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Government of Madras.

Public DEPARTMENT.

G.O. No.	1123	Press Mis.
Dated	21-7-	1871

Not to be folded or roughly handled

21st July 1871.

No. 191. Read the following Despatch from the Right Honourable the Secretary of State for India, to His Excellency the Right Honourable the Governor in Council, Fort St. George, dated India Office, London, 22nd June 1871, No. 5 (Sanitary):—

I forward, herewith, for the use of your Excellency's Government, one hundred copies of a Scheme of Analysis by Dr. F. N. Macnamara, for the use of Medical Officers on tour for the purpose of examining the Potable Waters of Military and other stations. The Analysis has been sent to the Army Sanitary Commission, and the remarks of that authority will be forwarded to your Excellency in Council.

No. 192. ORDER THEREON, July 1871, No. 1123.

Ordered that the Analysis referred to in this Despatch be distributed as follows:—

	Copies.
Inspector-General, British Medical Service	40
Acting Inspector-General, Indian Medical Department ...	50
Chemical Examiner	1
Sanitary Commissioner	1
Board of Revenue	1
Military Department	1
Public	6
Total...	100

(True Extract.)

Chief Secretary.

To the Inspector-General, British Medical Service.
" Acting Inspector-General, Indian Medical Department.
" Chemical Examiner.
" Sanitary Commissioner.
" Board of Revenue.
" Military Department.
" Public Department,

SCHEME OF ANALYSIS

FOR USE BY
MEDICAL OFFICERS ON TOUR FOR THE PURPOSE
OF EXAMINING THE POTABLE WATERS OF MILITARY
AND OTHER STATIONS,

BY
F. N. MACNAMARA, M.D.,
CHEMICAL EXAMINER TO THE GOVERNMENT OF INDIA.

The history of this compilation is as follows:—In the commencement of 1866 the Government of India decided that the water supply of military cantonments of India should be examined and reported on, and ordered that the work should be carried out by Medical Officers visiting the different stations for the purpose.

In consequence of this order, six Civil Surgeons, who volunteered for the duty, were sent down to Calcutta for instruction in water analysis, and I was directed to prepare a scheme of analysis for use by the analysts when on tour, and to make such other arrangements as might occur to me to be necessary for their guidance and for the furtherance of the duty in hand.

The analysis of the potable waters was begun in September 1866, and the reports of the work done in that and the following six months were published in my first report to the Government of India on the potable waters of military cantonments of the Bengal and adjacent Presidencies.

However, great inconvenience was found in practice to result, in consequence of the temporary removal of Civil Surgeons from their stations, a difficulty which was happily met by employing, in their stead, Assistant Surgeons who had recently come out to India, and who had benefited from the full instruction in the most recent methods of water analysis given by Dr. Parkes at the Army Medical School, Netley. Several of these gentlemen have been employed, and their reports upon their work have been regularly published in the reports to the Government of India, upon the subject of the analysis of potable waters.

In the last report, published in January 1869, when I was on the point of leaving India, I could state that the waters of fifty stations in Bengal, the North-West Provinces, the Punjab, Oude, and the Central Provinces, had been examined and reported on.

On reaching England in September 1869, I learnt that these reports had been submitted by Government to Dr. Angus Smith, whose criticisms and suggestions on them appeared in the annual blue book, published at the India Office, on the Progress of Sanitary Works in India. The perusal of Dr. Angus Smith's remarks led me to request that I might be officially placed in communication with him, with a view to the compilation of a scheme of analysis for general use under Government, for the examination of potable waters throughout India. Subsequently, Government suggested that I should also communicate on the subject with Dr. Parkes, under whom the Medical Officers receive at Netley the instructions necessary to enable them to carry out the scheme of water analysis.

The result of my communication with Dr. Angus Smith, Dr. Parkes, and Dr. de Chaumont, and of the help which I received from those gentlemen in the way of criticism and suggestions, is the scheme for water analysis now published, a scheme which I believe to be well suited for the object in view, namely, the examination, for hygienic purposes, of the water in use in military and civil stations.

With reference to recent controversies regarding the most effective methods of water analysis, it should be clearly understood in regard to the details of the present scheme, that it is not insisted that the scheme embraces exactly those processes which might be recommended as the best for use if the analytical work

GOVT. OF INDIA

PUBLIC.

TO THE INSPECTOR-GENERAL,
BRITISH MEDICAL SERVICE,
&c.,

2 copies only, 21/11/69

Q. Q. 111 100/1871, No. 1123

JULY
Nos. 191-193.

Directing the distribution of the hundred copies received from the Secretary of State of the Scheme of Analysis by Dr. Macnamara for the use of Medical Officers on tour for the purpose of examining the Potable Waters of Military and other stations.

were to be done in a well fitted laboratory, but such as are considered to be best suited for use by Medical Officers who have to carry their apparatus with them, and who must extemporise their laboratories in such accommodation as a dák bungalow or spare barrack room can furnish.

I fear we must acknowledge that the information which our processes of water analysis give us is of positive rather than negative value. I mean that, though we may be able to assert that one water is dangerous, or that another water is naturally good, we are unable to assert from the results of any mode of examination that a water is not dangerous, for we are as yet quite unable to recognize the presence or affirm the absence of special animal poisons, such as those which we have good reason to believe may be active agents in producing many diseases, and on this account I have always strongly insisted, in the instructions which have been from time to time issued to our water analysts, upon the urgent necessity for making their examinations as particular as possible into the circumstances of each water source. This fact being always borne in mind, that it is possible by proper arrangements to eliminate from a water source any danger of artificial contamination. The form of report drawn up for the use of the analysts, which will be found in the Appendix to the scheme, is intended to guide the analyst in his investigations upon this point. It has been my plan to send a supply of these forms to each analyst, requesting him to fill one of them up in reporting upon each water which he may have to analyse.

I believe it to be the wish of the Indian Government that the work of water analysis should be extended to Civil stations, and that it should be actively carried on throughout India. One might dwell at much length upon the advantages which must result to the advancement of sanitary measures in that country from the completion of such a work. Looking to the case of the European Army only, this must be evident, that sanitary measures designed for its benefit must very much fail of their object, so long as cantonments are surrounded, at a greater or less distance, by villages and towns crisscrossed with all sorts of insanitary abominations, and notably with water sources which invite and receive artificial contaminations in every form. The aim of this compilation is to serve as a guide to the detection of danger in the special form of contaminated or impure water; and we may confidently hope that the time has passed away when such dangers, once detected and pointed out, will be lightly thought of, or be allowed to continue.

EXAMINATION AND ANALYSIS OF A POTABLE WATER.*

1. Examination of Physical Properties.

- A. Of fresh water from the source.
- B. Of water after standing in a loosely covered vessel 72 hours:
 - a. Colour.
 - b. Smell.
 - c. Taste.
 - d. Turbidity or clearness.
 - e. Presence of animal or vegetable forms; their nature, and how far detectable by the naked eye.
 - f. Presence of mechanical impurities, whether in large or small quantity, and their nature.

* In some cases it may not be possible to make an analysis *in extenso*. The following points should then be attended to:—

- I. The examination should in all cases comprehend—
 - A. Qualitative analysis.
 - Physical.
 - Chemical.
 - a. Unconcentrated water.
 - b. Concentrated water.
 - B. Quantitative:
 - I. Hardness:
 - a. Total.
 - b. Permanent.
 - c. Removable.

2. Qualitative Analysis of the Water.

A. Of unconcentrated water:

- a. Reaction.
- b. Free carbonic acid.
- c. Sulphuric acid.
- d. Nitric acid.
- e. Nitrous acid.
- f. Chlorine.
- g. Sulphuretted hydrogen.
- h. Lime.
- i. Iron.
- k. Ammonia.
- l. Lead and other metals.

B. Of water concentrated to one quarter of its original bulk:

- a. Reaction.
- b. Nitric Acid.
- c. Iron.
- d. Magnesia.
- e. Phosphoric acid.
- f. Silicic acid.

3. Quantitative Analysis.

A. Hardness:

- a. Total.
- b. Permanent.
- c. Removable.

B. For constituents of water:

- a. Total solids.
- b. Effects of ignition.
- c. Silica.
- d. Oxide of iron, alumina, and phosphates.
- e. Lime.

II. General analysis:

- a. Total solids.
- b. Observation of effects of ignition.
- c. Chlorine.
- d. Sulphuric acid.

III. Amount of Oxygen removed by—

- a. Readily oxidisable matters.
- b. Matters oxidisable at 60° cent. (= 140° Fahr.).

II. In addition to the above, the following may be added under the under-mentioned conditions:—

- (1) In the case of any exceptionally important water sources; (2) when the foregoing examination leaves any doubt as to the character of a water; (3) in the case of two water sources of any station and of a larger number, if many water sources have to be examined in any station; (4) in other cases which the Chemical Examiner at that Presidency thinks proper.

Determination of the amount of—

- Lime.
- Magnesia.
- Nitric acid.
- Nitrous acid.
- Free ammonia.
- Albuminised ammonia (by Wanklyn and Chapman's method).

III. At the discretion of the Analyst, or at the request of the Chemical Examiner at the Presidency, the examination may further be made of—

- Suspended matters.
- Gaseous constituents.
- Silicic acid.
- Oxide of iron, alumina, and phosphoric acid.
- Alkaline chlorides:
 - Chloride of potassium.
 - Chloride of sodium.
- Combined carbonic acid.
- Sulphuretted hydrogen.
- Phosphoric acid.

- f. Magnesia.
- g. Alkaline chlorides.
- h. Sodium.
- i. Potassium.
- k. Sulphuric acid.
- l. Chlorine.
- m. Combined carbonic acid.
- n. Nitric acid.
- o. Nitrous acid.
- p. Ammonia, free, and of salts and of urea.
- q. Albuminoid ammonia.
- r. Amount of oxygen removed from permanganate of potassium by—
 Nitrites.
 Iron (protoxide).
 Gases of putrefaction.
 Organic matter.
- s. Amount of oxygen removed from permanganate of potassium by acidified water heated to 60° cent. (= 140° Fahr.)

4. Determination of Gases in Solution.

- a. Carbonic acid.
- b. Oxygen.
- c. Nitrogen.
- d. Sulphuretted hydrogen.

Collection of the Water.

The water for examination ought to be collected and preserved in a clean stoppered glass bottle, which has previously been well rinsed out with some of the same water as that to be collected. If the bottle is a new one, or if it has been in use for any other purpose than the collection of water, it should be cleaned out with strong sulphuric acid, and then with water till the washings are no longer acid. The water ought to be collected from a spot where it is not subject to artificial disturbance, and in filling a bottle its mouth must be immersed about two feet below the surface of the water.

In taking river water, a spot for the purpose should be chosen in the middle of the stream, in a neighbourhood where the stream is not liable to pollution by the outlet of a sewer, or other source of impurity.

Colour.

To observe the colour of the water, fill two tubes, one with distilled water, the other with the water under examination, and having placed them side by side upon a white surface, compare the colour of the columns of water as the eye looks down through them. The tubes should be of colourless glass, two feet long, and about two inches in diameter. If such tubes are not procurable, then glass flasks may be employed instead. In reporting the colour of a water, state the height of the column of water used in the examination, and specify the shade of colour, whether brown, yellowish brown, greenish, or if any other tint.

Smell.

The smell, if any, of the water, may be best observed by shaking up some of the water in a wide-mouthed bottle, about one half filled, and then inhaling the air in the upper half of the bottle. Subsequently place the bottle, loosely stoppered, upon the sand bath, and raise the temperature of its contents to 100° Fahr. (= 37.78 cent.), then again observe the odour. If the water contains any quantity, the addition of a little solution of caustic potash will develop an unpleasant smell.

Taste.
The taste, if any, of the water, may be best determined when its temperature is about 100° Fahr. (= 37.78 cent.).

Clearness.
The turbidity or clearness of the water must be noted. The presence of suspended matter and its character may be recognized while the water is under examination as regards its colour.

Organized Forms.
Note must be taken of the presence of any organized forms. If any are present, and their nature cannot be learnt by the naked eye, the microscope should be used. In order to test for the presence of nitrogenized matter capable of developing into organized forms, place about a litre of the water in a loosely covered glass vessel. The vessel must be placed in a well-lighted place, and should be allowed to stand at least three days, when both the water and any sediment which may have collected must be examined for organized forms, both with the eye and with the assistance of the microscope. Attention must also be paid to any change in colour or smell which the water may have undergone while standing.

Chemical Analysis of the Water.

The water must be analysed as quickly as possible after its collection. If the specimen has been taken from the source, it must be filtered in the ordinary way prior to analysis, taking care to reject the first portions of the filtrate, which may have served to wash away any soluble matters from the filter paper. But if the specimen has been taken from a receptacle, as, for instance, from a barrack or hospital filter, in which the water has been placed for immediate consumption, it should be analysed at once, without further purification by filtration in the laboratory.

Matter in Suspension.

Let a measured quantity of the water stand in a tall vessel till the suspended matter has thoroughly subsided. Syphon off the clear portion, and transfer the remainder with the sediment to a weighed platinum capsule, in which the whole may be evaporated to dryness and the residue weighed. The weight, less that of the total solids calculated for the bulk of water with which the sediment was evaporated, will be suspended matter, if no hygroscopic salts are present. Otherwise add pure distilled water, and decant thrice, then dry and weigh. The amount of combustible (organic?) matter in the residue may then be determined by ignition. After igniting the residue it should be allowed to cool, and must then be moistened with solution of ammonium carbonate, and subsequently be heated to 190° cent. (= 366° Fahr.) before being weighed.

Qualitative Analysis.

Reaction.—Fill two test tubes with the water and place in one a piece of blue, in the other a piece of red litmus paper. After a lapse of ten minutes note if any change has occurred in the colour of either slip, and if so, note if the change is permanent, or if it disappears after exposure to the air for about ten minutes.

Free Carbonic Acid.—Pour a little of the water into about 8 C.C. of lime water contained in a test tube. If free carbonic acid is present, the precipitate of carbonate of calcium which first forms will redissolve on the addition of a large quantity of the water.

Sulphuric Acid.—Add to some of the water in a test tube a few drops of dilute hydrochloric acid, and then solution of chloride of barium. One and a half grains of sulphates to the gallon of water give no precipitate with the barium salt unless the water be allowed to stand some time. Three grains of sulphates per gallon cause an immediate haze, and after a time a slight precipitate.

Nitric Acid.—Mix cautiously about 5 C.C. of the water with an equal bulk of pure, concentrated, sulphuric acid, and allow the mixture to cool. Then gently drop in a crystal of ferrous sulphate. Nitric acid, if present, in notable quantity, will produce immediately, or within fifteen minutes, a brown discolouration, around the crystal.

Nitrous Acid.—Mix a few drops of acetic acid, solution of starch, and iodide of potassium, and, if the mixture is colourless, which will be the case if the ingredients are pure, add it to about 100 C.C. of the water. A blue colour indicates nitrites.

Starch solution may be prepared by rubbing up some starch in a small amount of cold water into a paste, then adding boiling water, and allowing the solution to stand till a sufficient quantity has been added and can be poured off.

Chlorine.—Add to some of the water a few drops of pure dilute nitric acid, and then solution of nitrate of silver. One grain per gallon of chloride or bromide causes a haze; four grains cause a marked turbidity on the addition of the silver solution. Ten grains per gallon cause a considerable precipitation.

Sulphuric Acid.—Add to some of the water in a test tube, a few drops of potassium dichromate solution. If sulphuric acid is present, the solution will develop a blue colour, or, with a few minutes, a brown colour.

Iron.—Test the water with potassium ferrioxalate solution. If iron is present, a brown colour will be developed.

Lime.—Add to the water with some of the water, a few drops of solution of potassium ferrioxalate and a few drops of solution of ferrioxalate and ferrioxalate of potassium.

With reference to the above tests, it should be noted that the presence of potassium solution for organic matter, etc., must be taken into consideration, as any ferrous salt in the water, as this would develop a brown colour, etc., etc.

Lead.—Boil about 100 C.C. of the water with a few drops of phosphoric acid, transfer to a long test tube, and add a few drops of hydrogen sulphide. A brown discolouration indicates the presence of lead. Other metals as copper, bismuth, or silver, would also cause a brown discolouration with sulphuric acid, but these metals need hardly be looked for in a drinking water, unless there are special circumstances requiring such examination.

Ammonia.—Add Nessler's test to some of the water in a large test tube or flask. The presence of ammonia or other ammoniacal salts is indicated by a yellow or brown colour, or brown precipitate, the intensity of the reaction varying with the amount of ammonia in the water.

Examination of Concentrated Water

About 200 C.C. of the water should be evaporated to one-fourth its bulk, and then tested as regards its reaction, and the presence of nitric acid, in the manner already described.

If the iron test fails to detect nitric acid, apply the Brucine test to some of the concentrated water. The solution of brucine to be used for the purpose contains one part of the alkaloid in 1000 of distilled water. Take 2 C.C. of the solution and mix it with 2 C.C. of the concentrated water; then take some pure concentrated sulphuric acid in a pipette; pass the end of the pipette through the mixture to the bottom of the test tube, and allow the acid to flow slowly out so that it may form a distinct layer beneath the mixture of water and brucine solution.

If nitrates be present, a fugitive pink zone will form at the junction of the two liquids. Prior to employing this test, the reagents themselves should be tested for nitric acid.

Magnesia.—To 10 C.C. of the filtrate, add a little solution of chloride of ammonium, and then sufficient solution of oxalate of ammonium to precipitate all the lime present. Filter, and to the filtrate add a few drops of solution of phosphate of sodium, and sufficient of solution of ammonium to give a strongly ammoniacal smell to the fluid. If magnesia is present, crystals of the phosphate of magnesia and ammonium will deposit within twenty-four hours.

The portion of concentrated water which remains must be tested for phosphoric acid. Add a slight excess of hydrochloric acid, evaporate to dryness in a platinum capsule, and ignite in order to render any silicic acid insoluble. Treat

the residue with about 1 C.C. of dilute nitric acid, filter, and mix the filtrate with an equal bulk of solution of molybdate of ammonium and about half the quantity of pure strong nitric acid. If phosphoric acid is present, a yellow crystalline precipitate will form immediately, or after a short time.

Silicic Acid.—The residue undissolved by nitric acid may be looked upon as silicic acid.

Quantitative Analysis of the Water

Hardness.—For the determination of the hardness of the water by the soap test, a standard solution of soap is required, and, in order to standardize this solution, a water of standard hardness must be made.

The standard water is prepared by dissolving 300 grains of pure selenite in a litre of pure distilled water. This solution is equivalent in hardness to water containing 16 grains of carbonate of calcium to the gallon. On Clark's scale, it is water of 16 degrees of hardness. Each degree of hardness on Clark's scale indicates that a gallon of the water would waste about 10 grains of hard soap. Thus, one gallon of water of 10 degrees of hardness would waste about 100 grains of hard soap. Pure carbonate of calcium (Island spar) may be used in the place of selenite in the preparation of this standard water. Thus: dissolve 230 grains of the carbonate in pure hydrochloric acid, evaporate to dryness, again dissolve in water, again evaporate to dryness. Repeat the solution and evaporation till a perfectly neutral solution of chloride of calcium is obtained. Then dilute with distilled water to one litre.

To prepare the standard soap solution: dissolve some soap (soft potash soap by preference) in a mixture of equal parts of alcohol and distilled water, and if necessary filter the solution. Put 100 C.C. of the standard water into an 8-oz. bottle, and run in some of the soap solution; shake the bottle well, run in more soap solution, shake again, and so on till a permanent lather is produced, a lather which will last unbroken during five minutes, even though the bottle is placed on its side. If the soap solution is too strong, it must be diluted with spirits of wine till 32 C.C. produce the permanent lather with 100 C.C. of the standard water.

In the actual estimation of the hardness of a water, should the hardness exceed 16 degrees, the water must be diluted with two, three, or four times its bulk of distilled water, in order to reduce the hardness below 16 degrees.

Put 100 C.C. of the water under examination into an 8-oz. bottle, run the standard soap solution slowly in, and continue the additions till vigorous shaking produces a lather which will remain permanent during five minutes. Then refer to the Table for the hardness corresponding to the amount of the solution which has been used. Then, if the water has been diluted prior to the employment of the test, multiply the degree of hardness found in the Table by 2, 3, or 4, according to the dilution employed. The number found will be the degree of hardness on Clark's scale.

Clark's Soap Test Table, extended by A. H. Elliott (Fresenius's Quantitative Analysis, 5th edition, 1876, page 252).

Degree of Hardness	0	1	2	3	4	5	6	7	8	9
0	1.4	1.58	1.76	1.94	2.12	2.30	2.48	2.66	2.84	3.02
1	3.2	3.42	3.64	3.86	4.08	4.30	4.52	4.74	4.96	5.18
2	5.0	5.2	5.42	5.64	5.86	6.08	6.30	6.52	6.74	6.96
3	6.8	7.0	7.2	7.42	7.64	7.86	8.08	8.30	8.52	8.74
4	8.6	8.8	9.0	9.22	9.44	9.66	9.88	10.10	10.32	10.54
5	10.4	10.6	10.8	11.02	11.24	11.46	11.68	11.90	12.12	12.34
6	12.2	12.4	12.6	12.82	13.04	13.26	13.48	13.70	13.92	14.14
7	14.0	14.2	14.4	14.62	14.84	15.06	15.28	15.50	15.72	15.94
8	15.8	16.0	16.2	16.42	16.64	16.86	17.08	17.30	17.52	17.74
9	17.6	17.8	18.0	18.22	18.44	18.66	18.88	19.10	19.32	19.54
10	19.4	19.6	19.8	20.02	20.24	20.46	20.68	20.90	21.12	21.34
11	21.2	21.4	21.6	21.82	22.04	22.26	22.48	22.70	22.92	23.14
12	23.0	23.2	23.4	23.62	23.84	24.06	24.28	24.50	24.72	24.94
13	24.8	25.0	25.2	25.42	25.64	25.86	26.08	26.30	26.52	26.74
14	26.6	26.8	27.0	27.22	27.44	27.66	27.88	28.10	28.32	28.54
15	28.4	28.6	28.8	29.02	29.24	29.46	29.68	29.90	30.12	30.34
16	30.2	30.4	30.6	30.82	31.04	31.26	31.48	31.70	31.92	32.14

To determine the permanent hardness of the water, boil 1 litre of the water for half an hour in a flask, the mouth of which is fitted with an upright glass tube, 2 or 3 feet long; the use of the tube will to a great extent prevent loss by evaporation. Allow the boiled water to cool, make up with recently boiled distilled water to the original volume, and then titrate. The temporary or removable hardness is the difference between the total and permanent hardness.

Dr. Parker ("Practical Hygiene," 3d edition, page 45) recommends the use of the soap test as a rapid method for the determination of certain constituents of a water. Should the analyst determine to use this method, he must prepare a soap solution by graduating it with the dilute of barium solution (26 grammes in one litre of water) so that 2 C.C. of the soap solution shall give a lather with 50 of the barytic solution.

The analyst will also require, for the determination of sulphuric acid (SO_4), by this method, a solution of nitrate of barium, containing 26 grms. of the pure nitrate in 1 litre of water.

The process is as follows:—the total hardness is first determined, and then the following processes are employed.

Lime by the Soap Test.—Messrs. Boutron and Boudet have proposed, after determination of total hardness, to precipitate the lime by ammonium oxalate, and then to determine the hardness again. The difference will be owing to lime removed. The difficulty here is to add enough, and not too much, of ammonium oxalate, which itself in excess gives hardness.

"I have found the best way to perform this process is to have a perfectly concentrated clear solution of ammonium oxalate, and to add to 50 C.C. of water one drop for every four measures of soap solution used; then, in other bottles, to add respectively one, two, and three drops more. Then determine hardness of all the bottles, and select the result which gives the least hardness. In this way we can hit on the bottle which contains enough but not too much ammonium oxalate. The water need not be filtered, but it should be allowed to stand at least for three or four hours, or, better still, twenty-four hours, before the hardness is taken.

"Then multiply the difference between the total hardness and the hardness after the addition of the oxalate by the co-efficient for lime; this is .00014, as each measure of the soap solution is equivalent to this amount of lime.

"Example—

Total hardness	52
After lime precipitated	10
Difference	42

$$42 \text{ measures} \times .00014 \times 20 \times 70 = 8.292 \text{ grains of lime per gallon.}$$

"Or, to save trouble, multiply the number of measures by 196; the result is grains per gallon. If carefully done, this result will be near the truth.

Magnesia by the Soap Test.—Boutron and Boudet propose to determine the magnesia by boiling the water from which the lime has been thrown down. All usual elements of hardness except the magnesia are thus got rid of. This is by no means so accurate a process as that of the lime; the lather is formed much less perfectly and sharply, and, in addition, the constitution of the magnesia and soap compound is variable. The result must be considered as quite approximative.

"Take 200 C.C. of water, add to it the number of drops of solution of ammonium oxalate known to be sufficient by the lime experiment; allow to stand for twenty-four hours; filter; boil for half an hour, replace loss by distilled water; allow to cool in the vessel, which should be well corked, and determine hardness in 50 C.C.

"As the lime has been thrown down and all iron removed, and carbonic acid driven off, the hardness is owing to magnesian salts of some kind.

"Calculate as magnesia, the co-efficient of which for each degree of soap solution is .0001, or as magnesium, the co-efficient of which is .00006.

"Example.—Hardness, after driving off carbonic acid by boiling and precipitating lime = 11.

$$(11 - 2) \times .0001 \times 20 \times 70 = 1.26 \text{ grains of magnesia per gallon.}$$

"Or, to save trouble, multiply the number of degrees by 14; the result is grains per gallon.

"Although this result is merely approximative, it is really nearer the truth than the determination by weighing, in the hands of a beginner.

Sulphuric Acid by Soap Test.—This plan was proposed by Boutron and Boudet, and is briefly as follows:—

"The hardness of the water being known, 50 C.C. of the weak barytic solution (= 26 grammes of barium nitrate in one litre of distilled water) are added to 50 C.C. of water, and the mixture is allowed to stand for 24 hours. The hardness (supposing no SO_4 were present) would be exactly equal to the original hardness of the water, and of the barytic solution combined. But SO_4 , being present, barium sulphate is precipitated, and there is a loss of hardness. Each degree of loss equals .0002 of sulphuric acid (SO_4).

"Example—

Original hardness	52
50 C.C. barytic solution	22
	74
After precipitation	71
Difference	12.8

$$.0002 \times 12.8 \times 20 \times 70 = 3.684 \text{ grains per gallon of } \text{SO}_4.$$

"Usually this process gives good results. Occasionally, from some cause of which I am ignorant, the barium sulphate does not precipitate. This does not depend on the amount of sulphuric acid. The ease with which this process is done renders it useful.

Free Carbonic Acid.—Determination by the soap test. In order to get rid of the fallacy from free carbonic acid acting on the soap, Clark recommended that the water should be well shaken in a bottle, so as to disengage some of the CO_2 , and then that the air should be sucked out. But this does not entirely remove the carbonic acid.

"By the soap test the free carbonic acid can be determined in the following way. Throw down all the lime carefully by ammonium oxalate, without adding be an excess, and determine the hardness in 50 C.C. as usual. The hardness will owing to magnesian salts, iron, if it exists (or alumina or baryta in mineral waters), and carbonic acid. If, now, the water, freed from lime, be boiled, and the loss of water replaced by distilled water, the carbonic acid will be driven off. The hardness should be then again determined. The difference between the first and second trials will (if no iron still exist in the water) give the amount of soap solution which had been previously acted on by the carbonic acid.

"Example—

1. Total magnesian and carbonic acid hardness = 12 measures.
2. Magnesian hardness = 7

$$\text{Carbonic acid hardness} = 5$$

"1 measure of soap sol. corresponds to .00022 grammes carbonic acid. Therefore, $.00022 \times 5 \times 20 \times 70 = 1.54$ grains per gallon. As 2.116 inches weigh one grain, multiply the number of grains by 2.116 to bring into cubic inches per gallon.

$$1.54 \times 2.116 = 3.25 \text{ cubic inches.}$$

"Or, to shorten the calculation, multiply the number of degrees of soap solution by .65; the result is the amount of cubic inches per gallon— $5 \times .65 = 3.25$ cubic inches per gallon.

"If much iron exists in the boiled water, it must be determined and its amount deducted, one measure of soap sol. corresponds to .00014 grammes of iron (Fe)."

Determination of Solid Constituents of the Water.

Total Solids.—Evaporate to dryness half a litre of the water*, dry the residue for an hour at 120 cent., (= 248° Fahr.), and weigh—this gives the total solid con-

* Or a litre or more, if special conditions render a larger quantity desirable, as is often the case with soft water containing only a small amount of solids.

residue of the water! The evaporation is preferably carried on from first to last in a platinum capsule, but the evaporation may be carried to a small bulk in a porcelain capsule, and the residue transferred, by the help of a wash bottle and a very flexible steel spatula to the weighed platinum capsule in which the operation is to be completed. Whichever plan is pursued the capsule should be protected during the operation with a piece of filtering paper to keep out dust.

The platinum capsule, with its contents, may be rapidly cooled by contact with a mass of iron, or the capsule may be allowed to cool under a bell glass, over strong sulphuric acid.

Burn off the organic matter at a low red heat, and while doing so, note the degree of blackening the residue undergoes, the rapidity with which the colour is destroyed, and the nature of the ash, which is evolved by the burning organic matter.

Blackness or darkness caused by heat indicates the presence of carbonaceous substances; a rapid whitening shows the presence of nitrates.

Silica.—Treat the ignited residue with dilute hydrochloric acid, taking care to prevent loss by effervescence, dilute with water, and if the silica is in any quantity, or if an exact analysis is required, collect it in a filter, wash, dry, ignite, and weigh.

Oxide of Iron, Alumina, and Phosphates.—To the filtrate add ammonia (free from carbonate of ammonium) in excess. The precipitate, which may be considered to consist of oxide of iron, alumina, and phosphates, may be collected and weighed.

Lime.—If it is not desired to collect the precipitated oxide of iron, add to the alkaline solution oxalate of ammonium in large excess (and when it is necessary some solution of chloride of ammonium), boil and filter, wash, dry, ignite, and weigh. During the ignition of the precipitate, great care must be taken that the temperature of the crucible does not rise above faint redness, scarcely perceptible in daylight.

Moisten the residue with a few drops of a saturated solution of carbonate of ammonium, dry, and apply a gentle heat by moving the crucible about in the flame of a lamp. Finally weigh. The weight of the carbonate of calcium multiplied by .56 will give the weight of lime, CaO , in the precipitate.

Magnesia.—The filtrate from the oxalate of lime should, if bulky, be evaporated down, then treated with ammonia in large excess, and with excess of solution of phosphate of sodium.

The mixture should be well stirred, without touching the side or bottom of the beaker with the stirring rod, and should be set aside for at least 12 hours, to allow the phosphate of magnesium and ammonium to precipitate. The precipitate is then collected in a filter, and thoroughly washed with dilute solution of ammonia (about 1 to 3) dried, and ignited strongly with the ash of the filter in a platinum crucible, and weighed. The weight of the pyrophosphate of magnesium multiplied by .36 (accurately, .36036) gives the weight of magnesia (MgO) in the precipitate.

Alkaline Chlorides.—Evaporate 1250 C.C. to about one sixth, add 2 or 3 C.C. of thin pure milk of lime, so as to give the fluid a strongly alkaline reaction, heat for a little while, then wash the contents of the dish into a quarter-litre flask. If a little insoluble matter adheres to the dish, it is of no importance. Allow to cool, fill to the mark, shake, allow to settle, filter through a dry filter, take 200 C.C. of the filtrate (= 1000 C.C. of the water), transfer to a quarter-litre flask, add carbonate and oxalate of ammonium, fill to the mark, shake, allow to settle, filter through a dry filter, take 200 C.C. of the filtrate (= 800 C.C. of the water), add some chloride of ammonium (to convert sulphate of sodium or potassium into the chloride), evaporate, ignite, and weigh the residual chloride of sodium, and potassium.

If the water contains only a small proportion of sulphuric acid, the addition of the chloride of ammonium will suffice for the conversion of the sulphates into

chlorides. If the proportion of sulphuric acid is large, it is necessary to add at once, before the addition of the milk of lime, a quantity of chloride of barium equivalent to the known amount of the sulphuric acid. Make sure that the tested chlorides when weighed are soluble in water, and give no precipitate with carbonate of ammonium (Fresenius).

Potassium Chloride.—Dissolve the residue in a small quantity of water, add an excess of strong solution of bichloride of platinum, evaporate in a water-bath nearly to dryness, treat the residue with alcohol of sp. gr. .83, cover the dish with a glass plate, and allow it to stand for three or four hours, with occasional stirring. If the liquid has become of a deep yellow colour, this is proof that a sufficient quantity of platinum solution has been added. When the precipitate has settled down, pour off the clear fluid, and wash several times by decantation with small quantities of alcohol. If the process has been properly performed, the precipitate will be in the form of a heavy yellow powder, consisting of minute octahedral crystals.

Finally, dry the precipitate in a water-bath, and weigh in the dish. The weight of the crystalline precipitate, if multiplied by .30507, gives the weight of the chloride of potassium. The weight of the chloride of potassium deducted from that of the mixed chlorides gives that of the chloride of sodium.

Sulphuric Acid.—Add hydrochloric acid and chloride of barium to half a litre of the water in a beaker. Allow the beaker and its contents to stand 12 hours, that the precipitated sulphate of barium may thoroughly settle. Collect the precipitate upon a filter, wash, dry, ignite, and weigh. The weight of the sulphate of barium multiplied by .343 equals the weight of sulphuric acid (SO_3);* multiplied by .611, the weight of sulphate of sodium.

Chlorine.—Evaporate half a litre of the water to about one fourth its bulk. If the water is very alkaline, almost neutralize by the cautious addition of dilute nitric acid. Then titrate, using pure chromate of potassium and a standardised solution of nitrate of silver.

The standard solution of nitrate of silver is made by dissolving .479 gramme of the salt in one litre of distilled water. 1 C.C. of this solution corresponds to 1 milligramme of chlorine.

Combined Carbonic Acid.—Evaporate half a litre of the water to a small bulk, add a few drops of solution of litmus, and run in standard hydrochloric acid till a purplish red tint appears, then boil to drive off carbonic acid, and if the blue colour of the litmus reappears add more of the acid, and so on till a purplish red tint not due to carbonic acid is obtained. The weight of hydrochloric acid in the standard acid used, multiplied by .6028, will give the weight of combined carbonic acid (CO_2 = 44) in the water operated on.

To prepare the standard acid, dilute with distilled water 100 C.C. of strong hydrochloric acid to 1 litre. Take 20 C.C. of the diluted acid, dilute with twice the bulk of distilled water, add solution of nitrate of silver in excess, wash the chloride of silver by frequent decantations. Finally, collect the precipitate in a small weighed porcelain capsule, evaporate to dryness, ignite till the chloride of silver is on the point of fusion, and weigh. The weight of chloride of silver, multiplied by .2544, gives the weight of pure hydrochloric acid in the 20 C.C. of diluted acid operated on.

Or the hydrochloric acid may be titrated by means of the solution of nitrate of silver used in the estimation of chlorine, 1 C.C. of which = 1.0282 milligramme of hydrochloric acid. Previous to the titration the acid should be carefully neutralized by the cautious addition of solution of carbonate of sodium.

Nitric Acid.—The following plan, which is the one recommended by Dr. Paul (Watt's Dictionary of Chemistry, Vol. V., page 1032) is probably, on the whole, the best test that can be employed for the approximative estimation of nitrates.

Take 1,000 C.C. of the water, add a little aluminium chloride and then a little carbonate of sodium, and evaporate slowly to about 100; volatile organic matter will be driven off, while the greater part of the non-volatile organic matter and the iron will be precipitated. Make up with distilled water to 200 C.C., allow to stand, and decant off 100 C.C.; add 450 C.C. of distilled water, and then add permanganate of potassium solution (see page 13), drop by drop, so long as the

* Professor Wanklyn advises a simple form of steam bath. A common two gallon tin can is taken, a perforated cork fitted on the mouth, and a funnel passed through the perforation; the crucible is placed in the funnel, a little roll of paper being placed between the funnel and crucible to let the steam pass. Water is boiled in the tin can.

* If taken as SO_4 , multiply by .411.

† If taken as CO_3 , multiply by .822.

colour is destroyed in a few seconds. The bleaching effects of nitrites upon permanganate of potassium is almost instantaneous, and may therefore be distinguished from the effect of any organic matter which may remain in the prepared solution.

Multiply the amount of permanganate solution used by two, to get the amount per litre. 1 C.C. of the solution of permanganate is equivalent to 2875 milligrammes of NO_x .

If aluminium chloride is not available, add a few drops of solution of chloride of calcium and carbonate of sodium to the water, then two or three grains of alum.

Phosphoric Acid.—The phosphoric acid in water can be best measured by the process described in Fresenius's *Quantitative Analysis* (5th English edition, page 159). One or more litres of the water are to be evaporated to dryness, and the residue, well dried, treated with dilute nitric acid. The acid solution, filtered from silicic acid, is then mixed with a considerable quantity of molybdate of ammonium solution (about 40 parts molybdic acid must be added for every part of phosphoric acid), stir, without touching the sides, and set aside for 12 or 24 hours in a warm place (not better than 40°C. , = 144°Fahr.), then remove a portion of the clear supernatant fluid with a pipette, mix it with an equal volume of molybdate solution, and allow it to stand some time at 40°C. If a further precipitation takes place, return the portion to the main quantity, add more molybdate solution, allow to stand again 12 or 14 hours, and test again. When complete precipitation has been effected, transfer the precipitate to a small filter, remove the rest from the beaker by means of the filtrate, and wash the precipitate with a mixture of equal parts of molybdate solution and water, which should be dropped on in small quantities. Then dissolve in ammonia the precipitate on the filter, wash the latter, and precipitate the solution by the addition of a clear mixture of sulphate of magnesium, chloride of ammonium, and ammonia; the solution should smell strongly of ammonia; allow to stand from 12 to 24 hours in the cold, filter, and wash the precipitate with weak ammonia (one part of the ordinary solution and three parts of water) until the washings are free from chlorine. Finally, dry, ignite, and weigh the pyrophosphate of magnesium.

The molybdate solution for use in the process is prepared as follows:—

Take of molybdate of ammonium	10 grm.
Solution of ammonia, s.g. 0.980	40 C.C.
Strong nitric acid	120 "
Water	40 "

Dissolve the salt in the ammonia by the aid of heat, pour the solution into the nitric acid and water, which have been previously mixed together, and keep the whole at 40°C. for nine hours. Finally, allow to settle and draw off the clear fluid.

Oxidised Nitrogen.—Nitrogen in the form of nitrates and nitrites (by Schulze's process, modified by Messrs. Wanklyn and Chapman). Introduce 100 C.C. of the water into a perfectly clean non-tubulated retort, and add 50 C.C. of a ten per cent. solution of caustic soda. The soda solution must be perfectly free from nitrates. Distil the contents of the retort till the distillate, as tested by Nessler's solution, is absolutely free from ammonia. After the retort and its contents have cooled, introduce a piece of sheet aluminium; now incline the neck of the retort upwards and close the mouth with a cork fitted with a small glass tube containing fragments of glass or porcelain moistened with dilute hydrochloric acid. The glass tube should be about the diameter of a quill, and between 1 and 2 inches long. The hydrochloric acid tube is connected with a second similar tube, but containing pumice stone moistened with strong sulphuric acid, to prevent ammonia from the atmosphere entering the apparatus. The retort thus fitted is allowed to stand about 12 hours. The contents of the first tube are then washed into the retort, the cork is removed, and the retort is connected with a condenser, which has been thoroughly purified. The end of the condenser should dip into a glass cylinder containing 70 or 80 C.C. of distilled water free from ammonia. About half the contents of the retort are distilled over, and the distillate is made up with distilled water to 150 C.C. 50 C.C. of the solution of ammonia thus obtained is now Nesslerised, but if the colour obtained in this first trial is too strong, the final determination must be made in another portion of the solution previously diluted with pure distilled water, and then the quantity of nitric acid and nitrogen may be calculated for the 100 C.C. of water operated on.

The following table (taken from Messrs. Wanklyn and Chapman's book on water analysis) will be of use in converting observed amounts of ammonia into nitrogen or nitric acid.

TABLE showing the amount of Nitrogen and Nitric Acid corresponding to different amounts of Ammonia.

Ammonia, NH_3	Nitrogen	Nitric Acid, HNO_3
1	0.89	3.71
2	1.78	7.41
3	2.67	11.12
4	3.56	14.82
5	4.44	18.53
6	5.33	22.24
7	6.22	25.94
8	7.11	29.65
9	8.00	33.35
10	8.89	37.06
11	9.78	40.76
12	10.67	44.47
13	11.56	48.18
14	12.44	51.88
15	13.33	55.59
16	14.22	59.29
17	15.11	63.00
18	16.00	66.71
19	16.89	70.41
20	17.78	74.12
21	18.67	77.82
22	19.56	81.53
23	20.44	85.24
24	21.33	88.94
25	22.22	92.65

The process of distillation should be used wherever it is possible to do so, but, should the necessary appliances not be at hand, the process without distillation contrived by Messrs. Wanklyn and Chapman, and described by them as follows, may be resorted to (Wanklyn and Chapman on "Water Analysis," p. 40—44).—

"In the process, as we have just described it, there is a *distillation*. Should there be any reason for avoiding a distillation, the following modification may be used:—

"Prepare a soda ley by dissolving 100 grms. of solid soda in water and diluting to a litre, and ensure its freedom from nitrates by taking the soda as free from them as can be obtained in the first instance, and subsequently dissolving a very little aluminium in the cold solution of the alkali.

"Take 200 C.C. of this soda ley and mix it with 200 C.C. of the sample of water, and place in the mixture a bit of metallic aluminium. Allow the aluminium to dissolve in the cold and the liquid to become clear. There will thus be got a liquid containing the original ammonia and the ammonia arising from the nitrates. Every 100 C.C. of this liquid will contain 50 C.C. of the sample of water.

"For comparison, take 200 C.C. of the soda ley and dissolve in it, in the cold, the same quantity of aluminium as before. When the aluminium has dissolved, add 200 C.C. of the sample of water and allow to subside. Every 100 C.C. of the mixture will contain 50 C.C. of the water and 50 of the soda ley. This *comparison liquid* will be like the first one in every respect except one, whereas the first one has the nitrate reduced to ammonia, it will have the nitrates in the unreduced state.

"Decant and determine the ammonia by the Nessler test, using the *comparison-liquid* instead of distilled water, and using the standard ammonia of strength 0.1 milligram., per 1 C.C.

"It will be observed that the *comparison-liquid* contains as much original ammonia as the liquid which is to be compared with it, and that therefore no correction is needed for original ammonia. Before mixing the sample of water with the soda ley, whether to make the liquid to be reduced or the *comparison liquid*, add a very little chloride of calcium so as to get an appreciable quantity of precipitate on mixing with the soda ley.

"It will be understood that when the ammonia originally present in the water is large relatively to that given by the nitrates, as occasionally happens, this plan, without distillation, would not be advantageous.
 "It is, of course, open to the analyst to expel the free ammonia more or less completely by boiling the water before commencing the operation of determining the nitrates."

Determination of Matter easily oxidised in the cold by solution of permanganate of potash.—(Nitrites, sulphuretted hydrogen, ferrous salts, organic matters of unstable constitution, such as those which are largely present during the putrefaction of both vegetable and animal azotized substances.)

Dissolve 395 grammes of permanganate of potash in 1 litre of pure distilled water, each C.C. of the solution is equivalent to .0001 of a gramme of oxygen. 100 C.C. of the solution should be exactly decolourized by 100 C.C. of a freshly prepared solution of crystallized oxalic acid, containing .7875 gramme of the acid in a litre of distilled water. The acid solution should be warmed with a little dilute sulphuric acid prior to the titration of the permanganate solution.

Process.—Place half a litre of the water in a glass beaker standing on a white surface, add 1 C.C. of strong sulphuric acid, and then drop in the permanganate solution, using either a small Bink's burette with a very open scale, or a Mohr's burette fitted with a glass stopcock. Add a drop of the permanganate solution, stir and wait till the colour disappears, then add another drop, stir and again wait, and so on till a colour is produced which is permanent for a minute or two. The quantity of permanganate solution used to produce this result is noted, and the number of C.C. used, less 3 C.C. the quantity which will give a red tint to half a litre of water, multiplied by .0001, gives the amount of oxygen needed to destroy the readily oxidisable organic matters.

Determination of less readily oxidisable Organic Matters.—Take half a litre of the water, add 1 C.C. of strong sulphuric acid, and as much solution of permanganate of potassium as has been needed in the foregoing operation for the destruction of the readily oxidisable organic matters, raise the temperature of the water to 60° cent. (= 140° Fahr.), remove the lamp, and then drop in the permanganate solution, stirring well. Stop the moment the least red tint is produced, when it disappears add a little more until the tint is permanent for half an hour. If any brown colour is produced a little more acid should be added and the process done more quickly, or, if this fail, the water must be diluted with an equal quantity of pure distilled water. Read off the number of C.C. used, inclusive of that added for the destruction of readily oxidisable organic matters, deduct 3 C.C. (the amount of permanganate needed to give a red tint to half a litre of water), and multiply by .0001 to get the quantity of oxygen needed for destruction of oxidisable matters of half a litre of water.

Before operating on the water for determination of the oxidisable matters, it should be allowed to clear itself by standing in a dark place in a covered glass vessel during 24 hours. If a litre of the water be set aside in this way, the upper three-fourth may be syphoned off; it should not be filtered off, at any rate not through a paper filter.*

Determination of the Ammonia obtainable from Water.—Free ammonia is that yielded when the water is distilled after admixture with carbonate of sodium. Albuminoid ammonia is that yielded by subsequently distilling the water with caustic potash and permanganate of potassium. (Messrs. Wanklyn and Chapman's method for determination of the nitrogenous organic matter in water.)

* Drs. Miller and Letheby's method for determination of organic matter by the use of permanganate of potassium:—

Required:—

Solution of permanganate of potassium, strength, .895 gramme in one litre of water.

Solution of hyposulphite of sodium, strength, one gramme in one litre of water.

Solution (fresh) of starch.

Solution of iodide of potassium, strength, about one part in four of water.

Process.—To half a litre of the water in a beaker add 2 C.C. of sulphuric acid and 10 or more C.C. of the permanganate solution. Allow the beaker with its contents to remain during three hours. Then add a few drops, say 10, of starch solution, and as many drops of the solution of iodide of potassium. Stir, and then run in with constant stirring the solution of hyposulphite. Read off the quantity used

Place half a litre of the water in a tubulated retort connected with a Liebig's condenser, the retort should hold from 1200 to 1500 C.C. of water, add 15 C.C. of a saturated solution of carbonate of sodium, and distil off, first, 100 C.C. of water, and then 50 C.C. The 50 C.C. last distilled should be Nesslerized, and, if found to contain more than $\frac{1}{100}$ th of a milligramme of NH_3 , a further 50 C.C. of distillate must be obtained. At the conclusion of the distillation, the 100 C.C. which first distilled over, must be Nesslerized in the manner to be described presently.

Now add to the water remaining in the retort 50 C.C. of a solution of caustic potash and permanganate of potassium, containing 200 grammes of solid caustic potash and 8 grammes of permanganate of potassium to 1 litre of water. Now distil off 200 C.C. Put this aside, distil off 50 C.C. more, and continue the distillation till 50 C.C. of distillate contains less than $\frac{1}{100}$ th of a milligramme of ammonia. Finally, Nesslerize the 200 C.C. first distilled off.

If the water is very bad, containing much organic matter, Messrs. Wanklyn and Chapman recommend that about 600 C.C. of recently distilled water, should be put into the retort with 15 C.C. of saturated solution of carbonate of sodium, and distilled till the distillate is free from ammonia. Then 100 C.C. of the specimen of water are added, and the operation proceeded with, as has been described.

Bad water is apt to bump while boiling with the alkaline solution of permanganate of potassium. When this happens it is well to introduce fragments of broken up tobacco pipe, previously ignited, into the retort.

Messrs. Wanklyn and Chapman (p. 55 of their work) recommend the following plan for the estimation of ammonia, present as such, in water:—

"There are some difficulties in estimating the ammonia, present as such, in water. As we have already remarked, the presence of many substances interferes with the Nessler test, and we have to elude this difficulty. This may be done by operating as follows,—

"Take 500 C.C. of water, add a few drops of solution of chloride of calcium, then a slight excess of potash. Filter, put it into a retort, and distil until the distillate comes over free from ammonia, then make up the contents of the retort with distilled water to their original volume, viz., 500 C.C. Now take 200 C.C. of the original water, treat it with chloride of calcium and potash as before. Then filter, care being taken to have the filter paper well washed before commencing the filtration.

"In this way two samples of water are obtained, the one with the ammonia, as in the original water, and the other without the ammonia, but in every other respect the same as the former.

"This second portion of water is to be used in the place of distilled water to

Foot note, or fly sheet, for Dr. Macnamara's Pamphlet on Water Analysis (page 15).

An adaptation as follows of Messrs. Wanklyn and Chapman's plan may be very conveniently employed for the approximative determination of organic matter in water.

Fit a flask of about 500 c.c. capacity with an india-rubber cork, through which passes one end of a doubly bent glass tube. The other end of the tube is connected by means of a piece of rubber tubing with a small glass or tin lined worm tube, passing through a tub about 9 inches high and 7 inches in diameter. Introduce into the flask 200 c.c. of the water for examination with 30 c.c. of Wanklyn and Chapman's solution of permanganate of potassium and potash, and distil over 100 c.c., which may then be Nesslerized in the usual way. The process thus conducted occupies far less of the operator's time than the old permanganate process, and undoubtedly gives more reliable results.

The same apparatus, using a flask of about 800 c.c. capacity, and 250 c.c. of water, may be very conveniently employed in carrying out Messrs. Wanklyn and Chapman's process for the separate determination of "free" and "albuminoid" ammonia, and the apparatus possesses this advantage, no small one to the travelling water analyst, that it is far more readily portable, and far less liable to breakage, than a Liebig's condenser and retorts.

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Place half a litre of the water in a tubulated retort connected with a Liebig's condenser, the retort should hold from 1200 to 1500 C.C. of water, add 15 C.C. of a saturated solution of carbonate of sodium, and distil off, first, 100 C.C. of water, and then 50 C.C. The 50 C.C. last distilled should be Nesslerized, and, if found to contain more than $\frac{1}{100}$ th of a milligramme of NH_4 , a further 50 C.C. of distillate must be obtained. At the conclusion of the distillation, the 100 C.C., which first distilled over, must be Nesslerized in the manner to be described presently.

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"In this way two samples of water are obtained, the one with the ammonia, as in the original water, and the other without the ammonia, but in every other respect the same as the former.

"This second portion of water is to be used in the place of distilled water to make the Nessler comparisons; as both samples contain the same impurities, they will affect the tint of the Nessler test in the same manner. Thus the error arising from the presence of salts, &c., is avoided."

In making the determinations of organic matter and nitric acid by the methods introduced by Messrs. Wanklyn and Chapman, the operator must ensure the absolute cleanliness of the apparatus which he employs. He should rinse out the vessels himself immediately before using them, employing for the purpose first some of the water under examination, and then pure distilled water. The distilled water should be free from ammonia, and from organic matter which might yield ammonia. All the re-agents also must be carefully tested. Any nitrates in the caustic soda may be destroyed by dissolving a small quantity of aluminium in the solution. The solutions of carbonate of sodium and of alkaline permanganate of potassium should be tested on a sample of pure distilled water before employing

at the instant the blue colour of the iodide of starch disappears. Next, add to half a litre of distilled water the same quantity of sulphuric acid, permanganate of potassium, starch solution, and iodide of potassium as employed with the water under examination. Titrate with the hyposulphite solution. The excess of hyposulphite solution used in the second experiment will represent the quantity corresponding to the permanganate destroyed by the organic matter of the impure water.

Example.—20 C.C. of hyposulphite and 10 C.C. of permanganate solution used in experiment with water. 32 C.C. used in trial experiment with distilled water and 10 C.C. of permanganate solution. 12 C.C. of hyposulphite represent the permanganate destroyed by the organic matter, and as we have learnt that 32 C.C. of hyposulphite solution correspond to 10 C.C. of permanganate solution,—

$$32 : 10 :: 12 : x = 3.75 \text{ C.C.}$$

the amount of permanganate consumed in three hours by the organic matter of half a litre of water. The trial experiment with distilled water must be made each time the process is employed.

them. The Liebig's condenser should be cleaned out immediately before it is used, by distilling a little pure distilled water through it. The operations must be, of course, carried on in a room the air of which is free from artificial contamination by ammonia.

Distilled water free from ammonia can be obtained by re-distilling ordinary distilled water, rejecting that which first comes over, and which will be found to be ammoniacal.

Measurement of Ammonia by Nessler's Test.—Preparation of Nessler's test fluid:—Dissolve 35 grammes of iodide of potassium in 100 C.C. of distilled water. Dissolve 16 grammes of corrosive sublimate in about 300 C.C. of water. Add the latter solution to the former, till the precipitate which forms refuses to dissolve on agitation. Filter, add to the filtrate strong aqueous solution of potash (about 160 grammes of potash will be needed), and make up with distilled water to 1 litre. Finally, add 5 C.C. of a saturated aqueous solution of corrosive sublimate. Put the fluid aside, and when it is clear, decant off the clear fluid from the precipitate. The Nessler fluid thus prepared should be kept in a stock bottle, from which from time to time a small service bottle for immediate use should be replenished.

In addition to the Nessler reagent, we require for the application of the test,—

Standard solution of ammonia, made by dissolving .0315 grammes of chloride of ammonium in water, and making up to 1 litre. One C.C. of this solution equals $\frac{1}{100}$ of a milligramme of ammonia. In practice it is convenient to keep a solution of ten times this strength, and to dilute it when required for use.

Test cylinders or test tubes on feet. These should hold about 160 C.C., and should be marked at 50, 100, 150 C.C.

The pair of cylinders or test tubes used in a measurement should be of the same size and height; the graduating marks must be at the same height in each.

A pipette to measure the standard solution of ammonia; it should be of very fine bore, having consequently a very open scale.

A pipette for the delivery of the Nessler reagent; it should be marked to deliver $1\frac{1}{2}$ C.C.

Process.—Fill one of the cylinders up to the 100 C.C. mark with the solution to be examined, run in $1\frac{1}{2}$ C.C. of the Nessler test, and place the cylinder on a white surface. Observe the tint. Place the second cylinder beside the first, and run into it so much standard solution of ammonia as may be judged necessary to produce the same tint as that already developed in the other cylinder. Then make up with distilled water to the 100 C.C. mark, and add $1\frac{1}{2}$ C.C. of Nessler test. Allow the liquids to stand for ten minutes before deciding that the colouration of both is the same. If it is not, another cylinder must be charged with standard ammonia, distilled water, and Nessler test, and this must be repeated till the colouration of the fluid in both cylinders is equal. Then the amount of ammonia used in the second cylinder represents the amount present in the 100 C.C. of solution under examination.

If the ammonia is too concentrated, precipitation will occur.

If the solution contains more than 1 milligramme of ammonia in 100 C.C. of water the colouration will be too dark for accurate estimation of the equality of the tint in the two cylinders. It is best to work with solutions which do not approach this limit.

If the solution of ammonia is very strong, it must be diluted before Nesslerizing, or the ammonia may be estimated by means of standard hydrochloric acid.

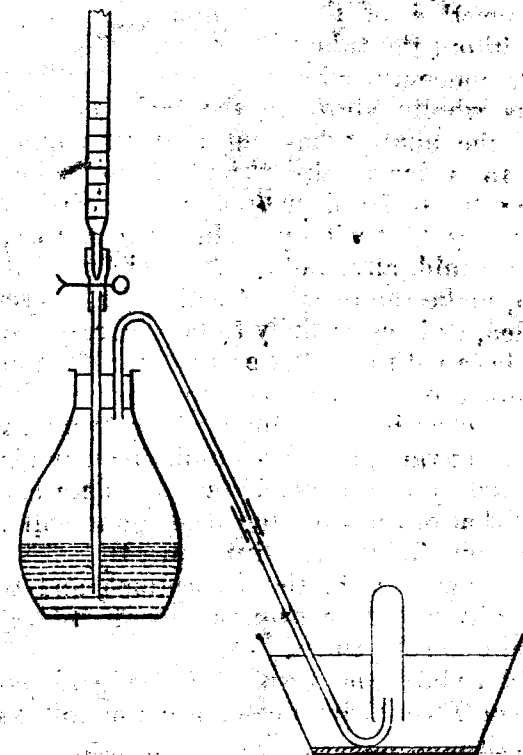
Determination of the Gases held in solution by the Water.—An accurate estimation of the gases (oxygen, nitrogen, and carbonic acid) held in solution by water can be made only by the help of such apparatus as that invented by Bunsen for the purpose, and described in his work on Gasometry, or by the apparatus described by Dr. Angus Smith in the 12th volume of the second series of the Memoirs of the Literary and Philosophical Society of Manchester, or by the apparatus still more lately constructed and described by Mr. Macleod in the August number of the Journal of the Chemical Society for 1869.

But in many cases it may suffice to make an approximative determination of the quantities of the gases held in solution by a water, and such a determination may be effected by the use of an arrangement constructed from the apparatus usually found in a chemical chest.

Take a flask of about 600 or 700 C.C. capacity, and provide it with a well fitting cork, through which two glass tubes pass. One of the tubes, about ten inches in length, is slightly drawn out at one end, which reaches nearly to the bottom of the flask. The other end of the tube is connected, by means of a piece of india-rubber tubing provided with a spring clamp, with a burette, the burette being supported in the ordinary way by a stand.

The other tube, about a foot in length, is bent to a V shape, having a short arm about two inches in length, which is inserted so as just to pass into the flask. The long arm is connected by means of a short piece of india-rubber tubing with another glass tube, similar in length and shape to the first. These two tubes serve for a delivery tube, and when the apparatus is put together, the lowest bend will be a foot or more beneath the level of the bottom of the flask. The lower end of the tube dips into a basin of the water under examination.

The gases which escape from the water in boiling are to be collected in a tube (preferably graduated) of about 30 C.C. capacity.



To use the apparatus.—Let the flask be about half filled with the water which is to be experimented on, make the water boil, and continue the boiling till all the gas has been driven out of the water, that is, till the vapour received into the collecting tube filled with cold water wholly condenses. When this point has been reached, close the mouth of the delivery tube, by pressing a cork down upon it, and at the same moment open the spring clamp; the water in the flask will be forced up into the burette; then the clamp must be closed, while the steam is again allowed to escape from the delivery tube. Invert the collecting tube filled with cold water over the mouth of the delivery tube, charge the burette with the water which is to be examined, open the clamp, and allow from 2 to 2½ C.C. of water to run down from the burette into the flask; owing to the partial condensation which will ensue, a little water may run into the delivery tube; so soon as this has been again completely driven out by the steam, let in another portion of water, and so on till the operator sees he has collected sufficient gas for the purposes of analysis, and till the whole of the water thus far introduced into the flask has been freed of its gases. He will learn from the quantity of water he has introduced into the burette the amount which has yielded the gas which has been collected. The analyst should be careful not to let the whole of the water run out of the burette during the continuance of the operation. In the first instance, it may be well to let the water drawn up from the flask stand at the lowest mark on the burette, and this stage of the operation may be ended by allowing the water to run down to the same point.

If the operator is careful to keep the water in the flask in a state of active ebullition, and is careful not to introduce at one time more than 2½ C.C. or

thereabouts of water into the flask, and if he does not repeat the charges too rapidly, there need be no fear of absorption taking place to such an extent as to risk the entrance of water from the collecting tube into the flask.

To determine the proportion of carbonic acid, nitrogen, and oxygen in the gas thus collected, the following process may be resorted to, if only a small quantity of mercury and no graduated tubes, excepting burettes, are at hand. The mercury bath may be made by cutting a well about one and a half or two inches in diameter, and two inches deep in a block of close-grained wood. When the gas in the collecting tube has fallen to the temperature of the air, the quantity within must be marked, as by a scratch with a file, the water within and without the tube having been in the first place brought to the same level. Next, a small test tube, holding about 10 or 15 C.C. may be filled with the gas by transference from the collecting tube. The open end of the test tube is closed with the finger, and the tube transferred to the mercury bath, a few drops of very strong solution of potash must be thrown up into the tube, and the tube with its contents allowed to stand till the absorption of the carbonic acid gas is complete; then the mercury within and without the tube is levelled, and the point at which it stands marked. Next a little concentrated solution of pyrogallic acid is thrown up, and when the oxygen has wholly absorbed the level of the mercury in the tube is again marked. If the analyst has but a small supply of mercury, it may happen that, owing to a large absorption of carbonic acid or oxygen, the absorption tube cannot be so far depressed as to bring the mercury within the tube to the same level as that without. In such a case, if the analyst has a barometer at hand, he would, after noting the height of the mercury in the tube above that in the well, make the usual correction for difference of pressure. But if he is not so provided, as is very likely to be the case under the circumstances which this scheme is designed to meet, he must transfer the tube with its contents to the water bath, allow the mercury to escape, and mark the quantity of gas in the tube. Then, after charging the tube afresh from the collecting tube, the carbonic acid, with the oxygen, may be simultaneously absorbed by the addition of solution of potash and pyrogallic acid, when the difference between the amount of this absorption and that of the carbonic acid alone will indicate the proportion of oxygen in the gaseous mixture. Subsequently, the quantities of the gas collected in the tube, the amount of the absorptions, and the remaining nitrogen, may be learnt by running in water from a burette to the height of the marks which have been made upon the tubes.

The analyst should reduce the results of the gaseous determination to the standard temperature of 0° cent., by a calculation as follows:—

As 273 + observed temperature : 273 :: observed : volume at 0° cent.
in degrees of cent. volume

For example: What is the corrected volume of 30 C.C. of a gas at 15° cent.?

$$273 + 15 : 273 :: 30 \text{ to } x = 28.44 \text{ C.C.}$$

Professor Miller found that Thames water contained the following amount of gases in C.C. per litre in its flow down stream:—

	Kingston.	Hammer-smith.	Hammer House.	Greenwich.	Woolwich.
Carbonic acid - -	30.3	—	45.2	55.6	48.3
Oxygen - - -	7.4	4.1	1.6	.25	.25
Nitrogen - - -	15	15.1	16.2	15.4	14.6

Sulphuretted Hydrogen.—This can be determined by a solution of iodine in iodide of potassium. Dissolve 6.35 grammes of iodine in a litre of water by the aid of a little iodide of potassium. Make a solution of starch by pouring a little boiling water on good starch, then rubbing it well with cold water and filtering so as to get an almost clear solution. Take one litre of the water to be examined, put in some starch solution, and drop in from a burette the iodine solution until there is the slightest possible blue colour. Read off amount of iodine solution used,—

1 C.C. of iodine solution = .0008 grammes of sulphur.
" " " = .00085 grammes of S.H.

ANALYST'S REPORT.

GENERAL.

1. Period during which the waters were examined
2. Average yearly rainfall
3. Rainfall of the week preceding the commencement of the analyses
4. State of weather at the time of commencing the examination of the water
5. State the general features of the adjacent country, noticing—
 - a. Rivers and watercourses
 - b. Undulation or flatness of the ground
 - c. Contiguity of any hills
 - d. Direction of the watershed with reference to the source from which the water is taken
 - e. Geological formation of the hills and adjacent country
 - f. Fertility or barrenness of the soil
 - g. The existence of forest land
 - h. Is the country well wooded with large trees?

Note.—Answers to the questions in the "General Report" will be inserted in only one of the Reports sent from each station.

* The Analysts should be provided with a sufficient number of copies of this Report to allow of one being filled up in the case of each water examined.

6. Remarks of the people regarding the water of the station as to its purity, wholesomeness, sufficiency, &c.

7. Special prevalence of any endemic or epidemic diseases among the people who use the water, e. g., diarrhoea, dysentery, cholera, malarious fever, spleen diseases, typhoid fever, diseases of the skin, calculi, goitre, tenia, lumbricoid, or guinea-worms

DETAIL.

*I. SOURCE OF WATER.—A WELL. ITS POSITION, DATE ON WHICH ANALYSIS OF THE WATER WAS BEGUN.

1. A shaft without any masonry, commonly called a "kutchra well"

2. Pucka brick without lime mortar

3. Pucka brick with lime mortar

4. The existence of any ridge to prevent spilled water from falling back into the well

5. Is there a masonry platform around the well to throw off the water which is spilled during drawing, ablutions, &c., and thus prevent it from immediately trickling down the outside of the masonry and returning to the well?

6. Give the diameter of the platform, and state if there is any drain in or around it to carry off the water

7. Date when the well last dried up, or when the supply was so much diminished that it became dry in the middle of the day, while drawing water for usual consumption

8. Does the depth of water in the well diminish very much during the hot dry months?

* Each water source should be so described as to be readily identified on a subsequent visit by the same or another Analyst; and a record should be left in the station of the sources from which water has been taken for analysis.

9. Method of drawing water -

10. Date when the well was last cleaned -

11. Is the well covered in so as to exclude dust, leaves, &c.; if so, by what?

12. Distance from the surface of the ground to the water -

13. Depth of water in the well -

14. Diameter (interior) of the well -

15. By whom is the water used, and for what purposes?

16. Remarks of those using the water of this well, as to its purity, wholesomeness, &c. -

17. Note the proximity of—

a. Any dwelling-houses, barracks, coolie houses, latrines, or urinals, and state their probable distance from the well -

b. Also of any trees, or accumulation of refuse matter, filth, mud, water, or manure -

18. c. The existence of any saucer-shaped depressions (in which water is liable to lodge), ponds, tanks, drains, water-courses, or rivers in the vicinity, and give their respective distances from the well -

19. Nature of the soils through which the well has been sunk -

a. Surface soil -

1. Alluvium -

2. Marl -

3. Clay -

4. Calcareous -

5. Sandy

6. Humus

7. Oosur, or soils which have a saline efflorescence on the surface

b. Subsoil:—

1. Granite, metamorphic, or trap

2. Clay

3. Limestone or Kunkur

4. Sandstone

5. Sand

6. Gravel

II.—SOURCE OF WATER.—A TANK: LOCATION, AND DATE ON WHICH THE WATER WAS BROUGHT.

1. Give the measurement of the tank, side, or, if square, of one side, and depth in centre

2. Are the sides of the tank of earth, or of pukka masonry?

3. The presence of water plants

4. Their nature

5. The existence of fish

6. Supply of water how maintained, by rain, or by water derived from rain draining from the adjacent land?

7. Is the supply perennial, and does it continue good throughout the year?

8. State the depth of the water in the centre of the tank at the time of your visit, and also so far as you can learn it, the depth when the water is at its highest and lowest level in the tank

9. Purposes for which the water is used, viz, ablution and washing clothes, for drinking or culinary purposes, or all

10. Are cattle bathed or watered in the tank, and is jute, sunn, or any of the fibres, or other products steeped or washed in it?

Remarks of those using the water of this tank regarding its purity, wholesomeness, &c.

By whom is the water used?

Note the proximity of any dwelling-houses, cattle sheds, latrines, large trees, accumulations of refuse matters, filth, manure, and state the probable distance

Do any drains open into the tank, to how far from it do they extend; do they run in the neighbourhood of any bazaar or other possible source of pollution?

5. Nature of the soil in which the tank has been made

a. Surface soil:—

1. Alluvium

2. Marl

3. Clay

4. Calcareous

5. Sandy

6. Humus

7. Oosur, or soils which have a saline efflorescence on the surface

b. Sub-soil:—

1. Granitic, metamorphic, or trap

2. Clay

3. Limestone

4. Sandstone

5. Sand

6. Gravel

III.—SOURCE OF WATER*—A RIVER OR SPRING.

Position of the source with reference to townships, and date on which the analysis was begun.

1. Name of river -
2. Tidal or otherwise -
3. Geological formation at the source -
4. Nature of bed -
5. Depth of water at the time of commencing the examination -
6. Velocity of the stream -
7. Distance from any native city at which the water was drawn, and whether it was taken from above the city or below it -
8. By whom and for what purposes is the water used? -
9. Is the suspended matter in large amount, and is it allowed to subside before use, or is the water usually drunk in the condition in which it is drawn? -
10. Remarks of those using this water as to its purity, wholesomeness, &c. -
11. Is the water equally good throughout the year? -

ANALYSIS OF THE WATER.

1. Qualitative Analysis.

A. Physical examination:

a. Of fresh water from the source,—

1. Colour -
2. Smell -
3. Taste -
4. Turbidity or clearness -

* The water of any river that may flow within a distance of five miles from the station should be analysed. The water for analysis should be taken from a point above any city or town on its banks, and from a spot where it is not liable to be contaminated by sewage.

5. Presence and nature of animal, or vegetable forms -

6. Presence of mechanical impurities, whether in large or small quantity, and their nature -

b. Of water after standing 72 hours,—

1. Any change in physical properties of water -
2. Development of organized forms, their nature, and if in large or small quantity -

B. Chemical analysis:

a. Of unconcentrated water,—

1. Reaction -
2. Free carbonic acid -
3. Sulphuric acid -
4. Nitric acid -
5. Nitrous acid -
6. Chlorine -
7. Sulphuretted hydrogen -
8. Lime -
9. Iron—ferrous or ferric salt -
10. Lead, or other heavy metals -
11. Ammonia -

b. Of concentrated water,—

1. Reaction -
2. Nitric acid -
3. Phosphoric acid -
4. Silicic acid -
5. Iron—ferrous or ferric salt -
6. Magnesia -

Quantitative Analysis.

A. Of suspended matter :

1. Total amount per litre -
2. Combustible matter per litre -

B. Of dissolved constituents of the water :

a. Hardness,—

1. Total - - - -
2. Removable - - - -
3. Permanent - - - -

b. General analysis,—

1. Total solids per litre -
2. Effects of ignition -
3. Silicic acid - - - -
4. Oxide of iron, alumina, phosphoric acid - - - -
5. Lime - - - -
6. Magnesia - - - -
7. Alkaline chlorides -
8. Potassium chloride -
9. Sodium chloride -
10. Chlorine - - - -
11. Sulphuric acid - - - -
12. Combined carbonic acid -
13. Nitric acid - - - -
14. Nitrous acid - - - -
15. Phosphoric acid - - - -
16. Sulphuretted hydrogen -
17. Ammonia, free, and of salts

18. Albuminoid ammonia -

19. Amount of oxygen removed by readily oxidisable matters -

20. Amount of oxygen removed by oxidisable matters of water heated to 60° cent. (=140 F.) -

c. Measurement of gaseous constituents,—

1. Of nitrogen (corrected for 0° cent.) from one litre - - - -
2. Of oxygen - - - -
3. Of carbonic acid - - - -
4. Oxygen and nitrogen, taken at 100. state proportion of oxygen

*Report on comparative examination of Water
from source, from upper vessels and from
lower vessels of filters in Hospitals and
Barracks.*

Date of Analysis.	Water source, its position, and by whom used.	Physical Properties of Water.

No-5.

Copy Scheme of Analysis by
Dr. F. W. Macnamara

LONDON:
Printed by GEORGE E. EYRE and WILLIAM SPOTTISWOODE,
Printers to the Queen's most Excellent Majesty.
For Her Majesty's Stationery Office.

Referred to in Sanitary Letter to
Madras, No. 5, dated 22nd June 1871

P.G. 7 21 July 1871
In B.C.N. 191.

Sanitary
No 5

London

India Office

22 June 1871

My Lord

I forward herewith
in the use of your
Excellency's Government,
One Hundred Copies of
a Scheme of Analysis
by Dr. F. W. Macnamara
in the use of Medical
Officers on tour for the
purpose of examining
the Potable Waters of

His Excellency
The Right Honourable
The Governor in Council
Fort St. George

Military

100 copies

[Signature]

[Signature]

186

~~Mr. Secretary~~ The Committee
has been sent to the
Army Sanitary
Commission, and the
remarks of that Authority
will hereafter be forwarded
to your Excellency in
Council. -

I have the honor to be
My Lord
Your Lordships most
Obedient humble Servant

Chas. B.

Sanitary
22nd June 1871

81-1

21 July 1871 No 1123

Ordered that

Analysis referred to in the
Despatch be distributed
as follows.

Dispersed sent District heads:
"ent. ~~Dispersed~~" — 400
- 8. - D. Hoim. Dept. 50
General Officer — 1-
Sanitary Commr — 1-
House of Revenue — 1-
Military Dept. — 1-
Public Department — 6
100

L.B.

Forwards for the use of the Madras
Govt. 100 copies of a Scheme of
Analysis by D. Leucanumera
for the use of Medical officers
on tour for the purpose of
examining the Potable Water
Supply of the District.

17th July 1871