My invention relates to a new and improved chrome assistant in a mordanting bath for bottom chrome dyeing where bichromate of soda is to be used. I have discovered that by the use of a phosphate, preferably in the form of acid sodium pyro-phosphate, it is possible to obtain an action on bichromate of soda when used for bottom chroming of wool. The pyro-phosphate acts as a chrome assistant, as is sometimes referred to by operators of bottom chroming operations, and as this action takes place in the mordant bath, the bichromate is reduced to lower oxides of chromium without allowing it to be deposited on the wool fiber in the form of higher oxides, chromium chromate, or chromium hydroxide.

My object is to produce brighter colors from correspondingly proportional quantities of dye when pyro-phosphates are used as assistants, than is possible when lactic or tartaric acid salts are used for such purpose. A further object is to reduce the cost of the dyeing operation by the substitution of acid sodium pyro-phosphate for lactic and tartaric acid salts.

Heretofore it has been the practice to use organic assistants for bottom chrome dyeing, where the bichromate is reduced to lower oxides before deposition on the fiber. The salts of lactic and tartaric acid are commonly used, as well as numerous mixtures containing salts of these acids, as active ingredients.

Since there are many combinations of dye-stuffs which may be used with various assistants and varying results may be obtained, those that I will use are illustrative, and are not intended to limit the use of a phosphate, preferably sodium acid pyro-phosphate, except as specified in my claims.

Many dye-stuffs are very sensitive to the condition of the chrome on the fiber of the wool. The basis for the particular utilization of my process is that the bichromate of soda is more perfectly reduced to the lower oxide, or that the sensitive dye-stuffs give brighter and more even colors on wool which in the mordant bath were chromed with bichromate and pyro-phosphate. Further, I have observed that with certain dye-stuffs small quantities of pyro-phosphate produce colors which are much brighter than obtained from larger quantities of the same dye-stuffs when other assistants are used. This belief is supported by comparison of wool which had been chromed using pure sodium acid pyro-phosphate with wool chromed by using half refined tartaric acid.

I believe the pyro-phosphate is converted to pyrophosphoric acid, and it is the action of the pyro-phosphoric acid which reduces the bichromate. I have found that bisulphate of soda when mixed with pyro-phosphate will accelerate the action of the bichromate. The bisulphate of soda should be used preferably in quantities corresponding to the chemical equation.

Since some dye-stuffs are best suited to the condition of the chromium oxide resulting from certain mordanting baths, they will produce more satisfactory colors than will other dyes using the same bath. For instance, anthracene blue—Badische W. G. G. is typical of a dye which will produce an improvement of shade when used in connection with pyro-phosphate mordanting baths. Moreover, the increased brightness in color is obtained from it with less pyro-phosphate than if tartaric acid salts were used, and this leads me to believe that there may be some catalytic action of the pyro-phosphate in the chrome mordanting bath, or that some constituent in the wool is removed, which results in a material brightening of the color.

In order that my process may be illustrated, I will now give as an example the results of tests made by myself. A sample of white worsted yarn was treated with weak ammonia water to remove oils, after which treatment, the yarn was washed. Seven different mordanting baths were prepared and samples of yarn were treated in each bath, which contained 3% of sodium bichromate, based on the weight of the wool treated, and in addition each bath contained percentages of the different assistants, on the same basis as the bichromate, as follows:

- No. 1 bath contained as an assistant 1% sulphuric acid.
- No. 2 bath contained as an assistant 1¼% pure argols with 1¼% bisulphate of soda.
- No. 3 bath contained as an assistant 1¼%
pyro-phosphate with 1 1/4% bi-sulphate of soda,

No. 4 bath contained as an assistant 3% pure cream of tartar,

No. 5 bath contained as an assistant 3% pyro-phosphate of soda,

No. 6 bath contained as an assistant 3% "Lactozine"

No. 7 bath contained as an assistant 3% pure commercial lactic acid (26%).

After the mordanting bath, the skeins were washed very thoroughly before dyeing, and each skein was then dyed in a separate beaker of which there were five, each containing the dye-stuff in the percentage based on the wool dye, as follows:

Beaker No. 1 alizarine brown B—powder—calcio—1%,
Beaker No. 2 anthracene blue W. G. G.—paste—extra—Badische—4%,
Beaker No. 3 alizarine red—20% paste—2%.

Beaker No. 4 American dyewood logwood—fully oxidized—1%,
Beaker No. 5 American dyewood logwood—1/2 oxidized—1%.

One skein for each mordanting bath used was dyed with each of the first three dye-stuffs; while the skeins from the first five mordanting baths were dyed with the fourth dye-stuffs; and skeins from the mordanting baths No. 1 and No. 5 were dyed with the fifth dye-stuff only. In all cases the dye baths were brought to a boil in one half hour and continued to boil for fully one hour thereafter. In the case of No. 1 dyestuff, it showed very little difference, as all of the baths were practically exhausted after dyeing was completed. The skeins of wool treated in the pyro-phosphate mordanting baths seemed to exhaust the dye bath more quickly than those skeins treated in the other mordanting baths. With the No. 2 dyestuff, the dye bath was not exhausted after dyeing; however, this is often true with this particular dye-stuff and accordingly no color comparison was made on the bath. During the dyeing process it could be plainly seen that the skein from mordanting bath No. 5 produced a brighter color which continued so during the entire dyeing process. It was estimated that this sample of dyed yarn was 40% brighter than any other except No. 3, which was almost as bright. In order to produce such a bright color by the use of other assistants in the mordant bath, it would be necessary to use a great deal more dye-stuff.

Both pyro-phosphate treated skeins were brighter than any others and had a clearer tone in the case of No. 3 dye-stuff. The dye-stuff baths were all very well exhausted in the case of No. 3 dye-stuff. With the No. 4 dye-stuff the skeins from the pyro-phosphate mordanting baths were brighter than any of the skeins from the other mordanting baths and it seemed as if the dye-stuffs bath was more completely exhausted in the case of such skeins. In making the dye testing with No. 5 dye-stuff, it is expected that a better color would be produced from the No. 1 mordanting bath than from the mordanting baths where pyro-phosphate was used because the dye is an unoxidized logwood, and therefore requires an oxidizing assistant such as sulphuric acid. The result of this test was as expected and in the case of No. 5 the better colors were produced with the skein from No. 1 mordanting bath.

I claim therefore, broadly, the newly discovered property of a phosphate, and particularly pyro-phosphate, which makes it desirable as an improved assisting agent for sodium bi-chromate in bottom chroming of wool.

Having thus described my invention, what I claim as new and desire to secure by Letters Patent, is:

1. A mordanting bath for the bottom chroming of wool, comprising a bi-chromate of soda solution and a pyro-phosphate.
2. A mordanting bath for the bottom chroming of wool, comprising a bi-chromate solution and a water soluble pyro-phosphate.
3. A mordanting bath for the bottom chroming of wool, comprising a bi-chromate solution and sodium acid pyro-phosphate.
4. A mordanting bath for the bottom chroming of wool, having sodium acid pyrophosphate and sodium bisulphate added thereto.
5. The improved process for the bottom chroming of wool, which consists in adding sodium acid pyro-phosphate to the mordanting bath, then subjecting the wool to the depositing action of the reduced chromium oxide, then washing the wool, then subjecting the wool to the action of the dye-stuff in the dye bath.
6. The improved process for the bottom chroming of wool, which consists in adding pyro-phosphate to the mordanting bath, then subjecting the wool to the depositing action of the reduced chromium oxide, then washing the wool, then subjecting the wool to the action of the dye-stuff of the dye bath.
7. The improved process for the bottom chroming of wool, which consists in adding as an assistant in a mordanting bath sodium acid pyro-phosphate and a salt capable of converting said pyro-phosphate to pyrophosphoric acid in the mordanting bath.
8. The improvement in the bottom chroming of wool, which consists in adding pyrophosphoric acid as an assistant to the mordanting bath, then subjecting the wool to the depositing action of the reduced chromium oxide, then washing the wool, then subjecting the wool to the action of the dye-stuff of the dye bath.
9. The method of assisting the action of the bichromate of a mordanting bath for bottom chroming which includes adding a pyro-phosphate to the chromate solution.

10. The method of assisting the action of the bi-chromate of a mordanting bath for bottom chroming by the use of a water soluble pyro-phosphate.

11. The method of assisting the action of the bi-chromate of a mordanting bath for bottom chroming which includes adding sodium acid pyro-phosphate to the chromate solution.

In testimony whereof I affix my signature.

NEIL NEVILLE.