CHAPTER XIII

Coagulation: Ancient and Modern

Coagulation as an aid to sedimentation for the clarification of individual household and small industrial water supplies has been practiced from ancient times. At large industrial plants its use seems to have begun in the first third of the nineteenth century. It was seldom if ever used for municipal supplies until the fourth quarter of that century. In most cases, coagulation was not adopted as a separate process but was used in conjunction with filtration; it was rarely employed with slow sand filtration but was widely used to precede rapid filtration from 1885 onward. Before this combination came into general acceptance in the early 1900's, coagulation as an aid to sedimentation only was adopted by a few American cities, most of them large ones, and all using water highly turbid at all times and excessively so at intervals. Sooner or later, these cities built rapid filters, with precoagulation followed by sedimentation. As time went on, coagulation was introduced where prejudices against it had led to the adoption of slow sand rather than mechanical filtration. Finally, slow sand filtration was almost wholly replaced by rapid filtration, of which coagulation is an essential part. Thus coagulation and filtration, each used in a small way through the ages, were wedded. The practice spread over the world but less rapidly and completely in Great Britain and France than elsewhere.

The agent most generally used as a coagulant is aluminum sulfate—commonly known as alum, filter alum or sulfate of alumina. Lime and iron are frequently used together as coagulants; lime is sometimes employed alone; iron has been used in comminuted metallic form but is more generally employed as an iron salt. Other materials, improvised for time and place, have included almonds, beans and nuts, toasted biscuits and Indian meal.

The underlying principles of coagulation were scientifically defined earlier than those upon which filtration is based and might earlier have been utilized for the improvement of public water supplies had the public demand for clear water been more insistent and had it not been for the influence of ill-informed or prejudiced persons

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whose word was respected—notably Arago of France in 1837, the Massachusetts State Board of Health a half century later, as well as medical men who protested frantically against both coagulation and rapid filtration in the late nineteenth and early twentieth centuries.

Almonds, Beans, Nuts and Alum

Egypt.—Although coagulation must have been practiced from ancient times in Egypt, no record of it has been found until the close of the sixteenth century. Prospero Alpino, Italian botanist and physician, in a late sixteenth century book on Egyptian medicine, gives an eyewitness account of the use of almonds to aid sedimentation. After noting that "Galenus said in Book I, 'simp. med. facult.,'" that Egyptians once used water filtered through earthen jars, Alpino added:

> I saw another method, too, employed by them and frequently made use of, too, by which they made water clear and pure. For as soon as they had brought home water from the stream in camel skin bags they put it away in oblong vessels having a rather wide and round belly and holding two amphoras of water each. When the water had been placed therein and immediately settled they smeared the edges of the vessel with five sweet almonds properly crushed and grasping the almonds in the hand suddenly plunged hand and arm into the water up to the elbow and moved elbow and hand vigorously and violently this way and that through the water stirring it up until they had made it far more turbid than before. Then they withdrew the arm from the vessel, leaving the almonds in the water. They let it clarify and it was properly clear in three hours' time. They put the water from the great vessel into little earthen jars in which it became more clear and cooled. (1)

Felix D'Arcet, French chemist, industrialist and writer on scientific subjects, after a "sojourn in Egypt," wrote a notable paper describing filtration through porous jars in that country, and coagulation with almonds in northern Egypt and with beans in the Soudan. He also described a method of coagulation by alum that he had devised (2). The water of the Nile, he said, was always muddy but during the inundation contained eight grams per liter of suspended matter. Clarification was always employed but rather to avoid mud than as a hygienic measure. The poor, who could not afford the large, costly filter jars used by the rich, used almonds, ground and made into cakes. After placing the turbid water in jars, they rubbed by hand a cake of almonds on the inside of the jar, circularly from top to bottom. Next, the water in the jar was shaken vigorously in all directions.

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The jar was then covered and allowed to stand four or five hours during which time it became clear. The operation was performed by the "sacer" or water porter who brought the Nile water to the houses daily. In all the bazaars of Egypt small cakes of almonds could be bought for five parats or about four French centimes. Their average weight was about 64 grams and one served for a month. In the process of clarification the particles of almonds formed a sort of emulsion with the water; the oil united with the earth and the latter settled to the bottom of the jar. At Sennar and Dongola in the Soudan, instead of almond cakes, beans were used—broad, kidney or castor oil—but they did not give full limpidity.

Seeing the imperfection of water clarification by almonds, D'Arcet tried alum which both he and his father had used with success in France for clarifying the water of the Seine. With 0.1 gram of alum per liter of muddy water he obtained complete coagulation within an hour.

Alum, says D'Arcet, gave no cause for alarm on account of health. In its decomposition the excess of acid was taken up by the carbonate or bicarbonate of lime in the water, while the alum itself was entrained in the earthy particles thrown down. Large pieces of crystals of alum were preferable. These were attached to a thread and moved about in all directions. Formation of voluminous flocs and precipitate were signs that the alum had been dissolved. Powdered alum might be used more easily than lump. It was sprinkled on the surface of the water, avoiding pronounced agitation. Or a solution of alum might be employed, in which case the surface of the water was agitated lightly. In Egypt alum with a potassium base was used but an ammonia base would give the same results (2).

India.—The nut of the *Strychnos potatorum* was one of the seven substances used to purify water named in the *Sus'ruta Samhita,* according to a manuscript of 400 A.D. which summarized ancient Aryan and Indic lore (see Chap. I). This nut, wrote James F. W. Johnston in 1854, was at that time used to clarify the marsh waters of India. A supply of the nuts, he said, was often carried by travelers in that country. One or two of them "rubbed to a powder," on the sides of the earthen vessel containing the water, soon caused it to subside (3).

China.—Despite many references to the use of alum for small-scale water clarification in China, almost no writers go beyond mentioning that it was an ancient practice. The earliest eyewitness account of

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this custom is given by Navarette, a Spanish missionary of the latter part of the seventeenth century. He also mentions the use of coca for the same purpose and refutes theories of Chinese philosophers as to why the Yellow or Red River keeps its color from its source to the sea, attributing the color to the earth over which it runs. The Spaniard wrote:

> The yellow or red river is a remarkable thing, and is therefore called *Hoang Ho*. It springs in the west, runs many leagues without the wall, fetches a great compass about it, and returning again courses through *China* until it comes into the province of *Nan King*, where it falls into the sea. Its course is about eight hundred leagues, it is very rapid, and from its sources keeps a bloody hew [*sic*], without changing, or altering its colour in any place. When we went to court, we sailed on it two days and a half, and were surprised and astonished to see its whirlpools, waves and colour; its water is not to be drunk, and therefore we laid in our provision before-hand. Afterwards we observed a secret in nature, till then unknown to us, which was that the waterman and servants filled a jar of this water, putting in a little allum [*sic*], they shaked about the jar; then letting it settle two hours, it became as clear and fair as could be wished, and was so delicate, that it far exceeded the other we had provided, though it was extraordinarily good. In *Canton* I learned another easier and wholesomer cure for it, and it is only putting some small grains which make fish drunk (and in Spanish are called *coca*) into a jar, and the water will become clear in a very short time. (4)

General William Sibert, U. S. Army, of Panama Canal fame, saw water being coagulated in China in or about 1914. He stated:

> When first I boarded our houseboat I saw a Chinaman moving a cane back and forth in a large earthen vessel of water. I saw that the water gradually was being cleared and I asked the Chinaman what he was doing. I found that the cane had been pierced with small holes and that it was full of powdered alum. This alum, in dissolving, clarified the water. . . . This means of clarifying water I found had been used in China for centuries. (5)

England.—The earliest-known reference to the use of alum for water clarification in England is found in Dr. John Rutty's treatise of 1757 on mineral waters (6). Rutty states that "in Kent, where they have little but muddy pond water, it is cleared by throwing into it a little alum." Rutty also says: "I have found several spring waters to become more limpid upon the admixture of alum."

In London, wrote Dr. John Bostock (7), the water served by the New River Company in December 1827 was "very turbid and dark colored." After some hours of sedimentation the water was nearly transparent "but the dark colours still continued." Neither boiling

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nor filtration through sand and charcoal removed the color but "alum and certain metallic salts (sulfate of iron the most effective), especially when heated, threw down a precipitate and left the water without colour." Three years later, Abraham Booth (8) wrote that alum "clears the foulness of water readily. The salt is decomposed by the carbonate of lime and the alumina carries down all impurities."

The horror of an early nineteenth century Englishman over putting alum or any other "foreign matter" into water to clarify it at a time when most of the London water companies were serving turbid and heavily polluted water from the Thames was cited by Arago in 1837 as follows:

——speaking one day of the aluming of water to an English engineer, whose extensive experience had given him much practical acquaintance with the habits and feelings of the public, and who was lamenting the imperfection of the means now in use for purifying water, . . . "what are you proposing," said he immediately, "Water, like Caesar's wife, ought to be beyond all suspicion." * (9)

In 1843, James Simpson stated that "he had in some instances accelerated precipitation by previous mixture of alumina or pipe clay and other materials and had succeeded in throwing down the colouring matter so that the filters produced perfectly pellucid water" (10). No earlier use of coagulation before filtration has been found. Apparently these "instances" were temporary experiences only.

The possibility of clarifying the water of the Thames at London was noted in 1851 by a chemical commission in a report to the General Board of Health (11). The pronouncement was notable because it stated the cause and character of the turbidity concerned, noted the complete disappearance of the "alum" employed and also the nature of chemical and physical reactions. At that time, it may be added, all of the London water companies serving water from the Thames had or were about to have slow sand filters. Quoting now from the report:

Like rivers generally, the Thames is liable to turbidity from floods. It then acquires a yellow colour, well known as the flood tinge, which is of an unusually persistent colour, and only very partially removed by sand-filtration. . . . This clay tinge, which resists the action of acids and does not even fall down with carbonate of lime precipitated in the water, is known to be

* The shades of Caesar's wife have often been invoked to emphasize the importance of water from an unpolluted source but I have never seen the invocation credited to Arago's English engineer.—*M. N. B.*

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removable by alum. We were informed by an officer of one of the companies, that seven grains of alum per gallon [Imp.] would be sufficient in general to precipitate the clay completely, and to produce a perfect discolouration. The alumina is itself entirely removed, but sulphuric acid is introduced which, by converting lime into sulphate, would induce a hardness permanent on boiling. In floods also the water often tastes disagreeably of vegetable matter. (11)

England's earliest adoptions of alum as a coagulant have been summarized in substance as follows by Peter Spence, Managing Director of Peter Spence and Sons, Ltd., chemical manufacturers in Manchester:

As far back as 1881 a reservoir containing the water supply of Bolton was so turbid that it was objectionable. It was treated at the intake at the rate of 1½ grains per gal. [Imp.] and this gave perfectly clear and colourless water. [At Bacup, early in the twentieth century,] a reservoir was purified by applying aluminoferric and chalk from a raft sailing from bank to bank. (12)

Despite the clear understanding of the nature and possibilities of coagulation in England, as shown above by quotations from seventeenth to nineteenth century writers, and by its successful application in at least two cases, it has been practiced there in comparatively few instances. Not even since the tardy and limited rise of rapid filtration there has it been widely used. Under the emergency conditions of the first World War it was introduced on a large scale by Sir Alexander Houston, Director of Water Examinations, Metropolitan Water Board, and his engineering coadjutors, but the purpose was primarily to reduce the cost of pumping to settling reservoirs.

French Experiments and Opinions

Some industrial works in Paris used alum to clarify the waters of the Seine as early as the 1820's. Evidence of this is found in two paragraphs in a memorial on water purification by Raymond Genieys (13). His clear but cautious remarks on coagulation, freely translated, follow:

Clarification by the use of reagents, notably alum.—Divers means have been employed to hasten the separation of suspended matters. In some establishments at Paris they have tried the use of salts that, by a double decomposition with those contained in the water, form salts of a high enough specific gravity to deposit promptly and carry down with them the matters in suspension. But this means has to be modified according to the almost continual and unexpected changes of dissolved salts in the water, thus requiring many precautions. The only application that we cite is its use in

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many manufactories and hospitals that, for their service, cannot employ the water of the Seine as pumped directly from the river when the latter is suddenly charged with muddy particles. The means used consists of alum, or sulfate acid of alumina, and of potassium or of ammonia; this salt acts with much efficiency for separating foreign matters suspended in the waters. They do not yet clearly explain the mode of action in this operation; they know only by experience, that if to a hectoliter of very muddy water they add about 5 grams of alum, the water becomes very limpid in a short time.

They think that the elements which this process introduces in the water are in too small proportion to be harmful in the ordinary uses. But this means is not yet in common use, and filtration, which in other respects has all the advantages, without presenting the inconveniences, is today the process more generally prevalent.

A specific instance of an experiment on the use of alum at Paris, in 1828, follows (14).

"Tainted animal matter" was added to water to give it a bad taste and smell. A drachm of powdered alum was added to 2 gal. (Imp.) of this water. After stirring the water, then allowing it to stand for 24 hours, a deposit of 1 in. was found, the water above which was "perfectly pure in taste and smell" and "more clear and sparkling" than a sample that had passed through a thin layer of animal charcoal. A test showed that at least a third of the alum had been neutralized and that the remainder had not caused "astringency which could at all interfere with the valuable properties" of the water or injure its consumers. An equal weight of carbonate of soda was subsequently introduced to neutralize any acidity remaining in the water.

The earliest disapproval of the use of a coagulant in water treatment was expressed by Arago (9). In a report written in 1837, approving the filter of Fonvielle, there is a long passage, heretofore overlooked by historians of coagulation. It says in substance:

Science, or rather chance, brought to light the means of hastening considerably, or rendering almost instantaneous, the precipitation of earthy matters held in suspension by water. This means consists in adding powdered alum to the turbid water. . . . If it is true that water after having been alum'd, still requires filtration, we can easily conceive why the employment of alum, as a means of clarification, has not become general. Besides, in the large way, the price of the salt in addition to other means, might be objectionable. Another more serious objection is that it affects the chemical purity of river water, that it introduces a salt which it did not before contain,—that in supposing this salt wholly inactive in certain proportions, consumers might fear that at times these proportions might be very materially exceeded, and that this might easily happen through the negligence, or mistake, of a workman.

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FIG. 62. DOMINIQUE FRANCOIS ARAGO (1786–1853)

Secretary, French Academy of Sciences, from 1830 until his death; although he condemned use of powdered alum to hasten settlement of turbid water in 1837, by the following year he helped in advancing the water works art by promoting the Fonvielle filtering apparatus (see Chap. IV)

(From print lithographed at Paris after drawing from life by Maurier)

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. . . [These and other reasons not cited here led Arago to condemn] every means of clarification which would introduce into river water any new substance, that it does not originally contain; and therefore the most recent trials of engineers have all been directed to the employment of inert materials, or those which cannot add anything to the water. These materials are gravel of different sizes, . . . sand of different degrees of fineness and pounded charcoal.

French aversion to coagulation was taken advantage of by the promoters of the Puech multiple filters (see Chap. IV) who claimed that by means of their system not only coagulation but also sedimentation was unnecessary. Despite this assertion both have been introduced in some of their installations.

Outstanding among the nineteenth century studies of coagulation by alum was one made by M. C. Jeunnet, at the Central Chemical Laboratory of Algeria, at Algiers, and reported by him in 1865 (15). These studies seem to have been overlooked by all writers on coagulation except Austen and Wilber, twenty years later (16). Had they been seen and studied without preconceived notions there might have been much less opposition to the use of alum or sulfate of alumina in England, France and the United States in the early years of rapid filtration. "The clarifying action of alum, applied at the rate of 200 to 500 ppm., upon muddy waters," wrote Jeunnet, "is an established fact and has been known for a long time; yet it would seem that this method has always been looked upon with misgiving, even when any other method is difficult to practice. . . . I am now in position to show upon what slight grounds the apprehensions of the hygienists rest." Jeunnet experimented with waters ranging from distilled water to liquid mud. In no case was it necessary to add more than 400 ppm. of alum to produce clarification in seven to seventeen minutes. Subsidence unaided by coagulation required from one to eight days "and the supernatant was still cloudy." Humus bodies and organic matter seemed to be removed completely. Summarizing his findings, Jeunnet said that

——turbid water, no matter what be the nature or the quantity of the earthy material it holds in suspension, can be made potable within a period of from seven to seventeen minutes by adding to it finely powdered alum at the rate of 400 ppm., provided that the whole bulk of the liquid is, at the time when the reagent is added, in vigorous agitation. In this operation, the alum dissociates instantly into its constituent salts; the potassium sulfate is found quantitatively in the treated water; the aluminum sulfate decomposes and thereby brings about clarification; its base separates in in-

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soluble form and carries down with it the suspended solids and the humus bodies, while the acid attacks the alkaline and alkaline earth carbonates and converts them into sulfates. Therefore the purified water shows enrichment in the sulfates of potassium and calcium, and in free carbon dioxide and sometimes also in bicarbonate; and it has lost its organic content.

Alum in quite a large excess will react in just the same way; it will be completely decomposed without any ill consequences to the water, other than trifling additional quantities of the sulfates of potassium and calcium. When the latter salt already is present in concentration approaching saturation, the reaction is not interfered with, but some calcium sulfate is precipitated.

Sodium alum reacts exactly like ordinary alum, there being no appreciable gain in time from its greater solubility. Aluminum and ferric acetates react only slowly and incompletely and are therefore unsuitable. Although slower in action than alum, aluminum biphosphate might be preferable if it were not that the liberated carbon dioxide redissolves notable quantities of earthy phosphates, which cannot be got rid of, even by boiling.

Aluminum sulfate is equally effective with alum; less of it is needed in ratio of 7 to 10; and it introduces no alkaline sulfate. (15)

Frank Hannan, translator of the above passage, and former chemist for the water works at Toronto, Ont., makes the following comments on some features of Jeunnet's paper:

The pH concept might help to explain certain things. Owing to temperature, pH of Algerian natural waters is probably high. By "alum," Jeunnet always means $KAl(SO_4)_2 \cdot 12H_2O$. We have drifted into calling aluminum sulfate "alum." Superiority of aluminum sulfate as a coagulant is clearly recognized.

The Netherlands and Belgium

A report on the efficiency of alum as a coagulant when applied to water carrying both finely divided clay and organic matter was made by a Netherlands Commission in 1869 or a little earlier. Some members of the commission proved, according to an abstract in a London journal, that the turbidity of Netherlands waters was "due to extremely minutely divided clay," which so held up organic matter that the combined material would "pass through filters and not deposit, even after many days of rest." Alum at the rate of 10 to 20 ppm. produced "a flocculant precipitate" which took up the turbidity of the water and left it perfectly clear (17).

The commission had been instructed to find means of improving the potability of waters where needed, particularly those drawn by villages and towns on the lower Maas, where "from time immemorial"

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persons not accustomed to using the water daily suffered from diarrhea. Repeated chemical and microscopical analyses had never "revealed the precise cause of this peculiar property, which is not possessed by the water of the same river, higher up" (see also *Iron and Its Salts* below).

Groningen, Holland, seems to have led the way in the use of alum for a municipal water supply. It built water works in 1879–80. While plans for a filter plant were under way, B. Salbach, engineer of Dresden, Germany (18), realized that pretreatment would be needed to remove all color from the peaty water of the River Aa. With the approval of Professors Huizinga and Van Calker, of the University of Groningen, he decided to use coagulation in fill-and-draw settling basins before the water went to slow sand filters. Tests showed that a minimum of 120 ppm. of "sulfated potassium aluminate" was necessary for complete decolorization. At each stroke of the pump which delivered water to the settling basins a solution of alum was also delivered by a small pump, ensuring the mixing of a proportionate amount of the coagulant to the raw water.

Allen Hazen, in the first edition (1895) of his book on filtration (19), stated that alum had been used repeatedly at Leeuwarden, Groningen and Schiedam, Holland, where the river waters are "colored by peaty matters which cannot be removed by simple filtration" but its use had been "generally abandoned, or at least restricted to time when the raw water is unusually bad." At Antwerp, Belgium, he added, resort was made to alum during the drought of 1893 to obtain "a better filter effluent, especially as there was some fear of cholera."

The Leeuwarden plant, apparently built in 1891, combined alum-coagulation with sedimentation, cascade-aeration and filtration (20).

America

Two early American improvised uses of a coagulant illustrate what has probably been done many times in other countries with means readily available. Abraham Booth, in his little book of 1830 (8), states: "Some toasted biscuits put into the water of the St. Lawrence were found serviceable in preventing its bad effects in the fleet of Sir Charles Saunders." Apparently Booth had in mind the large British fleet that arrived near Quebec in June 1759 before Wolfe fell in his gallant exploit. Use of Indian meal, sprinkled on the surface

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of a pail of water by boatmen on the Mississippi, was reported in 1819 by Schoolcraft, an American mineralogist, who was, however, better known for his writings on the American Indian (21).*

Use of coagulation on a public water supply in America was begun in 1885 by the Somerville & Raritan, N.J., Water Co. The process was firmly established as an adjunct to rapid filtration by that company's project (see *The Hyatt Mechanical Filter,* Chap. VII). Since then it has been, with few exceptions, part and parcel of rapid filtration in America.

Notable American studies of alum as a coagulant were made in January 1885, by Professors Peter T. Austen and Francis A. Wilber of Rutgers University, New Brunswick, N.J. (16). Theirs was the first scientifically conducted American investigation of the subject; it followed closely the granting of the Hyatt patent on simultaneous coagulation and filtration; it fixed a reasonable limit on the alum dosage; it suggested that coagulation as an aid to sedimentation without filtration was a possible treatment method but would require a long period of sedimentation; it also indicated, but with inadequate proof and without emphasis, that alum could be applied to filters without intermediate coagulation basins. The authors stated that of the many reagents suggested and tried for water purification none seemed to offer the advantages of alum. They also noted Jeunnet's paper of 1865, already summarized above, and mentioned the use of alum in the Hyatt filter. They added that

> ———by the addition of a minute amount of alum, water is rendered capable of a most perfect mechanical filtration. . . . *If it can be proven that alum not only clarifies water, but also removes from it disease germs and ptomaines, its use will prove of incalculable value to the human race, for facts begin to indicate that a vast number of diseases are communicated through drinking water* [author's italics] (16).

In an article by Austen it is stated that alum might be used where purification of the water is needed only at some seasons of the year

* At my suggestion, A. V. Graf, Chief Chemical Engineer, St. Louis Water Works, in 1936 tested the efficiency of corn meal in reducing the turbidity and bacteria in Mississippi River water. Two tablespoonfuls of corn meal were added to 2 l. of water, the water stirred, allowed to settle for 30 minutes, and then the top liter siphoned from the bucket. The turbidity of the river water was reduced from 1,900 to 150 ppm., the bacterial count from 5,600 to 2,500 per ml. and the *Esch. coli* index from 10,000 to 9,000. A parallel test with 2.5 g. of prepared mustard per liter gave less reduction of turbidity and total bacteria, but higher reduction of the *Esch. coli* index.—*M. N. B.*

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and where the expense of large pumps and filters would be a burden (22).

Soon after Austen and Wilber suggested the use of coagulation without subsequent filtration, this practice was followed at several water works, in some cases years before filters were built.

At Omaha, Neb., the water company introduced coagulation 34 years before rapid filtration. On August 1, 1889, when beginning to draw water from a new intake, upriver from the old one, the use of sulfate of alumina was begun. "It was used somewhat crudely for several years," wrote Theodore Leisen (23) long afterwards, "by injecting it into the discharge lines of the low-service pumps and dumping additional amounts into the weirs between the settling basins. Manufacture of aluminum sulfate at the plant was begun in August 1916 by the Metropolitan Utilities District. Rapid filters were put into full operation in October 1923."

The next known American use of a coagulant before filtration was in 1901, at Chester, Pa., when the New Chester Water Co. built a pumping station on the polluted Delaware River and two 7-mil.gal. settling reservoirs on Harrison Hill. Rapid filters were added the next year (24). In 1902, a coagulant was applied to settling reservoirs at Kansas City, Mo. (see Chap. XII).

The next recorded instance of the use of coagulation before filtration, and in some respects the most notable, was at St. Louis, Mo., early in 1904; but instead of sulfate of alumina, lime and sulfate of iron were used (see Chap. VII). Coagulation without filtration was put into use at Selinsgrove, Pa., in 1905 (25); Iola, Kan., 1906 (26); and Nashville, Tenn., in 1908 (27). Thirty-seven instances of coagulation without filtration, scattered through America, were listed in 1915 (28). By that time, rapid filtration was in full swing, generally combined with coagulation (see Chap. VII).

Antedating these plants, there appeared what seems to be the first American water works to employ coagulation as an aid to sedimentation. Although the water was passed through a filter, the filter was so small that it may be ignored, except as a curiosity.

The Vicksburg, Miss., Water Co. built works in 1887–88. Clarence A. Delafield, New York City, was engineer. Water was taken from the Mississippi River. Writing of the character of the water from the point of view of a Northerner, accustomed to clear or relatively clear water, Delafield stated, in a description of the water works:

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"This turbid water is thoroughly enjoyed by those who are accustomed to it . . . ; but to those unfamiliar with its use the appearance of the liquid is disgusting, and a clear bright water is insisted on" (29).

The Vicksburg settling reservoir was of the flowing-through type, in three compartments, with combined capacity of 1 mil.gal. "Alum," at the rate of about 1 grain per gallon, was pumped to a long, narrow influent chamber and passed through numerous port holes, 4 ft. above the bottom of the chamber, into the first settling compartment. From the top of this compartment the water went down through passages in the division wall and was discharged into the

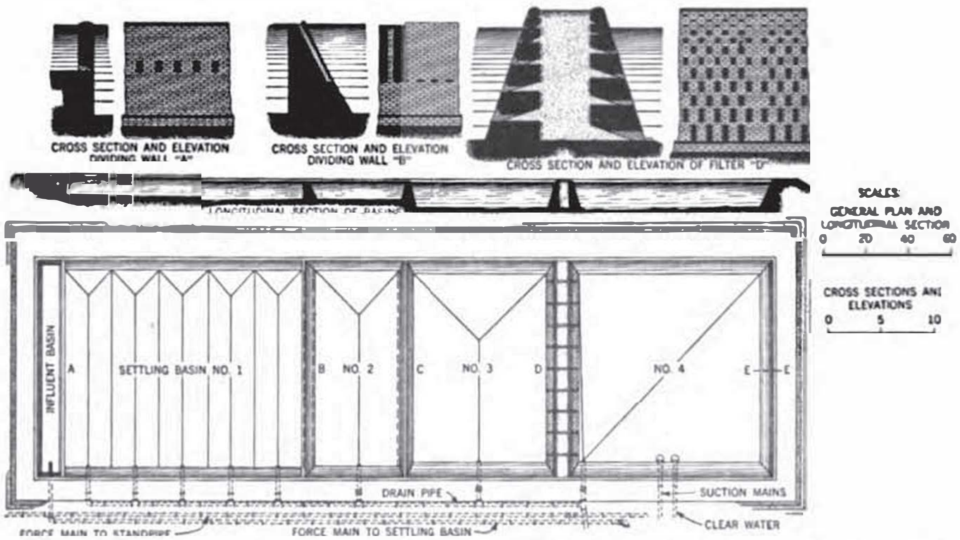

FIG. 63. COAGULATION AND SEDIMENTATION BASINS AND LATERAL-FLOW FILTER AT VICKSBURG, MISS.

Plant designed and built in 1887–88, by Clarence Delafield, Engineer (From Delafield's "The Vicksburg Settling Basin," *Trans. A.S.C.E.*, August 1889)

second compartment 3 ft. above its bottom. Discharge from the second to the third compartment was in a thin sheet over the division wall. From the final settling compartment, water passed to a small lateral-flow filter located in a wall and thence to a clear-water chamber. Although Delafield stated that after the treatment plant had been in use several months the final effluent was giving "perfect satisfaction," four Jewell rapid filters were installed in 1890. Postchlorination was begun in 1918. The city acquired the works in 1915. At an unstated date the clear-water basin was covered to prevent algae growths (30, 31).

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Lime for Clarification

Notwithstanding the extensive use of lime during the heyday of chemical precipitation of sewage, it has seldom been used, alone, for water clarification, although it has been used with sulfate of iron at a relatively few water treatment plants, large and small. Abraham Booth, in his book of 1830 (8), states: "At Senegal [West Africa] where the water is extremely unwholesome, unslaked lime has been used to purify it." At Sandhurst, Victoria, Australia, about 1873, use of lime was adopted to precipitate clay before slow sand filtration; it was used also at a second plant, without filtration (32). William Ripley Nichols (33) wrote in 1883 that "at several other works [than Sandhurst] lime is used . . . sometimes followed by filtration." No hint as to the location of these works is given. Studies of the excess-lime method of water treatment are detailed by Sir Alexander Houston in his London Metropolitan Water Board reports for 1912 (34) and later. In the following year (35), he notes various conditions in which the use of lime "appears to be especially attractive" and mentions the adoption of "liming" by Aberdeen, Scotland, in 1913. In his 1930 report (36), he tells that the excess-lime method was used by the Southend Water Co., England, in 1929—and the company appeared to be still using it in 1939 (37).

In 1943, Wattie and Chambers studied the relative resistance of coliform organisms and certain enteric pathogens to excess lime treatment in order to evaluate the frequent claim that this treatment will make the water bacteriologically safe. They reported that disinfection can be accomplished by such treatment within definite limitations of contact time and pH value (38).

Iron and Its Salts

Iron, either as an element, or as a part of a salt, was covered in many treatment patents and was employed as a coagulant at several water treatment plants in the last half of the nineteenth century. Outstanding among the patentees were: Medlock in 1857, with a proposal to produce iron oxide by merely suspending iron in water (see below); Spencer, also in 1857, with his magnetic carbide of iron as an oxidizing filtering material; Bischof in 1870, with spongy iron, also as a filter medium; Anderson, in the 1880's and 1890's, with his revolving cylinder in which metallic iron was constantly dropped into water

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as it passed through, the water then being aerated and filtered; and Sellers, who followed Anderson closely in time and method, using, perhaps unwittingly, the suspended iron rods of Medlock's patent, but making the rods revolve and rub on each other. All these schemes, except Medlock's, were put into use, but the total number of plants was small and all were soon abandoned. Better success attended perchloride and some other salts of iron.

Henry Medlock's British patent of January 21, 1857, on treating water with metallic iron, was notable because it aimed at oxidizing organic matter in solution in accordance with a growing conception of the dangers from organic matter or its products of decomposition. He proposed to put "scrap iron, iron turnings, iron wire, or sheet iron" in a vessel, using about 1 lb. of iron per gallon of water. After contact of 24 to 48 hours, the precipitate would be removed by filtration through sand. His specifications describe the chemical reaction of the iron on the water.

Six months after Medlock's patent was granted (June 19, 1857) one was issued to Thomas Spencer, of London, for the removal of "organic color and deleterious gaseous bodies" from water, using an oxide of iron either independently or in combination with subsequent filtration. A second British patent (June 23, 1858) covered the manufacture of "magnetic carbide of iron by heat." Ground to the size of ordinary sand, the product was to be used as a filtering material. If turbidity as well as color were to be removed, fine sand was added to the magnetic carbide. A dissertation on coloring and organic matters in water and their removal was embodied in the specifications of the first patent. A paper by Thomas Spencer, "On the Supply and Purification of Water," published in abstract in 1859 (39), told how he came to develop magnetic oxide or protocarbide of iron and explained its action on water. His correlation of the oxidation by iron oxide and by ozone is noteworthy in a paper published more than 85 years ago.

Unlike most of the multitudinous methods of water purification patented in England and elsewhere, Spencer's filter was adopted for several water works. A brief trade report in *Engineering* (London) (November 16, 1866) under the title, "Filtering Water," stated that filters of magnetic oxide of iron "overlain by sand, are now in use at Wakefield, Southport, and Wisbeach" (England). A week later, Spencer stated (39) that his process was to be applied to the Hooghly River

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water supply of Calcutta, India. Kirkwood's St. Louis report of 1869 (40) describes the Spencer filters at Wakefield, which he said produced satisfactory results with the water drawn from the Calder, characterized as "of a dark, inky hue, slightly offensive to the senses." A less favorable picture of the Wakefield water supply, with condemnation of the source rather than the filters, appeared in a report of the Rivers Pollution Commission (41). The filters were used for two decades.

Gustav Bischof, Professor of Technical Chemistry, Andersonian University, Glasgow, devised the spongy iron process of water treatment in 1870. Like Spencer, he placed a layer of his patented material below a layer of sand. Bischof, who had the advantage of the early knowledge of bacteria, claimed that his spongy iron destroyed disease germs. After establishing a large business in domestic filters he or his associates organized the Bischof Spongy Iron Water & Sewage Purifying Co. Seven British patents were granted to Bischof in the period, 1870–87. The first and basic patent (September 19, 1870) concerned the use of spongy iron for filtering and purifying water—preferably spongy iron made from purple or spent ore of iron pyrites. Notwithstanding high praise for the spongy-iron process, based on laboratory tests made in 1873–74 by the Rivers Pollution Commission (41), the material seems to have been used by only one municipal water supply in Great Britain—Stamford. A more important adoption was by a British-controlled water company of Antwerp, Belgium, in which William Anderson of London held an important position (42). After laboratory experiments, spongy iron was included in a permanent plant put into use on June 21, 1881. Water from settling reservoirs was passed through three prefilters and three final filters, the former containing spongy iron. Provision was made for aeration between the two sets of filters. In a lengthy discussion on Anderson's paper (42), Dr. Edward Frankland, whose opinion carried great weight in England for many years, emphasized the removal of bacteria by the filters but gave no supporting data. Anderson, in closing the discussion, said it had centered mainly on whether the "bacteria and their germs were or were not destroyed by the spongy iron; but what was wanted was a substance which would take out the colour of the water, which would remove the turbidity and which would reduce the organic contamination to an amount which would admit to be reasonable." Strange doctrine to relegate germs to the rear, but that was early in 1883!

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Cementation of the gravel and spongy iron layers of the Antwerp filters, only two years after they were put into use, led Anderson to make experiments which resulted in the "Anderson Process." This was merely a device for producing by attrition finely divided particles of metallic iron, which were to be dropped into water flowing to a slow sand filter. It eliminated the spongy iron prefilter of the Bischof process, served the same general purpose as Spencer's carbide of iron, and was a more efficient method of producing oxide of iron than suspending iron wire idly in water as patented by Medlock in 1857. That the Anderson process did not originate with Anderson, although the revolving purifier did, is shown by the words of both Anderson and his latter-day partner, Easton Devonshire. In a paper read in 1883 (43) Anderson said that he, in association with Henry Ogston, studied the possibilities of agitating finely divided iron and water. It was decided to follow a method suggested by Sir Frederick Abel, "who in Medlock's time had already had considerable experience in the use of iron for purifying water," and had decided in "favor of simple agitation of a comparatively small quantity of iron with the water to be treated." No sooner did Anderson adopt Abel's suggestion than he began to take out patents, first in England, then in the United States. His first English patent, dated November 23, 1883, covered the purification of water by passing it through spongy iron placed in a horizontal cylinder, revolving on hollow trunnions which served as water inlets. To keep the iron constantly falling, longitudinal shelves were attached to the inside of the cylinder. In a second British patent, as also in most of the eight American patents granted to Anderson or his associate, Easton Devonshire, in 1885–91, plain rather than spongy iron was specified.

A permanent installation was put in use by the Antwerp Water Works Co. early in 1883. The entire supply of about 2.6 mgd. (U.S.) passed through three revolving cylinders before going to slow sand filters (42). By May 1904, stated Devonshire (44), the number of revolving cylinders had been increased to five. Air was forced through holes in a false bottom of the cylinders "thus aerating the water and changing the ferrous to ferric oxide." Owing to variations in the character of the water, the iron did not always "precipitate completely before passing through the filters, making it necessary to throw the revolving purifiers out of service." Rusting tanks were tried as a substitute for the cylinders (44). Apparently this arrange-

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ment was unsuccessful for in 1908 Puech-Chabal multiple filters were installed (45). On January 1, 1930, a second Puech-Chabal plant was also in use, much farther up the Nethe. At each plant precoagulation with sulfate of alumina was being used—unusual with Puech-Chabal filters (46). The Anderson process was used at a few other places on the continent of Europe and a plant was installed at Worcester, England, in 1892. Allen Hazen, in his book on filtration (1895), reviewed the Anderson process and expressed himself unfavorably regarding it (19). Judging from opinion expressed late in 1899 by several European water works men in discussing a paper by Weyl, the Anderson process had by then nearly run its course on the Continent (47).

A campaign to introduce the Anderson "revolving purifiers" in the United States, extending through several years, was a complete failure. In the early 1890's, demonstration cylinders were set up at Boston, Philadelphia, Allegheny (48), and St. Louis, and what was intended to be a permanent plant, which never functioned, was installed at Poughkeepsie, N.Y. A notable account of the trial at St. Louis, with reasons for its failure, appeared in the annual report of the water works for 1893–94.

By far the most ambitious attempt at coagulation with metallic iron was made at Wilmington, Del., by George H. Sellers, beginning in the 1890's. Following the construction of emergency plants built for the Tacoma, Wash., Water Co., of which Sellers was chief engineer, he obtained a contract for an elaborate plant at Wilmington, which was operated until 1903 and then abandoned as useless when Theodore Leisen became chief engineer of the Wilmington water works. Bundles of revolving iron rods were supposed to produce a coagulant applied to an upward-flow filter working at a high rate. The process was combined with aeration. The general idea both at Tacoma and at Wilmington was suggested by the Anderson process. Sellers, whether knowing it or not, harked back to the Medlock patent of 1857 in using iron rods, but went one better by having them revolve. He took out four United States apparatus patents and one process patent on July 24, 1896, and a British patent three days later, and organized the United States Filtering & Purifying Company of which he was manager.

Perchloride of Iron.—Perchloride of iron for the purification of water was studied in Europe and America between 1850 and 1900.

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Its use was proposed and may have been adopted by Amsterdam in the late 1860's. L. H. Gardner induced St. Louis to try it in a large reservoir in the early 1880's. He experimented on a small scale and, at his suggestion, it was tried by Isaiah Smith Hyatt at New Orleans in 1883. This trial seems to have led to the Hyatt coagulation-filtration patent of 1884 (see *The Hyatt Mechanical Filter,* Chap. VII).

The earliest study of perchloride of iron for water treatment found was reported by Peligot early in 1864 (49), in a memoir on the composition of water. After two paragraphs describing the waters of the Seine and the Canal de l'Ourcq at or near Paris, Peligot included perchloride of iron in a discussion of several coagulants that would clarify swamp or putrescent waters. The passage reads:

> Copper sulfate, ferrous sulfate, ferrous chloride and especially ferric chloride produce in [water], when added in suitable quantity, flocculent precipitates which settle out more or less rapidly at the bottom of the vessel. In the case of ferric chloride, the deposit, in the form of ochreous floc, settles out in a few minutes. In the case of copper sulfate, settlement of the greenish precipitate which is formed is not complete until after standing for twelve to fifteen hours.

Seventy-three days after the publication of Peligot's memoir, Carl J. A. Scheerer, of Freiburg, Saxony, obtained a British patent (July 7, 1864) on "an improved process for cleansing and clarifying water by adding to it a very dilute solution of the neutral sulfate of peroxide of iron ($Fe_2O_3 + 3SO_3$)." Shortly after this agent is added to the impure water

> ———it becomes decomposed, and forms, with some of the impurities contained in the water, a basic salt, which is insoluble in water. The solid and insoluble particles of this new salt are precipitated, and, together with the impurities in the water, form a sedimentary deposit from which the purified water may be allowed to run off, leaving the sedimentary deposit in the tank or reservoir.

Perchloride of iron was given much attention in a report on water purification made by a Netherlands Commission in 1869 (17). Dr. J. W. Gunning of Amsterdam is cited as having shown that 0.032 g. of perchloride of iron, added to 1 l. of water from the River Maas, or to even fouler water, made it "perfectly wholesome and even agreeable for use." After setting forth more testimony of the same tenor the report says that water treated with perchloride of iron and carbonate of soda, then filtered through fine sand, would be supplied to

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Amsterdam through the water works then proposed for that city. The report also stated that the committee felt justified in issuing an order for the use of the process on the bad waters existing in many parts of the kingdom (17). (For a portion of the report dealing with alum, see above.)

Overlooked by previous writers on coagulation are the studies of the 1880's on perchloride of iron as an aid to sedimentation made by L. H. Gardner, Superintendent of the New Orleans Water Works Co. for many years before water works were built by the New Orleans Sewerage and Water Board. In May 1883, he briefly outlined his small-scale experiments with perchloride of iron on the muddy Mississippi at New Orleans (50). In October 1885, he published a summary of methods of water clarification used or proposed during the two decades past, including his office experiments at New Orleans and his tests on a 12-mil.gal. reservoir at St. Louis. He outlined Spencer's magnetic carbide and Bischof's spongy iron filters and the precoagulation methods of Anderson (see above). After trying all known precipitants, he "was convinced that a solution of iron is of easier mechanical application to water" than any of the others tried; that the "rapidity of action narrows the necessary area of settling basins materially"; that "mechanical devices of the simplest character" would admit to the influent water $\frac{1}{8}$ lb. of iron to 1,000 gal. of water; and that "settling reservoirs can be made self-cleaning." In this paper (51), Gardner related his earlier suggestion to Isaiah Smith Hyatt that led to the coagulation-filtration patent of 1884 (see Chap. VII).

Gardner's advocacy of iron as a coagulant went unheeded. Alum or sulfate of alumina was adopted in the Hyatt and other rapid filters and for two decades held undisputed sway. Then sulfate of iron, supplemented by lime, entered the field and gained a strong foothold. In limited use today are other iron compounds such as ferric chloride, chlorinated copperas (ferrous sulfate reacted with chlorine) and ferric sulfate, but sulfate of alumina still dominates the field.*

The earliest explorer that the author has found in the sulfate of iron field was somewhat casually mentioned in 1872 by Dragendorff †

* The nature, reactions, advantages and disadvantages of the iron compounds and of sulfate of alumina, as understood in 1940 by specialists in water treatment, are concisely reviewed in *The Manual of Water Quality and Treatment*, American Water Works Association, New York (1940).

† G. Dragendorff was a professor in the university town of Dorpat, 165 miles southwest of Leningrad.

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thus (52): "Years ago, as is well known, Scheerer proposed for purification of water, especially when it contains a large quantity of organic substances, the use of a solution of ferric salt, especially of the sulfate."

"About two years ago," said Dow R. Gwinn, Superintendent of the Quincy, Ill., Water Co., in 1900 (53), "William Jewell of Chicago, with the assistance of the writer, began some experiments on the use of iron as a substitute for alumina at Quincy." The coagulated and settled water was pumped to mechanical filters. Among eight patents granted to William M. Jewell on July 17, 1900, three were for producing a coagulant by the reaction of sulfurous acid, iron and water. Looking back in 1930 upon the early events at Quincy, Wolman, Donaldson and Enslow (54) wrote that "as early as 1903 William B. Bull used ferrous sulfate and lime coagulants at Quincy, Ill." Bull was one of the owners and the leading figure at the Quincy water works for many years.

The first large city to adopt ferrous sulfate and lime was St. Louis, Mo., where an installation was put into use on March 22, 1904. This was done in great haste in order that visitors to the St. Louis Exposition might not be repelled by the turbid water which St. Louisans had put up with since Kirkwood's recommendation for sedimentation and slow sand filtration was ruthlessly thrust aside in 1866. A permanent plant was soon built. Next among the large cities to use sulfate of iron and lime were Cincinnati, beginning on November 1, 1907, and New Orleans, commencing in February 1909.

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Chapter XIII

Coagulation: Ancient and Modern

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CHAPTER XIV

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