

points marked *D* being the attaching points to the flue-sheet, eight of these are used and the trouble is practically overcome. It will be seen that with the ordinary fire box, as shown in fig. 2, the distance between the top stay-bolt in the front flue-sheet and the bottom tube is considerable, and that as the tube is held by the friction of the expanded portion and the bead, the strain at that point must be far greater than that which should, in good practice, be put upon the tube. By distributing these stays along the bottom of the sheet, as we have shown them, the strain on the tube-sheet is brought down to the normal point. The additional expense of doing this work is simply that involved in the making of the braces and putting them in position, for no more iron would be required for the deep flange, as shown on the improvement, than on the old, except that there would be a little less scrap left from the sheet.

Fig. 5 gives a partial outside view of the throat-sheet, showing the cracks about the upper stay-bolts *F*, which are caused by the strain on the sheets when the braces at *D* are not used. Every master mechanic knows the trouble which he has with leaky throat-sheets, and the cracks which are so liable to form around the upper stay-bolts, so that the braces which are used here serve the double purpose, not only of preventing the tube-sheet from becoming distorted and cracked, but also protects the throat-sheet from the annoying cracks that are likely to appear around the upper stay-bolts.

CONTRIBUTIONS TO PRACTICAL RAILROAD INFORMATION.

Chemistry Applied to Railroads.

• SECOND SERIES.—CHEMICAL METHODS.

VI.—METHOD OF DETERMINING PHOSPHORUS IN PHOSPHOR BRONZE.

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(Continued from page 578, Volume LXVII.)

OPERATION.

Put 1 gram of fine borings in a beaker about 2½ in. in diameter by 8½ in. high, and add 25 c.c. of aqua regia. Cover the beaker and allow to dissolve; then heat to near boiling point of the solution for half an hour. Add 25 c.c. of distilled water and then 20 c.c. of concentrated C. P. ammonia, specific gravity 0.90. Then add 50 c.c. of ammonium sulphide. The principal portion of the copper and lead is precipitated as sulphides, and the tin and phosphoric acid are in solution, which is a clear yellow. Digest at a temperature near the boiling point of the solution for 20 minutes. Allow to settle, then filter through a 9-cm. filter into an Erlenmeyer flask, holding about 800 c.c. The filtration and washing is best managed as follows: Pour the clear liquid on the filter, and allow most of it to run through. Then pour the remaining liquid along with the precipitate on the filter, and allow everything that will to run through. Put the filter with the precipitate on it back into the beaker, and add 50 c.c. of the ammonium sulphide wash water. Warm and stir occasionally for 10 minutes to secure as complete solution and diffusion of the soluble material as possible; then pour everything on another filter and allow all that will to run through. Then wash on the filter with about 50 c.c. more of the ammonium sulphide wash water. This gives a volume of filtrate of about 200 c.c. Add now to this filtrate 10 c.c. of magnesia mixture, and shake to secure uniform diffusion of the precipitant. Put the flask in ice water and allow to stand in same with occasional agitation for two hours. Filter through a 7-cm. filter, and wash with ammonia wash water only until the washings react slightly with silver nitrate. Then add 10 c.c. of dilute hydrochloric acid to the flask, and so manipulate that the liquid touches all parts of the inside of the flask and dissolves any adhering precipitate. Then with the liquid in the flask dissolve the precipitate on the filter, allowing the solution to run into a small beaker. Wash the flask and filter with the same dilute hydrochloric acid until the volume of the filtrate is about 80 c.c. Add now 5 c.c. of magnesia mixture and 10 c.c. of concentrated C. P. ammonia 0.90 specific gravity; agitate by stirring, put in ice water, and allow to stand with occasional stirring for two hours. Filter on a 5 cm. filter, wash with same ammonia wash water, until the washings react

only slightly opalescent with silver nitrate. Smoke off the filter, ignite until the precipitate is white, and weigh.

APPARATUS AND REAGENTS.

The apparatus required by this method needs no special comment.

The aqua regia is made of equal parts nitric and hydrochloric acid by volume, both concentrated C. P.

The C. P. ammonia is obtained in the market, specific gravity 0.90.

The ammonium sulphide solution is made by treating C. P. ammonia, specific gravity 0.90, with H_2S until no further absorption takes place, and then adding two-thirds as much by volume of the same ammonia to the solution. It is only slightly yellow in color, and may usually be obtained in the market.

The ammonium sulphide wash water is made by adding three parts of distilled water to one part of the above solution, both by volume.

The magnesia mixture is made by dissolving 66 grams of crystallized C. P. magnesium chloride and 168 grams of C. P. ammonium chloride in 780 c.c. of distilled water, and adding 420 c.c. of C. P. ammonia, specific gravity 0.96. Allow to stand two days and filter.

The ammonia wash water is made by adding to 800 c.c. of distilled water 200 c.c. of C. P. ammonia, specific gravity 0.90, and 25 grams of crystallized C. P. ammonium nitrate. Filter before using.

The dilute hydrochloric acid is made by adding 1 part concentrated C. P. acid, specific gravity 1.20, to 4 parts distilled water, both by volume.

CALCULATIONS.

The atomic weights used are magnesium, 24; phosphorus, 31; oxygen, 16. The molecular formula of magnesium pyrophosphate used is $Mg_2P_2O_7$.

Since 27.93 per cent. of the magnesium pyrophosphate is phosphorus, the amount of phosphorus in the precipitate may be obtained by the proportion $a : b :: 0.2793 : x$, in which a represents the amount of phosphor-bronze taken to start with, expressed in grams; b , the magnesium pyrophosphate obtained, also expressed in grams; and x the phosphorus sought, which will likewise be in grams. Then, since the above proportion gives the actual amount of phosphorus in 1 gram or part of phosphor-bronze, it is obvious that the per cent. of phosphorus—that is, the amount in 100 grams or parts, will be 100 times this amount. Where 1 gram is taken to start with, the following brief rule may be used: Express the weight of magnesium pyrophosphate found in grams, move the decimal point two places to the right, and multiply by the decimal 0.2793. Thus if the magnesium pyrophosphate found is 0.0304 gram, the per cent. of phosphorus is $(3.04 \times 0.2793) 0.849$ per cent.

NOTES AND PRECAUTIONS.

It will be observed that this method oxidizes the phosphorus by means of nitric acid, with hydrochloric acid present to hold up the tin; separates the phosphoric acid from the lead and copper by means of ammonium sulphide, and precipitates the phosphoric acid as ammonium magnesium phosphate in presence of tin and ammonium sulphides, the precipitate being purified by a second precipitation before weighing.

The aqua regia used contains more nitric acid than is customary for this reagent. Nitric acid alone would possibly be as good or perhaps better to oxidize the phosphorus, but experience shows that the meta-stannic acid formed when nitric acid alone is used dissolves in the ammonium sulphide with some difficulty. Incomplete solution would of course result in loss of phosphorus. Accordingly some hydrochloric acid is used to bring the tin into solution. With the method as recommended, there is no difficulty with the tin.

Heating the aqua regia solution for half an hour after the metal is in solution secures complete oxidation of the phosphorus, and the addition of the water dilutes the acid sufficiently so that the strong ammonia can be added without too violent reaction.

If the ammonium sulphide used is made as recommended, very little if any of the copper sulphide is dissolved. Strong yellow sulphide of ammonium gives more difficulty from this cause; and if the yellow sulphide is used, the first ammonium magnesium phosphate precipitate may be contaminated with copper sulphide, which has separated during the two hours in the ice water. This copper sulphide may not cause subsequent difficulty, but it is better not to have it present.

The 20 minutes' digestion after the ammonium sulphide is added, and the filtration and washing recommended, success-

fully remove the phosphorus from the lead and copper sulphides. An examination of these sulphides by decomposing them with nitric acid, separation of the lead as sulphate, then separation of the copper as sulphide in acid solution by means of H_2S after partial neutralization of the free acid with ammonia, concentration of the filtrate to small bulk, and testing with molybdate of ammonia solution shows only a trace of yellow precipitate.

It is desirable to have the bulk of solution in which to precipitate the phosphoric acid by magnesia mixture as small as may be, on account of the possible solubility of this precipitate. At the same time the lead and copper sulphides are so gelatinous that complete washing on the filter is difficult. The procedure recommended apparently secures the result desired with the least amount of wash water. The following experiment has been made on this point. When the ammonium sulphide solution is ready to filter, the total bulk due to reagents added should be 120 c.c.; but on account of evaporation and decompositions the actual bulk was 109 c.c. Of this 101 c.c. ran through the filter after the clear liquid and precipitate had been put on the filter as directed. After putting the precipitate and filter back into the beaker, adding 50 c.c. of ammonium sulphide wash water, and digesting covered for 10 minutes, the bulk of solution, including the filter, was 59 c.c. Of this 48 c.c. ran through the second filter. Neglecting the volume of the precipitate, and assuming that the phosphoric acid is uniformly disseminated in the liquid, it is evident that eight-one hundred and ninth of the phosphorus is left behind after the first filtration and eleven fifty-ninths of this after the second filtration. Reducing these fractions, it appears that after all that will have run through the second filter, only 1.37 per cent. of the phosphorus is left behind. As phosphor-bronze usually contains less than 1.00 per cent. of phosphorus, it is obvious that only about one hundredth of a per cent. of phosphorus remains to be washed out. The 50 c.c. of wash water recommended is apparently abundant for this purpose. In the above experiment the measurements, when the precipitate was present, were made in the beaker, and must be regarded as close approximations only.

The use of a flask for the first precipitation offers some advantages over a beaker, the principal one being that the ammonium sulphide is less exposed to the air, and consequently undergoes less change, with resulting less probability of throwing down free sulphur or traces of sulphide of copper than if a beaker is used. The flask should be covered with a small watch glass, but it is not necessary to use a cork or a glass rod for stirring.

Over-washing of the ammonium magnesium phosphate is to be avoided as carefully as under-washing. The directions given should be closely followed.

The precipitation of the phosphoric acid by excess of magnesia mixture, in presence of the tin and ammonium sulphides, seems to be fully as satisfactory as in the presence of chloride of ammonium alone. An examination of the filtrate and washings from the first precipitation by evaporation nearly to dryness, taking up with nitric acid with just enough hydrochloric to hold up the tin, and testing with molybdate solution, shows only a trace of yellow precipitate.

It is not advisable to weigh without the second precipitation. Although careful manipulation and the use of almost colorless ammonium sulphide may avoid contamination from copper sulphide, there is always danger of free sulphur in the ammonium magnesium phosphate. For good work the second precipitation should never be omitted.

Ammonium magnesium phosphate is liable to be reduced during the ignition of the filter, and thus lead to slightly low results. To obviate this difficulty, the filter and precipitate are put into the crucible wet, and the filter "smoked off" and then burned. The "smoking off" consists in applying the heat to the wet material in the crucible so slowly that the volatile matter of the filter passes off without ignition, free access of air being maintained at the same time. To accomplish this, fold up the wet filter with the precipitate in it, and place it in the crucible. Put the crucible on the triangle as in ordinary ignitions, and leave the cover off. Then heat the open end of the crucible slowly. The filter and precipitate gradually dry, and soon the parts of the filter in contact with the crucible begin to distill off the volatile matter at low heat, even before the whole is dry. This process goes on if the flame is properly adjusted, until in a little while everything that is volatile at a low temperature has passed away, and the precipitate, with a black envelop of carbonaceous matter, is left. When this is the case the temperature can be raised, the lamp moved back to heat the bottom of the crucible, and the carbon burned off completely. Usually when the temperature is raised, the black envelop of carbonaceous matter falls away from the precipitate and is rapidly consumed. By this method

of ignition the material is a little longer time in the crucible than with the old method of previously dried precipitates, but the danger of reducing the precipitate is believed to be very much diminished. The small amount of nitrate of ammonia in the ammonia wash water left in the filter paper facilitates this operation.

When a bronze contains only small amounts of phosphorus, it is advisable to start with 2 to 5 grams. The manipulation and proportions of reagents are, however, the same except that 75 c.c. of ammonium sulphide should be used for the first addition, and 75 c.c. of ammonium sulphide wash water for the second addition, and about 100 c.c. of the same wash water for washing on the filter. This gives a bulk of about 300 c.c. for the first precipitation.

It seems probable that the method described above in careful hands will give results accurate to about one hundredth of a per cent., although it is not rare that duplicate determinations on the same sample differ two hundredths. Where proper care is given to each point, it takes about seven hours to get a result.

MORE ROOM NEEDED.*

SECRETARIES of the Interior and Commissioners of Patents again and again have given voice to the crying need for more breathing space for the men and women who work in the Patent Office; and however much of sameness it may entail, an ever-present menace to the health and safety of these people makes the imperative duty of this report to present this matter again and first of all. On high authority an office occupant needs 4,000 cub. ft. of air space in a room having "ordinary ventilation," which he occupies two successive hours. Two hundred and seventy people in the examining force of this Bureau have but 900 ft. of air space each, in rooms which they occupy for seven consecutive hours, and 110 persons in the assignment and draftsman's divisions have less than 500 ft. of air space each, in rooms which they occupy for the same length of time, and the ventilation is not "ordinary;" it does not rise to that dignity.

Originally the corridors in the Patent Office building ran to the exterior walls, where there are windows admitting light and air; but supposed necessity has since located a room at each extremity, converting the corridors into dead-air spaces, needing artificial light at noonday. The corridor-walls are lined on both sides with unsightly wooden closets and file-cases filled with record-papers. A great number of the force work in basement and sub-basement rooms, intended simply for storage purposes in the original planning of the building.

There are stored more than 1,000 tons of copies of patents on five different floors, tucked into every nook and corner where an eager eye can discover a few feet of available space, so disconnected in order and arrangement that it not infrequently happens that, to select two copies standing next each other in number, one must travel from the sub-basement to the galleries, four stories above. These copies are stored upon these galleries beyond the limit of safety, the worst overloading being directly over the Commissioner's room, and in that near vicinity the cracking of the roof-supports gives daily evidence of the danger which constantly threatens all below.

The situation is serious. It is one which in reason demands immediate relief. The present Secretary of the Interior, commenting in appreciative and generous phrase upon this matter in his letter to the President of the Senate, dated March 18, 1892, says:

It is imperatively necessary that the Department of the Interior should be granted a public building in which to do its works and preserve its archives commensurate with the important service demanded and the great national services devolved upon it. As it is, burdens of material are not only heaped upon the buildings the Department occupies beyond their strength, but burdens of labor are imposed upon the officials, without regard to human endurance.

It would seem that no reasonable question can be made but that the permanent solution of the difficulty is thus correctly stated; but the Patent Office ought to have relief meanwhile. The immediate relief which is possible, and which Congress has apparently approved in the past, is the present and entire removal of the General Land Office from the structure commonly known as "the Patent Office building."

By act approved March 3, 1887, it was enacted:

That as soon as practicable after the completion as provided for in the sundry civil act approved August fourth, eighteen hundred and eighty-six, and not later than December first, eighteen hundred and eighty-eight, the Secretary of the Interior shall cause to be removed to the Pension Building the General Land Office, Bureau of Education, Office of Commissioner of

* Report of the Commissioner of Patents for 1892.