

THESIS.

A Study of the Methods of a Sanitary Analasis of Water, with
Special Reference to the Ammonias.

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A Study of the Methods of a Sanitary Analysis of Water,
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The analysis of water, as to its potability, is very different from that of an ore. The Chemist determines the various percentages of iron, zinc, aluminum, etc., and from these determinations he can render an opinion as to the quality of the ore, and need not know anything of the local surroundings from where the ore was taken.

On the other hand, a water analysis is not complete when the parts per million of chlorine, free and albuminoid ammonia are determined but the history of the water must be known. Whether the chlorine and ammonia comes from animal or vegetable origin, where the water is taken from, the local surroundings, etc., must be known to the Chemist before he is called upon for an opinion as to whether the water is potable or not. As Nichols has well said, "It is a great mistake to suppose that the proper way to consult a Chemist is to send a sample of water in a sealed vessel with no hint as to its source. On the contrary, the Chemist should know as much as possible as to the history and source of the water, and, if possible, should take the samples himself."

In this work the writer has followed the methods as given in, Examination of Water, Mason and Water Analysis, Wanklyn.

Collection of Samples of Water.

The water was taken from tap of the City water supply. The faucet was opened and the local lateral emptied before sampling.

Total Solids.

The determination of the amount of total solids in water is a very simple operation. A known quantity of water is evaporated to dryness in a weighed platinum dish, and the residue weighed.

Determination:- The platinum dish is thoroughly cleaned and weighed. Then 70^{cc} of the water is carefully measured into the dish by means of a graduated burette. Evaporate to dryness on a water-bath. Distilled water was used in the water-bath so that no residue would form on the outside of the platinum dish. When dry, the dish was placed in an air bath and heated at 130^{cc} for an hour. It was then cooled in a desiccator and weighed.

If the weight of the empty dish be subtracted, the weight of the water residue will be obtained. One gallon of water weighs 70,000 grains, and 70^{cc} of water weighs 70,000 milligrammes; so that 70^{cc} of water is a miniature gallon, wherein the milligramme corresponds to the grain. Thus knowing the number of milligrammes of residue which 70^{cc} of water leave, we know the number of grains of solids in a gallon of water.

Results:

Total Solids.

#1.		#2.
33.4376	Weight of platinum dish	33.4376
<u>33.4452</u>	Weight of dish and subst.	<u>33.4450</u>
.0076	Weight of substance	.0074
7.6	Total solids, grns. per gal.	7.4
$.058318 \times 7.6 \times 1000 = 443.22$ parts per million.		
$.058318 \times 7.4 \times 1000 = 431.55$ parts per million.		

Chlorine.

Water generally has chlorine in it and it occurs usually in combination with sodium, occasionally with calcium and magnesium. An excessive amount of chlorine in water is generally looked upon as an indication of sewage contamination. Uncontaminated natural water has very little chlorine in it while mine and sewage are comparatively highly charged with chlorine in the form of common salt. Then if a sample of water is found to contain very little chlorine it is an indication that it is not charged with sewage. Where the water is known to come from salt regions an excessive amount of chlorine does not indicate pollution, but in general, when a water shows much chlorine it should be regarded as suspicious.

The determination of chlorine in water depends upon the fact that if to a chloride solution, colored yellow by a little potassium chromate solution as an indicator, a solution of silver nitrate is added, white silver chloride will be produced until the last trace of chlorine disappears, whereupon red silver chromate will appear.

Reagents required:-

Standard Silver Solution: Prepared by dissolving 4.8022 gr. of crystallized silver nitrate in one liter of water. Each cubic centimeter of such a solution is of a strength sufficient to precipitate one milligramme of chlorine.

Potassium Chromate Indicator: Dissolve 2 gr. of the pure salt in 100^{cc} of distilled water.

Determination;- One hundred cubic centimeters of the water is measured into a beaker by means of a pipette. About 1^{cc} of the potassium chromate solution is added, which will give a distinct yellow color, and the standard silver solution is run in from a burette, until the red tint of silver chromate just appears. From the known amount of silver solution used the amount of chloride present is obtained, and this, multiplied by ten, will give the chlorine in milligrammes per liter or parts per million.

Comparates(Mason).

Wanklyn considers 140 as possibly suspicious.

Frankland places the permissible limit at 50.

Leed's standard for American rivers, 3 to 10.

Ordinary sewage, about 110 to 160.

Human urine(av. 24 samples) 5872.

Results:

Chlorine.

The sample used was 100^{cc}. Value of the standard silver nitrate solution used was .00237.

Sample.	AgNO ₃ .	Parts per Million.	Grains per gallon.
1.	1.0 ^{cc}	23.7	1.3821
2.	.7	16.6	.9680
3.	.6	14.2	.8281
4.	.6	14.2	.8281
5.	.6	14.2	.8281
6.	.6	14.2	.8281
7.	.6	14.2	.8281
8.	.6	14.2	.8281
9.	.6	14.2	.8281

Organic Matter---Free and Albuminoid Ammonia.

The determination of solids and of chlorine are only secondary in importance in comparison with the determination of organic matter.

The following chemicals and apparatus are required for the analysis:-

- 1.The Nessler reagent.
- 2.Dilute standard solution of ammonia.
- 3.Solution of potash and permanganate of potash.
- 4.Carbonate of soda.
- 5.Distilled water.
- 6.Retort and Liebig's condenser.
- 7.Bunsen burner and retort stand.
- 8.Nessler glasses.(Graduated cylinders,50^{cc} and 100^{cc}).
- 9.Half liter flask,100^{cc} pipette,25^{cc} and 10^{cc} pipette.
- 10.Graduated burets, and bottles.

The reagents necessary are:-

"Nessler's" Solution:- Dissolve 16 gr.mercuric chloride (HgCl_2) in half a liter of pure water.Dissolve 35 gr. potassic iodide (KI) in 200^{cc} pure water. Pour the first solution into the second until a faint show of excess is indicated. Add 160 gr. solid potassium hydrate (KOH) .Dilute to one liter, and finally

add strong solution of mercuric chloride, little by little, until the red mercuric iodide just begins to be permanent. Do not filter from excess of mercuric iodide, but let it settle to the bottom of the vessel. The finished reagent should have a pale straw color.

"Nessler's" solution will give a distinct brownish-yellow coloration with the most minute trace of ammonia. The reaction is,
 $2(2KI, HgI_2) + NH_3 + 3KOH = NH_4OH + 7KI + 2H_2O$.

Pure Water:- The distilled water furnished in the laboratory was tested and found to be free from ammonia and this was used.

Alkaline Potassic Permanganate solution:- Dissolve 200 gr. solid potassic hydrate (KOH) and 8 gr. crystallized potassic permanganate ($K_2Mn_2O_8$) in 1250 cc of pure water. Boil down to one liter.

Sodic Carbonate Solution :- Dissolve 50 gr. of the pure salt in 300 cc of pure water.

Standard Ammonia Solution:- Dissolve 1.5706 gr. of pure dry ammonium chloride in half a liter pure water. Dilute 5 cc of this solution to half a liter with pure water. This second solution will represent a strength of .01mg. of ammonia per cubic centimeter, and is the standard solution used.

Nesslerizing:- is the process of finding the strength of dilute solutions of ammonia by means of the Nessler test. To 50 cc of the distillate in a "Nessler" jar 2 cc of Nessler reagent is dropped from a graduated burette. If ammonia is present the liquid in the

jar will be a brownish color. The next step is to imitate the depth of color in the distillate. A clean jar or tube is taken and a certain volume of the standard solution of weak ammonia is measured from a burette. This is diluted up to 50^{cc} with pure water and 2^{cc} of the nessler reagent added. After standing five minutes if the colors are the same, then the amount of ammonia in the distillate is known.

Determination of Free Ammonia (Mason).

Place 200^{cc} pure water in the retort and add 10^{cc} of the sodic carbonate solution. The retort must be fitted to the condenser so that the joints will be air tight. Distil off two 50^{cc} jars of water and "Nesslerize", if no ammonia is present in the second jar then the apparatus is clean.

To the contents of the retort add half a liter of the water to be examined.

Distil and catch the distillate in 50^{cc} "Nessler" jars. The rate of distillation should be about 15 minutes for each 50^{cc} jar. "Nesslerize" each jar of the distillate and continue the operation until four jars have been distilled and "Nesslerized".

Albuminoid Ammonia (Mason).

Clean the retort thoroughly after the distillation of the free ammonia and refit it to the condenser. Place in the retort 200^{cc} pure water and 60^{cc} of the alkaline permanganate solution. Distil off three 50^{cc} jars and "Nesslerize", the third one in order to be sure that the apparatus is free from ammonia. Add half a liter of the water to be examined, and proceed with the distillation, and the "Nesslerizing" of the successive 50^{cc} portions of the distillate until six 50^{cc} jars have been distilled. This determination gives the total ammonia (free plus albuminoid); subtract the free ammonia from the total ammonia and the difference will be the albuminoid ammonia.

Results;

Free and Albuminoid Ammonia. (Mason)

Free Ammonia.

1st. "Nessler" Jar	.020 mg.
2nd. " "	.004
3rd. " "	.001
4th. " "	<u>.000</u>
	.025
	<u>2</u>
.002916 gr. per gal. Parts per million-	.050

Albuminoid Ammonia.

1st. "Nessler" Jar	.022 mg.
2nd. " "	.003
3rd. " "	.002
4th. " "	.0005
5th. " "	.000
6th. " "	<u>.000</u>
	.0275
	<u>2</u>
.003208 gr. per gal. Parts per million-	.055 Total NH_3 .
	<u>.050</u>
.000392 gr. per gal. Parts per million-	.005 Albuminoid NH_3 .

Free Ammonia.

1st. "Nessler" Jar	.020 mg.
2nd. " "	.003
3rd. " "	.002
4th. " "	<u>.000</u>
	.025
	<u>2</u>
.002916 gr. per gal. Parts per million-	.050

Albuminoid Ammonia.

1st. "Nessler" Jar	.022 mg.
2nd. " "	.002
3rd. " "	.002
4th. " "	.001
5th. " "	.000
6th. " "	<u>.000</u>
	.027
	<u>2</u>
.003116 gr. per gal. Parts per million-	.054 Total NH_3 .
	<u>.050</u>
.000199 gr. per gal. Parts per million-	.004 Albuminoid NH_3 .

Determination of Free Ammonia (Wanklyn).

The retort having been washed out with a little strong acid, and thoroughly rinsed with pure water until free from acid. Half a liter of water under examination is next measured into the retort and the retort fitted to the condenser.

When 50^{cc} of the distillate have distilled over, the jar is changed and the first 50^{cc} is "Nesslerized". The distillation is continued until 150^{cc} have distilled over, and the 150^{cc} of distillate are thrown away. The recommendation has been given to "Nesslerize" only the first 50^{cc} of free ammonia, and then to add one-third. The first 50^{cc} invariably contains three-quarters of the total amount of free ammonia.

Albuminoid Ammonia.

Fifty cubic centimeters of the solution of alkaline potassium permanganate is added to the contents of the retort and the distillation proceeded with. (The writer has found it profitable to add a few pieces of broken glass to the retort at this stage to prevent bumping). The distillation is continued until three 50^{cc} jars have been distilled and "Nesslerized". This determination gives the total ammonia (free plus albuminoid); if the amount of free ammonia found above be subtracted from this, the difference will be the albuminoid ammonia.

Free and Albuminoid Ammonia (Wanklyn).

Free Ammonia.

1st. "Nessler" Jar	.020 mg.
plus one-third	<u>.006</u>
	.026
	<u>2</u>
.003033 gr. per gal. Parts per million-	.032

Albuminoid Ammonia.

1st. "Nessler" Jar	.023 mg.
2nd. " "	.003
3rd. " "	.002
4th. " "	<u>.000</u>
	.028
	<u>2</u>
.003266 gr. per gal. Parts per million-	.056 Total NH_3 .
	<u>.052</u>
.000233 gr. per gal. Parts per million-	.004 Albuminoid NH_3 .

Free Ammonia.

1st. "Nessler" Jar	.020 mg.
plus one-third	<u>.006</u>
	.026
	<u>2</u>
.003033 gr. per gal. Parts per million-	.052

Albuminoid Ammonia.

1st. "Nessler" Jar	.023 mg.
2nd. " "	.003
3rd. " "	.002
4th. " "	<u>.000</u>
	.028
	<u>2</u>
.003266 gr. per gal. Parts per million-	.056 Total NH_3 .
	<u>.052</u>
.000233 gr. per gal. Parts per million.	.004 Albuminoid NH_3 .