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CHEMICAL ANALYSES OF SURFACE WATERS
OF MISSOURI.

BY

HERBERT W. MUNDT.

A

THESIS

submitted to the faculty of the
SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI
in partial fulfillment of the work required for the
Degree of
CHEMICAL ENGINEER

Rolla, Mo.

1927.

Approved by

W. D. Turner

33975

TABLE OF CONTENTS.

	Page
Introduction.....	2
Method of Sampling	3
Dissolved Mineral Constituents.....	4
Methods of Analysis	6
Expression of Results	11
Accuracy of Results	12
Tables of Analyses	13-28
Industrial Applications.....	29
Summary	38
Index to Analyses	40-45
General Index	46
Drainage Map	In pocket
Bibliography	39

CHEMICAL ANALYSES OF SURFACE WATERS OF MISSOURI.

INTRODUCTION

This investigation was made by the Missouri Bureau of Geology and Mines. The results of the analyses of surface waters of Missouri are published elsewhere.

The object of the investigation was to obtain information as to the chemical character of the river and spring waters of the state, such information being valuable and interesting to the industries and geologist as well as to the consumer of such waters.

The work for this investigation was started in June 1925 and was continued intensively during the entire summer, a few additional samples being analyzed during the spring and summer of 1926.

The samples were collected by members of the Water Resources Branch of the United States Geological Survey, and the field staff of the Bureau of Geology and Mines. The turbidity determinations were made by the State Board of Health.

The entire state was covered in an attempt to secure at least one sample from each section. Fewer

analyses are reported for the area north of the Missouri River than the area south of the Missouri River for several reasons, one of which is that there are fewer springs. Of the 92 springs sampled, only two are located north of the Missouri River. Practically all of the springs sampled are located in the area underlain by Mississippian and Cambro-Ordovician rocks. Furthermore, there are fewer large rivers north of the Missouri River. In several cases, samples of the same river or spring were taken but at different times.

Of the 237 analyses reported, 114 are of river waters, 92 are of spring waters and 34 are summaries. Seven analyses are by W. D. Turner, 26 are by Herbert W. Mundt, and 173 are by W. D. Turner and Herbert W. Mundt.

SAMPLING.

The samples were collected in one-gallon glass bottles which were rinsed with the water to be sampled before the sample was taken. Samples of streams were taken as nearly as possible in the center, by holding the mouth of the bottle against the flow, about four inches below the surface. Samples of the springs were secured at the mouth of the spring whenever possible. The samples were packed four to a case, and shipped to the laboratory

as soon as possible after being collected.

On reaching the laboratory, the samples were filtered to remove suspended matter, but a portion for the determination of suspended matter was first removed, if the quantity was large. To insure uniformity, the sample was shaken vigorously before each succeeding part was removed. About two liters were filtered and portions were taken as follows, the constituents written together all being determined on one portion:

<u>Constituent determined.</u>	<u>Amount taken.</u>
Silica, calcium, and magnesium	- 500 cc
(Sodium and Potassium) as sodium	- 250 cc
Carbonates, bicarbonates, and sulphates	- 200 cc
Chlorides	- 200 cc
Nitrates	- 100 cc
Iron	- 50 cc
Total solids	- 100 cc

If the constituent to be determined was present in large amounts, the portion taken for the determination was smaller than given in the above table. For the determination of suspended matter, 100 cc was taken if the waters were excessively turbid or muddy. From waters which contained but small amounts of suspended matter, 1000 cc was taken for the determination of suspended matter, the filtered portion being used for the other determinations.

DISSOLVED MINERAL CONSTITUENTS.

Silica:

Silica is assumed to be present in colloidal

state. Summaries by drainage basins show about 5 to 15 parts per million.

Iron:

Iron occurs in all the waters but rarely in quantities greater than two parts per million.

Calcium and Magnesium:

Calcium and Magnesium are dissolved in all the waters in appreciable quantities, the amount varying with locality of the state from which the sample was taken. They are responsible for the general high hardness of the waters analyzed.

Sodium and Potassium as Sodium:

Sodium and potassium are dissolved in nearly all of the waters analyzed, the amounts usually being smaller in samples of spring water and larger in the samples of river water but varying with the locality of the state from which the sample was taken. Large amounts promote foaming and priming in boiler waters.

Chlorides:

Chlorides are usually low, occurring in increased amounts in more thickly populated districts. Spring waters in general do not contain much chloride and some of the river waters have but small amounts. Except in waters from the larger rivers, the content is too small to have any effect on industrial processes.

Nitrates:

Nitrates occur in negligible quantities in practically all of the waters.

METHODS.

Turbidity:

Turbidity where reported was determined in the State Board of Health Laboratory.

Total Suspended Matter:

For waters that contained suspended matter in large amount, (principally Missouri River, Mississippi River and rivers in the northern part of the state) 100 c.c. of unfiltered water were evaporated to dryness in a tared dish and the amount of suspended matter plus total dissolved solids, determined on a filtered sample, was deducted leaving the amount of suspended matter. This method was necessary because of exceedingly slow filtration when the usual method was used, i.e. filtration through an asbestos padded Gooch crucible. For waters having but a small amount of suspended matter, the usual method, filtration of 1000 c.c. of water through an asbestos padded Gooch crucible, was used.

Volatile and Non-volatile Suspended Matter:

After obtaining the weight of total suspended matter, the crucible was ignited, cooled, and re-weighed and amounts of volatile and non-volatile suspended matter derived from the weights so obtained.

Coefficient of Fineness:

Coefficient of fineness is the ratio of the suspended matter to the turbidity and is a measure of the size of the particles in suspension. As the coefficient becomes greater so will the removal of suspended matter be accomplished with greater facility.

Silica:

Silica was obtained by evaporation with hydrochloric acid, dehydration and filtration; ignition, cooling and weighing before and after adding sulphuric acid and hydrofluoric acid was required to determine true silica.

Iron:

Iron was determined by the colorimetric sulphocyanide method, the comparison being made in a Duboscq colorimeter. Nitric¹ acid was used in place of hydrochloric acid since the color produced by the addition of sulphocyanide is deeper and does not fade so rapidly. Also the necessity of using potassium permanganate to oxidize the ferrous to ferric iron is obviated.

Alumina:

Alumina is not reported because of the generally minute quantity present. In such small quantity its condition and effect would be negligible in most industrial processes.

Calcium:

After removing silica and combined oxides, calcium

was determined by precipitation with ammonium oxalate which was added in excess to the faintly ammoniacal boiling solution containing calcium and magnesium. After filtering and thoroughly washing, the calcium oxalate was dissolved in dilute sulphuric acid, heated and titrated with a standard solution of potassium permanganate.

Magnesium:

Magnesium was precipitated in the cold filtrate from the calcium by the addition of a solution of microcosmic salt. Concentrated ammonia was added equal to one-tenth² the volume of the solution. Filtration, washing, ignition, cooling and weighing of the precipitate was carried out after the solution had stood over night or at least twelve hours.

Sodium and Potassium as Sodium:

In as much as the quantity of water was sufficient to warrant it, a separate portion was taken for this determination instead of using the filtrate from the sulphate determination. Magnesium was removed with barium hydroxide and after filtration and concentration, calcium and barium and other impurities were removed by repeated treatments with ammonia, ammonia oxalate, and ammonium carbonate. Ammonium salts were expelled by evaporating to dryness and heating after each treatment. The treatments were repeated until no apparent impurities remained. The small volume of solution containing the sodium and potassium together with a few drops of

² Large amounts of ammonium hydroxide added here are disadvantageous --Bulletin U.S. Geol. Survey 176--P.65--1900.

concentrated hydrochloric acid, was evaporated to dryness, heated to about 600°C., cooled and weighed as sodium and potassium chlorides. Because of the small quantity of potassium in most of the waters, it was thought unnecessary to separate sodium and potassium. They are therefore calculated and reported together as sodium. The error involved in thus reporting is negligible.¹

Carbonate and Bicarbonate:

Carbonate was determined by adding phenolphthalein to the sample. If a pink color was produced, titration with standard acid to a colorless end point was required. After recording the amount of acid required for carbonate, or if no color was produced with phenolphthalein, three drops of methyl orange was added and the titration continued to the usual end point. From the size of the sample, the normality of the acid and relation between alkalinity by phenolphthalein and by methyl orange, the amount of carbonate and bicarbonate were calculated.

Sulphate:

Sulphate was determined on the portion used for carbonate and bicarbonate. After the addition of a few c.c. of hydrochloric acid, the sample was heated to boiling and the sulphate was precipitated as barium sulphate by the slow addition of barium chloride. The precipitate was filtered, washed, ignited, cooled and weighed.

¹ Dole, R.B. Analyses of Waters East of the One Hundredth Meridian, Water Supply Paper No. 236, p. 33--1909.

Chloride:

Chloride was determined by adding 1 c.c. of 5% potassium chromate solution and titrating with standard silver nitrate to the customary end point.

Nitrate:

For the determination of nitrate, the water was evaporated to dryness with two c.c. of a 10% solution of sodium carbonate. After adding two c.c. of phenosulphonic acid and making alkaline with ammonium hydroxide, the yellow color developed was compared with similarly developed shades of known nitrate content. The comparison was made in a Dubosca colorimeter.

Blanks:

Blank determinations were made on all the regular methods. The amounts found were deducted, where required, from the regular determinations.

Hardness:

Total hardness has been calculated as calcium carbonate from determined calcium and magnesium, assuming magnesium to be present as calcium. This is, in general, a more reliable figure than the soap hardness result which was found to be at variance with the calculated hardness in at least one third of the analyses made.

Alkalinity has been calculated as calcium carbonate from the carbonate and bicarbonate computed as carbonate, and represents temporary hardness only in those cases where sodium and potassium carbonates

occur in small amounts or not at all, since residual alkalinity after boiling was not determined.

Sum of Constituents:

The sum of constituents has been reported instead of the customary total dissolved solids. As the total dissolved solids were dried at 120°C., it is probable that all of the occluded water and the water of crystallization was not driven off. Further, if there were much organic matter, part at least would be carbonized and some of it would be completely destroyed if a higher temperature (180°C.) had been used. It would seem, therefore, that the sum of the constituents would more accurately represent the amount of mineral matter dissolved in the water if the quantity of organic matter were small. The bicarbonate, of course, has been calculated to carbonate and is included as such in the sum of the constituents but the negligible amount of combined oxides was not included.

Residue after Ignition:

Residue after ignition represents the amount of material remaining after the total dissolved solids were ignited at 650° to 700°C.

EXPRESSION OF RESULTS.

The results are expressed in parts per million of the radicals determined. For the convenience of those who may desire the results in other forms, it may be stated that for practical purposes, multiplying parts per million by 0.058 gives the equivalent in grains per U. S. gallon

and multiplying by 0.070 gives the equivalent in grains per imperial gallon.

The springs and rivers are tabulated separately and are further classified according to drainage basins. The location from which samples of the rivers or springs were taken appears on one page with such information as is relevant and the analysis appears on the page directly opposite. Each location is given a number which corresponds to the analysis on the opposite page having a like number. The drainage basin and sources are arranged alphabetically. Summaries of individual drainage basins are reported.

ACCURACY.

Established methods were used in making the analyses and the manipulative technique was accompanied by the necessary care. The analyses have been carefully checked for indications of errors. Stabler's ¹ method for computing per cent of error was applied to all analyses and together with Dole's ² table of criteria for accepting analytical data, formed the standards to which all the analyses conform.

¹ Stabler, Herman, Some Stream Waters of the Western United States, U.S. Geol. Survey, Water Supply Paper No. 274 p. 167 -- 1911.

² Dole, R. B. Analyses of Waters East of the One Hundredth Meridian, Water Supply Paper No. 236 p. 38 -- 1908.

Tables of Analyses
of River and Spring Waters.

NOTE:

Pages 14 to 28 inclusive - Analyses of the waters outlined in index. These analyses are being published as Chapter Five of Volume XX, Water Resources of Missouri. They will probably be returned by the printer about July 1st, 1927, and placed on the pages above mentioned.

INDUSTRIAL APPLICATIONS.

General Uses of Water:

Water is consumed in large quantities for domestic use, laundering, steam making, and various industrial processes. The requirements are not alike in all cases. Generally, as the amount of dissolved mineral matter in a water decreases, its acceptability for all uses increases. For domestic use and the preparation of food products, potable water is required. For laundering, steam making, and many other industrial processes soft water is required.

Potability:

A water to be potable should be free from disease germs and organic or inorganic matter in suspension, possess no color or disagreeable odor, and should not contain excessive amounts of dissolved mineral matter. The following requirements for potable water are taken from Reprint No. 1029, U.S. Public Health Service, pp. 24-25.

PHYSICAL CHARACTERISTICS.

1. Turbidity should not exceed 10 (silica scale), and in general it should not be more than 5.
2. Color should not exceed 20 (standard cobalt scale), and preferably should be less than 10.
3. There should be no odor or hydrogen sulphide, chlorine, or any other substance, and the water should be free from odors caused by the presence of

microscopic organisms.

CHEMICAL SUBSTANCES WHICH MAY BE PRESENT IN NATURAL WATERS.

1. Lead (Pb) shall not exceed 0.1 p.p.m., and copper (Cu) shall not exceed 0.2 p.p.m.
2. Zinc (Zn) shall not exceed 5.0 p.p.m.
3. Sulphate (SO₄) should not exceed 250 p.p.m.
4. Magnesium (Mg) should not exceed 100 p.p.m.
5. Total solids should not exceed 1,000 p.p.m.
6. Chlorides (Cl) should not exceed 250 p.p.m.
7. Iron (Fe) should not exceed 0.3 p.p.m.

Most of the spring and some of the river waters analyzed would be potable without treatment. Coagulation, filtration, sterilization, and softening could be used separately or in combination where required to produce water suitable for most purposes.

Hardness:

Hardness is undoubtedly the most important property of a water with reference to its industrial use. This is especially true with reference to laundering and steam making. Hardness is indicated by the increased amount of soap necessary to produce a lather, by the scum which accumulates before a lather is produced, due to the formation of insoluble calcium and magnesium compounds, and by the formation of scale in boilers or any other vessel in which the water has been heated.

Hardness is usually expressed as parts per million of calcium carbonate. For example, if a water had

a hardness of 100 parts per million of calcium carbonate, it could be stated in a different way that 100 pounds of calcium carbonate were dissolved in 1,000,000 pounds of water, or on the basis of liquid measure that 100 pounds of calcium carbonate were dissolved in approximately 120,000 U. S. gallons of the water. With this particular water about 612 pounds of pure anhydrous or about 1000 pounds of laundry soap would be used without producing a lather in the 120,000 gallons of water as this amount of soap would be required to react with and effect the removal of the calcium. In laundering, it would thus seem necessary to remove the hardness by more economical methods.

Hardness in the waters analyzed is due to calcium and magnesium. They may be in solution as bicarbonates, sulphates, nitrates or chlorides. Other elements could cause hardness but the quantity in the waters analyzed was not sufficient to contribute much, if anything to the hardness.

There are two kinds of hardness, temporary and permanent. Temporary or carbonate hardness is indicated when the reacting value of carbonate and bicarbonate is equal to or greater than the reacting value of calcium and magnesium. Such hardness may be almost completely destroyed by boiling or heating as the bicarbonate is decomposed and the calcium and magnesium are thrown out to the extent of their solubility in the water. Alkalinity reported in terms of calcium carbonate would be a measure of this

hardness if there were no sodium and potassium carbonates in the water. If the sodium and potassium carbonates were present, residual alkalinity would have to be determined and deducted from the total alkalinity and the difference would be equal to the true temporary hardness. Residual alkalinity determinations were not made on the waters analyzed as the quantity of sodium and potassium was usually small. Permanent or non-carbonate hardness due to sulphates, nitrates or chlorides of the alkaline earths is indicated when the reacting value of the carbonate and bicarbonate is less than the reacting value of the calcium and magnesium. In such waters, the hardness cannot be completely destroyed by heating or boiling and recourse must be had to chemical means for completely removing the calcium and magnesium.

Softening of Hard Water:

The hardness of water may be destroyed by several different methods. For temporary or carbonate hardness, heating is usually sufficient to render the water soft enough for most purposes. A water treated under ideal conditions may still have in solution 5.2 parts per million of calcium and 3.8 parts per million of magnesium. This would represent hardness of about 27.7 parts per million and it would be safe to say from a practical standpoint that a water could not be softened to less than 30 to 40 parts per million of hardness calculated as calcium carbonate.

Lime and soda ash are the chemicals usually used to soften water, principally because of their cheapness. Lime is added as limewater equivalent to the free acids,

free carbon dioxide, bicarbonates, iron, aluminum, and the magnesium. Sodium carbonate in solution is added equivalent to the calcium present as sulphate and chloride, or introduced by addition of limewater. Naturally if the softening is conducted at an elevated temperature with agitation, there will be greater efficiency and the reaction will take place more rapidly. Barium carbonate could be used in some cases in place of sodium carbonate but the poisonous effect of even small amounts of barium in the water would not balance its usefulness in the removal of sulphates. Its increased cost over sodium carbonate would also have to be considered.

Among the other substances sometimes used to soften water are sodium silicate, hydroxide and phosphate, barium oxide and hydroxide, aluminum and iron sulphate, boiler compounds, which are principally strong solutions of soda ash, and the zeolites¹ or exchange silicate softeners in which use is made of the property of some silicates of exchanging sodium for calcium and magnesium. When the silicate begins to lose its exchange capacity, it may be regenerated by passing through it a solution of common salt, which now exchanges sodium for the calcium and magnesium in the silicate. "Permutit" (an artificial silicate whose formula is approximately $2 \text{SiO}_2 \cdot \text{Al}_2\text{O}_3 \cdot \text{Na}_2\text{O} + 6 \text{H}_2\text{O}$) is an example of such silicate. The advantages and efficiency

¹ Gans, R., On Zeolites and Similar Compounds. Abstracted in Jour. Soc. Chem. Ind. Vol. XXVI pp. 1091 and 1251 -- 1907.

of this type of softener are apparent.

Analyses of water from the City well at Rolla before and after being passed through a "Permutit" water softener are given below. Results are in parts per million.

	<u>Before</u> ¹	<u>After</u> ²
SiO ₂	8.0	10.2
Fe	0.34	0.2
Ca	49.6	0.4
Mg	28.5	0.4
(NaK)Na	7.7	135.3
CO ₂	8.4	24.6
HCO ₃	261.0	272.8
SO ₄	19.9	25.1
Cl	3.2	5.0
NO ₃	0.0	trace
Alkalinity as CaCO ₃ ...	228.0	264.8
Total hardness as CaCO ₃ (Calculated)..	240.8	2.6
Sum of constituents...	253.8	335.1
Total suspended matter	0.1	8.4
Error in per cent	+0.8	-0.08

Foaming, Priming and Corrosion:

Foaming, or the formation of bubbles upon or above the surface of the water which is being used in a boiler is usually attributed to suspended or organic matter and excessive amounts of sodium and potassium. In this connection, it would seem that the injudicious use of the boiler compounds sometimes used to treat the water within the boiler, would increase the tendency of the water to foam and their beneficial effect, usually that of producing soft scale could easily be offset by the introduction of foaming. Small pieces of scale, which have become detached

1 This sample was taken from water tap at 1111 State Street and was analyzed July 30, 1925.

2 This sample was taken directly from the soft water tap of the softener and was analyzed Feb. 28, 1927.

from the deposit in the boiler, may also promote foaming. As there is no practical way to remove the sodium or potassium from the water, frequent blowing off or change of feed water supply would seem to be the only remedies. Foaming, due to suspended matter in the feed water, can easily be remedied by coagulation or filtration before the water enters the boiler. According to Stabler¹ waters may be classed according to their tendency to foam in a locomotive boiler as follows:

- Non-foaming - Can be used one week without foaming.
- Semi-foaming - ~~One complete change of water necessary,~~ to avoid foaming oftener than every two days.
- Foaming - - - - Foams in less than two days, if changing water or blowing off is not resorted to.

Priming occurs, if foaming proceeds to the extent that water is carried from the boiler with the steam in the form of a spray. It is therefore intimately related to foaming.

Foaming and priming due to sodium and potassium would probably not occur in most of the waters analyzed. Some of the river waters contain large amounts of suspended matter and moderate amounts of sodium and potassium. After removal of suspended matter, however, little difficulty should be experienced because of foaming.

Corrosion and pitting of a boiler may be caused by free acids which may be present in the original water, or which may be developed after the water has been heated. Substances which undergo hydrolysis, as the salts of sodium, potassium, and magnesium cause corrosion. Organic acids

¹ Stabler, Herman, Some Stream Waters of the Western United States, Water Supply Paper No. 274 p. 174 -- 1911.

formed by the decomposition of organic matter contribute to the difficulty. Carbon dioxide either free or evolved by the decomposition of calcium and magnesium bicarbonates upon heating the water also is important. In other words, corrosion will occur if the hydrogen in any of the acids which might be formed as suggested above, is replaced by the metal of the boiler.

Corrosion may be prevented by allowing a thin film¹ of scale to form in the boiler. This will protect the boiler, partly because of the coating of scale formed and partly because of the alkalinity introduced. Free acidity or acidity which may develop when the water is heated may be counteracted by increasing the alkalinity of the water. The high alkalinity of most of the waters analyzed indicates that corrosion would not be probable.

Scale:

Scale deposited within the boiler is due to settling out of suspended matter, to precipitation of sparingly soluble compounds, or to concentration of the water. Such scale may be hard or soft, depending on the insoluble compounds which are precipitated. Calcium and magnesium are the predominant constituents of both hard and soft scale. As sulphates, they would produce a hard, and as carbonates a soft scale. Sodium and potassium have no effect on scale formation as their salts are soluble. Silica, iron, and aluminum all contribute to the formation of scale but the quantities are unimportant as compared with

¹ Kent, Wm., Eng. News Vol. 52, p. 198 --1904.

calcium and magnesium which predominate as scale forming constituents in the waters analyzed. For formation of soft scale, which can be removed with greater facility and is more desirable than hard scale, sodium carbonate seems to be efficacious¹. Boiler compounds (essentially solutions of sodium carbonate), or tannin, starch, molasses, and other organic substances which produce soft scale are apt to be injurious because of the possibility that by their decomposition, corrosive organic acids will be formed and the metal of the boiler will be attacked.

The most reliable practice is to soften the water before it enters the boiler and in this connection pre-heating or the soda-lime method is suggested because of economy. Sulphates could be removed with barium carbonate but the expense would probably be prohibitive.

Other Industrial Considerations:

In the paper making industry pure water is essential for the manufacture of a uniform high-grade product. Alkaline waters which contain much iron, suspended matter or calcium and magnesium bicarbonates interfere with the pulping, sizing and bleaching of the paper. Acids and vegetable coloring matter also have a deleterious effect. In dyeing and bleaching, suspended matter, iron, calcium, magnesium, and bicarbonate seriously interfere with the production of colors and the efficiency of bleaching. In

¹ Peter, Robert, Well water-Geological Survey Kentucky Vol. 5 new series 1880 pp. 188-190. Cited by Chase Palmer U.S. Geol. Survey Water Supply Paper No. 233, p. 190.

tanning, bicarbonate interferes with the removal of hair from the hide and decreases the absorption of tannin because of precipitation of calcium carbonate in the tissue. Iron produces black spots which are undesirable except on black leather. Organic matter causes leather rot and chlorides interfere in the swelling process. Softening applied to the waters analyzed will effect the removal of the harmful constituents and permit their use in any of the above mentioned processes.

SUMMARY

Analyses of 114 river waters and 92 spring waters and 34 summaries by drainage basins are given, together with the methods used for determining the various constituents. The industrial application of the analyses and suggestions for treatment of water for different purposes are described.

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Index to Analyses.

Source	Rivers Town	Page
	-B-	
Big	Byrnesville	16
Big	E. Bonne Terre	16
Black	Leeper	14
Black	Poplar Bluff	14
Black	Poplar Bluff, 3 mi. S.	14
Blackwater	Blue Lick	16
Blue	Kansas City	18
Bourbeuse	Union	16
	-C-	
Castor	Zalma	16
Cedar Creek	Pleasant View	20
Chariton	Elmer	14
Chariton	Keytesville	14
Chariton	Rockford	14
Chariton, Little... ..	Forest Green	14
Crooked Creek	Montauk	14
Guivre	Troy	14
Current	Doniphan	14
Current	Eminence	14
Current	Owls Bend	14
Current	Van Buren	14
	-D-	
Dry (or North) Fork of Spring.....	Neck City	20
	-E-	
Eleven Point	Bardley	14
Elk	Noel	20
	-F-	
Fabius, Middle	Lewistown	14
Fabius, North	Monticello	14
Fabius, South	Taylor	14
Flat Creek	Sedalia	16
	-G-	
Gasconade	Hazlegreen	14
Gasconade	Hooker	14
Gasconade	Jerome	14
Gasconade	Mt. Sterling	14
Gasconade	Waynesville	14
Grand	Brunswick	16
Grand	Summer	16

	-H-	
Headwater Diversion Channel	Dutchtown	16
	-I-	
Indian Creek	Simmons	14
Indian Creek	Anderson	20
	-J-	
Jacks Fork	Eminence	14
James	Galloway	16
	-K-	
Kansas	Kansas City	18
	-L-	
Lamine	Clifton City	16
Little Piney	Arlington	14
Little Osage	Horton	18
Little River Ditch No. 1....	Kirk	20
Little River Ditch No. 66...	Kirk	20
Little River Ditch No. 81...	Kirk	20
Little St. Francis..	Fredericktown	20
Loutre	McKittrick	18
	-M-	
Marmaton	Horton	18
Meramec	Eureka	18
Meramec	Steelville	18
Meramec	Sullivan	18
Meramec	Ten Brook	18
Mississippi	Burlington Park	16
Mississippi	Cape Girardeau	16
Mississippi	Caruthersville	16
Mississippi	Hannibal	16
Mississippi	Jefferson Barracks	16
Mississippi	Louisiana	16
Mississippi	New Madrid	16
Mississippi	Ste. Genevieve	16
Mississippi	St. Louis	16
Missouri	Boonville	18
Missouri	Chesterfield	18
Missouri	Glasgow	18
Missouri	Hermann, opp.	18
Missouri	Jefferson City.	18
Missouri	Kansas City	18
Missouri	St. Charles	18
Missouri	St. Joseph	18
Missouri	Washington	18
Missouri	Waverly	18
Moreau	Jefferson City.	18
Musselfork	Keytesville	14

-N-		
Niangua	Roach	18
Nodaway	Burlington Junction.....	18
Nodaway	Nodaway	18
-O-		
Osage	Bagnell	18
Osage	Jefferson City	18
Osage	Osceola	18
Osage	Rich Hill	18
-P-		
Piney	Cabool	14
Piney	Devil's Elbow	14
Platte	Agency	18
Platte	Ravenwood	18
Pomme de Terre	Buffalo, West of	20
Pomme de Terre	Hermitage	20
-R-		
Roche Perche Creek	McBaine	18
Roubidoux	Waynesville	14
-S-		
Sac	Collins	20
Sac	Stockton	20
Salt, Middle Fork of.	Paris.....	20
Shoal Creek	Grand Falls	20
Shoal Creek	Neosho	20
South Grand	Archie	20
South Grand	Brownington	20
Spring	Carthage	20
Spring	Waco	20
St. Francis	Fisk	20
St. Francis	Patterson	20
-T-		
Tarkio	Fairfax	20
Thompson	Trenton	16
-W-		
Wakenda Creek	Carrollton	18
White	Beaver, Ark.....	20
White	Branson	20
White	Forsyth	20
White, N. Fork of....	Tecumseh	18
Whitewater	Whitewater	16

Springs

Source	Town	Page
-A-		
Alley	Alley	22
Amsden	Centerville	22
-B-		
Bartlett Mill	Waynesville	22
Beaver	Steelville	24
Bennett	Brice	26
Big	Van Buren	22
Big	Neosho	26
Big	Mt. Vernon	26
Blue	Eminence	22
Blue	Alton	22
Blue or Shanghai.	Hooker	22
Blue	Bourbon	24
Blue or Sweet ...	Eldridge	26
Boiling	Hooker	22
Boiling	Licking	22
Boze Mill	Alton, 15 mi. E.....	22
Bryant	Bryant	26
-C-		
Carter	Piedmont	22
Cave	Pearl	26
Chesapeake	Chesapeake.....	26
Clarkson	Pierce City	26
Coppedge or Relfe	Relfe	22
Creasy	Waynesville	22
Crystal	Larissa	26
-D-		
Double	Dormis	26
Dunnegan	Dunnegan	26
-E-		
Eidson	Bolivar	26
-F-		
Falling	Waynesville	22
Faulkenberry	Lesterville	22
Fullbright	Springfield, 4 mi.N.....	26
-G-		
Gaines Ford	Rolla	24
Glencoe Hollow...	Eureka	24
Graham	Thomasville	22
Gravois Mills....	Gravois Mills	26
Greer	Greer	22

-H-		
Hahatonka.....	Hahatonka	26
Hazleton	Hazleton	24
Hodgson Mill	Sycamore	26
House	Eureka	24
Humansville	Humansville	26
-I-		
Idlewild	Cuba	24
-J-		
Jones	Springfield, 4 mi. E...	24
-K-		
Keener	Keener	22
Kratz	Stanton	24
-L-		
Lake	Lake Springs	24
Leeper	Leeper	22
-M-		
Mammoth or Prewett	Edanville	24
Marbut or Verona...	Verona	26
Markham	Williamsville	22
Mark Twain	Hannibal	24
Meramec	St. James	24
Mill	Mill Spring	22
Miller (Ebb and Flow)	Big Piney	24
Montauk	Montauk	22
Morris	Rockbridge	26
Morse Park.....	Neosho	26
-O-		
Onandaga Cave	Leasburg	24
Ousley	Spring Creek	24
-P-		
Paris	Paris Springs	26
Paydown	Paydown	24
Phillips	Barren	22
Pierce City	Pierce City	26
Piney	Yancy Mills	24
Prewett or Mammoth.	Edanville.....	24
Pulltight	Ink	22
-R-		
Randolph	Ellington	22
Reed	Centerville	22
Reeds	Reeds Springs	24

-R-Cont.-

Relfe or Coppedge	Relfe	22
Roaring	Stanton	24
Roaring River.....	Cassville	26
Rollins	Columbia	24
Roubidoux	Waynesville	24
Round	Owls Bend	22

-S-

Sands	Sands	24
Schlicht	Schlicht	24
Shanghai or Blue..	Hooker	22
Siloam	Siloam Springs	26
Slabtown	Edanville	24
Stockton	Stockton	26
Stone Mill	Spring Creek	24
Sugar Tree	Newburg	24
Sweet	Sweet Springs, 1 mi.S....	24
Sweet or Blue	Eldridge	26

-T-

Taylor	Elijah	26
Thomasson Mill....	Couch	22
Thox Rock	Newburg	24
Turner Mill	Alton	22

-V-

Vaugt	Couch	22
Verona or Marbut..	Verona	26

-W-

Warner Bay	Lesterville	22
Welch	Cedar Grove	22
Wilder	Elijah	26

-Y-

Yancy	Yancy Springs	24
Youngs	Rolla	24

General Index.

	Page
Accuracy of Results	12
Dissolved Mineral Constituents.....	4
Expression of Results	11
Foaming, Priming and Corrosion	34
General Uses of Water	29
Hardness	30
Index to Analyses	40
Industrial Application	29
Introduction	2
Methods of Analysis	6
Other Industrial Considerations	37
Potability	29
Sampling	3
Scale	36
Softening of Hard Water	32
Summary	38
Tables of Analyses	13-28
Table of Contents	2

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DRAINAGE MAP OF MISSOURI

DRAINAGE AREAS COMPILED BY
 H. C. BECKMAN, DISTRICT ENGINEER
 COMPILED IN COOPERATION WITH THE
 UNITED STATES GEOLOGICAL SURVEY

- LEGEND
- Stream flow gauging station.
 - ▲ River stage station of U. S. Weather Bureau.
 - ▲ Present rainfall station of U. S. Weather Bureau.
 - Boundary of primary drainage basin.
 - - - - Boundary of secondary drainage basin.
 - 580 Red figures below name give total area of drainage basin.
 - 264 Red figures with arrow give drainage area of stream above that point.

1927

Note
 Boundaries of drainage areas as shown are approximate. Areas were measured on more detailed maps.

