanks and the pipe line running over the top of all the tanks carried the effluent from the settling tanks to the weir boxes set over the filters.

## **OPERATION OF TESTING STATION.**

The experimental studies covered a period of about one and onehalf years, from January 1, 1915, to June 30, 1916. These dates include only the time during which the testing station was operated and exclude the preliminary construction period.

The actual tests may be divided roughly into two periods. During the first one the operation was based on previous studies made by Phelps and by Sackett, and was confined to the use of mechanical filtration following sedimentation, with or without the aid of chemical coagulants. During the second period biological treatment on filters of coarse media, in conjunction with sedimentation without coagulation, was studied. The main tests in this period were prefaced by small-scale studies to justify the change which involved rather extensive remodeling of the plant.

### PERIOD 1.

# PLAIN SEDIMENTATION—CHEMICAL PRECIPITATION—MECHANICAL FILTRATION.

Period 1 covered seven months, January to July, 1915, inclusive. During this period the mechanical filter and four settling tanks were operated.

Sedimentation and chemical precipitation.—Tanks 1 and 2 were connected in parallel, and the waste which was pumped for treatment was equally divided in a head box with one-half going to each tank. The effluents were collected and combined in a common box and applied to tank No. 3, which was followed by tank No. 4, so that each of these two received the total pumpage or twice that applied to either tank No. 1 or No. 2. All tanks were operated as Dortmund tanks.

The volume of waste treated in the tanks during this period is recorded in Table No. 4.

		Wast treate		Tan	ks 1	and 2.			та	nk 3.		-		Tank	4.	hours, all ries.
Month, 1915.	Total waste, gallons per	ber day.	of total ste.		period, ch tank.	e treated, ns per day, tank.		ing period, hours.	gra	emic uns j allor	per	treated, per day.		Settling period, hours.	treated, per day.	period, ks in se
	24 hours,1	_	Per cent of waste.	Influent.	Settling period hours each tank.	Volume gallons each tai	Influent.	Settling	Alum.	Iron.	Lime.	Volume gallons	Influent.	Settling	Volume	Settling
January. February March. April. May. June.	1, 110, 000 1, 110, 000 1, 110, 000 1, 110, 000 1, 110, 000 1, 110, 000	13.640 14,270 15,900 14,730 11,070	$     \begin{array}{r}       1.2 \\       1.3 \\       1.4 \\       1.3 \\       1.0 \\       1.0     \end{array} $		2.1 2.0 1.9 2.1 2.2	6,820 7,145 7,950 7,360 5,535		$\begin{array}{c} 0.59 \\ 0.59 \\ 0.54 \\ 0.54 \\ 0.59 \\ 0.63 \\ 0.63 \end{array}$	10 10 20			$\begin{array}{c} 11,470\\ 13,640\\ 14,270\\ 15,900\\ 14,730\\ 11,070\\ 14,590 \end{array}$	(+) (+) (+) (+) (+) (+) (+) (+) (+) (+)	1.1 1.1 1.0 1.0 1.1 1.2 1.1	$\begin{array}{c} 11,470\\ 13,640\\ 14,270\\ 15,900\\ 14,730\\ 11,070\\ 14,590\end{array}$	3.8 3.4 3.4 3.8 4.0
July	1, 110, 000				2.1		(3) 	0.59	_	5.7	0.1	14, 590		1.1	13,667	-

[Jan. 1 to July 31, 1915.]

<sup>1</sup> Average during operation from August, 1915, to July, 1916.

<sup>2</sup> Raw waste. <sup>3</sup> Effluent tanks 1 and 2.

4 Effluent tank 3.

As arrangements had not yet been made to measure the total flow from the mill, the value given does not represent actual conditions. It is, however, very nearly correct, being an average of actual measurements taken over the period of 11 months following those months contained in the table. On the basis of this average figure the test tanks were able to settle 1.2 per cent of the total waste from the mill for an average total detention period of 3.7 hours.

The main preliminary settling of 2.1 hours was afforded in tanks Nos. 1 and 2. These were connected in parallel to give a sufficiently long detention without a too large and too expensive tank and yet to treat a sufficient volume of water to make the tests representative. With this detention period the upward velocity of flow was 0.05 foot per minute.

Tank No. 3 served mainly as a mixing tank for the chemicals which were added to the waste as it entered, and the storage period was consequently short, 35 minutes, corresponding to an upward velocity of 0.19 foot per minute. This point of application was chosen in preference to the inlet of the first tanks, because it was desired to operate with economical chemical treatment, and this seemed most practical when the bulk of the suspended cellulose had been removed.

In tank No. 4 there was afforded a detention period of 1.1 hours to settle the chemical precipitate formed in tank No. 3. The upward velocity of flow was 0.1 foot per minute.

The results obtained as shown by the alkalinity and suspended solids determinations are recorded in Table No. 5.

# TABLE No. 5.—Monthly average analyses of raw and settled waste during period 1.

	Raw w	aste.	E ffluer 1 a	fluent <sup>1</sup> tanks 1 and 2.				Efflu	ent tan	k 3.			Effluent tank			s 4.
		total.	Susper solid	nded s.	total.	g	nemic rains p gallor	per	Susp so	ende lids.	d	total.	Sus	pend olids,	led	total.
Month, 1915.	is, total.	CaCO <sub>3</sub> , to		al.	CaCO <sub>3</sub> , ti					Per	cent oval.	CaCO <sub>3</sub> , to		Per	cent oval.	03,
	Suspended solids,	Alkalinity <sup>2</sup> as (	Total.	Per cent removal	Alkalinity as <sup>2</sup> (	Alum.	Iron.	Lime.	Total.	Over influent.	Over raw.	Alkalinity <sup>2</sup> as C	Total.	Overinfluent.	Over raw.	Alkalinity <sup>2</sup> as (
farch pril fay une uly	1,810 2,050 2,120 1,220 1,110	470 400 300 370 310	970 1,260 1,000 550 530	47 39 53 55 52	490 390 350 330 320	10 10 20	10.0 5.7	5.7	810 1,050 960 600 570	16 $17$ $4$ $-9$ $-8$	55 49 55 51 49	460 350 300 330 360	740 890 790 560 530	9 15 18 7 7	59 57 63 54 52	46 32 32 36 31
Average	1,660	370	860	48	380				800	7	52	360	700	12	58	35

[Jan. 1 to July 31, 1915.]

<sup>1</sup> Tanks in parallel. Samples taken of mixed effluents. <sup>2</sup> Analyses made on filtered samples.

As would be expected, the alkalinity content remained practically constant throughout the whole series of tanks, due allowance being made for sampling error. Even when 5.7 grains per gallon of lime were added there was no appreciable increase, indicating that the precipitant was largely removed as sludge.

Tanks Nos. 1 and 2 removed about 48 per cent of the suspended solids. While this result compared well with the results of similar tank treatment of domestic sewage, it is too low when viewed in the light of the actual amount of suspended solids left in the effluent.

There was only a small amount of suspended solids removed by tanks Nos. 3 and 4. Tank No. 3 brought the total average removed up to 52 per cent. This value is lowered by the negative values or increases in suspended solids during June and July, which were due to the development of septic action and the unloading of sludge. The results accomplished by tank No. 4 are but little better than those by tank No. 3. The total removal was increased to 57 per cent. The average amount of suspended matter in the effluent was 700 p. p. m.

It may be concluded that the economically settleable solids are removed during a detention period of two hours and that the advantage of chemicals, used within practical limits, is negligible. These conclusions confirm the results obtained by Sackett and by Phelps.

Mechanical filtration.—The combined effluents from the settling tanks were treated on a mechanical filter similar to those used in water purification. Table No. 5 contains the operating data of this filter.

TABLE No. 6.—Operation data for mechanical filter during period 1.

	Sched- uled rate			Volume	Loss of h	ead feet.	
Month.	in million gallons, per acre per 24 hours.	Number of runs, daily average.	A verage duration of run, in hours.	treated, million gallons, per acre per day.	Initial.	Final.	Per cent of wash water.
January February March April May June June	39 50-	$1.21 \\ 1.27 \\ 2.72 \\ 1.5 \\ 1.16 \\ 1.16 \\ 1.89$	3.8 3.4 1.0 1.0 3.0 3.4 2.9	$\begin{array}{r} 2.30\\ 3.42\\ 4.42\\ 3.12\\ 3.63\\ 4.12\\ 5.72\end{array}$	0.67 .67 .67 .67 .67 .67 .67	10.98 10.98 11.37 7.71 8.57 6.99	161 176 110 80 55 55 71
Average	28	1.56	2.6	3.82	. 67	9.43	101

[Jan. 1 to July 31, 1915.]

The average rate was 28,000,000 gallons per acre per 24 hours, which, however, varied from 12,000,000 to 50,000,000. As it was impossible to operate continuously, the actual volume treated was but 3.82 million. Throughout the whole period the filter clogged very rapidly, requiring frequent washings, and, as a consequence, operated on an average but four hours a day.

After each wash or at the beginning of each run the initial loss of head was 0.67 foot. The maximum loss attainable was 12.2 feet, but this was seldom obtained, as other factors necessitated washing before that head was reached. Frequently the rate fell below that required, but more frequently the sand broke, leaving cracks through which the waste flowed without any treatment, requiring that the filter be shut down and washed.

The most noticeable item in the table, however, is the amount of wash water used. The filter was, as a rule, washed at a rate of 1 vertical foot per minute for a period of 5 to 7 minutes. Even the minimum used was 52 per cent, while the average was 101 per cent.

These computations are based on the volume of waste filtered as measured at the outlet of the filter. There is, however, a large correction which must be applied when filters of this type are washed at frequent intervals. Owing to the shortness of the run the clean wash water left in the filter is a considerable proportion of the effluent during the next run. It is estimated that this amounts to about 36 per cent on the basis of these average figures. That is, the actual net yield of the filter is 36 per cent less and the percentage of wash water, based upon net performance, correspondingly greater.

The results of chemical and biological analyses made of the influent and effluent of this filter are contained in Table No. 7.

TABLE NO.	7Monthly	average	analyses	of	influent	and	efluent	of	mechanical
		filt	er during	pe	riod 1.				

and the second second	(effl	uent uent k 4). \	Effluent.									
THE REAL			1. No. 1	Suspended solids.				Putres	hours			
Month.	as pe	Sus- pended solids.	Alka- linity <sup>1</sup> as CaCO <sub>3</sub> ,		Per cent removal.		Oxy- gen de- mand, 24	to reduce M. B at room tempera ture. <sup>2</sup>				
		sonus.	total.	Total.	Over influ- ent.	Over raw.	hours.	Dil. 1:3.	Dil. 1:5,	Dil. 1:10,		
March. April May fune	460 320 320 360	740 890 790 560	370 300 310 360	230 220 310 190	-69 75 61 66	87 89 85 84	75 90 80 100	35 20 45 40	55 35 65 50	88 88 88 88		
fuly A verage	370	530	380	160	- 70 68	86	100	35	50	8		

[Jan. 1 to July 31, 1915. Results in parts per million.]

<sup>1</sup> Analyses made on filtered samples

<sup>2</sup> Maximum incubation period, 96 hours.

While this table shows an apparent reduction in suspended solids of 68 and 87 per cent of the influent and raw waste, respectively, an oxygen demand for 24 hours of 89 p. p. m., and a stability of 35 hours with a 1:3 dilution, it must be noted that the same error, due to the wash water retained in the filter, enters into these results, as the samples were composites covering the whole operating period. They contain, therefore, about 36 per cent of wash or diluting water.

Applying this correction, the suspended solids in the undiluted effluent would increase about 55 per cent to 344 p. p. m., giving the reduced removals of 50 and 79 per cent for the influent and raw waste, respectively. The oxygen demand would increase to 139 p. p. m. and the corrected dilution for the putrescibility results would be 1:5.3 instead of 1:3.

When the filter was dismantled there was about 1 inch of sand at the surface that contained a large amount of material resembling clay, and below this the sand was found to be clean and free of foreign matter. The grading of the material had remained practically the same as originally placed except that some sand had worked down into the top layer of finer gravel. The screens separating the two gravel layers and the sand from the gravel were undisturbed, which was true also of the strainer plate. Many of the holes in this plate were found to be filled with a muddy deposit consisting of iron rust and solids from the waste, which also collected on the under side of the plate around the holes. The drain space was about onehalf filled with sand that worked down through the joints or around the outer edge of the strainer plate, which did not fit closely enough to the side of the tank. The filling of a part of the space under the strainer plate with sand does not affect the computations for the correction of the wash water used and had no effect on the operation of the filter. The reduction in the volume of water retained in the underdrains would be compensated by the increase in the voids in the sand or in the depth of water standing on the surface of the filter due to the settling of the sand away from the wash-water drain above the filter.

At best, however, it is apparent that the results of mechanical filtration can not improve the character of the waste beyond the point indicated by the removal of suspended matter, and in view of the high oxygen demand values of the waste under investigation it appears that biological treatment for the oxidation of organic matter may be demanded in many cases.

### PERIOD 2.

#### BIOLOGICAL TREATMENT.

The high oxygen demand results for the filtered waste obtained from the operation of the testing station during period 1 suggested that further studies should be made to develop a process that would give a final effluent suitable for discharge into a stream with a relatively low dilution. A slow sand filter and a small cinder filter were, therefore, constructed on a small experimental scale to treat the settled waste. These were put into operation August 1, 1915. This date was the beginning of period 2, which continued to the end of the tests, June 30, 1916.

On the basis of results obtained from the small cinder filter the plant was remodeled. Tanks Nos. 3 and 4 were converted into trickling filters (filters Nos. 1 and 2) with stone as media, and the mechanical filter and tank No. 5 into trickling filters (filters Nos. 3 and 4) with cinders as media. Over filter No. 4, contained in tank No. 5, there was placed a small gravel roughing filter. These filters have already been described in detail. (See pp. 29 to 34.)

Sedimentation.—Tanks Nos. 1 and 2 were retained as settling tanks of the Dortmund type and were operated in parallel as before, followed during August, September, and October by tanks Nos. 3 and 4 operating as settling tanks, and the smaller cinder and slow sand filters receiving as influent the effluent of tank No. 4. When the plant was rebuilt in October with the larger cinder and stone filters, tanks Nos. 1 and 2 were converted into Imhoff tanks and continued to be operated in parallel through February. After that month a Dortmund dosing tube was installed in tank No. 1, and the two tanks were operated independently, the effluent from tank No. 1 being applied to the small cinder filter, with excess wasted, and that from tank No. 2 going to the remaining filters. From this tank also there was an excess of effluent which was wasted.

Table No. 8 contains the monthly average volume of raw waste together with the volume treated in the tanks.

TABLE No. 8.—Monthly average volumes of waste treated in tanks during period 2.

nitorpodition niti do minine do minine	Total	Was treat		and par infl	nks 1 1 2 in rallel uent, waste.	infl	nk 1 uent, waste.	infl	ank 2 luent, waste.	1.10	uent, ta 2 in pa	anks	l and
Month,	waste, gallons per 24 hours.	Gallons per day.	Per cent of total.	Settling period, hours each tank.	Gallons per day, each tank.	Settling period, hours.	Gallons per day.	Settling period, hours.	Gallons per day.	Settling period, hours,	Gallons per day.	Settling period, hours.	Gallons per day.
August September October November December January February March April May June Average	$\begin{array}{c} 1, 100, 000\\ 1, 050, 000\\ 1, 130, 000\\ 1, 040, 000\\ 1, 070, 000\\ 960, 000\\ 980, 000\\ 980, 000\\ 950, 000\\ 1, 150, 000\\ 1, 150, 000\\ 1, 300, 000\\ 1, 450, 000\\ 1, 110, 000\\ \end{array}$	16,500 13,900 6,800 5,100 4,800 4,700 7,100 10,200 9,200 10,600 10,000 9,000	1.5 1.3 .6 .5 .4 .5 .7 1.1 .8 .8 .7 .8	2.9 4.0 3.0 3.0 3.0 	3, 400 2, 550 2, 400 2, 350 3, 550	3.0 3.0 3.0 3.0 3.0	4,600 5,300 5,000	3.0 3.0 3.0	4,600 5,300 5,000	. 59	16,500 18,700		16,500

[Aug	. 1, 1	915,	to Ju	ne 30,	1916.]
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1 Tanks 3 and 4 discontinued Sept. 11, 1915.

During period 2 only about two-thirds as much waste was treated as during period 1. It amounted to 0.8 per cent of the total waste from the mill, and the period of detention in tanks Nos. 1 and 2 was increased from 2.1 to 3 hours. As, however, tanks Nos. 3 and 4 were operated during only the first two and one-half months, the total settling period was slightly reduced.

The total volume of waste was above 1,000,000 gallons, except for the first three months of 1916. During this time one of the machines was running mostly on chip board, thereby eliminating beater waste for the stock for that machine. Different stocks produced different volumes of waste, but for the same stock the waste, as a rule, varied with the amount of product.

The amount treated varied from 16,500 and 13,900 gallons per day during August and September, before the plant was remodeled, to as low as 4,700 after remodeling, and later to an average of 10,000 gallons, when the station was operated 16 hours per day. These amounts were determined by the storage period desired in the tanks, and variations for the same storage period were due to abnormal operation, such as closing the plant down for making repairs or alterations.

Because of the limitations of the field laboratory at Noblesville, samples were sent to the central laboratory at Cincinnati after December 1. It was possible in that way to obtain much more complete analyses during this period than during period 1.

In cases where the same determinations were made at both laboratories the results given are for the average. In the case of the suspended solids determinations it should be noted that differences of 10 per cent or more in the monthly average results of analyses made at the two laboratories were exceptional. The alkalinity results showed variations as much as 25 per cent, which is to be expected, considering the color in the waste and the difficulty in obtaining the true end point in the titrations. No other determinations were made at both laboratories, as the oxygen demand and putrescibility determinations could be made only at Noblesville and the nitrogens and oxygen consumed only at Cincinnati.

The monthly average analyses of the raw waste and tank effluents are included in table No. 9.

TABLE No. 9.—Monthly average analyses of raw waste and tank effluents during period 2.

RAW WASTE. [Results in parts per million.] Alkalinity as Suspended solids. Putrescibility. Oxy-CaCO<sub>2</sub>. gen Oxydegen mands, Per cent re-Nitro-Hours con-24 gen total. Month. moval. to Red. M. B. hours. sumed. Car-Bicar-30' 96' room Dil. Total. bonbon-C. room tem-Over ate. ate. pera-ture. tem-Over influpera-ture.1 raw. ent. 1,490 115 615 August .... September ..... 1,000 20 500 .... .... October ..... 710 0 350 ... 2,0802,2001,250205 November .....  $780 \\ 590$ 45 23 830 +96 25 1:25 December ..... 280 26 560 50 1:25 91 409 +96 January .... 320 120 610 20 1:10 -96 18 488 February ..... 1. March..... 250 30 203 520 1,200 25 65 1:5 29 860 230 35 1:5 42 38 2, 080 164 743 April..... 1:5 May..... 1 45 3 330 42 197 804 30 1,090 2,220 775 60 June ..... 34 158 910 29 141 598 40 Average ..... 1,810 EFFLUENT TANKS 1 AND 2 IN PARALLEL (INFLUENT, RAW WASTE). August ..... 710 0 700 Õ 600 520 300 September ..... 48 .... 58 Ö 600 October ..... ...... 970 53 100 635 60 November ..... 1:25 1:20 +9624 650 50 200 515 110 1,110 December ..... 530 58 60 400 50 +96January..... February..... 18 500 1:10 30 455 +96 660 50 80 53 21 63 558 575 62 680 Average .....

<sup>1</sup> Maximum incubation period, 96 hours.

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[Aug. 1, 1915, to June 30, 1916.]

TABLE No. 9Monthly	werage analyses of raw wa	ste and tank effluents duri	ng
	period 2—Continued.		

PROBAT TOTAL	Susp	ended so	lids.	inthe	Alkalinity as CaCO <sup>3</sup> .		ods a	Oxy-	Putress	eibility.
Month.	Total.		Per cent re- moval.		Car-	Bicar-	Oxy- gen con- sumed,	gen de- mands, 24 hours, room		Hours to Red. M. B.
		Over influ- ent.	Over raw.	total.	bon- ate.	bon- ate.	30′ 96° C.	tem- pera- ture.	Dil.	tem- pera- ture.
Mareh. April. May June.	1,220 1,090 1,280 1,340		46     47     62     43	27 28 30 30	165 125 115 135	445 525 675 670	680 780 840	45 90 65 75	1:10 1:10 1:10 1:10	78 45 45 45
Average	1,230		50	29	132	579	767	70		

EFFLUENT TANK 1 (INFLUENT, RAW WASTE).

EFFLUENT TANK 2 (INFLUE, RAW WASTE).

March April May June	1,070 1,200		22 27 32 31	170 125 120 120	400 570 615 700	$^{1,160}_{\begin{array}{c}670\\760\\810\end{array}}$	40 70 55 70	1:10 1:10 1:10 1:10	72 66 50 42
Average	1,185	 52	28	134	571	850	60		

EFFLUENT TANK 3 (INFLUENT, TANKS 1 AND 2).

August	$710 \\ 580$	0 11	$52 \\ 42$		720 660		
Average	645	- 6	48	 18	690	 	 

EFFLUENT TANK 4 (INFLUENT, TANKS 1 AND 2).

August September		· 17 17					
Average	535	17	57	 10	650	 	 

These results are very nearly the same as those obtained for the corresponding constituents during period 1. The suspended solids in the raw waste are slightly higher in period 2, but the difference is less than 10 per cent. The alkalinity as shown is over twice that given for period 1, but this is due to the fact that the determinations were made on samples filtered through a filter paper in period 1 while they were made on the unfiltered sample during period 2. The difference between the alkalinity values during the two periods was 440 p. p. m., equivalent to 3,670 pounds of alkalinity per million gallons of waste. This alkalinity was doubtless in the form of calcium carbonate in suspension and was therefore removed by filtration. The variations of the alkalinity in the table are attributed to the different grades of straw and the corresponding variation in the amount of lime used in the digesters during the time covered by the samples,

The efficiency of the tanks in removing suspended solids checks with the results obtained during period 1. The average for the period when tanks No. 1 and 2 were in parallel was 48 per cent as compared with 53 per cent during the seven months when operating conditions were the same, and after the two tanks were operated independently the reductions were 50 and 52 per cent for tanks No. 1 and 2, respectively.

In other respects the effluent from the tanks is only slightly different from the influent. The nitrogen content is, within sampling errors, almost identical with that in the raw waste for corresponding months.

That the suspended solids removed in the tanks were largely mineral matter and cellulose is evidenced by the comparatively low reduction in the oxygen consumed value coincident with a removal of 50 per cent of the suspended solids.

Reduction of the organic matter in the waste during tank treatment with the producton of less stable compounds is to be seen in the 24-hour oxygen demand results which are about twice as high in the tank effluents as in the raw waste. This is further indicated in the putrescibility results where a 1:10 dilution of tank effluents resulted in but a very short prolongation of the period of color retention over the raw waste when diluted 1:5 or one-half as much.

There is no noticeable increase in efficiency in either Tanks 1 or 2 after November when they were converted from Dortmund to Imhoff tanks. Nor does tank No. 2 show any different results following the change back to the Dortmund type in March.

The fact that the reduction in suspended solids was almost as great for tanks 1 and 2 during period 1 when the detention period was but 2.1 hours as it was during period 2 with a 3-hour period, is confirmation of the statement previously made that the economically settleable solids were removed in two hours. The results from tanks No. 3 and 4 also bear this out. Tank No. 3, owing to septic action accompanied by rising of the sludge, showed a zero or negative removal. Tank No. 4 effected a slight additional removal over the two primary tanks, but it was too small to be of practical value. These two tanks together added 1.7 hours to the detention period of the first two tanks.

The results of tank treatment were, the removal of roughly 50 per cent of the suspended solids of which the bulk was mineral matter and cellulose, and the preliminary decomposition of the organic matter thereby rendering the effluent less stable though clarified.

Filtration.—The effluent from the settling tanks was treated by filters of various types. During July, August, and September two small test units were operated, one made of sand and the other of cinders. On the basis of results obtained from these filters, four large units were installed, two of stone and two of cinders, which were operated as trickling filters. Preliminary to one of the cinder filters, a small roughing filter was installed. These filters have already been described in detail.

The operating data of these filters are contained in table No. 10.

TABLE No. 10.—Average rates of operation of filters during period 2, in thousand gallons per acre per day.

Filter.	Small cinder.	Sand.	No. 1.	No. 2.	No. 3.	Gravel rough- ing.	No. 4.	Gravel ing fi	
wal glaviterasjans	Rates	of opera	tion. T	housand	gallons	per acre pe	r day.	Length	Wash water,
Receiving effluent from-	Tank 4 (1).	Tank 4.	Tanks 1–2 (²).	Tanks 1-2 (2).	Tanks 1–2 (²).	Tanks 1-2 (²).	Rough- ing.	of run, hours.	per cent.
August September October	209 197 204	69 52							
November December	185 200 185		175 175 175	175 175 175	175 175 175	1,978 1,978 1,978	175 175 175	39 24	4.5 10.1
January February March	218 168		112 181 170	112 181 170	112 181 170	1, 970 1, 271 2, 050 1, 920	112 181 170	66 75 71	5.5 5.0 5.3
April. May. June	$     \begin{array}{r}       172 \\       381 \\       365     \end{array} $		388 367	388 367	388 367	4,380	388 367	36	5.4
Average	226	60	218	218	218	2,222	218	52	6.0

[Aug. 1, 1915, to June 30, 1916.]

<sup>1</sup> Changed to effluent tank 1, November, 1915. <sup>2</sup> Changed to effluent tank 2 alone, March, 1916.

As the testing plant was operated only 8 hours a day up to February, the schedule for the filters was arranged to give the full dosage in that time. There was, therefore, a 16-hour rest period during each day. In February, 1916, arrangements were made to prolong the dosing period to 16 hours, with 8 hours' rest. The same daily rate was maintained but the time of application doubled. As shown in the table, the rate was doubled during May and June, but the dosage period of 16 hours maintained as before.

During August, September, and October the operation of the small cinder filter was quite satisfactory, while that of the slow sand was very unsatisfactory. The surface of the sand became clogged very quickly after the waste was applied and, within slightly over a week of operation, the waste, which was standing over the filter at night when the plant was shut down, failed to filter through by the next morning. The surface of the filter became coated with an almost impervious mud layer, the effluent came through colored, and the rate was so reduced by August 19 that it was necessary to discontinue the filter operations. The mud layer dried and was removed August 31, and the filter was put into operation again on September 1. At the very start the effluent was somewhat turbid. While there was some improvement later, the general results were so unfavorable and the rate so low that the impracticability of filters of this type was appreciated and the tests were discontinued September 11.

The favorable results obtained from the small cinder filter served as a basis for remodeling the plant in October. Sand filters had been eliminated principally on account of the surface clogging, but there was no evidence that material coarser than cinders would not give as satisfactory results as cinders, and the necessity of screening the cinders had not been determined. Accordingly four large filters, already described, were constructed, one filled with limestone, average size 1½ inches; one with limestone screenings, one-fourth-inch size; one with cinders unscreened; and one with cinders screened over a one-fourth-inch mesh screen.

From November to January, inclusive, the dosage period was 8 hours out of the 24, during which time the normal rate of application was 200,000 gallons per acre per day. Unavoidable interruptions, such as the closing down of the plant or the mill for repairs, cut these rates down as shown in Table No. 10. In February the period of operation, nominally changed to 16 hours, averaged in actual fact only 9 hours. In May and June, with scheduled rates of 400,000 per 16-hour day, the actual daily net rates for all the filters except the small cinder filter were 388,000 and 367,000 gallons, respectively. The net rates on the small cinder filter for the two months were 381,000 and 365,000 gallons, respectively.

The rate on the gravel roughing filter was controlled by the rate on filter No. 4, as this filter took all the effluent from the roughing filter. The periods of dosage were the same and the scheduled rates figured 2,260,000 and 4,530,000 gallons, respectively, for the 200,000 and 400,000 gallon rates of filter No. 4. The actual net rates were lower, owing, as in the case of the other filters, to unavoidable periods of idleness which reduced the net rates below those scheduled.

The coarse material of which the filter was composed made much longer runs possible than in the larger mechanical filter operated during period 1. On an average during the six months of operation the period between washings was 52 hours. These long runs had their effect on the wash water, which averaged but 6 per cent of the volume treated, including the figure of 10.1 per cent used in January, and 5.1 per cent omitting January.

The results accomplished by these filters are contained in Table No. 11.

TABLE No. 11.-Monthly average analyses of influents and effluents of filters during period 2.

[Aug. 1, 1915, to June 30, 1916.]

SLOW SAND FILTER.

Results in parts per million except those for putrescibility.

	Putresci- bility. <sup>2</sup>	e M. B.	Hours to reduc at room tempe	30	09	1000
	Putr	11 201	Dilution.	00	0	1 12
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TABLE No. 11.-Monthly average analyses of influents and effluents of filters during period 2-Continued. GRAVEL ROUGHING FILTER.

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	Putresci- bility.2	e M. B.	Hours to reduc				8888258888
			Dilution.				2222222222222
	ne. hours,	ind, 24	Oxygen dema				8 II 2 4 1 2 2 2 3 3 8
-	-aim (	96° C, 3	oxygen consu	009 900	630		150 150 155 155 155
	Alkalinity as CaCO <sub>2</sub> .		Bicarbonates.	585 390 520 520 520 520 520 520 520 520 520 52	585		410 220 230 230 230 230 250 250 250 250 250 250 250 250 250 25
and and	Alka as Ce		Carbonates.	140 160 150 150 85 85	115		0.640886689
Effluent.	13-		Nitrates.				0.2
EM	Nitrogen as-		Nitrites.				$\begin{array}{c} 0.016\\ 0.016\\ 0.008\\ 0.021\\ 0.026\\ 0.056\\ 0.056\end{array}$
	Nitr		Total.	26.0 30.0	28.0		6.8 5.6 10.0 10.1 6.8
	olids.	Per cent removal.	Over raw.	1832588	62		88888888888
	Suspended solids.	Per	.tasufini 1970	- 10 83 88 88 88 88 88 88 88 88 88 88 88 88	18	RS.	2222222222
	Suspe		Total.	920 920 920 920	782	CINDERS.	155 145 100 100 155 130 125 125
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-	Put		Dilution.	1100 1100 1100 1100		SCREENED	1:10
	e. ponts,	nperatur and, 24	CXYgen demi	8588855	22	4, SC	37
	-uju 0	96° C. 3	essin uses utes,	650 1,160 690	734	TER	810 810
	Alkalinity as CaCO <sub>3</sub> .		Bicarbonates.	635 515 400 455 400 570 675	504	FII	585 3900 580 580 580 580 580 580 580 580 580 5
Influent.	Alka as Ci		Carbonates.	100 2000 100 1125 110	121	•	140 160 150 150 120
II		ı.	Nitrogen, tota	24 18 22 26 26	24		2888
	T	ids, tota	los bebneqsus	1, 110 1, 110 1, 130 1, 110 1, 110 1, 110	940		$     \begin{array}{c}             980 \\             740 \\             740 \\             550 \\             956 \\             840 \\             840 \\             1,340 \\         $
merender saft		Source.		Tanks 1 and 2. do do Tank 2. do Tank 2.			Gravel roughing filter to May 13 <sup>1</sup> . do do do (1). do
		Month, 1915-16.		November. December. January. February. March. April.	Average		November. December. January. February. March. April. April. May 14-31. June.

<sup>1</sup> After May 13, tank 2.

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Average......

The results given in table No. 11 show in condensed form the average analyses by months for the entire time covered by Period Two.

There were only a comparatively few analyses made of the effluent from the slow sand filter during the two months of operation, as the operation difficulties became apparent from the first and it was evident that beds of this type could not be operated without excessive attention. The results given show about the same amount of removal for suspended matter as was obtained by the cinder filter for the same period, while the alkalinity, all in the bicarbonate form, is considerably higher than the values given for the cinder filter effluent. The number of hours required to reduce the methylene blue in the samples was less than that for the small cinder filter for the same period covered by both filters.

The monthly average results of the analyses of samples from the small cinder filter are fairly uniform for the entire period of 11 months. The suspended matter after the first month was low until March, when, with the exception of April, it increased with that in the influent. It should be noted, however, that even with the higher values in May and June, oxidation, as indicated by the nitrates, was at its maximum. That this improvement was possible, in spite of the increase of rate during these two months from 200,000 to 400,000 gallons per acre per day, is strong evidence of increasing efficiency of the filter. The relative removal for the suspended matter, when referred to either the influent or the raw waste, is uniform for the monthly averages, and the average for the 11 months is probably what can be expected in the normal operation of a treatment plant.

The alkalinity results in the form of carbonate and bicarbonate show considerable variations in the filter effluent from that in the influent. The reason for the decrease of the carbonates was that the light flocs of calcium carbonate, resulting from digesting the straw with lime, did not settle out in the tanks, but were removed by the filters, but it is not evident why the bicarbonates were reduced by the filters. It is possible that the color in the waste from the settling tanks masked the end point, thereby giving too high values, but even this explanation will not hold for the last two months of the tests, for the alkalinity increased in the filter effluent at this time, with the color remaining the same as during the earlier period. This subject will be given further study in the operation of the small permanent unit to be constructed.

The oxygen-consumed values are rather high for an effluent showing such a satisfactory degree of nitrification. The only explanation that can be given is that there is present in strawboard waste a large amount of cellulose material, part of which is very stable and is not readily broken down, except by the action of strong chemicals such as sulphuric acid in the nitrogen determination or permanganate in the hot acid solution in the oxygen-consumed determination.

As it was impossible to use an incubator because of the lack of heat other than an oil lamp, which fire risk prohibited, the putrescibility results are recorded as the number of hours to reduce methylene blue at room temperature, instead of relative stability numbers. During the winter months this temperature was about 70° F. and in the summer about the same as the air temperature. Because of limited equipment samples were held but 4 days, 96 hours.

In but very few instances was methylene blue reduced in less than 90 hours, and even then there was no odor of hydrogen sulphide present. This fact and the presence of nitrates indicates that the organic matter was in a stable condition, and that by reason of the character of the suspended solids less significance attaches to their presence in this waste than in a sewage effluent. The oxygen demand values for 24 hours were low throughout the entire period, varying from 7 to 23 p. p. m., and averaging 12. The dissolved oxygen in the samples was, on an average, 5 p. p. m.

Tests to be described later under filter No. 4, show that fish could live in the effluent of this filter with a dilution of 1:1.

Though the filter gave no evidence of becoming clogged, it was washed on May 13, to ascertain to what extent solids were being retained. In washing the filter by applying water through the effluent pipe but very little material was removed, and when the water was applied to the surface, after the first flush which washed the underdrains and outlet pipe, the water passed through practically clear. The deposits that were removed were black and smelled like loam, and were fairly alive with worms, small bugs, and other organisms.

This loamy deposit was found to a depth of 2 feet when the filter was finally dismantled. It appeared to have been thoroughly worked over into humus by the worms within the filter and was well oxidized. From the character and amount of this material it seems safe to conclude that the filter would have maintained the high quality of its effluent for some time. It, doubtless, would have deteriorated eventually, but a period of good service for two years can reasonably be expected between washings, and the washing can be readily and economically accomplished by playing a 2-inch hose over the surface.

The small cinder filter was operated three months longer than any of the other filters, and more significance is attached to these results than to any of the others. Briefly stated, the important features are: (1) The suspended matter in the influent was reduced 91 per cent by the filter, and the 80 p. p. m. remaining in the effluent did not have any material effect on the oxygen demand or putrescibility; (2) nitrification was active throughout the tests notwithstanding the large amount of suspended matter removed by the filter; (3) the oxygen demand in 24 hours was very low, and on the average did not exceed the average amount of oxygen present; (4) the putrescibility samples were as a rule stable for more than 90 hours with a dilution of 1:3 and without dilution for more than 50 hours.

The results obtained from treating settled waste on limestone screenings were not as satisfactory as those obtained from cinders. The influent was the same, but the material of the filter was too coarse and the voids so open that the suspended matter readily passed through. The organic nitrogen was considerably higher for the entire period and nitrification generally much lower. The alkalinity results show the same variation that was found in the small cinder filter, but the oxygen consumed and oxygen demand values were two and three times higher, respectively, in this filter, and the putrescibility test upon samples diluted 1:5 gave an average stability of less than 76 hours.

The condition of this filter when dismantled at the close of the tests, and when examined during the progress of the tests, was not satisfactory. The material retained by the filter had some odor, there was a slimy growth on the stones, and there were no worms or higher organisms such as were found so abundantly in the screened cinder filters. When this filter was washed the solids removed were stringy, yellowish in color, and highly putrescible on incubation, as indicated by odors.

The results obtained by treating the settled waste on limestone  $1\frac{1}{2}$  inches in diameter were even less satisfactory than those from the stone screenings. This material was entirely too large, as indicated by the low removal of suspended matter, the high oxygen demand, and the low stability figures. There were no growths of worms and it was felt that even if they should develop the solids would not be retained long enough to permit any action by them.

The effluent from cinder filter No. 3 was considerably better than the filtered waste from the limestone screenings or the coarse limestone, but it was not quite so satisfactory as that from the small cinder filter. The cinders used in this filter were unscreened, and as a result of the fine material present considerable pooling took place on the surface, which no doubt interfered with the proper operation of the bed. Compared with the small cinder filter, the suspended matter was on an average 35 p. p. m higher, the organic nitrogen 1.9 p. p. m. higher, the oxygen consumed 23 p. p. m. higher, the nitrates 0.82 p. p. m. lower, the oxygen demand 7 p. p. m. higher, and the relative stability lower for the last three months of operation. With dilutions of 1:3 this effluent at room temperature was stable, on the average, for 86 hours.

The analyses of the effluent from this filter show very clearly the effects of using unscreened cinders for filtering the waste. Pooling on the surface and freezing prevented the proper aeration, and as a result the solids stored in the voids of the bed were not reduced to humus to the same extent as occurred in the washed cinders in the small cinder filter and the screened cinders in filter No. 4.

When this filter was washed the material removed more nearly resembled that from the beds filled with stone, and there was a notable absence of the worms and other organisms so abundant in the other two cinder filters.

The gravel roughing filter, already described, was placed immediately over filter No. 4 for the purpose of removing a part of the suspended matter and to act as a colloider in gathering together the fine particles which constituted the major portion of the suspended matter in the effluent of the settling tanks. The results obtained, however, proved that such treatment was not practical. Only 19 per cent of the applied suspended matter was removed for the entire period, the total organic nitrogen was not reduced, and the oxygen consumed was only reduced 16 per cent.

The results obtained from the operation of filter No. 4, screened cinders, were practically the same as those obtained from the small cinder filter. The suspended matter was 5 per cent higher, the organic matter 1.4 p. p. m. lower, the nitrates 0.5 p. p. m. lower, the oxygen consumed 13 p. p. m. lower, the oxygen demand 4 p. p. m. higher, and samples diluted 1:3 were stable for 89 hours in the average for the entire period covered by the test.

There was no pooling on the surface of this filter, and the solids removed by washing were more of the nature of humus, and the bed was alive with worms, bugs, and other smaller water organisms. When dismantled there was considerable deposit in the upper half of the bed, but the analyses would indicate that this deposit was thoroughly worked over in the lower half and was not interfering with the oxidation processes.

Studies were made with the effluent of this filter to determine its effect upon fish life. On April 13, 1916, a box was constructed  $3\frac{1}{2}$ feet long by  $2\frac{1}{2}$  feet wide by 1 foot deep and placed just beneath the outlet of the filter. All the waste from the filter flowed through the box, and in addition river water from the mill supply was added. Twenty minnows were placed in the box, and in a dilution of one part purified waste to three parts of water they lived without being affected in any way. On April 24 the ratio of the purified waste to the river water was reduced to 1:2 and this mixture was maintained until May 8, when the dilution was again changed to 1:1. Up to this time all the fish were alive and as active as when placed in the box. The dilution of one part filter effluent to one part of river water was maintained from May 8 to July 1, and during this time one minnow died, the others remaining as active as when taken from the river.

It is quite possible the fish would have lived in the filtered effluent without dilution, but this was not attempted, because the testing station was operating only 16 hours per day during the week and was closed down on Sundays. In streams or rivers receiving the waste it is reasonable to expect a dilution of at least 1:1, and a continuous flow, so that the tests outlined above approximated as nearly as possible the most unfavorable conditions that could exist in a river. Practically no food was given the minnows in the 78 days of the test except the minute organisms that were naturally found in abundance in the filtered waste.

Reviewing the results in the table as a whole it is obvious that filters filled with coarse limestone or limestone screenings will not produce a satisfactory effluent, and further that a roughing filter preceding filtration through cinder filters is not practical. Of the three cinder filters operated two gave satisfactory results, while the third. made of unscreened cinders, pooled badly on the surface and gave less satisfactory chemical results. The small cinder filter was composed of unscreened cinders, but they were washed to remove the very fine material, so that this filter can be more properly described as a screened cinder filter. The large screened cinder bed No. 4 gave a satisfactory effluent throughout the eight months of operation, the effluent having less total organic nitrogen, more nitrates, and a less oxygen demand in the average for the last month than the grand average for the entire period of operation. This shows definitely that the bed was in good working condition when the tests were discontinued, and it is upon the results obtained from the operation of this filter, together with those from the small cinder filter, that recommendations are made for designing a complete plant for filtering settled strawboard waste.

Treatment with activated sludge.—In order to study the effects of aerating the waste mixed with activated sewage sludge, two glass tubes, each 1 inch in diameter (inside) and 4 feet long, were set up and cylinders of filtros inserted at the bottom and cemented to make them water-tight.

In tube No. 1 activated sludge, from the Cleveland test unit, diluted with tap water, was placed and aerated to serve as a control and as a source of sludge for starting the tests using the waste. In the second tube activated sludge from tube No. 1, after aeration, and raw strawboard waste were placed in the ratio of 20 per cent sludge to 80 per cent waste. No attempt was made to measure the volume of air drawn through the tubes by means of a Richards filter pump, but the suction was regulated to maintain a sufficient ebullition of bubbles to keep the sludge well distributed throughout the length of the tubes. The amount of air varied on account of the varying water pressure, but, under the lowest pressure occurring during the daytime, the suction was arranged to maintain thorough aeration.

The object of these studies was to determine whether the higher organisms found in sewage activated sludge would live and multiply in strawboard waste aerated with sewage activated sludge, and whether results could be obtained similar to those obtained in treating sewage by this process, with particular reference to the removal of color. There was some question whether the high carbonate and bicarbonate alkalinity of the waste would interfere with the ordinary processes developed in activated sewage, and it was assumed that the behavior of these higher organisms in aerated waste would be an index of the effect the unusual character of the waste would have on the development of the process.

Samples of 1 c. c. were taken from each tube before the sludge settled to the bottom and diluted so that the number in the field of the microscope could be accurately determined. Duplicate counts were made of all samples. Where only one organism was found in the dilution a plus sign was recorded, but when two or more were found the dilution factor was multiplied by the number found to get the total number per c. c.

In addition to the record of the organisms, careful notations were made of the color and putrescibility, and oxygen demand determinations were made from time to time.

The following table gives the class and the number of organisms found in the tubes in which the activated sewage sludge and the waste were aerated. In columns marked "No. 1" are given the organisms in the activated sewage sludge, and in "No. 2" those in the wastes aerated with 20 per cent of the sludge. TABLE No. 12.—Table showing class and number of organisms in activated sewage sludge, column 1, and strawboard waste aerated with 20 per cent of activated sewage sludge, column 2.

1	tion.	C. c. raw wastes added.	63	250 250 390 390 390 390 390 390 390 390 390 39
	Operation.	C. c. super- natant removed.	2	250 250 250 250 250 250 250 250 250 250
100	1998	lea dard s.1	5	$\begin{array}{c} 7,600\\ 7,600\\ 20,000\\ 20,000\\ 20,000\\ 21,600\\ 337,600\\ 337,600\\ 337,600\\ 11,800\\ 11,800\\ 11,800\\ 11,200$
		Zooglea in standard units.1	1	$+ \\+ \\55,000 \\56,000 \\70,000 \\70,000 \\140,000 \\140,000 \\140,000 \\140,000 \\140,000 \\20,000 \\20,000 \\20,000 \\10$
	100	Molds.	5	
	0.23	Mo	1	+ + + ++
	12	fers.	63	$\overset{400}{\overset{12}{}}_{+++}^{+}$
		Rotifers.	1	++ 500000000000000000000000000000000000
	10	Euplotes.	3	\$00 400
	Organisms, number per c. c.		1	3,000 2,000 2,000 2,000
		richa, cella, ylis, sium, c.	63	$\begin{array}{c} \begin{array}{c} & & & & \\ & & & & \\ & & & & \\ & & & & $
	isms, nu	Peritricha, vorticella, epistylis, carchestum, etc.	1	Few. Few. 30,0000 30,0000 30,0000 30,0000 30,0000 30,0000 20,0000 22,0000 22,0000 22,0000 22,0000 10,00000 10,00000 10,00000000
	Organ	a and gia.	5	800 800 400 400 400 800 800
	11.0	Arcella and diffugia.	1	$++\begin{array}{c} 0.00000000000000000000000000000000000$
	-	otus.	61	800 12,800 12,800 2,400 12,800 2,400 2,400 2,400 2,400
		Lionotus.	1	2,000 6,000 6,000
19		ites tified.	13	28,000 1
		Cillates unidentified.	1	++ 20,000 20,000 2,000 2,000 2,000 2,000 60 60
		Date, 1916.		February 17. 18 19 23 24 23 24 28 28 28 28 28 28 28 28 28 28

<sup>1</sup> A standard unit is a square, .02 mm. on a side.

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In this table it will be observed that the higher organisms did not appear in the activated sludge from Cleveland until the third day of aeration, and it was not until the third day that the sludge settled to the bottom of the tube with a clear supernatant. The different classes of organisms given are only those that were found relatively abundant at some time during the test. Minute worms were found on three occasions but not more than one in any one count, so it was assumed they did not grow to any extent, and they are not recorded. Other organisms were found occasionally, but they are not included, as they did not appear on consecutive days or in numbers sufficient to warrant any connection with the purification.

Unidentified *Ciliates* were present in the original sludge in numbers too small to count but showed a sudden increase to 18,000 after the third day of aeration. The second day after tube No. 2, containing the strawboard waste, was started they were present in large numbers. After the third day the number was greatly reduced, at times being absent altogether, but they appeared more frequently and in greater numbers in tube No. 2 than in control tube No. 1.

The organisms identified as *Lionotus* were evidently not present in the original sludge but were introduced with the sewage added to the control tube on March 3. At the end of three days they disappeared and were not found later. Evidently this organism is present in strawboard waste as it appeared in tube No. 2 much earlier than in tube No. 1 and continued to be present throughout the tests.

Arcella and Difflugia were present in the original activated sludge but only in small numbers until after the fourth day of aeration when they showed a count of 10,000. After that they were present continuously in numbers ranging from 2,000 to 8,000. They were present in very much reduced numbers in the tube containing the waste, and with the exception of one day when there was a count of over 1,000, they were absent altogether in 40 per cent of the samples analyzed. Their first appearance moreover was several days later than in tube No. 1.

The organisms classed as *Peritricha* were present in the original sludge and showed rapid development to a count of 90,000 on the fourth day of aeration. After this, with the exception of one day, February 26, they showed a steady decrease to a count of 2,000. After adding sewage on March 3 the number increased, and thereafter never fell below a count of 6,000. This increase was due probably to the addition of necessary food which they found in the sewage. In tube No. 2 this class of organisms was present throughout the entire test. The numbers show wide variations but at no time was there such a marked decrease as was observed in the control. The raw waste which was being continuously added to this tube evidently contained sufficient food to support these organisms.

*Euplotes* were found in the control tube on three consecutive days after the second day of operation. After this they were not found in any of the samples examined. In tube No. 2 they were present in the samples collected on the sixth and seventh days, after which they disappeared for the remainder of the tests. It is, therefore, quite evident that the conditions of the experiment were not favorable in either tube for the development of this organism.

Rotifers and molds were found in both tubes. In the control they were present in small numbers throughout the test but never in sufficient numbers to attach any importance to their presence. This is also true of tube No 2, although the *Rotifers* in this tube developed in larger numbers than in tube No. 1, on one day reaching a count of 18,000, whereas the maximum for tube No. 1 was but 80. *Molds* were never present in sufficient numbers in either tube to be recorded more than present.

The Zooglea masses, reported in standard units, were present in the original sludge and gave, with but few exceptions, high counts in both tubes. In tube No. 1 the counts ranged from 8,000 to 255,000 with apparently no regularity of either increase or decrease. In tube No. 2 these masses were present in much smaller numbers than in the control tube. With the exception of the two days in which they were absent in the control the count in tube No. 2 exceeded that in tube No. 1 on but one day and was equal to it on but one other day.

While counts of the organisms in the aerated waste were, as a rule, less than in the control it is well to bear in mind that the portions of supernatant removed from the waste had some influence on the total number found. While no counts were made to show how the organisms were distributed in the contents of the tube it seems safe to assume there was a fair proportion in the liquid apart from the suspended matter, and removing the supernatant, therefore, had the effect of reducing the total count. The conclusion to be drawn from this test is that the organisms found in activated sewage sludge will grow and develop in the aerated strawboard waste to the same extent they do in aerated sewage.

Chemical analyses were made of only a few samples collected throughout the run. In the average results the oxygen consumed was 107 p. p. m., nitrates 3.5 p. p. m., and suspended matter 184 p. p. m. These analyses were made of samples taken after the sludge had settled for a period of 30 minutes. The putrescibility samples in all tests made after 48 hours of aeration, and when not more than

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100 c. c. of the supernatant were removed, were stable, and the oxygen demand after five days' incubation averaged 15 p. p. m. For shorter periods of aeration the putrescibility samples were not stable, and the oxygen demand varied from 25 to 40 parts.

The efficiency of the treatment in the removal or reduction of color fell far below expectations. A sample of the original mixture of activated sludge and waste was filtered and kept for comparison with the filtered supernatant at different times during the tests. At no time during the period of operation was the color reduced more than 50 per cent, and when all the supernatant was removed each day, as was done toward the end of the run, there was not more than 25 per cent reduction.

Summing up the results of these tests, which covered the period from February 19 to March 19, 1916, it is evident that the organisms present in sewage activated sludge will not only live when strawboard waste is mixed with activated sewage sludge but will multiply in large numbers. This was shown in the last seven days of the test, when the number of organisms was practically the same in the waste as in the control, although 80 per cent of the waste in the tubes was removed each day. What relation these higher organisms have to the actual purification processes is not known, but their continual presence in all sewage effluents from oxidizing filters, when at least some oxygen is present, has led to the assumption that they are intimately associated with the breaking down of organic matter.

Throughout the test the activated sewage sludge added to the waste maintained its flocculent condition and settled rapidly when aeration was stopped, and the coarse suspended matter in the waste likewise was formed into flocs that settled rapidly. But the finely divided suspended solids, about 105 p. p. m., that remained in the supernatant at the end of 30 minutes' settling would not settle out after prolonged quiescence. The results in general were not sufficiently favorable to warrant carrying out any more extensive tests at the testing station at Noblesville.

### VOLUME, COMPOSITION, AND DISPOSAL OF SLUDGE.

In the treatment of industrial wastes, the sludge problem is, as a general rule, of prime importance. To the handling and disposal of this product of purification may be charged practically all of the operating cost of the plant, except where pumping of the waste is required in lieu of head for gravity flow. Volume and composition of sludge.—In the tests at Noblesville careful records were kept of the volume of sludge deposited, its composition, treatment, and possible ultimate disposal as a fertilizer.

In Table No. 13 are recorded the results of 17 runs made in tanks 1 and 2 to determine the volume and composition of the sludge. For comparison the sludge is reduced to tons of dry solids per million gallons and compared with the amount of suspended solids removed by the tanks.

TABLE No. 13.—Sludge accumulated in settling tanks compared with suspended matter removed.

augustas, tas	al ada	1.9		Sludge	e accun	aulated	1.	a line	Suspen	ded so	lids ren	noved.	- ·
Period covered (1915-16).		yards.		rds per s.	1	Analys	is.	r solids ons.	Suspended solids. p. p. m.			r solids	accumulated ids removed.
From—	То—	Deposited, cubic yards.	Waste treated, th gallons.		Per cent water.	Specific gravity.	Per cent volatile.	Deposited, tons dry solids per million gallons.	Influent.	Effluent.	Removed.	d, tons c	Ratio of sludge accumulate suspended solids removed
Feb. 25. Mar. 23. Apr. 12. June 7. June 28. Nov. 20. Dec. 1. Jan. 5. June 13.	Mar. 23 Apr. 12 June 7 June 28 Sept. 16 Dec. 1 Jan. 5 Feb. 1 July 1	4.80 3.51 5.97 .80 7.02 .78 1.54 .93 4.45	$\begin{array}{c} 111.6\\86.7\\82.5\\21.9\\376.2\\11.7\\53.2\\39.5\\66.2 \end{array}$	$\begin{array}{r} 43.0\\ 40.5\\ 72.4\\ 36.5\\ 18.7\\ 66.7\\ 28.9\\ 23.5\\ 67.2\end{array}$	92.7 90.7 93.2 97.5 91.2 191.0 191.0 191.0 91.0	1.04 1.03 1.01 1.03 1.04 1.04 1.04 1.04	64.3 65.3 58.8 54.3 44.8	$\begin{array}{r} 2.75\\ 3.31\\ 4.28\\ .78\\ 1.43\\ 5.25\\ 2.28\\ 1.86\\ 5.30\end{array}$	$\begin{array}{c} 1,664\\ 2,087\\ 2,160\\ 564\\ 1,336\\ 2,634\\ 1,582\\ 1,167\\ 2,362\end{array}$	906 1, 230 984 328 633 1, 182 844 665 1, 136	758 857 1,176 236 703 1,452 738 502 1,226	$\begin{array}{r} 3.16\\ 3.57\\ 4.90\\ .98\\ 2.93\\ 6.04\\ 3.08\\ 2.09\\ 5.10 \end{array}$	0.87 .93 .87 .80 .49 .87 .74 .89 1.04
Total period.		29.8	849.5	35.1				2.48				3.35	. 74
				1	FANK	NO.	2.						
Feb. 25 Mar. 23 A pr. 12 June 7 June 28 Dec. 1 Jan. 5 Feb. 1	Mar. 23 Apr. 12 June 7 June 28 Sept. 16 Jan. 5 Feb. 1 Feb. 29	4.49 4.91 6.58 .97 7.21 1.64 .92 .10	111.686.782.521.9376.253.239.541.3	$\begin{array}{r} 40.2\\ 56.6\\ 79.8\\ 44.3\\ 19.2\\ 30.9\\ 23.3\\ 24.2 \end{array}$		1.04 1.04 1.03 1.01 1.03 1.04 1.04 1.04 1.04 1.04 1.04 1.04 1.04 1.04 1.04 1.04 1.04 1.04 1.03 1.01 1.03 1.04 1.03 1.04 1.03 1.04 1.03 1.04 1.03 1.04 1.03 1.04 1.03 1.04 1.03 1.04 1.04 1.03 1.04	62.9 61.5 59.7 53.4	$\begin{array}{r} 2.72\\ 3.78\\ 4.37\\ .94\\ 1.53\\ 2.43\\ 1.84\\ 1.89\end{array}$	$\begin{array}{c} 1,664\\ 2,087\\ 2,160\\ 564\\ 1,336\\ 1,582\\ 1,167\\ 1,238\end{array}$	906 1,230 984 328 633 844 665 675	$758 \\ 857 \\ 1,176 \\ 236 \\ 703 \\ 738 \\ 502 \\ 563$	$\begin{array}{r} 3.16\\ 3.57\\ 4.90\\ .98\\ 2.93\\ 3.08\\ 2.09\\ 2.34 \end{array}$	0.86 1.05 .89 .96 .52 .79 .88 .80

TANK NO. 1.

<sup>1</sup>Assumed from other analyses.

2.30

2.39

2.88

3.24

.74

.74

812.9

33.0

34.1

26.8

Total period.

Average tanks 1 and 2..... These results show wide variations in the volume of sludge deposited, ranging from 18.7 cubic yards per million gallons for tank No. 1, from June 28 to September 16, 1915, to 79.8 for tank No. 2 from April 12 to June 17, 1915. Three factors are largely responsible for these differences. First, the period covered by the run has a direct bearing on the density of the sludge, as it compacts as the layer becomes deeper. Second, the character of the product is a factor, as the different products are made from different raw materials which give widely different amounts of settleable solids in the waste. And finally the season of the year has a direct bearing on the bacterial action and liquefaction of the sludge in the tank.

In comparing the sludge accumulated with the suspended solids removed, the agreement, as shown by the ratio in the last column, is remarkably good. There are naturally some results at considerable variance with the average, but when the factors mentioned above are considered, together with the difficulty of obtaining a truly representative sample of the sludge for analysis, and with the fact that the samples for the suspended solids determination cover but a relatively small proportion of the total run, the differences are less than would be expected. The ratio of the sludge accumulated to the solids removed of 0.85 is probably a very good figure.

In many investigations this ratio is over 1, but such results are generally found in wastes containing large particles of suspended solids which are excluded from the analyses. Where the solids are fairly uniform in size and small enough to be included in a representative sample for analysis, the ratio would be expected to fall below 1 as it did in these studies. A certain amount of liquefaction must be expected, together with occasional periods of unloading, which would not be proportionately represented in the samples of the effluent taken for analysis.

The volume of sludge deposited in tanks No. 3 and 4 was too small to be accurately computed. It was of no practical importance, as the studies demonstrated that such secondary tanks are of too little value to be included in a full-size plant.

The sludge removed from the tanks was homogeneous in character, flowed readily through a 4-inch pipe, and had a yellowish color somewhat darker than straw. As a rule it was free from gas or other evidence of fermentation. In June, 1915, however, sludge drawn off showed marked evidence of fermentation. It was full of minute gas bubbles, black in color, and in other respects resembled welldigested Imhoff sludge. On beds it dried much more rapidly than the sludge ordinarily obtained and was a much better sludge to handle. At no other time did this digestion take place in the tanks, and the reason for it at this time was never found.

Efforts were made in the laboratory to discover the cause, and a means to start or assist digestion. Fresh sludge was treated with sewage, horse and cow manure, and fresh fecal matter, for the purpose of seeding it with the necessary bacteria to start digestion.

The sewage was added at the ratio of 1:3 and the manure at the ratio of 1 pound per gallon of waste. The samples were well shaken and allowed to stand in the laboratory under observation for three months. During this time there was no action whatever and no reduction of the color similar to what takes place when domestic sewage is allowed to stand for a similar period. Other tests made by incubating mixtures of fresh fecal matter and the raw waste showed a pronounced gas formation as long as the fecal matter was undergoing digestion, but no further action after it was reduced.

From these tests and from the operation of the settling tanks it is obvious that bacterial action similar to that common in sewagetreatment tanks can not be expected to develop in a short time in treating strawboard waste in any form of settling tank, although it is possible for bacteria to develop after a long period of time that will reduce the organic matter in the waste.

Sludge drying.—During both periods the sludge removed from the various tanks was submitted to drying tests on three different kinds of beds.

No. 1 had an area of 100 square feet and was filled to a depth of 5 inches with unscreened cinders. Bed No. 2 had the same area as No. 1 and had 4 inches of straw over the bottom, and bed No. 3 had an area of 138 square feet, and was filled to a depth of  $7\frac{1}{2}$  inches with cinders screened over a  $\frac{1}{2}$ -inch screen. The sludge outlets from the tanks were at such an elevation that the sludge could be drawn from the outlet pipes direct to the filters by means of troughs. Only concentrated sludge from the bottom of the settling tanks was applied to the drying beds, and on the average 10 inches were added to each bed at a time.

The results of the investigations made in treating the sludge on open beds are contained in table No. 14.

	Bed No of c	o. 1, 5 in cinders		Bed No of	. 2, 4 in straw.		Bed No. of screet	. 3, 7½ i ned cin	nches iders.	ho ngbala add
Date.	Source.	Num- ber of days dry- ing.		Source.	Num- ber of days dry- ing.		Source.	Num- ber of days dry- ing.		Remarks.
1915	1							1		Landa and the second
Sept. 16. Sept. 17. Sept. 20. Sept. 21.				Tank 2	0 3 4	90.8 84.3 83.3	Tank 1 do do do		91.2	Rain Sept. 17. Rain Sept. 20.
Sept. 24.				do	5 7 12	81.8 80.9 79.2	do do		77.3 72.8	Rain Sept. 25 and 28 Rain Sept. 30 and Oct. 1.
Oct. 5				do	15 18 21	78.8 77.9 75.5	do do			Rain Oct. 4.
1916					-21	10.0				Bowleff of Deal
Feb. 25	Tank 2	0 1	92.7 84.4	do Tank 1	0	90.5	do			Temperature below freezing during mos of run.
Feb. 27	do	. 2	80.2 83.8	do	1	86.8 83.6	do			orrun.
Mar. 2	do	5	80.8	do	24	84.0	do			Theory Charge
Mar. 4	do			do	56	83.8 83.3	do			Rain Mar. 5 and 7.
Mar. 8 Mar. 24	do Tank 1	0	92.3	do Tanks 1	10 0	81.8 92.3	do		92.7	
Mar. 25	do	1	88.4	and 2.	1	87.7	do	1	86.9	Rain a. m., Mar. 25.
Mar. 26 Mar. 27	do do	23	85.7 85.7	do	23	86.2 86.4	do do	23	84.4 85.2	Temperature below freezing during mos of run.
	do	45	85.3 85.4	do	45	85.8 85.4	do	4	84.3	
Mar. 30	do	6	84.2	do	6	84.9	do	5 6	83.3 82.8	relieve storing
Mar. 31	do	78	83.4 82.9	do	78	83.9 83.3	do	7 8	81.4 81.3	E plany month
Apr. 2	do	9	82.8	do	9		do	9	82.8	about the
Apr.3	do	10	81.2	do			do	10 12	79.1 77.8	A La Sala a Maria
Mar. 31	Tank 2	0	89.0	do			do			and the state
	do	34	88.1 87.8				do			BIN SALE
Apr. 6	do	6	85.2	do			do			hour has a state
Apr. 11.	do	11 13	82.3 80.2	do			do			
Apr. 29	do Tank 2	0	88.1	do			do			To state the lite
May. 3	do	5	84.1	do			do			a manager and
may. 6	do	8	80.7				do			

TABLE No. 14.—Sludge drying on open beds.

As a rule the sludge in each of the nine runs recorded in the table was reduced to about 80 per cent water in 10 days. The weather conditions during most of the runs were very unfavorable, owing to extremely low temperatures and frequency of rains and snows. Beds 1 and 2 had an additional disadvantage in being shaded from the morning sun by the strawboard mill and from the afternoon sun by the testing station. The sludge on bed 3, which was better located, showed slightly the lower water content for the same drying period and under identical weather conditions.

The sludge applied to the beds contained from 90 to 93 per cent water, which was the best obtainable from the small units operated. Experience has shown, however, that larger tanks, which allow deeper sludge deposits and have larger sludge outlets, give better sludge than can be obtained with test units. It seems safe to assume that in a permanent plant sludge containing 87 per cent moisture can be obtained for drying on beds.

While it would be possible to remove sludge from the drying bed with 80 per cent water, especially during the winter months when frozen, it is better to allow further drying to obtain a sludge more readily handled. From the results in the above table, it would appear hardly safe to count on dosing a bed more than once a month throughout the year. This would allow time for drying and removing the sludge and preparing the bed for the next dose. It would be necessary to provide sufficient drying-bed capacity to handle the sludge on a uniform schedule throughout the year, as storage capacity could hardly be provided to hold sludge for favorable drying weather during the summer.

Table No. 13 indicates that the total volume of sludge to be expected is 2.75 tons dry solids per million gallons. This is equivalent to 21 tons with 87 per cent water, and, with a specific gravity of 1.05, to 24 cubic yards per million gallons.

On the basis of total mill product, the most satisfactory basis for sludge computations for general use, and taking the Noblesville figure of 38,300 gallons of waste per ton of product, this is equivalent to 0.92, or practically 1.0, cubic yard of sludge, containing 87 per cent water per ton of product.

The drying beds, therefore, to take an accumulation of 30 days will have a capacity of 27 cubic yards per daily ton of product, which, flooded 10 inches deep, would require an area of about 875 square feet.

Use of the sludge as a fertilizer.—Tests were carried out on large plots of ground on the strawboard farm and on another farm owned by Dr. Booth to show the fertilizing value of the solids removed from the combined wastes from the strawboard plant.

These tests were carried out on corn ground only and cover the two seasons of 1915 and 1916. In each year and on both the farms where the sludge was used the soil selected was the poorest that could be found in the fields that were to be planted in corn, and in every case the strips of land fertilized, as well as the "controls," were accurately laid off and stakes set to mark the boundaries. The amount of sludge added to each plot was computed to a dry basis containing 10 per cent moisture, and the solids were applied to the land on the strawboard farm in 1915 by a lime spreader and in 1916 by shovels. The lime spreader was the ideal method of obtaining an accurate distribution of the sludge, although spreading by hand apparently gave the same results.

The experimental strips on the strawboard farm in 1915 consisted of plots of different sizes. One plot was 200 feet long by 23.5 feet wide, to which sludge from the settling tanks was added at a rate of 4 tons per acre; a second plot, 175 feet long by 22 feet wide, was used as a control; a third and a fourth plot, each 95 feet long by 23.5 feet wide, were treated with sludge from an old settling pond at the rate of 4 tons per acre. The part of the field containing the test plots was farmed in the same manner as the rest of the field and when the corn was harvested the amounts grown on each section were weighed in the field. The corn husked from the stalks grown on plot No. 1 weighed 421 pounds, equivalent to 3,895 pounds per acre. On plot No. 2, unfertilized, the weight of corn was 314.5 pounds, or 3,556 pounds per acre. Plots No. 3 and 4 received the same amount of fertilizer and are considered as one. The weight of ears husked from these two plots was 387.5 pounds, or 3,780 pounds per acre.

The increase per acre of plot No. 1, fertilized with sludge from settling tank, over Plot No. 2, unfertilized, was 339 pounds, or 9.6 per cent, and of Plots No. 3 and 4 together, 224 pounds, or 6.3 per cent.

The increase in yield on the fertilized sections was not large, but it must be borne in mind that only 4 tons of sludge per acre were added and that, allowing 70 pounds to the bushel, the unfertilized sections produced 50.8 bushels per acre. This soil is not greatly in need of a fertilizer and moreover the season of 1915 was favorable. For an average season and on land reduced in fertility to 30 or 40 bushels per acre the relative increase would no doubt have been much greater.

Another series of farm tests was carried out in 1915 on a farm owned by Dr. Booth and located about 6 miles out from Noblesville. The strips of ground selected for the tests were on the high ground in the field, which was no doubt the poorest soil, but like the test plots on the strawboard farm had received a coating of manure within three years. Three plots were staked off, each 195 feet long and 28 feet wide, with the control in the center. Eight rows of corn came within each strip. On two of the strips sludge obtained from a 10-acre abandoned pond that was formerly used for settling out the solids from the wastes at the strawboard plant, and at this time was filled up with dry sludge, was applied at a rate of 8 tons per acre. A manure spreader was used for applying the solids and a very even distribution was obtained.

The corn was husked from the stalks standing in the field on October 15, and was, therefore, practically dry. The amount harvested from the unfertilized section was 645 pounds, and for the two sections treated with strawboard solids, 1,445 pounds, or an average of 722 pounds to each strip. The increased yield was, therefore, 12 per cent.

While the strips of ground selected on Dr. Booth's farm were in the poorest section of the field, nevertheless the yield was high on the unfertilized section on account of the favorable season and the fact that the land was not run down. The increase of 12 per cent was much better than was obtained on the strawboard farm, as might have been expected from the application of 8 tons of sludge instead of 4.

In 1916 the tests were repeated on another set of test plots on the strawboard farm, three strips being laid off; No. 1, 40 feet wide by 443.5 feet long; No. 2, 50 feet wide by 373 feet long; and No. 3, 42 feet wide by 296 feet long. The sludge was applied at the rate of 16 tons per acre on a dry basis, 10 per cent moisture, to plots Nos. 1 and 3, and spread over the ground with a shovel. Plot No. 2 was used as a control. The corn was husked from the stalks standing in the field and weighed in the field. The yield from plot No. 1 was 780 pounds, or 1,915 pounds to the acre; from plot No. 2, unfertilized, 730 pounds, or 1,705 pounds to the acre; and from plot No. 3, 600 pounds, or 2,105 pounds to the acre. The increase was, therefore, 12.3 and 23, respectively, over the unfertilized plot, which yielded about 24.4 bushels to the acre.

The results given in the preceding paragraph were computed from the area of the strips as marked off by the stakes, and they were checked by adding together the length of all the rows in each strip and computing the increase for the fertilized sections over the unfertilized in terms of yield per linear foot. The total length of rows in plot No. 1 was 5,322 feet and the yield 0.1465 pound per linear foot; in No. 2, 5,595 feet, with a yield of 0.1305 pound per foot; and in No. 3, 3,757 feet, with a yield of 0.1600 pound per foot. The increase of No. 1 over No. 2 was 12.3 per cent, and of No. 3 over No. 2 was 22.8 per cent. These figures check very closely the increases of 12.3 and 23 per cent, computed from the total areas of the three strips.

The plots fertilized in the spring of 1915 were again planted in corn the following spring with no further addition of fertilizer in any form. The unfertilized section yielded 25.3 bushels per acre; plot No. 1, treated the previous year with 4 tons of sludge from settling tanks, 26.3 bushels per acre; and plots Nos. 3 and 4, similarly treated with 4 tons of solids from the abandoned settling pond, 27.4 bushels per acre. The fertilized sections gave an increased yield over the one receiving no treatment of 4 and 8 per cent, respectively.

It is interesting to note in comparing the results of the fertilizer tests for 1916 that the unfertilized plots in each separate set of plots, although a considerable distance apart, gave practically the same yield, 24.4 and 25.3 bushels per acre.

In addition to the field studies a series of box tests were made in the spring of 1916 at the testing station, using various grains and vegetables. The plants and seeds selected were uniform, and the same number of each kind, equally spaced, were planted in each of two boxes. There were 16 boxes, each 12 inches square and 10 inches deep, filled with earth thoroughly mixed, and free from foreign material such as rocks and hard lumps. The soil selected was from the poorest land of the strawboard farm, but it had been manured within three years and consequently had a fairly high degree of fertility. The odd-numbered boxes were fertilized with digested sludge from the settling tanks, at the rate of 6 tons, 10 per cent moisture, to the acre.

Field corn was planted in boxes 1 and 2 and sweet corn in 3 and 4. Twenty-one grains of corn were planted in each box, and, while it was not expected that the grain would mature, it was anticipated that any material difference in growth could be determined by cutting and weighing the stalks when they showed signs of drying up. In both boxes the field corn grew to a height of 12 inches and the sweet corn to 9 inches and then began to wither and die. The stalks cut at the ground and weighed showed no difference in weight between the fertilized and unfertilized boxes. The adverse conditions of growth in the small boxes probably outweighed the advantage due to the fertilizer which was found in the larger field tests and no significance is attached to these results.

Seventy-five grains of wheat were planted in each of boxes 5 and 6. There was no apparent difference in the growth up to June 26, when the stalks began to die. In the fertilized box there were 128 stalks and in the control 120. The increase in weight of the stalks in box No. 5 over No. 6 was 5 per cent. The wheat was growing less than one month, and, as it was not spring wheat, the abnormal conditions outweigh the value of the fertilizer.

Eighty-two grains of oats were planted in each of boxes 7 and 8. The growth in box No. 7 was better than in No. 8 throughout the growing period—May 13 to June 26—and there were 82 stalks in the fertilized box, when cut, as against 76 in box No. 8. The increase in weight was 16 per cent. Oats are normally a spring crop, and the conditions were much more favorable for a comparative test than with either the corn or wheat.

Boxes No. 9 and 10 were used for growing onions from sets, 27 sets being planted in each box. All came up in box No. 9, and all but one in box No. 10. There was a noticeable difference throughout the growing period between the plants in the fertilized and unfertilized boxes, and after the tops died down the onions with the tops removed were washed and weighed. There was an increase of 116 per cent in the fertilized box. The onions in box No. 9 were on an average about twice the size of those in box No. 8, and only one failed to mature in box No. 9, while six which started to grow failed to mature in the untreated box. Allowing for a reduction of the onions that came up but did not mature, the per cent increase is very large, and as the crop as a whole matured it is safe to assume that a very substantial increase can be expected by the use of this material in growing onions.

Tomato plants were set in boxes 11 and 12, three plants in each. The plants were all of about the same size and grown in the same beds. They were set out on May 13, and on May 26 there was a noticeable difference in the growth of the plants in the boxes.

On June 8 the tomatoes in the fertilized box were 7 inches high and those in the unfertilized box 6 inches. The stalks were much larger in the former box, the leaves thicker and larger and had a better color. June 22 the tomatoes in box No. 11 were 11 inches high and those in No. 12 were 10 inches, and the buds were much larger in the former. On June 26 the first blossoms were noticed, and by the 28th all the plants were in bloom in the fertilized box, but those in the unfertilized box did not blossom until the 30th. The plants began to die on July 2, and they were then cut close to the ground and weighed. Those in the fertilized box showed an increase in weight over the unfertilized of 23 per cent. Considering the history of the growth of the plants in the two boxes and the per cent increase in weight of the plants in the fertilized box, it seems safe to assume that the sludge had some beneficial effects on the growth of tomato plants.

Beans were planted in boxes 12 and 14, with 14 beans to each box. The only difference noted in the growths in the two boxes was in the number of blossoms, of which there appeared to be more in the fertilized box. The stalks began to dry before the pods were filled, so they were pulled up, the roots washed and dried and weighed with the vines. There was no difference in the weight of the vines from the two boxes.

Radish seeds were planted in boxes 15 and 16, 32 seeds in each box. Three days after planting 28 stalks were through the ground  $1\frac{1}{2}$  inches in box 15, and 24 in box 16, and the growth in the former box was slightly better. Twenty days after planting the radishes still showed a better growth in the fertilized box. At 46 days the tops began to wither and they were pulled, washed, dried, and weighed. Including the tops the per cent increase in weight for the fertilized box was 73 per cent. From the 32 seeds planted there were 24 radishes in the fertilized box and 22 in the other box, those from the former being much larger, on an average, than those from the latter.

Summing up the results obtained from the tests using the solids from the abandoned settling pond and from the settling tanks of the testing station as a fertilizer, it is seen that a variety of results were obtained, practically all showing more or less increase in the amount of grain and vegetables grown in soil treated with these solids.

In the field tests carried out in 1915 the increase in corn on the strawboard farm, using 4 tons per acre of the settling pond sludge, was 6.3 per cent, while the sludge from the settling tank of the testing station with the same rate of application gave an increase of 9.6 per cent. On Dr. Booth's land, using 8 tons per acre from the settling pond, the increase in yield was 12 per cent. This was an exceptionally good corn year as shown by the yield of 64.5 bushels for the unfertilized strip of the poorest soil in the field. In 1916 the amount of sludge added to the two plots in the strawboard farm was 16 tons per acre, and the increase obtained over a neighboring untreated plot was 12.3 per cent on one section and 23 on the other. The sludge used in this case was from an abandoned settling pond. This was an exceptionally dry season, and the effect of the drought is shown in the yield of 24.4 bushels on the same strip that produced 50.8 bushels per acre in 1915. It is probably safe to assume that for average years and on soil similar to that used in the tests an increase of 10 to 12 per cent can be expected when using 8 tons of the solid waste, 10 per cent moisture, from a strawboard plant. In addition to the increase in yield the first year there can reasonably be expected an increase during the following two or three years. This was borne out in corn harvested in 1916 from the strips treated in the spring of 1915, where the increase on the two plots under observation was 4 and 8 per cent, respectively. The amount added in these tests was undoubtedly too small. The physical condition of the soil was very much improved in every case where the sludge was used, and it was possible for the farmers to tell where the strips began and left off by the condition of the soil when plowing it.

The Ohio Agricultural Experiment Station Bulletin No. 274, published in 1914, gives some tables showing the per cent increase obtained by using different fertilizers.

In Table No. III, part 2, page 294, the increase in the yield of corn on land producing 61.93 bushels per acre was 8.1 bushels when using 8 tons of yard manure. The increase on Dr. Booth's land using 8 tons of strawboard waste sludge was 7.8 bushels per acre on land where the unfertilized soil yielded 64.5 bushels. While it may not be said without further study that the strawboard sludge is equal in value to manure under all conditions of soil and climate, these comparative results are of undoubted significance, and show that upon certain land, normally productive, an increase in yield of corn was obtained by the application of this sludge equivalent to the increase obtained upon other land of equal normal productiveness by the application of equal quantities of yard manure. On the strawboard farm, using the same kind of material as was used on Dr. Booth's land, but only one-half as much per acre, the percentage increase was about one-half as great.

The growing tests carried out in the boxes at the testing station gave much better results when using 6 tons per acre of digested sludge from the settling tank than those for corn in the field tests using 8 tons per acre of sludge from the old settling pond. Special precautions were taken to make the tests comparable, such as selecting the same number, and a good average of the seeds or plants for each box, and cultivating the plants in the same way while growing. In determining the per cent increase, the plants were weighed on a chemical balance and proper precaution taken to see that each plant was free of dirt. The corn, wheat, and beans in the box tests showed no increase in the fertilizer plots, but this was probably due to their beginning to dry up before the growth had developed to any extent. The oats showed an increase of 16 per cent, and there was a noticeable difference in the growth in the two boxes all through the growing period. Onions showed the most remarkable gain of all the test boxes, the increase being 116 per cent. The number of onions that sprouted and came through the ground but did not grow to maturity in the unfertilized box was greater than in the fertilized box, and as similar results were found in practically all the tests it does not seem fair to assume that the difference was due to the size of the sets planted. Even allowing 30 per cent for the difference in the number of onions gathered from the two boxes, there still remains a high per cent increase for the onions fertilized with the solid waste from the strawboard plant. Tomato plants of the same size were planted in two boxes, one with solids from the settling tank, as in all the other tests, and one serving as a control. There was an increase of 23 per cent in the weight of the vines in the box treated with sludge. This confirmed the earlier blossoming of these plants, and the color and heavier growth noticed throughout the period. Radish seed planted in two boxes, following the same procedure as outlined for all the other tests, showed a gain of 73 per cent in favor of the strawboard sludge. Fewer seeds grew to maturity in the unfertilized box than in the fertilized, as was the case with the onions and the oats. The radishes were much larger in the fertilized box and, as all the plants grew from seeds, there was no chance of the difference being due to a difference in selection as might possibly be true with the onions or tomatoes.

It is realized that tests on such a small scale as those carried out in the boxes are subject to wide variations, but considering the fact that all the tests using spring crops, except the corn and beans, gave increases varying from 16 to 116 per cent, it is reasonable to assume that by using not less than 6 tons, 10 per cent moisture, of the strawboard sludge per acre on soil planted in vegetables, a very decided increase can be expected in the amount of product raised.

In connection with the fertilizer tests samples of the sludge used were taken for analysis. In table No. 15 are recorded the results of these analyses. The sludges were those used in the tests and the analyses recorded are the average either of several samples or of duplicate determinations of the same sample. For purpose of comparison an analysis of horse manure is included in this table.

TABLE No. 15.—Analyses of strawboard waste sludge and horse manure.

training the second second second second	Per cent (dry basis 100° C.).					
Source.	Total nitrogen.	Calcium as CaCO <sub>3</sub> .	Total phosphorus as P <sub>2</sub> O <sub>5</sub> .	Total potassium as K <sub>2</sub> O.		
Fresh sludge settling tank. Digested sludge settling tank. Sludge from old settling pond. Horse manure <sup>1</sup> (including litter).	0.69 1.0 .66 1.7	21 23 37	0.38 .48 .29 .26	0.54 .42 .25 1.50		

<sup>1</sup>Data from Bulletin No. 246, Ohio Agricultural Experiment Station, p. 726.

These analyses do not show the sludge to be a high-grade fertilizer, but it should be remembered that a very important effect of the sludge is its mechanical action on the soil, in keeping it in a loose condition through the fermentation of the cellulose material.

The lime also is of great value to many soils. This, reported as calcium carbonate, is lowest in the freshly deposited settling tank sludge, and highest in that from the old settling pond which was well digested. The digested settling tank sludge comes between the two. These differences are to be expected, as digestion, exposure to weather, and washing by rain all tend to reduce the organic matter without affecting at all, or in a much less degree, the mineral constituents.

Compared with horse manure the strawboard sludge contains less nitrogen and potassium and more phosphorus. The presence of the lime also gives it a slight advantage.

### CONCLUSIONS.

The investigation of methods for the treatment and disposal of strawboard waste, here reported upon, was made on a sufficiently large scale, and continued for a sufficient length of time, to justify definite conclusions regarding the character of the waste and the results of treatment in a permanent plant designed on the basis of the data obtained.

Supplementing these tests a survey was made of seven other strawboard mills to ascertain the uniformity of the wastes produced in this industry. This survey demonstrated that within comparatively narrow limits the strawboard industry is well standardized and the waste from the Noblesville plant is fairly representative of this entire industry. It is believed, therefore, that the conclusions based upon the data obtained from the studies at Noblesville are applicable to the industry as a whole. They apply, however, only to the waste produced in the manufacture of strawboard. While other grades of paper were made at Noblesville, straw was the principal stock used, and the essential conclusions are based on long runs when only strawboard was made.

## CHARACTER OF THE RAW WASTE.

The strawboard waste is a composite of two separate portions from two distinct processes. The most concentrated and polluting of these two is from the beaters where the straw is washed after digestion in the rotaries. Of this waste there are about 20,000 gallons per ton of board, and it contains about 3,800 p. p. m. of suspended solids. This is equivalent to about 600 pounds of solids per ton of board. While a large part of these solids is inorganic, consisting of lime used in the digesters and dirt or silica on the straw, there is still considerable organic matter requiring oxidation to render it nonputrescible. The oxygen consumed value is about 3,400 p. p. m., and the 10-day biochemical oxygen demand value, 3,300 p. p. m.

The other portion of the waste is from the paper machine, including the felt wash, and is much less concentrated. In volume it is about the same as that from the beaters, being approximately 20,000 gallons per ton of board. The concentration of suspended matter is about one-sixth of that found in the beater waste, so that there are approximately 100 pounds of solids per ton of board. The solids in this waste are almost entirely fiber and it is believed that a large proportion of this could be recovered by using the paper machine waste for wash water in the beaters, as is actually done at some of the mills. The oxygen consumed and 10-day biochemical demand of this waste have similar values, averaging about 200 p. p. m.

These two wastes combined make a total of about 40,000 gallons, containing about 2,500 p. p. m. of suspended matter (see Mixed wastes, Table No. 2), or about 800 pounds per ton of board. This is 14 per cent more than is given above for the average of the solids in the individual wastes. There is an oxygen consumed value of 1,800 p. p. m. and the total alkalinity, made up of carbonates and bicarbonates, is about 1,000 p. p. m.

#### SEDIMENTATION.

Experimental studies made on this waste included treatments by sedimentation, filtration, and the activated sludge process. Periods of sedimentation ranging from two to four hours were employed in one tank and in a series of three or four tanks. Chemical precipitants (lime, sulphate of alumina, and sulphate of iron) were added during certain periods of the tests.

In almost every case about 50 per cent of the solids were removed by these treatments. The advantage of the longer sedimentation periods over the shorter ones, as measured by removal of solids, was practically negligible. The economically settleable solids were removed by a two-hour detention period. No further improvement resulted from the addition of chemicals up to the limit of what would be economically feasible.

At only one time during the tests was there any evidence of active fermentation within the tanks. This occurred early in the tests and the tanks were then converted into Imhoff tanks in anticipation of fermentation during the warm summer months. This did not occur, however, so that the ordinary horizontal flow sedimentation tank was designed for the larger unit to be installed by the strawboard company, provisions, however, being made to convert the tank to an Imhoff tank if active decomposition should begin after the tank is placed in operation. The experiments do not justify definite conclusions upon this point, although it is believed that the plain sedimentation tank will be found satisfactory.

The removal of 50 per cent of suspended matter by the tanks was not accompanied by the same ratio of reduction in the oxygen consumed values. The highest removal of the latter was 37 per cent in one period, and in another the removal was only 7 per cent. Carbonates showed some slight reduction.

#### FILTRATION.

The settled waste was treated on filters of seven different types. A mechanical sand filter was operated during the first six months of the tests but gave very unsatisfactory results. While there was an apparent removal of 68 per cent of the suspended solids, the actual removal was considerably less, as a large part of the effluent consisted of the water retained in the filter after washing, owing to the short runs. The mechanical features of operations were also unsatisfactory. The length of runs which could be obtained were very short, averaging less than three hours, and the wash water required reached the prohibitive amount of over 100 per cent of the waste treated. It was obvious early in the tests that this filter would not give satisfactory results, and it was abandoned in favor of biological filters.

These experiments with the mechanical filter were not conclusive as to this type of treatment. In view of the highly satisfactory results previously reported by Phelps, it is not clear whether the failure of mechanical filtration at Noblesville was due to differences in the physical or chemical characteristics of the wastes in the two investigations, or to differences in the construction and operation of the filters.

A slow sand filter was operated at the rate of 100,000 gallons per acre per day for a little over one month. The effluent was unsatisfactory, and the filter clogged too rapidly to permit continuous operation.

Two limestone filters were also tested. One contained limestone screenings and the other broken limestone averaging about 1½ inches in diameter. These filters were each operated at both 200,000 and 400,000 gallon rates, but it was evident that the filtering material was too large to retain the fine suspended matter in the effluent from the settling tanks. The effluent of the screening filter contained over 200 p. p. m. and that of the coarser limestone filter over 400 p. p. m. of suspended solids. While the stone became coated with a slimy, stringy growth, the filters during eight months of operation apparently did not ripen sufficiently to give satisfactory biological oxidation.

Unscreened cinders were used as the filtering medium in one filter, and while the suspended matter was reduced to about 100 p. p. m., the filter soon clogged and pooled on the surface.

The most satisfactory results were obtained with screened cinders as the filtering medium. Two filters of this type were operated and both gave very satisfactory results. Rates of 200,000 and 400,000 gallons per acre per day were maintained on each of these filters and at no time was there any indication of pooling or deterioration. The effluents, with a 1:3 dilution, were stable for a period of at least 89 hours in the average for all the samples covering the entire eight months, and contained more nitrites and nitrates than any of the other filters. It was noticed that within these filters there developed large masses of worms, bugs, and higher organisms and, coincident with their presence, the suspended matter retained was black and inoffensive, and the odor resembled that of loam. Fish were placed in a small box receiving the effluent from one of the filters

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diluted with an equal volume of fresh water, and they lived for over a month with apparently no distress.

These filters, as was also the case with all the others, removed practically all the carbonate alkalinity. The small amount of calcium carbonate left in suspension would not be sufficient to cause a deposit of the suspended matter when coming in contact with domestic sewage in a stream.

As a preliminary to one of the screened cinder filters a small gravel roughing filter was operated to act as a colloider. This filter, however, was not of sufficient benefit to justify its use in a permanent plant.

#### ACTIVATED SLUDGE.

The activated sludge process of purification, recently developed for the treatment of sewage, was tested in connection with this waste. Laboratory studies were made over a period covering one month to determine whether the organisms present in activated sludge could exist in the highly alkaline strawboard waste, and what degree of purification could be obtained by this method. It was thoroughly demonstrated that certain of the larger organisms not only lived but multiplied rapidly in the waste, but the degree of purification with a reasonable period of aeration was not sufficient to warrant extensive tests. The color reduction, which it was hoped to obtain by this method, was never over 50 per cent, and for the most part not over 25 per cent.

#### SLUDGE ACCUMULATION AND DISPOSAL.

Large volumes of sludge were deposited, amounting to 2.75 tons of dry solids per million gallons of waste treated. Computed to sludge having 87 per cent moisture, which it is believed can be obtained in a large plant, this would amount to 24 cubic yards per million gallons, or 0.9 cubic yards per ton of product, on the basis of an average discharge of 38,300 gallons of waste per ton of product.

Sludge treatment was studied in connection with open drying beds, and while a reduction to 80 per cent moisture was accomplished, as a general rule, within 10 days, the reduction below this point was more gradual and it was felt that under adverse conditions of temperature and rain it would not be possible to apply sludge to any one bed more than once a month throughout the year.

The disposal of the dried sludge will be the most serious problem in the operation of a plant treating this waste. Increased yields of corn were obtained comparable with those reported by the Ohio State Agricultural Experiment Station, from the application of yard manure in similar quantities to soils of similar normal productivity.

#### PLANT DESIGN.

On the basis of the data that have been presented, the following features are recommended for the design of a plant to treat strawboard waste. The computations are all referred to the basis of one ton of daily capacity or production of the mill. While the figures refer primarily to the data obtained at Noblesville, it is believed that they may be applied to other mills of this type, as the variations in volume and character of the wastes found in the survey were not sufficient to materially change the general design of the plant based on the data obtained in the tests.

The treatment should consist of sedimentation and filtration. The settling tank should be designed for a two-hour sedimentation period. On a basis of 38,300 gallons per ton of product, this would require a settling capacity of approximately 425 cubic feet per daily ton. This feature, however, will change with varying water consumption. As septic action did not develop during hot weather, the ordinary horizontal flow sedimentation tank is considered satisfactory, subject to further studies to be made in the permanent unit the strawboard company is constructing. In addition to the settling capacity ample provision should be made for the large volume of sludge which will be deposited in the tank. Withdrawal of sludge more frequently than twice a month can hardly be expected, especially during the winter months. As the average amount of water will be higher in the entire accumulation than that estimated for sludge to be applied to the beds, by reason of the thin top sludge, it will not be safe to provide storage on a basis of less than 89 per cent water. This gives an accumulation of 1.1 cubic yards of sludge per ton of product, so there is required, for 15 days' storage, approximately 450 cubic feet per ton of daily output. It is impossible to withdraw all the sludge at any one time, so that provision should be made for a residual of about 20 per cent. The total required sludge capacity therefore is about 550 cubic feet per daily ton of product.

For the drying of the sludge, open beds appear to be the most practical. The sludge should be withdrawn regularly twice a month throughout the year, as the volume is so great that it is impracticable to provide longer storage in the settling tank. By withdrawing small quantities at frequent intervals to small unit beds, it is estimated that sludge containing 87 per cent water can be obtained. This, as has been shown, represents a deposit of about 0.9 cubic yard per ton of product. As each unit of the sludge bed can not be expected to receive more than one dose per month during the winter, and as no provision is made for holding sludge for favorable drying weather, there should be provided 875 square feet of sludge-bed area per daily ton of product. The sludge must be removed from the beds after drying and disposed of. Under ordinary conditions the beds should, in the time allowed, reduce the water content to about 60 per cent, at which the sludge is spadable and readily handled. At this moisture content the total volume of sludge to be removed from the beds and disposed of is about a quarter of a ton per daily ton of product.

As an alternative for sludge beds requiring frequent cleaning there might be considered the use of ponds similar to those at present in use for settling the waste. These would require far less area and should be underdrained like farm land so that eventually the deposits in the ponds would drain dry and could be readily removed. Pending the development of a sufficient agricultural demand for the entire output of sludge from the plant, disposal by dumping may be employed.

The actual volume of dried sludge to be disposed of is about equal to that of the cinders produced under the boilers, and its disposal does not in reality present a much more serious problem. The cinders at present are used at many of the plants for fill and for dikes around the settling ponds. The sludge from the beds mixed with an equal volume of cinders could be dumped at any place where it is feasible to dump cinders.

Finally any plant installing either test units or a full treatment plant should make careful studies and experiments with a view of finding other possible uses to which the sludge may be put. The manufacture of fiber products, wall boards, etc., seems to present a possible field of development. Several very small experiments were made along this line during these studies. It was also attempted to use sludge in the mill by mixing in small proportions with regular stock. While the results were not satisfactory, they were sufficiently suggestive to show the desirability of further investigation along these lines.

The filters that were found best suited for purifying settled strawboard waste were those made of cinders from which the fine material was removed, and were 5 feet deep. Those placed in the filters in the test units were either washed or screened over a 4-inch screen. Later experience has indicated that the fine material can be removed at a low cost by washing the cinders in a small volume with water under pressure. This can be done either by applying water to the cinders in wagons while being hauled, if a dump wagon is used or if the bottom of the wagon bed has slats, or they can be washed on a sloping platform and the cinders removed with coal forks with the teeth close together. Under no circumstances should the horses and wagons be driven over the beds.

The underdrainage for the filters is very important. Farm tile, if properly laid and the filtering material carefully placed over it, is satisfactory and is recommended on account of the short lengths which afford more open spaces for drainage. The main drain under the beds should be 6-inch and the laterals 4-inch, and the laterals should be spaced not more than 8 feet apart, the bottom of the bed to be sloped so that each lateral drains 4 feet on each side. The slope of the tile should be such that all the filtered waste coming into it will drain freely from the beds. The bottom of the filter should be thoroughly compacted and smoothed so that there will be no pooling. This is very important and will justify very strict precautions to secure a smooth surface in preparing the underdrainage for the filters. The underdrainage system should be carefully inspected after 2 feet of cinders have been placed to see whether any of the tile have been broken.

The tile, laid in trenches with the sides sloping 4 feet each way, should be covered with 8 inches of coarse stone graded from 4 to 6 inches in diameter immediately over the tile to sizes averaging 1 to 2 inches.

The filters gave satisfactory results when operating at rates varying from 200,000 to 400,000 gallons per acre per day. The net rates in the average by months were slightly lower, but the filters were operated continuously at a rate of 400,000 for a long enough period to justify the recommendation that this figure be used in computing the area required to treat the waste from a strawboard mill. On the basis of 38,300 gallons of waste per ton of product there will be required 4,171 square feet of filter area per ton of product, and with a depth of 5 feet there will be required 772 cubic yards of cinders per ton of product.

The waste should be applied to the filter beds by means of a siphon and the distribution effected by means of troughs placed over the surface of the beds.

#### COSTS.

No detailed cost estimates can be given at this time because the cost of this plant is more than ordinarily dependent upon local conditions. Where there is no urgent need of complete purification immediately, tanks could be installed for removing the suspended matter and the cinders produced at the mill used for constructing the filters. For plants discharging not more than 1,250,000 gallons of waste per day and burning 100 tons of coal a day it would require about 8 years to build enough filters to treat all the wastes. Contractors who handle large quantities of cinders in Cincinnati state that cinders could be delivered in normal times within a radius of 40 miles of Cincinnati at an approximate cost of 35 cents per cubic yard f. o. b. siding within the 40-mile zone, and for longer hauls the extra freight would be in proportion to the distance. The cost of screening or washing the cinders would have to be added to the estimate given above. That would not add materially to the cost, especially if the cinders are washed either in the wagons hauling the material to the beds or on platforms.

Rectangular, hopper-bottomed concrete tanks, for plants of greater daily capacity than 7 tons of board, can be constructed with about 9 cubic yards of reinforced concrete per daily ton of plant capacity. Circular tanks are more economical of concrete, but call for somewhat higher unit cost of placing. Careful engineering design should be worked out in each case. Underdrainage of sludge beds may be estimated from similar local farm drainage costs. Land values and incidental costs are purely local.

The cost of such a plant is well within the means of any strawboard mill, and would in general represent an economy when the area and value of the land at present used for settling ponds is taken into consideration. The cost and supervision of the operation of such a plant would be but a very small burden and well worth while in view of the removal of the large settling ponds with their continual danger of becoming a nuisance, and the expense of replacing them as they become filled with sludge.

Observations made at several mills have suggested the possibility of numerous economies. These apply principally to the water consumption with the attendant cost of pumping, but there is good reason to believe that an appreciable saving could be made in the stock which is at the present time escaping with the waste. It is believed that in many cases the economies necessitated by the installation and operation of a treatment plant would eventually more than pay for the treatment of the waste.

Entirely aside from any questions of possible economies, the time is rapidly approaching when the strawboard-waste problem will have to be faced and disposed of in a more progressive and scientific manner than has hitherto been the practice. While the investigations here reported are in no sense complete or final, the information given will indicate the type of plant that is required and the approximate construction and maintenance charges involved.

A plant recommended on the basis of the tests was to be tried out on a larger scale, treating 20,000 to 30,000 gallons per day, the American Strawboard Co. building the plant and operating it under the general supervision of the United States Public Health Service. The filters have been constructed but the unusual business activity due to the war has thus far prevented the completion of the unit.

# THE DETERMINATION OF THE BIOCHEMICAL OXYGEN DEMAND OF INDUSTRIAL WASTES AND SEWAGE.

EMERY J. THERIAULT 1 and HARRY B. HOMMON.3

## INTRODUCTION.

In the investigation of methods for purifying industrial wastes made by the United States Public Health Service during the past three years a large number of analyses have been made to determine the biochemical oxygen demand of industrial wastes and sewage.

During the early part of the work considerable difficulty was encountered in obtaining reliable data, using the methods then available; i. e., the excess nitrate (Lederer), the relative stability and excess oxygen methods. It was found, using the excess nitrate method, that the ease in preparing the sample for incubation was largely offset by the later determinations of the residual nitrites and nitrates. It was also found that when the reduction method was employed the reduction of nitrites and nitrates to ammonia was frequently imperfect and that it was necessary to estimate both nitrites and free ammonia after reduction to determine the total residual available oxygen. The impossibility of observing intermediate stages in the deoxygenation limit the usefulness of the relative stability method. It did not seem permissible, moreover, to employ the conversion factors applicable to both the relative stability and nitrate methods to all types of industrial wastes without confirmation. The general applicability of the excess oxygen method as well as its freedom from empirical constants, closer simulation of natural conditions and the necessity of using it in any case for the calibration of other methods, prompted an intensive study of its shortcomings. The results of this study were so highly satisfactory that it seemed advisable to prepare and present an outline of the development of the oxygen demand studies.

The method adopted at the beginning of the work for determining the amount of oxygen required to completely oxidize the organic matter in a waste has been in use for some time in this country<sup>3</sup> and may be briefly described in its essential features as follows: Dilutions of the waste are made with aerated water, the degree of dilution being such that not less than 30 or more than 60 per cent of the

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<sup>\*</sup> Phelps, E. B., and Black, W. M. N. Y. Harbor Report 65 (1911).

oxygen initially present <sup>1, 2</sup> is used up during the subsequent incubation. If the diluting water has an appreciable oxygen demand, as is generally the case, its magnitude is determined by a blank determination and the value thus found is deducted from the value of the diluted waste. This corrected depletion, multiplied by a factor for the concentration of waste incubated, gives the oxygen demand of the straight waste for the period of incubation. Most of the difficulties mentioned in the literature bearing on the subject were encountered in the course of the work. A discussion of these difficulties and of the technic developed to minimize them is given in the following paragraphs.

## SOURCES OF ERRORS IN OLD METHODS AND TECHNIC DEVELOPED TO REMOVE THEM.

## 1. REAERATION DURING THE MANIPULATION AND DURING THE INCUBATION.<sup>3</sup>

It has not been found that any of the inconsistencies of the excess oxygen dilution method can be traced to reaeration during the manipulations if the oxygen content of the diluting water is within one part per million or thereabouts of the saturation value corresponding to the laboratory temperature. The liability of error from reaeration decreases rapidly as the oxygen content of a liquid approaches saturation at the temperature to which it is exposed, since the rate of absorption of oxygen is proportional to the saturation deficit of the liquid. If 37° C. incubation is used it is necessary that the initial oxygen content of the dilutions should be less than 6.6 p. p. m. to avoid loss of oxygen due to supersaturation. The fact that this saturation value differs so much from that of equilibrium at ordinary laboratory temperature renders the likelihood of reaeration great. Incubation at 37° is open to the further objection that, since the oxygen content of a water in equilibrium with air at that temperature is only about 6.6 p. p. m., the range of dilution is quite restricted. The fact that the reaction proceeds much faster at 37° than at 20° is in favor of 37° incubation; the results obtained, however, are not as reliable as those obtained at a lower temperature. An incubation temperature of 20° C. was adopted at the start and followed throughout the course of the studies presented later on in this paper. This temperature more nearly satisfies stream conditions with regard both to temperature and types of bacteria at work, and any error, due to reaeration during the manipulations, is elimi-

<sup>&</sup>lt;sup>1</sup> Fifth Report of Royal Sewerage Commission, App. IV.

<sup>&</sup>lt;sup>2</sup> Lederer, A. The Biochemical Oxygen Demand of Sewages, Jour. Ind. Chem. 6 (1914), 888.

<sup>&</sup>lt;sup>9</sup> Bruckmiller, F. W. Oxygen Demand of Sewages. Jour. Ind. Eng. Chem. 8 (1916), 403.

nated or at least minimized. If tightly fitting stoppers are used there is little chance for reaeration during incubation.

2. (a) LOSS OF OXYGEN AS EVIDENCED BY THE COLLECTION OF GAS UNDER THE STOPPERS OF INCUBATED BOTTLES—(b) VARIATIONS IN THE INCUBATOR TEMPERATURE.

Hale and Melia<sup>1</sup> have claimed that oxygen is liberated during incubation, even if the dilutions are undersaturated with dissolved oxygen at the start of a test, the oxygen being lost on removing the stoppers to add the reagents. Our experience, however, has not borne out this contention. Saturation for distilled water in the presence of air is 9.17 p. p. m. at 20° C. and 760 mm. pressure. For tap water, which was used in all the determinations made, it should be slightly less, due to the presence of inorganic salts. This value is still lowered in the dilutions owing to the presence of the waste itself. Moreover in an incubated bottle CO<sub>2</sub> (or even free N) is probably formed, tending to drive some of the oxygen out of solution. The investigation was started in the month of July, 1915. At this time of the year the temperature of Cincinnati tap water is about 25° C. corresponding to a saturation value of 8.3 p. p. m. for distilled water. Open tubes of a type described later were used in all the earlier determinations. No difficulties, due to the liberation of gases during incubation, were experienced until the following winter. The temperature of the tap water, as drawn, then gradually dropped to about 5° C., the oxygen content at this temperature being over 12 p. p. m. Large air bubbles were then constantly observed. This difficulty, plainly due to supersaturation, was remedied by diluting the tap water with boiled water in quantity sufficient to reduce the oxygen content below saturation at 20° C. A careful examination of 370 incubated bottles, before and after incubation, failed to reveal a single case where a gas bubble was formed when the oxygen content of the diluting water was less than 9.0 p. p. m. at the start of the test. The time of incubation of the above samples varied from 1 to 10 days. It was found later, when the use of tubes was discontinued, that gas bubbles would rarely be found if the initial temperature was adjusted as well as the initial saturation. Those occasionally found were ascribed in part to a lowering of the incubator temperature. The volume change of 300 c. c. of water from 20 to 17° C. is about 0.2 c. c., enough to cause a perceptible gas bubble, but too small, however, to vitiate the results even on the assumption that the entire bubble consists of oxygen, which is probably not the case. If the temperature rises above 20° C.

<sup>&</sup>lt;sup>1</sup>Hale, F. E. and Melia. A Comparison of Methods for Determining Putrescibility or Oxygen Demand. Jour. Ind. Eng. Chem. 7 (1915), 762. Lederer, A. Notes on the Practical Application of the "Saltpeter Method" for Determining the Strength of Sewages. Am. Jour. Pub. Health, 5, 355.

no air bubble will be found, since even if gas is liberated it is displaced by the liquid. The formation of a gas bubble due to evaporation of the liquid incubated may be prevented by keeping the incubator saturated with moisture.

3. OCCASIONAL ANOMALOUS RESULTS NOT READILY EXPLAINABLE,<sup>12</sup> LACK OF AGREEMENT BETWEEN DUPLICATE DETERMINATIONS, AND ERRORS IN MAKING THE DILUTIONS.

The failure to obtain concordant results when duplicate concentrations of a waste are incubated has been found to be due largely to imperfect technic. It was noted in the early experiments that the agreement between the biochemical oxygen demand values of the diluting water was, in general, quite poor. The cause of this poor agreement was definitely ascribed to the use of imperfectly washed bottles. Organic matter adhering to the sides of bottles previously used with greasy wastes is not all removed by simply rinsing with tap water and allowing to drain. The initiation of a routine of washing all bottles with concentrated sulphuric acid (or cleaning mixture), followed by a thorough rinsing with tap water, was accompanied by a marked decrease in the indicated oxygen demand of the diluting water as well as a much closer agreement between duplicate determinations of its value. The necessity for strict cleanliness in all glassware employed need not be further emphasized.

The results obtained with the diluting water were thereafter much more consistent, but poor agreement between duplicate dilutions of the same waste was frequently noted. This was afterwards traced almost wholly to poor mixing in preparing the dilutions. The adoption and rigid adherence to a method of mixing described later led to such close agreement between duplicate determinations that the incubation of duplicates was discontinued.

4. (A) LACK OF AGREEMENT BETWEEN DIFFERENT CONCENTRATIONS: THE LOWER CONCENTRATIONS GENERALLY GIVING MUCH HIGHER OXYGEN DEMAND VALUES 3 4 5-(B) DIFFICULTIES IN GAUGING THE DILUTIONS SO AS TO SECURE DEPLETIONS WITHIN 30 TO 60 PER CENT OF THE OXYGEN PRESENT AT THE START.6

The agreement between different concentrations was not improved, however, by the adoption of proper precautions relating to the use

<sup>1</sup> Lederer, A. The Biochemical Oxygen Demand of Sewages. Jour. Ind. Eng. Chem. 6 (1914), 887

<sup>&</sup>lt;sup>2</sup> Hale and Melia, loc. cit., page 763.

<sup>3</sup> Ibid.

<sup>\*</sup> Bruckmiller, loc. cit., page 404.

<sup>&</sup>quot; University of Illinois Bulletin. Water Survey Series No. 13, volume 14, page 322.

e Hale and Melia, loc. cit., page 763.

of diluting water slightly undersaturated at the laboratory temperature; the rinsing of bottles with acid; and the securing of perfect mixing in making the dilutions. It was found that the lower concentrations gave greater biochemical oxygen demand values than the higher concentrations in practically all cases. It seems reasonable to assume that the amount of oxygen used up during any given interval is directly proportional to the amount of organic matter present at the start. Therefore, the depletions found with any of the concentrations should, when multiplied by the proper concentration factor, give identical results. Whenever five or six concentrations of the same waste were incubated and removed on the same day an ascending series of values was invariably obtained. So consistently did this occur that an effort was made to determine the factors which might tend to cause the lower concentrations to give higher results, assuming that the anomaly was not due to any inherent defect in the method. Apart from the fact that any germicidal effect, due to the waste itself or to products formed during incubation, would be accentuated in the higher concentrations, nothing could be found for a long time that would explain the phenomenon.

Errors, such as loss of iodine during the titrations, dissolved oxygen in the reagents, etc., are largely counterbalancing and negligible. Loss of oxygen during the incubation would have the opposite effect, since this loss would be the greatest in the water blank. A clue to the explanation was found when an analysis of the results revealed the fact that these could be readily harmonized if the concentrations were increased by a constant. Thus in an actual case the concentrations incubated were 0.5, 1, and 2 per cent and the corrected depletions 2.2, 4.2, and 8.2 per cent, the corresponding oxygen demand values being 440, 420, and 410 p. p. m. If 0.05 is added to each concentration, these become 0.55, 1.05, and 2.05 per cent and the biochemical oxygen demand values are all alike; i. e., 400 p. p. m. It is evident that any constant common to each concentration would cancel out if the concentrations were subtracted from each other. The subtraction of the above values gives two independent concentrations: (2-1)=1 per cent and (2-0.5)=1.5 per cent. The subtraction of the corresponding depletions gives (8.2-4.2)=4 and (8.2-2.2)=6 p. p. m. The factor in the first case (concentration 1) per cent) is 100 and in the second case (concentration 1.5 per cent), 100/1.5; the corresponding oxygen demand values are 400 and 400 p. p. m. From another viewpoint a 2 per cent concentration of waste may be regarded as a 1 per cent concentration diluted with a water containing also 1 per cent of the same waste; the system may be extended so that a 5 per cent concentration is regarded as a 4 per cent concentration of waste in a diluting water containing 1 per

cent, etc. In other words, instead of using tap water, as drawn, for making the dilutions, a low concentration of the waste itself might be used for dilution and for the blank. The depletion found for this low concentration would be subtracted from the depletions found for the higher concentrations, these being diminished by the known strength of the low concentration thus used as a blank. In this manner any constant error would cancel out. In practice this method is quite cumbersome, since three concentrations must be incubated to derive two independent values. Moreover, the depletions must be such that the values obtained by their subtraction are quite large or else the percentage error is great. It was found possible, however, to secure a large number of such results. A few typical illustrations are given in the following table:

#### TABLE No. 1.-Table showing the oxygen demand values derived by two different methods of computation.

(a) Computing each analysis separately with a water blank.
 (b) Subtracting the concentrations and the corresponding depletions of two dilutions and computing the oxygen demand on the differences.

Water blank. Computations for first method.					Computations for second method.					Oxygen demand.				
ii.	Ш.	. p. m.	Conc. of waste per cent.	Depletion of con- centra- tion- p. p. m.			nand p. p. m.	Concentration subtracted per cent.	subtracted . m.	Concentration obtained per cent.	Depletion obtained p. p. m.	on factor.	hod p. p. m. <sup>1</sup>	d p. m.
Initial p. p. m.	Final p. p.	Depletion p.	Conc. of wa	As found.	Less blank.	Concentration factor.	Oxygen demand	Concentrati	Depletions p. p.	Concentrati per c	Depletion p.	Concentration factor.	Second method p. p.	First method p.
9.3	8.2	1.1	$     \begin{array}{r}       0.50 \\       1.25 \\       2.50     \end{array}   $	$2.8 \\ 3.9 \\ 5.6$	1.7 2.8 4.5	200 80 40	340 224 180	$\begin{array}{rrrr} 1.25 & -0.50 \\ 2.50 & -1.25 \\ 2.50 &50 \end{array}$	$3.9-2.8 \\ 5.6-3.9 \\ 5.6-2.8$	$.075 \\ 1.25 \\ 2.00$	$1.1 \\ 1.7 \\ 2.8$	133 80 50	147 136 140	340 224 180
8.8	8.6	.2	$   \begin{array}{r}     1.25 \\     2.50 \\     5.00 \\   \end{array} $	2.0 3.3 5.9	$     \begin{array}{r}       1.8 \\       3.1 \\       5.7     \end{array} $	80 40 20	144 124 114	$\begin{array}{rrrr} 2,50 & -1.25 \\ 5.00 & -2.50 \\ 5.00 & -1.25 \end{array}$	3.3-2.0 5.9-3.3 5.9-2.0	${\begin{array}{c} 1.25 \\ 2.50 \\ 3.75 \end{array}}$	1.3 2.6 3.9	80 40 27	104 104 104	144 124 114
8.4	7.9	.5	.125 .250 .500	$2.4 \\ 3.6 \\ 5.9$	1.9 3.1 5.4	800 400 200	$1,520 \\ 1,240 \\ 1,080$	$\begin{array}{r} .250125\\ .500250\\ .500125\end{array}$	3.6-2.4 5.9-3.6 5.9-2.4	$.125 \\ .250 \\ .375$	$1.2 \\ 2.3 \\ 3.5$	800 400 267	960 920 933	1,520 1,240 1,080
8.4	7.1	1.3	.050 .125 .250	$3.2 \\ 4.3 \\ 6.2$	$   \begin{array}{r}     1.9 \\     3.0 \\     4.9   \end{array} $	2,000 800 400	$3,800 \\ 2,400 \\ 1,960$	$\begin{array}{c} .125050\\ .250125\\ .250050\end{array}$	$\substack{4.3-3.2\\6.2-4.3\\6.2-3.2}$	$.075 \\ .125 \\ .200$	$1.1 \\ 1.9 \\ 3.0$	1,333 800 500	1,470 1,520 1,500	3,800 2,400 1,960
8.8	8.4	.4	$.625 \\ 1.25 \\ 5.00$	$2.2 \\ 3.0 \\ 7.6$	$     \begin{array}{r}       1.8 \\       2.6 \\       7.2     \end{array} $	160 80 20	288 208 144	$\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	3.0-2.2 7.6-3.0 7.6-2.2	.625 3.75 4.38	.8 4.6 5.4	160 27 23	128 123 123	288 208 144
8.8	8,2	.6	$.125 \\ .250 \\ .500$	2.3 3.4 5.7	$     \begin{array}{r}       1.7 \\       2.8 \\       5.1     \end{array} $	800 400 200	$1,360 \\ 1,120 \\ 1,020$	. 500 250	3.4-2.3 5.7-3.4 5.7-2.3	$.125 \\ .250 \\ .375$	$   \begin{array}{c}     1.1 \\     2.3 \\     3.4   \end{array} $	*800 400 267	880 920 907	1,360 1,120 1,020
8.6	7.8	8	.010 .020 .040	$3.0 \\ 4.0 \\ 6.0$	2.2 3.2 5.2	$10,000 \\ 5,000 \\ 2,500$	22,000 16,000 13,000	.040020	$\begin{array}{c} 4.0 - 3.0 \\ 6.0 - 4.0 \\ 6.0 - 3.0 \end{array}$	.010 .020 .030	$   \begin{array}{c}     1.0 \\     2.0 \\     3.0   \end{array} $	10,000 5,000 3,333	10,000 10,000 10,000	22,000 16,000 13,000

<sup>1</sup>Only 2 of the 3 values obtained are independent values.

It is evident that in the examples given the same results could have been obtained by assuming slight errors in the determination of the water correction. The excellent agreement, however, between duplicate determinations of the water correction offered no justification for such an assumption. It is possible, however, that the oxygen demand of the diluting water is not the same by itself (in the blanks) as in the dilutions. A plausible explanation of this lies in the fact that the organic matter in the diluting water is already highly oxidized and is subjected to the action of but small numbers of attenuated bacteria; in the dilutions, however, this organic matter is subjected to the action of a greater variety of less attenuated bacteria. If this be the true explanation, the use of the diluting water having no oxygen demand would eliminate errors of this sort. This has been found to be the case and is the basis of the method at present used. Distilled water would appear to be suitable, but that available for the work showed appreciable quantities of ammonia and nitrites; these forms of nitrogen would have a definite oxygen demand in the dilutions and this oxygen demand would be much less in the blanks, the distilled water being presumably sterile. Water drawn from deep wells may behave like distilled water. Moreover, the quantity of water required where much work is being done makes the use of distilled water undesirable. When Cincinnati tap water is stored for a sufficiently prolonged period its oxygen demand is reduced to such a figure that, when it is used for making dilutions, the biochemical oxygen demand values derived from different concentrations of a waste generally agree within a few per cent. It is not necessary, using this method of procedure, to obtain depletions of between 30 and 60 per cent of the oxygen initially present. Concordant results are obtained whenever the depletions are between 20 and 90 per cent if the diluting water has a 10-day oxygen demand of less than 0.6 p. p. m. If the corrected depletions determined upon concentration of 1 and 2 per cent are 0.4 and 0.8 p. p. m., respectively, it is apparent that an error of 0.2 p p. m. would make the biochemical oxygen demand values derived 20 and 30, or 60 and 50, instead of 40, in each case. To obviate errors of this nature the depletions should be greater than 1.8 p. p. m.; i. e., about 20 per cent of the oxygen intially present. Values derived from concentrations showing depletions up to 90 per cent of the initial oxygen are by no means less reliable than those derived from lower depletions. In fact they are probably more reliable as the percentage error is less. This simplifies the method considerably since fewer concentrations need be put up and the same concentration can be used to derive values for different periods of incubation.

## METHOD FOR THE DETERMINATION OF THE BIOCHEMI-CAL OXYGEN DEMAND OF INDUSTRIAL WASTES AND SEWAGE AS DEVELOPED FROM THE EXCESS OXYGEN METHOD.

As a result of these studies there has been developed and employed in the study of sewage and industrial wastes an improved procedure for the determination of the biochemical oxygen demand by the excess oxygen method. The procedure appears to overcome the more serious difficulties that have hitherto been experienced in the analytical details of this method and to give results that are concordant among themselves and sufficiently accurate for the purposes for which they are required. The details of this procedure will be given somewhat fully because of the importance that has been found to attach to a close adherence to detail in every essential operation.

#### APPARATUS.

Twenty-liter carboys .--- To store water for dilution purposes.

Bottles for incubating samples.—Two hundred and fifty c. c. or preferably 300 c. c. capacity, provided with ground-glass stoppers. Since ground-glass stoppers are generally not interchangeable, they should be tied to the necks of the bottles.

Devices to provide for volume changes during incubation or for entrapping liberated oxygen.—If it is desired to use devices of this character, it will be found convenient to use "open tubes" as devised by H. W. Streeter at the Baltimore Sewage Laboratory and later improved by him in connection with the Ohio River Investigation. These tubes are 6 inches long, one-half inch in diameter, of thick glass with edges smoothed in a flame. They are fitted at the center with a piece of heavy rubber tubing 2 inches long, slipping snugly over the tube, and of such thickness as to completely close the neck of the bottle. This device acts, practically, as a one-hole rubber stopper and a piece of glass tubing. To insert, place the moistened tube in the bottle until the rubber touches the liquid, then, holding the index finger over the open end of the tube, depress same into position.

Dilution flasks .- One thousand and five hundred c. c. capacity.

Siphons.—Two siphons are necessary, one for filling the dilution flasks and the other for filling the bottles from the flasks. The first one consists of a piece of glass and rubber tubing (one-fourth inch) bent so that one arm will extend down to the bottom of the carboy containing the diluting water and the other end extending down far enough to work as a siphon. A rubber tubing clamp serves to stop the flow when the flasks are full. The other siphon is used for filling the bottles to be incubated from the dilution flasks. This one consists of three parts—first, the siphon proper; second, a two-hole rubber stopper No. 6 set on the short arm of the siphon so that the end is one-fourth inch above the bottom of the flask; and, third, a short piece of glass tubing bent into a "U" form, one end extending through the rubber stopper and the other end connected with a piece of rubber tubing used to start the siphon.

Incubator.-Capable of regulation within one degree of 20° C.

Measuring flasks.-Graduated to correct for liquid displayed by reagents added.

*Pipettes.*—For adding the reagents below the surface of the liquid, pipettes may be improvised by cutting a 10-c. c. pipette near the end and inserting a rubber gasket in it to hold a small glass tube of the desired length.

#### REAGENTS.

As given in Standard Methods, A. P. H. A. 1917.

#### DILUTING WATER.

As already explained, it is essential that the biochemical oxygen demand of the diluting water should be less than 0.6 p. p. m. at the time of use. Moreover, it should not be supersaturated with dissolved oxygen at the temperature of incubation, and, if tubes are not used, the temperature of the diluting water should be adjusted to that of the incubator. Once the routine is established, diluting water answering the above requirements is easily kept on hand. Distilled water generally has an appreciable oxygen demand due to ammonia and volatile organic compounds and contains nitrites which interfere with the accuracy of the titrations. If the biochemical oxygen demand of the water is greater than 0.6 p. p. m. after 10 days of incubation it should be stored until it is reduced to that figure. Cincinnati tap water, as drawn, has a 10-day oxygen demand of about 1.3 p. p. m. If stored for 7 or 8 days and then used for making the dilutions, the 10-day oxygen demand is reduced to about 0.4 p. p. m. Stored tap water, saturated with dissolved oxygen at the start, may become undersaturated due to its oxygen demand, but will again approach saturation if storage is prolonged. The change in the oxygen content of a water due to gain from or release to the air is very slow if the water is within one part per million or so of saturation. The changes in oxygen content also lag considerably behind the temperature changes. It is desirable that the water contain at least 7.5 p. p. m. of dissolved oxygen at the start of the incubation. Water containing less may, of course, be employed but gives a narrower range of depletion.

Supersaturation is to be guarded against, since, even if devices for entrapping oxygen liberated during incubation be employed, the recovery of gaseous oxygen by the Winkler reagents is slow and uncertain. Contact with the precipitated Mn (OH)<sub>2</sub> can be secured only by the entrance of the dissolved oxygen into solution unless prolonged and emulsifying shaking be resorted to.

Saturation for distilled water, as stated above, is 9.17 p. p. m. at 20° C. in the presence of air. For seawater with 10,000 p. p. m. of chlorine, under the same conditions, saturation is only 8.3 p. p. m. Ordinary tap water should fall within these limits. For Cincinnati tap water saturation at 20° C. is probably 8.9 or 9.0 p. p. m. If the diluting water is stored at a temperature slightly above 20° C. super-saturation is easily prevented. If it must be stored at a temperature below 20° C. it may be brought to saturation at 20° C. before use by adding stored tap water recently boiled and cooled.

The adjustment of the temperature may be dispensed with if tubes are substituted for ground glass stoppers. Tubes should be used unless a carefully regulated incubator is available, as they provide for volume changes during incubation; any contraction meaning an air bubble. Tubes were not used in any of the analyses given in Table No. 3. The temperature of the diluting water was adjusted to about 20° C. using hot water or clean chipped ice as conditions required, allowing for temperature changes during the manipulations. Slight air bubbles were occasionally observed; these, however, as already explained, were too small to influence the results. If tubes are used it is quite unnecessary to use an oil seal, since reaeration through the open end of the tube is negligible.

#### PRELIMINARY DETERMINATIONS.

If the sample is acid or possesses hydrate alkalinity, bacterial activity will be inhibited. Caustic alkalinity may be removed by adding enough NaHCO<sub>3</sub> to form Na<sub>2</sub>CO<sub>3</sub> or else all the alkalinity may be neutralized with HCl and about 50 p. p. m. of bicarbonate alkalinity added. Acidity is neutralized with Na<sub>2</sub>CO<sub>3</sub>. If the diluting water has a high bicarbonate alkalinity this may be sufficient to neutralize the acidity or hydrate alkalinity of the sample in the concentration incubated. In the above cases the sample should be seeded with sewage bacteria.

The initial dissolved oxygen content of the sample should be determined as it furnishes an index of the biochemical oxygen demand of the sample. If no oxygen is present the indication is that the sample is heavily polluted. Wastes which have been heated, however, may be quite free from dissolved oxygen and still show a very low oxygen demand and dissolved oxygen may be present in heavily polluted liquids if the pollution is but recent. If the initial oxygen present is sufficient to take care of the biochemical oxygen demand of the sample, it is incubated without dilution. This is usually the case with river water and frequently so with effluents of good quality for short periods of incubation. If the sample is supersaturated with dissolved oxygen, tubes, as described above, should be used or preferably the excess of oxygen is removed by applying suction to a bottle partly filled with the sample. Supersaturation may be due to the presence of certain oxygen-forming algae or to collection of the sample at a temperature lower than that of incubation. An apparent supersaturation may be obtained if nitrites are present in large quantities and the Rideal modification is not employed. In the presence of atmospheric oxygen the NO formed reacts catalytically to liberate iodine from the excess of KI added. The Rideal modification should be employed in the presence of high nitrites or much organic. matter.

## CHOICE OF CONCENTRATIONS TO BE INCUBATED.

Other things being equal, the choice of the concentrations to be incubated will evidently depend on the biochemical oxygen demand of the sample. If no data are available as to its probable magnitude, a preliminary set may be prepared of the concentrations 5, 1, and 0.2 per cent. This trial set need be incubated but 10 hours to give an approximate upper limit of dilution. Once the upper limit has been decided upon, the number of concentrations to be incubated will depend on the certainty with which the behavior of the sample is known and the number of intervals at which it is desired to remove bottles for titration. The concentrations are to be made each 50 per cent of the next highest until the desired range has been compassed. For most wastes, four concentrations (ratio of highest to lowest = 1:8) are sufficient to insure 1, 5, and 10 day results. Once the method is well in hand, two, or if 5 and 10 day results only are desired, a single concentration is all that need be put up.

#### METHODS OF PREPARING THE DILUTIONS.

(a) By adding definite amounts of the sample to the bottles directly. This requires that the volume of each bottle be accurately known and introduces complications in the computations. If the dilutions to be prepared are small, the sampling error may be considerable. The agreement between duplicates is much better if the following method is used:

(b) A solution is prepared of the desired concentration and the bottles filled with it, using a siphon. If it is desired to titrate an initial sample and to incubate duplicates, a liter flask is partly filled

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with the diluting water and the sample added without special precautions against aeration. The liter flask is then made up to the mark with diluting water, moving the siphon up and down to secure good mixing. The contents of the flask are then transferred to three bottles, using a small siphon, the first portion going over being rejected in order to clean the siphon. The three bottles are first each filled half full, then nearly to overflowing. Stoppers or tubes are now placed in the bottles, which are then ready for incubation.

If several concentrations of the same waste are to be incubated together, it is convenient to put up one stronger than actually desired and to prepare the others from it. Thus, if it is desired to incubate 1, 0.50, 0.25, 0.125, 0.05, and 0.025 per cent concentrations, 25 c. c. of the samples are made up to 500 c. c. with precautions as above. The required concentrations may be obtained by making up to 1 liter 200, 100, 50, 25, and 10 c. c. portions of this solution. The mixture in the 500 c. c. flask may be made more uniform by blowing back the first 50 or 100 c. c. pipetted.

The procedure of first partly filling the bottles and of raising and lowering the siphon while filling the liter flasks is necessary for thorough mixing. In all the manipulations the siphons and pipettes are kept below the surface of the liquid, not so much to avoid reaeration as to prevent mechanical entrainment of air bubbles. If the diluting water has a measurable oxygen demand, blanks should be incubated to determine its magnitude. This demand, as explained elsewhere, is afterwards applied as a correction to the depletions found for the diluted waste. Bottles of the same size as those used for the dilutions are filled with the diluting water, rinsing the siphon before use in order to avoid contamination.

## DETERMINATION OF THE INITIAL OXYGEN CONTENT OF THE DILUTIONS.

The dissolved oxygen content of the diluting water must be determined in all cases, Standard Methods being followed. If the water has been stored for a long time, it is not necessary to employ the permanganate treatment. Given the dissolved oxygen content of the sample, the initial dissolved oxygen content of the dilutions may be computed from the amount of waste added and the known dissolved oxygen content of the diluting water. The three following cases should be borne in mind:

(a) The oxygen content found analytically is the same as that computed; i. e., there is no dissolved oxygen in the sample and the waste has no initial demand due to  $H_2S$ , ferrous compounds, etc. In this case if the concentration is 10 per cent and the initial dissolved oxygen content of the diluting water is 8 p. p. m., the initial dissolved oxygen content of the dilution is obviously 7.2 p. p. m.

(b) The dilution shows a higher dissolved oxygen content than computed; i. e., the waste has some dissolved oxygen in it. The

amount found analytically represents what is present in the bottle at the start of the incubation.

(c) The dilution shows a lower dissolved oxygen content than computed; i. e., the waste has an initial oxygen demand. If the Rideal permanganate modification is not employed an apparent initial oxygen demand may be found owing to absorption of iodine by the organic matter if its concentration is great. The bottle chosen for the determination should be allowed to stand for a short while until this purely chemical part of the oxidation has taken place. If the dissolved oxygen content of a 10 per cent concentration is found to be 6.6 p. p. m., while the dissolved oxygen content of the diluting water is 8 p. p. m., the waste has an initial oxygen demand of (7.2-6.6) (10) or 6 p. p. m. This initial oxygen demand should be recorded separately, as it represents the immediate oxygen requirement of the waste on entering a stream.

If several dilutions are put up at the same time it is not necessary to determine the initial oxygen content of each one; the amount in one is found and the rest computed. If the concentrations are all less than 1 per cent, the value found for the diluting water is assumed unless the initial oxygen demand is being determined. This initial oxygen demand is usually negligible in comparison with the 5 or 10 day biochemical oxygen demand. In converting one-day results to total biochemical oxygen demand figures, the initial oxygen demand should be deducted before applying any factor. It may afterwards be added to the total biochemical oxygen demand value derived. In many cases the permanganate treatment need not be employed in determining the dissolved oxygen content of the dilutions, the amount of organic matter present being too small to interfere. This, however, is best determined for each individual waste.

An alternative method of procedure is to omit all initial titrations of the dilutions. The initial dissolved oxygen or the initial oxygen demand in the sample itself is determined and the dissolved oxygen of the dilutions computed. A little consideration will show that the two methods of computation give identical results.

If tubes are used these are removed after incubation when no air bubble is present. If the tubes are retained, the heavy  $MnSO_4$  solution added first is allowed to settle a few minutes before adding the NaOH and KI mixture to prevent the formation of a precipitate in the upper part of the tube. If the reagents are not added through the tubes the amount titrated is increased to allow for the sample displaced by the reagents. After adding the two reagents mentioned, the bottle is shaken and the precipitated  $Mn(OH)_2$  is allowed to settle until it is contained in the lower half of the bottle. The shaking is then repeated and the precipitate allowed to settle a second time. After removing the tubes, if these are present, acid is

added to liberate the iodine. Iodine diffuses but slowly, and careful mixing is necessary to distribute it uniformly. When the iodine appears to be uniformly distributed, a few c. c. of solution may be removed and the bottle shaken. If the color of the precipitated hydroxide is pure white the sample contains little or no dissolved oxygen. Unless the amount of iodine present is less than corresponds to 1 p. p. m. of dissolved oxygen, 100 c. c. of the sample is all that need be titrated. If a titration is over-run another 100 c.c. portion is available. Starch is not added until the amount of iodine remaining is equivalent to about one-half c.c. of N/40 sodium thiosulphate. If tubes are not retained for the addition of the reagents and 4 c. c. of reagents are added to 300 c. c. bottles, 101.3 c. c. are titrated instead of 100. Once the sample has been measured out the titration should not be delayed. Titrations of 100 c. c. portions from the same bottles, made immediately and after standing for one to two hours at room temperature, gave losses up to 1 p. p. m., or 12 per cent of the true value, the losses being greater with higher concentrations of iodine.

#### COMPUTATIONS.

Given the initial and final oxygen content of a diluted sample over any period of time the biochemical oxygen demand of the sample for that time is the corrected difference times a factor for the concentration. The correction is based on the fact that the diluting water itself has an appreciable oxygen demand and is determined by incubating the diluting water under the same conditions as the diluted sample. The correction should be calculated for the amount of diluting water present. If the concentration is 50 per cent, the correction applied should be but one-half of the oxygen demand value found for the diluting water. If the concentrations are less than 10 per cent the correction may be considered equal to the biochemical oxygen demand of the diluting water and the same for all concentrations.

The concentration factor is the reciprocal of the concentration when expressed decimally. Thus, for a concentration of 5 per cent, or, decimally expressed, 0.05, the factor is 20.

In order to indicate the degree of concordance that is obtainable with two dilutions of the same sample, the results of 115 pairs of determinations made upon tannery waste, and of 89 pairs made upon strawboard waste have been brought together in summarized form in Table 2. Each pair consists of one determination made at some suitable dilution and a second at twice that dilution. It is believed, upon very good theoretical grounds, that the depletion in the first case should be twice that in the second, or, in other words, that the biochemical oxygen demand values obtained by the incubation of any dilutions of the sample, within the range of the analytical procedure will be consistent.

The table is also arranged to bring out a second fact, namely, the independence of the result upon the final degree of oxygen depletion. For this reason the data have been compiled into groups of nearly the same final depletion, as determined by the depletion in the lower concentration. The comparative sample at twice that concentration would therefore have about twice as great a final depletion. The tannery waste samples tested were the crude waste, the partly purified and the final effluents. The biochemical oxygen demand of these samples varied from 20 to 4,000 p. p. m. The 230 determinations are all given the same weight, irrespective of the source of the sample. Organic matter in all stages of oxidation is therefore considered. The strawboard wastes examined were the "beater," "machine," and a mixture of all wastes. The biochemical oxygen demand varied from 15 to 3,000 p. p. m. The incubation period for all wastes was from 5 to 10 days, and the temperature of incubation was 20° C.

The table shows the proportionality between oxygen depletion and the concentration and the absence of any effect of the extent of final depletion on this result.

In the case of the tannery waste samples the sums of the depletions for the two concentrations are 281.8 and 565.8. For the strawboard waste samples the sums are 227.5 and 449.2, respectively. In each case the ratio is nearly 1 to 2, the same as the concentrations compared.

	Tannery wast	æ.		Strawboard waste.				
Numiber of pars.	Range of depletion		epletions to—	Number	Range of depletion	Sum of depletions due to—		
	in the low concordant.	Concord- ant A.	Concord- ant 2A.	of pairs.	low concordant.	Concord- ant A.	Concord- ant 2A.	
8 6 8 7 7 13 6	0.7 to 1.3. 1.5 to 1.7. 1.8 to 1.9. 2.0 only. 2.1 only. 2.2 only. 2.3 only. 2.3 only.	$     \begin{array}{r}       14.6 \\       14.0 \\       14.7 \\       28.6 \\       13.8 \\     \end{array} $	$18.3 \\ 19.2 \\ 30.1 \\ 29.5 \\ 30.1 \\ 57.4 \\ 29.0 \\ 29.0 \\ 29.0 \\$	97578757	0.9 to 1.6. 1.7 to 1.8. 1.9 to 2. 2.1 and 2.2. 2.3 only. 2.4 only. 2.5 only. 2.6 and 2.7.	18.4	22, 8 22, 8 19, 5 30, 0 37, 1 34, 3 24, 5 36, 9	
6 4 8 5 8 4 10	2.4 only. 2.5 only. 2.6 only. 2.7 only. 2.8 only. 2.9 only. 3.1 and 3.2	$ \begin{array}{r} 10.0\\ 20.8\\ 13.5\\ 22.4\\ 11.6 \end{array} $	19.4 41.3 28.2 42.4 22.9 61.1	7 9 10 8	2.8 to 3 3.1 and 3.2. 3.3 and 3.4. 3.5 to 4.3.	20. 4 28. 6 33. 6 30. 4	40.0 56.5 66.1 58.7	
6 9	3.3 and 3.4 3.5 and 4.0	$20.1 \\ 33.5$	40.8 67.1					
115		281.8	565.8	89		227.5	449.2	

TABLE No 2.—Summary of biochemical oxygen demand determinations upon industrial wastes.

 $\frac{281.8}{565.8}$  or  $\frac{227.5}{449.2} = \frac{1}{2}$ -nearly.

The depletions in all groups are nearly proportional to the concentrations and the agreement in any group is within 5 per cent. The individual agreements (not shown here) are somewhat better when the depletions are 2.5 p. p. m. or greater, and do not appear to be any closer when the depletions are between 30 and 60 per cent of the oxygen initially present than when they are between 60 and 90 per cent. In fact, the higher depletions seem to give the most concordant results. In any case the discrepancies are well within the experimental error of titration. The conclusion seems to be warranted that the biochemical oxygen demand values found with different concentrations, using the excess oxygen method, are in very close agreement and that this agreement is not altered when the depletions do not fall within the prescribed 30 and 60 per cent requirement.

## SUMMARY.

1. An intensive study of the excess oxygen method for determining the biochemical oxygen demand of liquid wastes has been made. The method has been modified to give consistent and accurate results with sewage and a variety of industrial wastes such as are produced by tanneries, strawboard mills, breweries, distilleries, canning establishments, dairies, abattoirs, etc., as well as with highly purified effluents.

2. The difficulties encountered are outlined and it is shown that failure to obtain concordant results by the use of the excess oxygen method is largely due to the use of improperly adjusted diluting water, and, in a lesser degree, to errors in making the dilutions.

3. There is presented the detailed method of procedure developed and used by the United States Public Health Service in the study of methods for treating industrial wastes.

4. Results of a large number of routine determinations of the biochemical oxygen demand of strawboard and tannery wastes are summarized. It is shown that the precision of the biochemical oxygen demand values obtained with different concentrations is within 5 per cent if the diluting water has an oxygen demand of less than 0.6 p. p. m. at 20° C. for 10 days and the temperature and oxygen content of the water are adjusted to the incubation temperature.

5. By using water with a very low oxygen demand and adjusting its temperature and oxygen content before making the dilutions it is not necessary to secure depletions within 30 to 60 per cent of the initial oxygen present. The results are perfectly reliable if the depletions are within 20 to 90 per cent of the initial oxygen content.

6. The technic of the method is also simplified considerably since fewer concentrations need be put up and the same concentration can be used to derive values over different periods of time.

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