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BY

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DEPARTMENT OF REGISTRATION AND EDUCATION  
STATE WATER SURVEY DIVISION

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### STATE WATER SURVEY DIVISION

A. M. BUSWELL, *Chief*

## A Carbon Study of Sludge Digestion-Gas from Water

BY T. E. LARSON, C. S. BORUFF AND A. M. BUSWELL

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Some time ago, Buswell and Boruff<sup>7</sup> reported in THIS JOURNAL the volume and weight of gas that are recovered during the quantitative anaerobic digestion of the various constituents found in sewage. These data indicate that the weight of gas produced always exceeds the weight of organic matter digested. (For a few exceptions, see references 7, 8 and 21.)

In earlier experimental plant studies, Buswell and Pearson<sup>9</sup> had obtained 1.25 pounds of gas per pound of volatile matter digested, but they had no definite proof at that time that their sampling and analytical errors were sufficiently low to justify their conclusions. To check these data and to determine possible sampling and analytical errors, they set up a 28-liter laboratory digester. All additions made to and withdrawals from this tank were carefully sampled and analyzed. The first attempt was somewhat of a failure as the tank contents became sour,<sup>10</sup> but the second time normal digestion was established. In summary the unpublished data show satisfactory balances, with a gas production, when corrected for dissolved CO<sub>2</sub> of 7.7 cu. ft. per pound of solids added, or 19.6 cu. ft. per pound of volatile matter digested, which amounts to 1.32 pounds of gas per pound of volatile matter digested. These data were obtained at an average feeding rate of 0.6 gram of total solids per day per liter of tank capacity (0.037 pound per cu. ft. per day).

The above data furnish additional proof that water enters the digestion reaction—the weight of the resulting gas being heavier than the solids removed by the amount of combining water. For further discussion of the mechanism of these reactions, the reader should consult references 7, 8 and 21.

Most of the gas data from large scale treatment plants, when calculated on a poundage basis, show the production of about a pound, or slightly less, of gas per pound of volatile matter digested. The writers feel that the reason for this is due to sampling and analytical errors in determining volume and solids content of the raw and digested liquors, as well as small errors in collecting, measuring and analyzing the gases produced. The difficulty of measuring and collecting representative samples from the large volumes of raw sludge added to, and the overflow and sludge liquors drawn from large digesters is still not recognized by some investigators. The errors involved in these measurements, plus the large amount of CO<sub>2</sub> gas lost to the air when liquors are withdrawn from the digesters, account for the fact that most of the treatment plants report only one pound, or slightly less, of gas per pound of volatile matter digested. Examination

of treatment plant data furnished by Sperry, Larson, Rumsey<sup>7</sup>, and more recently, Kraus<sup>20</sup> (see Table I), however, shows yields of gas the weight of which exceeds the weight of volatile matter digested (111 to 126 per cent).

Errors due to loss of ammonium acetate and ammonium carbonate during the total solids determination are significant<sup>3</sup> and are greater in the digested liquors (overflow and sludge) than in the raw solids, in which the ammonia nitrogen content is much lower; hence, data tend to indicate a greater loss of volatile matter through digestion than actually occurs. This error tends to lower the weight of gas collected per pound of volatile matter rather than to increase it.

Rudolfs,<sup>1</sup> in three laboratory experiments, recovered gas which weighed 91, 101 and 103 per cent, respectively, of the weight of the volatile matter digested. He reports the latter two values to be due to analytical errors, particularly the determination of volatile matter. The weight of a liter of CO<sub>2</sub> at 20° C. and 760 mm. pressure should have been computed at 1.84 grams ( $1.977 \times 273/293$ ) instead of 1.79 grams. Using the correct value increases the yields to 93, 105 and 105 per cent, respectively.

A carbon balance study of sludge digestion, using a digester of sufficient size that the data would be significant, and yet small enough that the sampling errors would not be great, such a study to include carbon determinations on all substances added to and withdrawn from the digester, accompanied by accurate total and volatile solids determinations and gas data, should definitely settle the question as to whether or not the digestion of sewage solids leads to the production of a greater weight of gas than the weight of volatile matter lost through fermentation. To this end, the following studies were made.

### **Wet Combustion Method for Total Carbon**

One of the first things found necessary in this study was to develop a wet combustion method capable of handling sludge. There are many wet combustion methods for determining carbon cited in the literature<sup>13-18</sup> but none was found suitable for samples of raw or digested sludge in which the carbon content may be as much as 40,000 p. p. m. In order to overcome sampling errors, it was thought advisable to use 20 cc. or more of sludge for analysis. This required greater CO<sub>2</sub> absorption capacity than given by any of the methods reviewed.

The apparatus developed and used is illustrated in Figure 1. The samples drawn were always immediately made strongly alkaline with solid NaOH in order to hold all dissolved CO<sub>2</sub>. These samples (20 to 50 cc.) were then slowly added to the warm oxidizing solution in the digester. The oxidizing solution was composed of 200 cc. of conc. H<sub>2</sub>SO<sub>4</sub>, 50 cc. of conc. H<sub>3</sub>PO<sub>4</sub>, 35 cc. of saturated H<sub>2</sub>CrO<sub>4</sub> and 1 cc. of CuSO<sub>4</sub> solution (10 per cent). It was noted that a much more uniform flow of CO<sub>2</sub> could be

obtained by adding the sample to the oxidizing solution rather than *vice versa*. The  $\text{CO}_2$  evolved and a stream of  $\text{CO}_2$ -free air were drawn by suction through a purifying train composed of one tube containing warm  $\text{H}_2\text{CrO}_4$ , to oxidize any small amount of  $\text{CO}$  that might be escaping, a second tube containing  $\text{KI}$  and a small amount of  $\text{H}_2\text{SO}_4$  to remove  $\text{Cl}_2$ , a third tube containing  $\text{Ag}_2\text{SO}_4$  in cone.  $\text{H}_2\text{SO}_4$  to remove  $\text{HCl}$  and any oxides of nitrogen or sulphur, then into a small bottle which acted as a spray trap, from which the  $\text{CO}_2$  was led into the gas scrubber containing 200 cc. of normal  $\text{NaOH}$ . The  $\text{CO}_2$ -free air was continuously drawn through the apparatus at the rate of 60 to 80 small bubbles per minute. After all of the sludge had been introduced, the oxidizing solution was heated to gentle boiling and the aspiration of  $\text{CO}_2$ -free air continued for two to three hours. As a safeguard against loss of  $\text{CO}_2$  through the absorber there were added

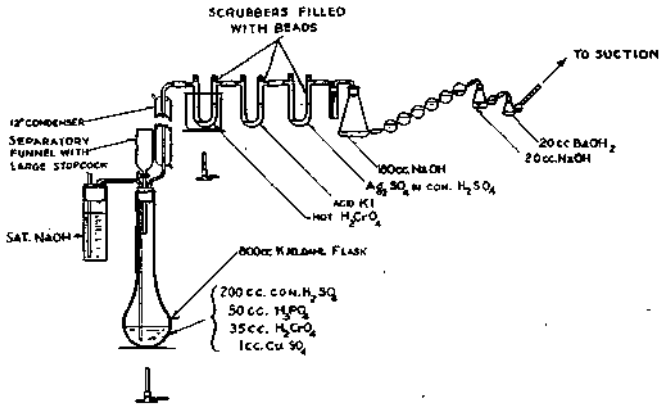


FIG. 1.—Wet Combustion Apparatus for Determination of Total Carbon.

at the exit end of the main absorber two small bulbs containing  $\text{NaOH}$  and  $\text{Ba(OH)}_2$ , respectively. The  $\text{Ba(OH)}_2$  served as an indicator as to the efficiency of the absorption, for if  $\text{CO}_2$  was passing the main absorber the  $\text{Ba(OH)}_2$  would become cloudy. Such being the case, the rate of addition of the sludge was decreased. The  $\text{NaOH}$  and  $\text{Ba(OH)}_2$  used in the small absorbers were always titrated and the amount collected in them, if any, added to that collected in the main absorber.

The carbonate formed in the  $\text{NaOH}$  absorber was precipitated by adding 50 cc. of saturated neutral  $\text{BaCl}_2$ . This mixture was heated almost to boiling and titrated slowly, while hot, with 0.5  $N$   $\text{HCl}$ , using phenolphthalein as the indicator. Thymolphthalein was tried as an indicator but the endpoint was hard to see in the  $\text{BaCO}_3$  suspension. The  $\text{NaOH}$ , to which was added neutral  $\text{BaCl}_2$ , was standardized against the 0.5  $N$   $\text{HCl}$ , which was, in turn, standardized against  $C. P. \text{Na}_2\text{CO}_3$ .

Overflow liquors were analyzed by the same procedure as used for sludges. Less NaOH was necessary to absorb the  $\text{CO}_2$ . The large amount of conc.  $\text{H}_2\text{SO}_4$  used in the oxidizer, namely, 200 cc, was necessary to overcome the effect of the water in the sludge.<sup>15</sup> The  $\text{H}_3\text{PO}_4$  aided in holding the  $\text{SO}_3$  in the hot solution.<sup>13</sup>

On weighed samples of pure dextrose, potassium acid phthalate and acetanilide, 98.6 to 99.9 per cent carbon recoveries were obtained. (See Table I.)

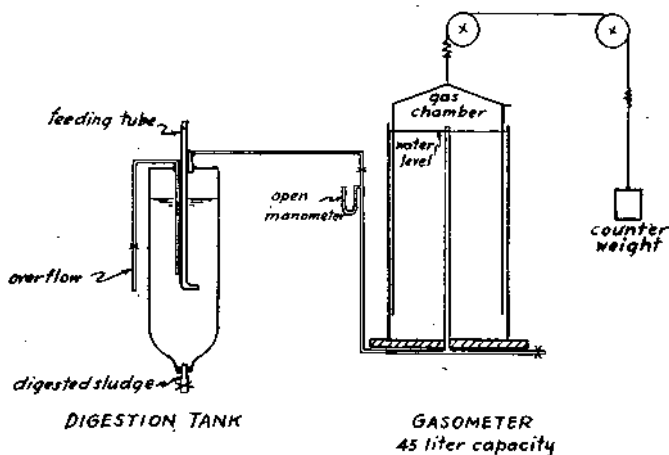


FIG. 2.—Experimental digester.

TABLE I

QUANTITATIVE CHECK ON WET COMBUSTION METHOD FOR TOTAL CARBON

Material Added	Carbon Added, Gram	Carbon Recovered, Gram	Per Cent Recovery
Dextrose	0.6293	0.6234	99.06
Potassium Acid Phthalate	0.8558 <sup>a</sup>	0.8441	98.63
Potassium Acid Phthalate	0.8500 <sup>a</sup>	0.8401	98.83
Potassium Acid Phthalate	0.8057	0.8045	99.9
Potassium Acid Phthalate	0.9885	0.9787	99.0
Acetanilide	0.0815	0.0811	99.57

<sup>a</sup> 0.5 Gm.  $\text{NH}_4\text{Cl}$  also added.

### Sludge Digestion—Experimental

In this investigation, two 10-liter glass bottles were used as anaerobic digestion tanks. They were equipped as shown in Figure 2. The tests were originally started by adding to the tanks digested sludge and overflow liquor from a sludge digestion tank. Fresh raw solids, collected at the Water Survey experimental treatment plant (Urbana-Champaign

sewage), were fed, a liter at a time, three times a week and overflow liquor or digested sludge withdrawn, two liters at a time, just prior to alternate feedings. The rate of feeding at the start was regulated through volatile acid control so that the volatile organic acid content at no time was over 800 p. p. m., calculated as acetic. After normal fermentation was estab-

TABLE II  
SLUDGE DIGESTION; GENERAL DATA

Continuous Feeding, 25-30° C.		
	Tank 1	Tank 2
Volume of Digesters, Liters	9.5	9.5
Days Operated	120	120
Total Solids Fed:		
Weight, Gm.	2242.6	2242.6
Volume, Liters	46	46
Fed, Gm. per Day per Liter Tank Capacity	1.97	1.97
Gas Recovered:		
Total Volume:		
Actual, Liters, S. T. P.	928.2	848.4
Corrected for Dissolved Gas, <sup>b</sup> Liters	970.3	890.5
Total Weight, CO <sub>2</sub> + CH <sub>4</sub> :		
Actual, Gm.	940.3	843.9
Corrected for Dissolved Gas, Gm.	1021.3	921.8
Analysis		
As Recovered, Per Cent by Volume		
CO <sub>2</sub>	25.9	25.0
CH <sub>4</sub>	70.0	70.0
N <sub>2</sub>	4.1	5.0
Corrected for Dissolved Gas, <sup>6</sup> Per Cent by Volume		
CO <sub>2</sub>	29.0	28.4
CH <sub>4</sub>	67.0	66.7
N <sub>2</sub>	4.0	4.9
Gas Produced per Gm. Volatile Matter Fed:		
Actual, Gm. per Gm.	0.632	0.567
	0.625 <sup>a</sup>	0.561 <sup>a</sup>
Corrected, <sup>b</sup> Gm. per Gm.	0.686	0.619
Actual, Liters per Gm.	0.624	0.570
	0.617 <sup>a</sup>	0.564 <sup>a</sup>
Gas Produced per Gm. Volatile Matter Digested:		
Actual, Gm. per Gm.	1.33	1.13
	1.35 <sup>a</sup>	1.15 <sup>a</sup>
Corrected, <sup>b</sup> Gm. per Gm.	1.44	1.23
Actual, Liters per Gm.	1.31	1.14
	1.39 <sup>a</sup>	1.16 <sup>a</sup>

<sup>a</sup> Corrected for probable loss of NH<sub>4</sub>HCO<sub>3</sub> during total solids-volatile matter determination.

<sup>b</sup> Corrected for gain in dissolved CO<sub>2</sub> and CH<sub>4</sub> in liquors withdrawn and gas liberated at end of experiment when tanks were treated with H<sub>2</sub>SO<sub>4</sub>.

lished the acid content remained about 200 p. p. m. and the pH between 7.1 and 7.2. The temperature varied between 25° and 30° C. (77° to 86° F.).

Gas was collected, measured, corrected to standard conditions and analyzed in a modified Orsat apparatus. All liquors fed and withdrawn were analyzed for total solids, volatile matter, ammonia and total nitrogen and total carbon. All analyses were made according to Standard Methods of Water Analysis.<sup>12</sup> Carbon analyses were made by the method already described in this article. A 100-cc. sample of all liquors added and withdrawn was dried and composited for cellulose, lignin and other special tests. (See Table VI.) After a preliminary run of 45 days, the digesters were emptied, sampled and a trial balance made to determine the accuracy of the sampling and analytical procedures. As this trial balance gave satisfactory results, the tank contents were returned to the digesters and the final 120-day digestion experiment reported in this article was started. The air in the small gas space at the top of the digester was swept out with sludge digester gas.

At the end of this run both tanks were dosed and mixed with dilute H<sub>2</sub>SO<sub>4</sub> (to neutrality) to drive off the dissolved and the combined CO<sub>2</sub>. The liquor was then withdrawn without loss of CO<sub>2</sub>. The tanks were washed clean with CCFree distilled water and the washings added to the liquor withdrawn.

**Feeding Rate.**—During the 120-day run, raw sludge was fed at an average rate of 1.97 grams total solids, containing 1.13 grams of carbon, per liter of tank capacity per day. (See Table II.) This is equivalent to 0.123 pound of total solids per cu. ft. of tank capacity per day and is of the general order of magnitude of the loading of digesters in most sewage treatment plants. In this respect the data are more comparable to plant operation data than those collected by Pearson and Buswell, in which solids were fed at an average rate of only 0.04 pound per cu. ft. of tank capacity per day. Rudolfs' carbon studies were made on batch digestions.<sup>1</sup>

**Gas Production.**—As noted in Table II, gas was produced in the two digesters at the average rate of 0.814 and 0.745 liter per day per liter of tank capacity, or 0.414 and 0.378 liter, respectively, per gram of total solids fed (6.6 and 6.1 cu. ft. per pound of total solids fed). This is equivalent to 0.632 and 0.567 liter, respectively, per gram of volatile matter fed (10.1 and 9.1 cu. ft. per pound of volatile matter) or 1.31 and 1.14 liters of gas, respectively, per gram of volatile matter digested (21.0 and 18.2 cu. ft. per pound of volatile matter digested). These gas production data are comparable to the mass of gas production data noted in the literature.<sup>2,11</sup>

**Carbon Balance.**—In the first tank, the weight of the carbon in the inoculum, plus that in the raw sludges fed, amounted to 1210.6 grams (Table III). The carbon left in the tank at the end of the run, plus that



TABLE III  
TOTAL CARBON BALANCE

	Tank 1, Gm.	Tank 2, Gm.
Added:		
Inoculum	190	175
Raw Sludge	1020.6	1020.6
Total	1210.6	1195.6
Withdrawn:		
Overflow Liquors	47.7	46.3
Digested Sludge	320.4	306.3
Final Mixture	331.0	310.0
Total	699.1	662.6
Removed through Digestion	511.5	533.0
Recovered in Gas	478.6	430.3 <sup>a</sup>
Unaccounted for	32.9	102.7
Per Cent Lost	2.7	8.6

<sup>a</sup> Thought to be low due to loss of gas.

found in all the overflow liquors, sludges and gases withdrawn, amounted to 1177.7 grams. This means that a negative error of 2.7 per cent was noted in the carbon balance. This error is within sampling and analytical limits. An 8.6 per cent loss was recorded for the second tank. All the data indicate that some gas must have been lost from the second digester. If the gas data are corrected for the probable gas analysis error mentioned in the next paragraph, the increase in weight due to the CH<sub>4</sub> might be as much as 20.4 and 22.6 grams of carbon, respectively. This would reduce the carbon balance error in the two tanks to 1.03 and 6.7 per cent, respectively.

**Nitrogen Balance.**—The weight of the nitrogen in the inoculum plus that in the raw sludges added to the first and second tank amounted to 91.2 and 89.7 grams, respectively, while the nitrogen left at the end of the run plus that found in the overflow liquors and sludges withdrawn

TABLE IV  
TOTAL NITROGEN BALANCE

	Tank 1, Gms.	Tank 2, Gms.
Added:		
Inoculum Mixture	23.0	21.5
Raw Sludge	68.2	68.2
Total	91.2	89.7
Withdrawn:		
Overflow Liquors	21.6	21.8
Digested Sludge	33.0	30.8
Final Mixture	40.0	35.0
Total	94.6	87.6
Change, Gain or Loss	+3.4	-2.1
Per Cent Change	+3.7	-2.3

TABLE V

## TOTAL SOLIDS AND VOLATILE MATTER DIGESTED

	Tank I				Tank II				Volume Liters
	Total Solids		Volatile Matter		Total Solids		Volatile Matter		
	As Determined, Gms.	Cor- rected, Gms. <sup>d</sup>	As Determined, Gms.	Cor- rected, Gms. <sup>d</sup>	As Determined, Gms.	Cor- rected, Gms. <sup>d</sup>	As Determined Gms.	Cor- rected, Gms. <sup>d</sup>	
Added:									
Inoculum	576.7	605.9	303.9	333.1	522.4	552.8	255.2	285.6	10.0
Raw Sludge	2242.6	2258.3	1488.5	1504.2	2242.6	2258.3	1488.5	1504.2	46.0
Total	2819.3	2864.2	1792.4	1837.3	2765.0	2811.1	1743.7	1789.8	
Withdrawn:									
Overflow	88.9	163.5	51.8	126.4	94.6	168.0	53.8	127.2	35.5
Digested Sludge	996.2	1028.0	506.3	538.1	972.5	1005.1	468.8	501.4	10.0
Final Mixture <sup>e</sup>	1025.0	1017.5	526.0	498.8 <sup>c</sup>	927.5	920.0	474.5	448.1 <sup>e</sup>	<sup>f</sup>
	(1017.5) <sup>b</sup>				(920.0) <sup>b</sup>				
Total	2102.6	2209.0	1084.1	1163.3	1987.1	2093.1	997.1	1076.7	
Digested	716.7	655.2	708.3	695.7 <sup>a</sup>	777.9	718.0	746.6	734.2 <sup>a</sup>	
Per Cent Digested	25.4	22.8	39.6	37.8	28.1	25.6	42.6	41.0	

<sup>a</sup> Corrected for gain in weight (1.30 times) of amino acid group during liquefaction to  $\text{NH}_4\text{HCO}_3$ .

<sup>b</sup> Corrected for conversion of carbonate to sulphate.

<sup>c</sup> Corrected for loss of ammonium sulphate during ignition.

<sup>d</sup> Corrected for probable loss of  $\text{NH}_4\text{HCO}_3$  during total solids-volatile matter determination.

<sup>e</sup> Treated with  $\text{H}_2\text{SO}_4$  to convert  $\text{NH}_4\text{HCO}_3$  and other bicarbonates to sulphates and release  $\text{CO}_2$ .

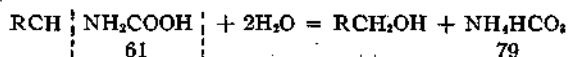
<sup>f</sup> 10.2 liters (Tank 1); 9.9 liters (Tank 2).

mounted to 94.6 and 87.6 grams, respectively. (See Table IV.) There was, therefore, a positive error of 3.7 per cent noted in the nitrogen balance data for the first tank and a negative error of 2.3 per cent in the second tank. These balances are also well within the limits of sampling and analytical errors. Only about 2 grams of the total 47 and 53 grams of nitrogen found in the gas can be accounted for as nitrogen gas dissolved in the sludges added. The rest is undoubtedly due to additive error produced by incomplete combustion in the 23 gas analyses made on the gas produced. If all of this apparent  $N_2$  were actually  $CH_4$  which was not burned in the combustion pipette, this error would account for 20.4 and 22.8 grams of the carbon unaccounted for in the carbon balance of the two digesters. The close balance noted for total nitrogen added and recovered in the liquors further substantiates the previous work of Buswell and Strickhouser that there is little or no free nitrogen ( $N_2$ ) produced during sludge digestion.

**Ash Balance.**—The ash balance shows an 0.8 per cent loss in the first and 3.1 per cent loss in the second tank. There were 1026.9 grams of ash added and 1018.5 grams recovered in the first digester while 1021.3 grams were added and 990.0 grams recovered from the second tank. These data are corrected for the  $H_2SO_4$  added.

**Solids Digested.**—As noted in Table V, 25.4 per cent of the 2819.3 grams of total solids added and 39.6 per cent of the total volatile matter added to the first tank were digested. Likewise, 28.1 per cent of the 2765.0 grams of total solids, or 42.6 per cent of the volatile matter added to the second tank, was digested. These percentage figures are somewhat reduced if the solids data are all corrected for the probable loss of all of the ammonia nitrogen as  $NH_4HCO_3$  during the process of drying of total solids sample. \* It is noted that this correction amounts to less

\* Data in "Corrected" columns (Table V) have been corrected for probable loss of all of the ammonia during the total solids determination as  $NH_4HCO_3$ . Since  $H_2SO_4$  was added to the "final mixture" liquors to convert  $NH_4HCO_3$  to  $(NH_4)_2SO_4$  prior to opening the tanks, and as the latter is non-volatile at  $103^\circ C$ , correction has been made in the total solids data only for the change of carbonate present to sulphate. The figure for volatile matter digested cannot be obtained by merely subtracting the corrected total solids withdrawn from those added, due to the fact that the gain in  $NH_4HCO_3$  in the final liquor represents 130 per cent of the weight of the amino and carboxyl groups originally present in the protein decomposed. The following reaction will serve to illustrate this particular step in the breakdown:



The additional  $NH_4HCO_3$  found in the digested sludges and overflow liquors in Tank I, over that present in the inoculum and raw sludges, was noted to be 94.0 grams. This represents the digestion of 94.0/1.30, or 72.3 grams of original amino and carboxyl groups. The "volatile matter digested" data must, therefore, be corrected for the gain

than 1 per cent for the raw sludge but runs from 2.2 to 3.4 per cent for digested sludges. The error in determining the total solids content of overflow liquors, which contain a high concentration of  $\text{NH}_4\text{HCO}_3$ , as noted in Table V, may be as much as 84 per cent. (Total solids: As determined, 88.9 grams; corrected for loss of  $\text{NH}_4\text{HCO}_3$ , 163.5 grams.)

Table VI gives an idea of the percentage composition of the solids added

TABLE VI  
PER CENT COMPOSITION OF SOLIDS ADDED TO AND WITHDRAWN FROM TANKS<sup>d</sup>

	Inoculum and Raw Sludge	Digested Sludge	Overflow Liquor	Final Tank Contents
1. Hot Water Soluble <sup>a</sup>	10.66	6.63	22.26	8.51
2. Petroleum Ether Soluble	20.8	6.9	1.4	10.5
3. Ethyl Alcohol-Benzene Sol.	7.4	1.35	3.34	1.9
4. Hemicelluloses <sup>c</sup>	1.21	0.73	0.68	1.75
5. Cellulose <sup>c</sup>	3.89	2.3	2.34	4.96
6. Lignin and Humus <sup>c</sup>	8.3	12.05	3.94	10.9
7. Protein <sup>b</sup>	16.5	10.9	17.9	15.8
8. Ash	36.6	50.5	42.4	47.4
9. Total Accounted For	105.36	91.36	94.36	101.72

<sup>a</sup> Corrected for ash.

<sup>b</sup> (Total N less ammonia N) X 6.25 = protein.

<sup>c</sup> Analysis according to Waksman and Stevens, *Ind. Eng. Chem., Anal. Ed.*, 2, 167 (1930).

<sup>d</sup> These analyses made on dried mixture of composites from both tanks; hence, some data vary slightly from individual tank data.

and removed. Analyses 1 to 6, inclusive, were made in succession on the same dried composite sample referred to earlier in this article. Table VII indicates the relative amounts and percentages of the various con-

TABLE VII  
GRAMS OF VARIOUS SUBSTANCES ADDED TO AND WITHDRAWN FROM DIGESTERS

	Inoculum and Raw Sludge	Digested Sludge	Overflow Liquor	Final Tank Contents	Digested	
					Grams	Per Cent
1. Hot Water Soluble	596.0	130.5	40.8	166.0	258.7	43.4
2. Petroleum Ether Soluble	1169.0	135.8	2.6	205.0	825.6	70.6
3. Alcohol-Benzene Sol.	413.5	26.6	6.1	37.1	343.7	83.1
4. Hemicelluloses	67.6	14.4	1.2	34.2	17.8	26.3
5. Cellulose	217.2	45.2	4.3	96.8	70.7	32.6
6. Lignin and Humus	464.0	237.0	7.2	212.8	7.0	1.5
7. Protein	922.0	214.5	32.9	308.4	366.2	39.7
8. Ash	2046.0	994.0	77.8	922.3	51.9	2.6
9. Total	5895.3	1798.0	172.9	1982.6	1941.8	33.0

in weight of 21.7 grams (94.0 — 72.3) before the loss of organic matter through digestion can be calculated. The correct "digested" figure for Tank I would, therefore, be 1837.3 — 1163.3 + 21.7, or 695.7 grams. The volatile matter digested (corrected) for Tank IT was calculated in the same manner.

stituents that were digested. The petroleum ether and alcohol-benzene-soluble fractions are composed mainly of fats and soaps with smaller amounts of waxes and resins. These constituents were removed to a greater extent than any other. They account for 61 per cent of the total loss in organic matter. Thirty-three per cent of the cellulose and 40 per cent of the protein added were removed through digestion. These constituents account for only 4 per cent and 19 per cent, respectively, of the total organic matter digested. These data again indicate that the greater portion of the gas formed during sludge digestion is due to the decomposition of the fats and soaps.<sup>8,9</sup> This is especially true of sewage from the hard water regions and certain other districts, where much fat, grease, or soap enters the sewage. Fatty acids and soaps, due to their low oxygen content, furnish large volumes and weights of gas per unit weight of material digested.<sup>7,8</sup>

The following reaction for the fermentation of glycerol stearate may serve to illustrate:



Each gram of this representative fat upon digestion furnishes 1433 cc. of gas which weigh 1.54 grams and contain 72 per cent methane. The other constituents found in sludge furnish lower volumes and less weight of gas per unit weight digested.<sup>7</sup>

### Summary

As stated in the introduction, the main object of this investigation has been to operate two moderately large laboratory digesters at a normal sludge digestion rate to determine how accurately a carbon balance could be obtained, and to obtain data on gas production. A satisfactory carbon balance has been obtained, especially on the first digester. This indicates that the sludge and liquor samples have been representative and that no excessive volumes of gas have been reported. The ash balances obtained in these studies also indicate that the solids data are accurate. Given this assurance, namely, that the data are accurate and representative, we are now prepared to present the gas data in terms of yield per unit weight of volatile matter digested. As noted in Table II, each gram of volatile matter digested in Tank No. I produced 1.33 grams of gas. This value is increased to 1.44 grams if the gas data are corrected for the  $\text{CO}_2$  held in solution by the liquors or to 1.35 if the solids data are corrected for the probable loss of ammonium salts, as  $\text{NH}_4\text{HCO}_3$ , during drying. (The latter includes only part of the dissolved  $\text{CO}_2$  correction.) Likewise, each gram of volatile matter digested in the second tank produced 1.13 grams of gas. This value, when corrected for increase in dissolved  $\text{CO}_2$ , becomes 1.23 which becomes 1.15 if the solids are corrected for the probable loss of  $\text{NH}_4\text{HCO}_3$ . All conceivable corrections have been

made in the total solids and volatile matter data, yet, as noted, these have little effect on the final data.

The production of 1.44 grams of gas (corrected for dissolved CO<sub>2</sub>) per gram of organic matter decomposed is to be expected from previous investigations.<sup>7,8</sup> Fats and soaps account for 61 per cent of the total loss in organic matter in these digestions; those of higher molecular weight, which are the ones found in sewage, furnish 1.50 to 1.55 grams of gas per gram of material decomposed.

The above data, backed by the experimental plant data already reported by Buswell and Pearson, the academic studies made on the digestion of pure compounds, and the data from a few large-sized plants, should definitely establish the fact that the anaerobic digestion of sewage solids produces carbon dioxide and methane, the weight of which exceeds the weight of the volatile matter removed due to reaction with water. The average weight of gas *collectable* per pound of volatile matter digested is probably about 1.2 pounds. As stated in an earlier communication, differences in the chemical make-up of the solids digested would make some difference. The concentration of the raw solids fed and the digested sludge drawn would also affect this figure in that thinner raw sludges would mean the withdrawal of more overflow liquor or sludge with corresponding greater losses of dissolved CO<sub>2</sub>.

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