Standard Sulphuric Acid.—A decinormal solution is used. Of this one cc. = 0.0006 gram carbon.

This method is best adapted for low carbon steels, because only small quantities of barium carbonate have to be filtered and washed.

When analyzing high carbon steels I have used smaller weights of steel (one or two grams) and have rinsed the barium carbonate and excess of barium hydroxide solution into a liter flask; after making up to one liter with pure water, I filter off 200 cc. and titrate by standard sulphuric acid. Steel 11920 gave 1.35 per cent. and 1.35. per cent. carbon by this method.

The work which I have been able to do seems to me to suffice to call attention to the merits of the barium hydroxide methods.

THE CONTRIBUTIONS OF CHEMISTRY TO THE METHODS OF PREVENTING AND EXTINGUISHING CONFLAGRATION.

By Thomas H. Norton.

[*Continued from page 147.*]

The interaction involved the formation of gypsum and aluminum chloride, which, in turn, precipitated the gelatin and converted it into a leatherlike insoluble substance. The object was to fill the pores of the fabric with gypsum and cover the surface with a hard binding material. Sir Frederick Abel, in reporting on the feasibility of this process found that it rendered the canvas very difficult of ignition, but that it also gave to it a degree of rigidity and harshness which forbade its use. In 1856 Maugham[1] patented the use of ammonium phosphate and starch; and in 1857 Thouret[1] patented the use of a mixture of three parts of ammonium chloride and two parts of ammonium phosphate, adopting these proportions on account of cheapness, although the ammonium phosphate alone gave most excellent results.

During this period the importance of better protection for the scenery of theaters was recognized. After a serious fire in the Berlin opera house, the custom was inaugurated of soaking all scenery in a strong alum solution.[2] In 1857 a commission in

[1] Amer. Arch., 13 and 14.

[2] Frémy, *Dict. de Chimie*, 10.

Paris carefully examined the subject, and in accordance with their recommendations the order was issued to have all scenery in theaters impregnated with waterglass.[1] After the lapse of some years it was found that scenery thus treated possessed but slight resistant power. The explanation advanced is that the waterglass, on drying, contracts steadily until the solid particles finally sit very lightly on the yarn of the canvas. Another is that a solvent action is exercised by the water in the water-colors often used by scenic artists. It has been suggested by Hexamer that the impregnation with waterglass could be advantageously followed by treatment with hydrochloric acid, thus precipitating silicic acid directly in the fibers of the yarn, a process practically similar to that of the use of mordants in dyeing.

Versmann and Oppenheim,[2] in 1859, reported to the British Association for the Advancement of Science the results of an elaborate and extended study on the use of salts in treating fabrics, including some forty different substances in the range of their experimentation. Their tests were made on muslin free from starch, weighing 33.4 grams to twelve square inches; and more in the direction of ascertaining the strength of the most effective solution, rather than the weight of a given salt absorbed. After immersion in a solution the excess was removed by pressing and not by wringing. Tests were conducted on a large scale in muslin works and laundries. None of the salts recommended to that time were found available where the operation of ironing was to be performed. Either a smooth surface could not be obtained, or the material was injured on the application of heat.

The valuable results of Versmann and Oppenheim may be briefly summarized as follows:

KCN—very effective in a ten per cent. solution, but poisonous and expensive.

Na_2CO_3 and K_2CO_3—both very effective in a ten per cent. solution, but the one is efflorescent and the other deliquescent.

$NaHCC_3$—very effective in a six per cent. solution, but carbon dioxide is rapidly lost and the protective power disappears.

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[1] Frémy, *Dict. de Chimie*, 10.

[2] Loc. cit.

$Na_2B_4O_7$—very effective, but on warming, boric acid is liberated and attacks the fabric.

$NaOH$—effective in eight per cent. solution.

Na_2SO_4—no effect.

$NaHSO_4$—twenty per cent. solution is protective, but the stuff is gradually attacked.

Na_2SO_3—twenty-five per cent. solution is protective, but the stuff is gradually attacked.

Na_2HPO_4—a saturated solution is effective, but the fabric becomes perfectly stiff.

Na_2SiO_3—the fabric is strongly attacked and the appearance affected.

Na_2SnO_3—protective, but attacks the fabric.

$(NH_4)_2CO_3$—too volatile.

$(NH_4)_2C_2O_4$—renders the fabric combustible.

$(NH_4)_2B_4O_7$—a five per cent. solution gives good protection, but the acid is easily liberated and corrodes.

$(NH_4)_2SO_3$—a ten per cent. solution is very effective, but the salt is deliquescent.

NH_4Cl—a twenty-five per cent. solution gives excellent results, but stiffens the fabric.

$(NH_4)_2HPO_4$—gives excellent results alone or when mixed with ammonium chloride as in Thouret's patent. Maugham's mixture of this salt with starch was not available on account of uneven distribution throughout the mass.

$(NH_4)_2SO_4$—when rendered perfectly neutral by a little ammonium carbonate, this yields the best results of all the ammonium salts. Chevalier's mixture of this salt and borax attacks fabrics at a summer temperature.

$$\left.\begin{array}{l}SnCl_2\\ SnCl_4\\ SrCl_4 2NH_4Cl\end{array}\right\}\text{All these are good protectives, but attack the material.}$$

The following salts give good protection when used in solutions of the strength indicated, but are not available on account of price, or corrosive, or other properties; $BaCl_2$ fifty per cent. solution, $CaCl_2$ ten per cent., $Al_2 3SO_4$ seventy-seven per cent., $KAl 2SO_4$ thirty-three per cent., $NH_4Al 2SO_4$ twenty-five per cent., $FeSO_4$ fifty-three per cent., $CuSO_4$ eighteen per cent., $ZnSO_4$ twenty per cent., and $ZnCl_2$ eight per cent.

Unsuccessful attempts were made to fix upon the fibers such protective salts as $BaSO_4$, $Al_2(PO_4)_3$, and various silicates. Zinc oxide and aluminum oxide gave good results, but would not adhere when washed. Antimony chloride was effective and withstood water, but not soap or soda. Stannous borate, phosphate, and arsenate gave good protection and withstood washing, but gave a yellow tinge to the fabric. Zinc and calcium stannates, while efficient protectors, would not withstand soap or soda. Stannic oxide was fixed permanently but imparted a yellow color. It seemed to be well adapted for coarse material, sail-cloth, canvas, etc.

For light stuffs, to be ironed, sodium tungstate was found to be the best agent, and the most effective solution is one of 28° Tw. or 1.14 sp. gr., containing also about three per cent. of sodium phosphate in order to prevent the formation and precipitation of the acid tungstate.

Where the hot iron is not to be applied directly, ammonium sulphate can be advantageously employed in a ten per cent. solution. The fabric is to be dried in chambers; the ordinary colors on prints, except madder purple, are unaffected.

Versmann and Oppenheim at first sought to produce, artificially, conditions similar to those existing in animal fibers such as silk and wool, which are not inflammable, and which contain about eighteen per cent. of nitrogen. Experiments to incorporate nitrogenous substances such as glue or albumen into vegetable fiber were without practical result, although it was found possible by the use of urea to introduce into muslin thirteen per cent. of nitrogen, rendering it thereby uninflammable.

Essentially the same object is obtained, as we have seen, by the use of ammoniacal salts.

Sir Frederick Abel[1] made reports in 1859 and 1860 to the English Ordnance Department on Versmann and Oppenheim's results, especially with reference to the protection of canvas. The advantages of sodium tungstate for light fabrics were fully confirmed by him. The availability of stannic oxide for sail-cloth was likewise confirmed. Sail-cloth was most effectively deprived of its inflammability thereby, and the protective agent was permanently fixed in the fabric, being affected neither by friction nor

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[1] Amer. Arch., 13 and 14.

by repeated washing, while the strength of the canvas was not diminished. The only objections to the use of stannic oxide were: First, the notable increase in weight amounting to fifty per cent. of the original weight of the canvas; and, second, the comparative costliness. While the first objection was regarded by Abel as practically inseparable from the permanent fireproofing of fabrics, the second he considered much more serious. Accordingly, we find him, shortly after, patenting and submitting to the Ordnance Department, a cheaper process for fireproofing canvas, consisting of the deposition in the fiber of a double sodium and lead silicate. Boiling solutions of basic lead acetate and of waterglass were used. I have not been able to ascertain how extended a use was made of this process. Abel in his reports states that he finds saturation with a solution of waterglass alone an efficient protective; but that its value is temporary only. He sums up the difficulties to be encountered in fireproofing fabrics as follows:

(1) The protective material renders the fabric harsh and rigid; or (2) it absorbs moisture and keeps the fabric damp; or (3) it affects the strength and durability of the fabric; or (4) it is easily detached by rubbing or shaking; or (5) it is soluble in water and must be renewed after wetting.

In 1870 Abel[1] recommended to the Ordnance Department treatment with calcium chloride for rope mantelets on board warships in order to keep them damp and prevent ignition during the firing of guns.

In 1871 the Austrian chemist Patera[2] introduced the use of magnesium borate as a protective for fine fabrics and delicate colors. The materials are soaked in a bath of three parts of borax and two and one-half parts of magnesium sulphate in twenty parts of water with the necessary amount of starch, then wrung between cloths and dried. For coarser stuffs he found mixtures of ammonium sulphate and gypsum effective.

In 1882 a special committee of the Franklin Institute of Philadelphia reporting on safety devices for theaters, gave the results of their experiments on the fireproofing of scenery and gauze.[1] They obtained the best results with the material devised by Dr.

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[1] Amer. Arch., 13 and 14.

[2] Flammenschutzmittel, Wien, *1871*.

J. Papen, of Frankfort, Germany. The details of its composition are not given, but it has rendered excellent results in the Frankfort opera house, yielding efficient protection, not altering in time, nor affecting the strength or color of the fabrics, and producing no injurious effect on the voices of singers or actors. The committee obtained also very good results from the use of ammonium sulphate, of silica precipitated on the fiber, and from the processes of Gantsch and Judlin, details of which are also lacking.[1]

This question of fireproofing scenery, etc., in theaters had already been the subject of considerable investigation. In 1877 a committee of the House of Commons took evidence on the matter, and we find Mr. Henderson, of the Criterion theater testifying that he used regularly sodium tungstate in the preparation of new scenery; although it was not available for old scenery.

A committee of the London Society of Arts,[1] in 1883, made a report on the same subject, recommending sodium tungstate as the best safeguard for scenery. They found on investigation, that the scenery in nearly all London theaters was treated with some fire-proof preparation; most having as a basis a borate or a silicate. They add in their report: "these compositions do not prevent the evolution of gas when an article is exposed to sufficient heat; and the gas takes fire and burns. When the external source of heat is removed, no more gas is evolved, and combustion ceases. Prepared articles burn when exposed to sufficient heat, but do not support combustion. One effect of this is that it is very much more difficult to set such materials on fire; and this either prevents the breaking out of the fire, or renders it much more easy to deal with when broken out."

The theaters of Paris had already used largely for protecting scenery, a solution of the following composition devised by Martin and Tessier:[2]

Ammonium sulphate	8
Ammonium carbonate	2
Boric acid	3
Borax	1
Starch	2
Water	100

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[1] Amer. Arch, 13 and 14.

[2] Frémy, *Dict. de Chimie*, 10.

Among recently introduced preparations for the purpose in view, the efficacy of which has not been fully tested, are the following:

Vendt and Herard,[1] 1885. A solution of eight parts ammonium chloride, ten parts ammonium sulphate, two and one-quarter parts sodium thiosulphate, four and one-half parts borax, and seventy-two parts water.

Winckelman :[2]—	Manganese chloride	33	parts.
	Glacial phosphoric acid	20	"
	Borax	10	"
	Magnesium chloride	12	"
	Magnesium sulphate	25	"

Fabrics are boiled for six hours in this solution and become thoroughly impregnated with insoluble double salts.

Martin and Tessier :[2]—	Boric acid	6	parts.
	Borax	3	"
	Ammonium chloride	15	"
	Water	100	"

Used chiefly for cordage, sail-cloth, canvas, and straw, the materials being steeped in the solution.

Vogt :[2]—	Ammonium chloride	2	parts.
	Zinc sulphate	1	"
	Water	20	"

Starch as needed.

Subrath :[2]—	Alum	5	"
	Ammonium phosphate	5	"
	Water	90	"

Hattin :[2]—Calcium dihydrogen phosphate, ammonia, and gelatinous silica.

Pereles :[2]—Combined solutions of sodium phosphate, silicate, and tungstate.

Nicoll :[2]—Solution of alum, borax, sodium tungstate, and dextrin, or equal weights of calcium acetate and chloride in hot water.

These comprise the processes for the protection of fabrics introduced up to the present time. As is easily seen, the use of sodium tungstate, the borates, or ammonium salts alone or in mixtures is the striking feature.

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[1] *Génie civ.*, 6, 227.

[2] Sc. Amer. Cyclop. of Receipts, p. 217.

A few words with regard to the methods of manufacturing or protecting paper are here in place.

Martin and Tessier[1] used the following bath for paper whether printed or unprinted.

Ammonium sulphate	8	parts.
Boric acid	3	"
Borax	1.7	"
Water	100	"

The solution is placed in a vat, at the end of the paper-making machine and kept at 50° C.

Paper thus treated is non-inflammable. The value in many kinds of business of a totally incombustible paper is easily appreciated; and several varieties, all based on a large use of asbestos, are in vogue. L. Frobeen's paper is made from ninety-five per cent. of asbestos and five per cent. of wood-pulp mixed in water containing borax and glue. For ink he uses a mixture of platinum chloride and india ink.

Halfpennig's paper[1] is made from one part vegetable fiber, two parts asbestos, one-tenth part borax, and one-fifth part alum, formed in the ordinary way into a pulp to which waterglass is sometimes added. A paper of great flexibility and strength is obtained by coating sheets of linen on both sides with the incombustible paper. His ink is a mixture of graphite, copal, copperas, and indigo sulphate.

Paper is also made from pure asbestos and from asbestos mixed with alum, aluminum sulphate, zinc chloride, and resin soap.

A fire-proof writing-ink is an ammoniacal solution of silver nitrate, with a little india-ink, while platinum chloride mixed with lamp-black and varnish is employed as a fire-proof printing-ink.[1]

METHODS FOR RENDERING WOOD INFLAMMABLE.

In taking up next, the history of the efforts to render wood non-inflammable we encounter two distinct methods of procedure. The first is impregnation by the solutions of the chemical compounds which are to be operative, and includes such variations as the precipitation of insoluble salts within the wood by double

[1] Sc. Amer. Cyclop. of Receipts, p. 217.

decomposition ; the second is the covering of the exterior of wood by protective coatings. In the case of existing structures, the latter is evidently the only means available.

The first recorded effort to protect wood was made at Munich in 1823, during the rebuilding of the Royal opera house after destruction by fire.[1] On the recommendation of Professor Fuchs all of the woodwork then received several coatings of waterglass. The surface covered was 400,000 square feet, and the cost was $1,000, or at the rate of $1 for 400 feet. Professor Fuchs prepared his solution by treating ten parts of caustic alkali, fifteen parts of infusorial earth, and one part of charcoal, with water. A somewhat similar composition was also in vogue then in England. It was made by grinding in oil one part of fine sand, two parts of wood-ashes, and three parts of slaked lime, and was applied with a brush. Fuchs' protective kept well, and was regarded as effective for twenty years, but later tests showed that its chemical composition was materially changed, and it no longer afforded security. It may be mentioned here that it was not then found available for scenery on account of the gloss it imparted. During this period the following mixtures came into vogue, chiefly for external protection, as in the case of shingled roofs: three parts of wood-ashes and one part of boiled linseed-oil; three parts of alum and one part of copperas; ashes and lime with skimmed milk as a binding material.

In 1841 Payne[2] introduced his combined process for rendering wood not only uninflammable, but also proof against wet and dry rot and insects. It consisted in the precipitation throughout the mass of a piece of timber, of barium or calcium sulphate by double decomposition. In carrying out the process, wood is introduced into a capacious cylinder, the air is drawn out by steam, and a solution of barium or calcium sulphide is injected into the partial vacuum; the cylinder is exhausted again and then completely filled with the solution of the sulphide; pressure is increased to 140 pounds per square inch, and after an hour the solution is drawn off. The operation is then repeated, use being made of a solution of copperas; as a result the pores of the wood are largely

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[1] Rymer-Jones, *Ecl. Eng. Mag.*, **33**, 55, *1885*; Hexamer, *Jour. of the Frank. Inst.*, **114**, 125.

[2] Rymer-Jones, *Ecl. Eng. Mag.*, **33**, 55, *1885*.

filled with the insoluble sulphate and it becomes hard as stone. The soft, porous, cheaper grades of wood are thus rendered equal in point of durability and strength to the hardest varieties of timber. Wood prepared in this way is largely used in England in connection with public works and railways.

[TO BE CONTINUED.]

[CONTRIBUTIONS FROM THE CHEMICAL LABORATORY U. S. DEPARTMENT OF AGRICULTURE, SENT BY H. W. WILEY, NO. 12.]

NOTE ON THE ESTIMATION OF IRON AND ALUMINA IN PHOSPHATES.[1]

BY K. P. MCELROY.

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IN 1888 Dr. Crampton, then First Assistant Chemist of the United States Department of Agriculture, was engaged in the analysis of a lot of baking-powders and did me the honor to entrust to me a portion of the analytical work. Among other things I had to determine the alumina in a number of alum phosphate powders and met with the usual difficulties. By the method I was instructed to use, I found it impossible to get results that were concordant. I therefore systematically went through all the known methods for separating alumina from phosphoric acid and finally decided to use the one accredited to Mr. T. R. Ogilvie in Crookes' Select Methods (page 538, edition of 1886). Briefly stated the method consists in neutralizing the filtrate from the phosphomolybdate precipitate with ammonia, redissolving the precipitate of mixed sesquioxides thus formed, in nitric acid, once more precipitating, filtering, burning, and weighing. The process gave very good duplicate results, and a trial with a known amount of alumina in the form of alum mixed with sodium phosphate gave results sufficiently near theory. This, together with the fact of its having appeared in a work so standard as that of Crookes, seemed sufficient credentials for the method, and I placidly went ahead with its use. When Dr. Crampton prepared his manuscript (which I did not see prior to publication) for his bulletin on baking-powders (Bulletin 13, Part Five, Division of Chemistry, U. S. Department of Agriculture) he gave me credit for the minor modifications I had made

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[1] Read at the Boston Meeting, December 27, 1894.