Determination of Total Dissolved Solids in Water Analysis

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The determination of total solids is one of the oldest determinations in water analysis and has apparently always been taken to represent the amount of material in the water. The difference between the weight of the residue on evaporation of unfiltered and filtered samples of the water has sometimes been taken as a measure of the suspended matter. The loss on ignition has frequently been taken as a measure of the organic matter in the water. Comparison of reported determinations of total solids and loss on ignition with the results of analysis of the actual mineral content indicates, however, that in some analyses the figures for total solids and for loss on ignition have no real significance.

The determination of total dissolved solids is based on the weight of the residue on evaporation after drying at a temperature of 105° to 110° C. (*1*, *5*) or at 180° C. (*3*). The loss on ignition is determined on the residue used for determining total dissolved solids by careful ignition over a small flame or in a covered metal dish (*4*). In calculating the quantity of dissolved material, the figure for bicarbonate is divided by 2.03 to account for conversion to carbonate on evaporation, and the quantities of the other constituents as found by analysis are added to give the quantity of dissolved mineral matter. In most water analyses the determined figure for total dissolved solids is greater than the sum of the determined constituents, because of the retention of water of crystallization or water of occlusion and because of small quantities of undetermined constituents. In analyses of certain types of waters, however, the quantity reported for total dissolved solids is less than the sum of the determined constituents because of the volatilization of certain constituents during the evaporation and subsequent drying.

Natural waters contain varying quantities of silica and the carbonates, sulfates, chlorides, and nitrates of calcium, magnesium, sodium, and potassium. For most waters it is assumed that the silica is present in the colloidal condition and after evaporation is present as SiO_2, although in certain waters, especially some from volcanic regions, the silica in the residue is in combination with the basic elements. Natural waters can be grouped for study according to the predominating acid radicals and in this paper are discussed under four groups—carbonate, sulfate, chloride, and nitrate waters. The analyses in Table I were selected from over 5000 made in the Water Resources Laboratory of the U. S. Geological Survey to represent the different types of water.

Carbonate Waters. Natural waters of the most common type are those in which calcium and bicarbonate are the principal radicals. This type is represented by the first two analyses in Table I. The residues from certain of these waters in which magnesium is small, when dried at 180° C., represent fairly accurately the dissolved material, as shown by analysis 1. Many waters of this type, however, contain considerable magnesium, and in some of these waters the determined figure for total solids is less than the sum of the determined constituents, as shown in analysis 2. In over 1000 analyses of waters of this type the determined figure for total dissolved solids was equal to the sum of the determined constituents in 15 per cent, greater than the sum by about 4.1 parts per million in 54 per cent, and less than the sum by about 3.0 parts per million in 31 per cent. The total solids for the whole group of waters averaged 81 parts per million.

The dissolved material in another large group of waters consists chiefly of sodium and bicarbonate (*2*). This group is represented by analysis 3, which indicates that in a water of this type the determination of total solids, based on the residue dried at 180° C., agrees well with the sum of the determined constituents.

Sulfate Waters. Waters in which sulfate is present in considerable quantities yield residues on evaporation that contain sulfates of calcium, magnesium, and sodium, with water of crystallization. Most of this water is driven off from the sulfates of sodium and magnesium when the residue is heated at 180° C., but this temperature is not sufficient to dehydrate calcium sulfate completely. Waters of this type are represented by analysis 4 in Table I. A great many waters high in sulfate carry enough chloride to affect the relation between the weight of the residues and the amount of dissolved material.

Table I. Mineral Constituents of Natural Waters

	SiO_2	Fe	Ca	Mg	Na	K	HCO_3	SO_4	Cl	NO_3	Total Dissolved Solids at 180° C.	Sum	Fixed Residue[a]	Color
	Parts per million													
1	7.9	0.07	52	5.0	2.4	1.7	178	3.9	1.8	0.20	165	163	164	...
2	9.0	0.02	32	15	2.6	1.3	171	2.7	2.6	0.53	137	150	133	...
3	16	0.07	5.3	3.9	141	3.2	388	5.0	12	1.1	382	378	379	...
4	13	0.08	113	29	97	3.9	186	407	23	7.3	811	785	774	...
5	30	0.12	184	29	127		214	48	454	1.0	1076	979	1049	...
6	17	0.03	68	19	19	4.7	239	19	29	50	331	343	297	...
7	6.3	0.06	17	2.6	1.3	0.8	58	8.2	0.8	0.10	67	66	63	5
8	6.0	0.01	5.2	1.7	5.7	1.4	13	2.3	10	Trace	66	39	51	200

[a] Figure obtained by subtracting loss on ignition from figure for total solids.

1. Allan Springs at Price, Ala., 1928.
2. Spring at Village Springs, Ala., 1928.
3. Well, 28 feet deep, at Hendersonville, Pa., 1926.
4. San Juan River near Bluff, Utah, average, 1931.
5. Well, 162 feet deep, at St. Petersburg, Fla., 1923.
6. Well, 24 feet deep, at Bartonsville, Fla., 1931.
7. Cowpasture River at Clifton Forge, Va., 1930.
8. North Fork of Black Creek, Middleburg, Fla., 1924.

Chloride Waters. Natural waters containing large quantities of chloride also generally contain considerable calcium and magnesium, and the residues from these waters are weighed with difficulty because of the presence of calcium and magnesium chlorides. The determined quantity of dissolved solids in practically all waters of this type is considerably greater than the sum of the determined constitu-

TABLE II. LOSS OF CHLORIDE DURING EVAPORATION AND HEATING OF RESIDUES FROM NATURAL AND ARTIFICIAL WATERS

	Total Dissolved Solids at 180° C.	SiO_2	Fe	Ca	Mg	Na + K	HCO_3	SO_4	Cl	NO_3	Ca	Mg	Na + K	HCO_3	SO_4	Cl	NO_3	Loss of Chloride during Evaporation and Heating 1 hr. at 180° C.
	Parts per million										*Milligram equivalents*							*Parts per million*
1	1076	30	0.12	184	29	127	214	48	454	1.0	9.2	2.4	5.5	3.5	1.0	12.8	0.0	50
2	1262	24	0.27	115	61	206	154	129	504	3.2	5.7	5.0	9.0	2.5	2.7	14.2	0.1	86
3	2630	16	0.12	177	101	643	144	253	1342	1.7	8.8	8.3	28.0	2.4	5.3	37.8	0.0	102
4	5377	8.4	0.36	254	181	1418	85	580	2662	Trace	12.7	14.9	61.6	1.4	12.1	75.1	0.0	86
5	1480	26	0.09	44	40	409	259	425	375	Trace	2.2	3.3	17.8	4.2	8.8	10.6	0.0	0
6	..	..	..	43	244	398	253	1233	366	Trace	2.1	20.0	17.3	4.1	25.7	10.3	0.0	0
7	..	..	..	37	113	340	217	353	546	Trace	1.8	9.3	14.8	3.6	7.4	14.3	0.0	0
8	..	..	..	28	192	271	174	282	720	Trace	1.4	15.8	11.8	2.9	5.9	20.3	0.0	100

1. Well, 162 feet deep, at St. Petersburg, Fla., 1923.
2. Well, 325 feet deep, at Eau Gallie, Fla., 1923.
3. Well, 265 feet deep, at Cocoa, Fla., 1923.
4. Salt Springs near Norwalk, Fla., 1924.
5. Well, 750 feet deep, at Fort Pierce, Fla., 1924.
6. Natural water to which solid magnesium sulfate was added.

7, 8. Natural waters to which a solution of magnesium chloride was added.

ents. Analysis 5 in Table I represents a water of moderate mineral content of this type.

Natural waters of the chloride type often show loss of chloride on evaporation and during the subsequent heating. The amounts of chloride in eight typical waters and the losses found after evaporation and heating are shown in Table II. In each case the loss of chloride occurred with residues from waters in which the chloride was more than equivalent to the sodium plus potassium. This was also brought out in a series of experiments with natural waters to which sodium chloride, magnesium chloride, and magnesium sulfate were added in varying amounts. The results of such an experiment with natural waters are shown by analyses 6 to 8 in Table II. It was also found that the addition of sodium sulfate or sodium carbonate to a solution of magnesium chloride in such quantities that the sodium becomes more than equivalent to the chloride eliminates the loss of chloride from such a solution.

The loss of chloride is usually not apparent from the weight of the residue on evaporation, because the amount of water held by the residue as water of crystallization is often greater than the loss of chloride.

NITRATE WATERS. Most natural waters have only small quantities of nitrate. In a few, however, the nitrate is present in considerable quantities usually with considerable quantities of other constituents that affect the weight of the residue (analysis 6, Table I). Waters that contain much nitrate may lose as much as 5 or 10 parts per million of nitrate when the residue is left on the steam bath as long as 4 hours. The loss of nitrate is increased during heating at 180° C. for one hour. The losses for seven natural waters are shown in Table III.

TABLE III. LOSS OF NITRATE DURING HEATING OF RESIDUE FROM NATURAL WATERS

(Analyses made in the Water Resources Laboratory, U. S. Geological Survey)

	Total Solids at 180° C.	SiO_2	Fe	Ca	Mg	Na+K	HCO_3	SO_4	Cl	NO_3	Loss of Nitrate during Heating: 4 Hours on steam bath	Loss of Nitrate during Heating: 1 Hour at 180° C.
		Parts per million										
1	331	17	0.03	68	19	24	239	19	29	50	..	19
2	406[a]	..	0.10	88	35	2.2[a]	367	16	12	42	26	35
3	330[a]	..	0.21	72	17	15[a]	204	67	14	31	9	18
4	372[a]	..	..	102	18	4.6[a]	328	15	19	34	13	30
5	265[a]	..	0.05	42	26	12[a]	199	16	28	23	7	7
6	303[a]	..	0.05	70	19	11[a]	255	27	20	15	9	13
7	209[a]	..	0.04	50	15	3.5[a]	206	5	5.0	16	11	12

[a] Calculated.

1. Well, 360 feet deep, at Leesburg, Va., 1931.
2. Well, 81 feet deep, at Marlboro, Va., 1931.
3. Well, 70 feet deep, at Sudley Springs, Va., 1931.
4. Well, 122 feet deep, at White Post, Va., 1931.
5. Well, 100 feet deep, at Whitaker, Va., 1931.
6. Well, 118 feet deep, at Gainesboro, Va., 1931.
7. Well, 53 feet deep, at Goresville, Va., 1931.

COLORED WATERS. Natural waters are often colored by organic matter. Waters of this type are represented by analyses 7 and 8, Table I. The more highly colored water (analysis 8) showed a much greater loss on ignition than the slightly colored water, and the agreement between the total solids and the sum was better for the water having less color.

DRYING AND IGNITION OF TOTAL SOLIDS. Many natural waters of moderate mineral content give residues on evaporation which when weighed after drying on a steam bath, with no other heating, show a fair agreement between determined total solids and the sum of the determined constituents. For 288 waters whose residues weighed between 100 and 500 mg. the difference between the weight when dry on the steam bath and the weight after heating for an hour at 180° C. was less than 10 mg. on 166 waters, between 10 and 20 mg. on 56 waters, and more than 20 mg. on 66 waters.

More concentrated waters give residues on evaporation which may have to be dried at 180° C. for several hours to obtain a constant weight. For certain types of waters whose residues are hygroscopic and difficult to weigh, it is preferable to evaporate a suitable volume in a weighing bottle that can be stoppered during the weighing. Waters of this type can be evaporated in an open dish rather rapidly and with no appreciable loss through spattering by applying heat from a hot noncorrodible surface above the dish. The residue obtained in this manner gives a better weight for the determination of total solids than that obtained when the water is evaporated in a dish on the steam bath and the residue dried at 180° C. to constant weight, although the loss in chloride is greater when the residue is heated from above. For highly concentrated waters that contain considerable calcium, magnesium, and sodium chlorides, such as brines from oil fields, Zinzalian and Withrow (*9*) recommend drying the residues at 750° C., making a correction for the amount of calcium and magnesium chlorides in the residue on evaporation. For waters of high concentration Reistle and Lane (*6*) determined the specific gravity of a sample and used this determination as the basis of the choice of the size of sample to be analyzed. For waters whose specific gravity was greater than 1.025 the total solids were calculated from the determined specific gravity. These authors give a table for the computation of total solids from the specific gravity in a range from 1.020 (total solids, 27,500 parts per million) to 1.140 (total solids, 210,000 parts per million) and state that the total solids computed in this way should check within 2.5 per cent the determined figure for total solids, based on the residue dried at 105° C.

RELATION BETWEEN CONDUCTANCE AND TOTAL SOLIDS. The total salinity of irrigation waters has been estimated from the specific electric conductance of the waters by Scofield and Wilcox (*8*), who used the method recently described by Scofield (*7*) for estimating the amount of soluble salts in soils or soil solutions. These authors state:

> Where desirable, the total salt content may be estimated as parts per million from the conductance determination by the use of suitable conversion factors. For approximate estimates with irrigation waters having a conductance of 100 or more,

TABLE IV. ANALYSES AND SPECIFIC CONDUCTANCE OF DIFFERENT TYPES OF WATERS

(Analyses made in the Water Resources Laboratory, U. S. Geological Survey)

	SiO_2	Fe	Ca	Mg	Na	K	HCO_3	SO_4	Cl	NO_3	Total Dissolved Solids at 180° C.	Sum	Specific Conductance (Reciprocal Ohms × 10^5 at 25° C.)	Sum ÷ Conductance
	Parts per million													
1	12	0.05	6.2	2.4	4.8	0.7	28	4.7	3.0	0.15	49	48	6.6	7.3
2	12	0.02	8.6	2.3	4.4	1.5	23	15	4.8	1.6	60	62	12	5.2
3	16	0.06	9.4	4.0	6.5	1.0	46	5.7	6.0	0.20	70	72	11	6.5
4	27	0.10	26	5.4	8.5	5.6	94	21	3.0	4.0	143	147	23	6.4
5	16	0.02	37	14	17	1.0	82	101	7.9	1.4	241	236	46	5.1
6	15	0.04	108	16	28	4.4	306	25	55	47	440	449	69	6.5
7	8.2	0.10	67	19	62	4.8	160	143	76	2.0	468	461	79	5.8
8	19	0.04	144	19	16	1.4	409	105	16	3.8	539	526	86	6.1
9	6.2	0.02	70	9.0	132	2.6	44	446	4.5	0.10	720	692	105	6.6
10	16	0.20	209	60	16	3.2	380	445	4.0	0.30	1018	941	120	7.8
11	8.4	0.08	100	44	217	4.0	222	372	231	14	1139	1100	185	5.9
12	16	0.10	120	60	129	3.7	195	599	22	15	1129	1061	146	7.3

1. Sacramento River at Sacramento, Calif., April 20, 1932.
2. Northwest Branch at Takoma Park, Md., April 18, 1932.
3. Sacramento River at Sacramento, Calif., April 6, 1932.
4. Well, 704 feet deep, at Middleburg, Va., October 30, 1931.
5. Canoe Brook at Irvington, N. J., February 26, 1932.
6. Well, 300 feet deep, at Berryville, Va., October 29, 1931
7. Dolores River near Cisco, Utah, April 1, 1932.
8. Well, 95 feet deep, at Stephens City, Va., October 28, 1931.
9. Wells at Marion, Ohio, part of public supply, treated water, April 6, 1932.
10. Wells at Marion, Ohio, part of public supply, raw water, April 6, 1932.
11. Colorado River near Grand Junction, Colo., March 11 to 20, 1932.
12. Gunnison River near Grand Junction, Colo., March 11 to 20, 1932.

in which sulfates predominate, the conductance multiplied by 7 will give a figure fairly close to the total salt content in parts per million. If the conductance is much below 100 or if chlorides predominate in the salt complex, then the conductance multiplied by 6 will be nearer the true value for total salt content.

Natural waters of different types give inconsistent results when an attempt is made to determine the factor that should be used to estimate the amount of dissolved material from the specific conductance. This was pointed out by Scofield (7) and is shown by typical results given in Table IV. The factor that must be used to obtain the quantity of dissolved solids from the conductivity of these 12 waters ranges from 5.1 to 7.8 and is not consistent for any range of solids or any type of water. Analyses 10 and 9, which have quite different conversion factors for estimating the solids from the conductivity, represent the raw and treated water of a public supply. Analyses 11 and 12, also with quite different factors, represent waters of similar quantities of dissolved solids but different in nature. It is evident that the conductivity is not an accurate measure of the dissolved solids, though it may serve to give approximate results of some practical value.

SUMMARY

The figure for total dissolved solids, based on the weight of the residue on evaporation after heating for 1 hour at 180° C., is reasonably close to the sum of the determined constituents for most natural waters.

Waters of the carbonate type that are high in magnesium may give residues that weigh less than the sum. Natural waters of the sulfate type usually give residues that are too high on account of incomplete drying.

Residues from waters high in chloride, in which the chloride is more than equivalent to the alkalies, may lose from 50 to 100 parts per million of chloride on heating. Residues from water high in nitrate may lose as much as 30 parts of nitrate on heating.

Waters of high concentration give more consistent results for the total dissolved solids when a small volume is evaporated in a weighing bottle. Time may be saved by evaporation in a dish by application of heat from above.

The specific conductivity of a water may be used to calculate approximately the quantity of dissolved material if the nature of the dissolved material is known.

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RECEIVED September 22, 1932. Presented before the Division of Water, Sewage, and Sanitation Chemistry at the 84th Meeting of the American Chemical Society, Denver, Colo., August 22 to 25, 1932. Published by permission of the Director, U. S. Geological Survey.

An Inexpensive Flame Shield

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A NUMBER of excellent flame shields are available from the various scientific supply houses at fairly low prices, but even this low cost assumes importance when a relatively large number of students must be supplied. Then, too, in research laboratories, the investigator often needs a flame shield immediately and is inconvenienced if it must be ordered. For these reasons the device described in this article is worth consideration.

The construction of the device is extremely simple. A tin can of the size used for prepared soups is secured, the top cut off smoothly, and a hole punched in the bottom. This hole should be of a size such that it fits tightly the upright tube of the burner, on which the shield is to be used. Adjustment is secured by raising or lowering the can, friction maintaining the position if the hole is of the proper size.

Larger cans may be used if a larger shield be desired. In some cases, gallon cans with both the top and bottom removed and holes punched in the sides for air inlets are advantageous. The relatively short life of the device is compensated by its inexpensiveness.

RECEIVED October 1, 1932.