

ANALYSIS OF THE PIGMENT IN A PAINT WHEN IT CONTAINS A COMBUSTIBLE SUBSTANCE.

BY J. E. THOMSEN.

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THE separation of a pigment from the vehicle is often very difficult, especially in the analysis of a paint skin or a thoroughly oxidized and dried paint. George H. Hurst¹ recommends the use of strong nitric acid for this purpose, but this cannot be used in the case under consideration because some of the combustible substance will be oxidized and lost. The oil, too, is converted into a greasy substance which can be only partly removed and that with great difficulty.

Professor Stillman² recommends the use of petroleum ether, but this fails to dissolve completely an oxidized oil, even when applied under pressure in an autoclave.

We have tried several other solvents without success. Alcoholic potash gives a soluble soap but the pigment is affected in many cases. A graphitic carbon pigment analyzed in this way gives too low results and amorphous carbon cannot be separated from the soap by filtration. Decomposing the soap with an acid will not answer because the oxidized fatty acid is as insoluble as the oil.

We finally tried mixing the paint with alcohol and passing dry hydrochloric acid into the mixture, and found this successful.

About ten or twelve times as much of pure ethyl or methyl alcohol should be used as there is of oil in the paint. The process is best carried out in an Erlenmeyer flask and gentle heating aids the solution, which usually requires about half an hour. After the oil is dissolved it can be filtered from the insoluble pigment and the latter washed on a weighed filter with an alcoholic solution of hydrochloric acid. It is then a simple matter to determine the percentage of carbon or of other combustible matter present.

Iron and some other substances are dissolved and can be shaken out with water after the alcoholic filtrate has been mixed with petroleum ether.

If it is desirable to examine the pigment for graphite or amor-

¹ "Painters' Colors, Oils and Varnish," 2nd Ed., p. 48.

² "Engineering Chemistry," 3rd Ed., p. 526.

phous carbon, a weighed portion of the residue is powdered for the purpose. With amorphous carbon this is easy but graphite is best powdered by mixing it with sand or some sharp substance. This sand can afterwards be dissolved by hydrofluoric acid.

About 0.5 gram of the finely powdered carbon is mixed in a dry 100 cc. beaker with 10–15 cc. of strong nitric acid (sp. gr. 1.50), the mixture warmed to 60–70° and 3–4 grams of dry potassium chlorate added and the operation repeated till a yellow graphitic oxide is obtained.

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ANALYSES OF SPANISH PAPRIKA.

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THE question of grading these peppers has been giving some trouble to importers, as there have been no standards for various grades, and some peppers entered as low grade, and running up to and over 10 per cent. ash, have been considered as adulterated, when the fact is that they were simply the very lowest grade of goods, being the sweepings from the floors of the mills and containing stems, seeds and dirt which had dried and fallen from the peppers when brought to the factory. This grade of pepper is used as horse and chicken feed and incidentally to bring down a high paprika to medium grade.

The samples were analyzed by methods of the A. O. A. C. They were furnished by a large importer who vouches for them as being true samples of the various grades specified and imported into this country.

The last three analyses are of samples of hot and sweet paprika grown by the U. S. Department of Agriculture and were received by me from the same importer in their original condition, unground.

Ash.—It will be noticed that the water-soluble portion of the high-grade peppers is very high, while the part insoluble in acid is very low. As the grade deteriorates, the water-soluble portion is lowered, the acid-soluble and insoluble being correspondingly raised, thus showing the presence of extraneous dirt.

Ether Extract.—In regard to the ether extract, the peppers composed of pure shell are very uniform as regards volatile and