

which is not appreciably raised by the addition of small amounts of mineral matter. The end product is graphite in some cases and not in others. Pure petroleum coke, heated without addition of mineral matter, is converted into graphite of excellent quality, while lampblack, although it increases in density, does not reach the value corresponding to graphite, nor acquire any of its other physical properties, even when heated with various oxides.

The impure carbons show a similar behavior. The properties of these carbons after firing are characteristic for each variety of carbon and independent of the amount of ash present.

Anthracite coal is only imperfectly graphitized by heating. The specific gravity of the fired material was approximately the same for three samples having a range of ash content from 6.46 to 17.68 per cent. Moreover, coal from which most of the ash has been previously removed graphitizes better than the crude material.

Bituminous coal coke, which graphitizes fairly well, yields an even better product if a part of its ash has been removed before firing.

It must, therefore, be concluded that a small amount of mineral matter exercises no beneficial effect in the manufacture of graphite by the heating of carbon and that the quality of the product cannot be improved in this way. As to the effect of mineral matter on the rate of conversion, no data are yet available, although some experimental work along this line has been started. At 3000° the maximum density is reached in less than 15 minutes, for any variety of carbon.

We need some theory of the nature of graphite and of amorphous carbon which will permit a rational explanation of the changes which occur on heating. The following discussion is a tentative step in this direction.

THE NATURE OF GRAPHITE.

Graphite in the most restricted sense of the term is an allotropic form of carbon having a definite and perhaps not very complex molecular configuration. The molecule might, for example, be regarded as two benzene rings side by side, and joined at all six angles, the extra bonds being satisfied as in the usual centric formula. Such a formula might be used to explain the formation of mellitic acid and graphitic acid by oxidation of graphite.

AMORPHOUS CARBON.

When an organic compound is decomposed, there results a mixture of substances constantly increasing in complexity until finally carbon is obtained. This carbon need not be regarded as a simple substance, but may be considered to be a mixture of many varieties of carbon each with a different number and arrangement of atoms in the molecule. According to this view it seems better not to regard "amorphous carbon" as the name of a distinct allotropic form, but rather as a general term covering all varieties of carbon except graphite and diamond. In speaking of amorphous carbon, therefore, the source of each sample should be stated.

THE MODE OF TRANSFORMATION.

In a given sample of amorphous carbon, some of the molecules will be capable of easily undergoing rearrangement under the influence of heat to form graphite molecules, while others will not, and the proportion of molecules capable of such change will determine the character of the final product.

Petroleum coke would therefore consist almost wholly of graphitizable molecules, while anthracite coal contains a smaller proportion.

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A METHOD OF ANALYZING SOME COMMERCIAL GOLD ALLOYS. METALS PRESENT: GOLD, SILVER, COPPER AND OCCASIONALLY ZINC AND TIN.¹

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PREPARATION OF SAMPLE.

Use a sharp file. Remove particles of steel with a magnet. Weigh out 0.5 gram of filings in a 4-ounce beaker. Add 50 cc. of *aqua regia* (40 HCl and 10 HNO₃). Heat just to boiling for 15 minutes or until decomposed. The AgCl is almost all dissolved, when boiled with the strong acid. Boil down to 10 cc., add 25 cc. HCl and again boil down to 10 cc. or until the AgCl begins to separate.

SILVER.

Dilute to 150 cc. with water. Boil until the AgCl coagulates well. Cool, let stand until clear. Filter on a weighed paper, and wash the AgCl. Dry and weigh. ($\text{AgCl} \times 0.7527 = \text{Ag}$.)

ZINC.

Add sufficient HCl to the filtrate to make 5 per cent. of concentrated HCl by volume. Pass H₂S through the cold solution rapidly for fifteen minutes. Filter and wash with H₂S water. The filtrate contains only the zinc. Boil off H₂S and precipitate with Na₂CO₃. Boil for fifteen minutes. Filter and wash with hot water. Dry, burn off in porcelain and weigh ZnO after blasting. ($\text{ZnO} \times 0.8034 = \text{Zn}$.)

TIN.

For the separation of Sn from Au and Cu in the sulphide precipitate, we take advantage of its solubility in HCl. The precipitate (and filter paper) is placed in a beaker and covered with 50 cc. of a mixture of water (35 cc.) and HCl (15 cc.). Boil for ten minutes. Cool. Filter. Add HCl to make 25 per cent. of concentrated acid by volume. Pass H₂S to reprecipitate any dissolved Cu. Filter and wash with H₂S water. Neutralize filtrate with ammonium hydroxide. Acidify with 1 cc. of HCl. Pass H₂S to precipitate the Sn. Filter. Wash with H₂S water. Burn off in a porcelain crucible, igniting finally over a blast. Weigh SnO₂. ($\text{SnO}_2 \times 0.788 = \text{Sn}$.) The CuS recovered as above is converted into CuO and is then dissolved in 5 cc. conc. HNO₃, by warming. Any SnO₂ present is recovered and weighed, the amount being added to that already obtained.

¹ Paper read at the Indianapolis Meeting of the American Chemical Society before the Industrial Section, June 30, 1911.

The Cu solution is added to that recovered from the gold, by the next operation.

COPPER.

Burn off the sulphides of gold and copper in a porcelain crucible, avoiding a heat greater than that required to burn off the filter paper. This hinders the shrinkage of the metallic gold, and leaves it more porous so that the CuO is readily dissolved out. Too high a heat causes CuO to unite with the glaze of the crucible. Place the Au and CuO mixture in a 4-ounce beaker. Add 10 cc. of concentrated HNO₃. Boil for 10 minutes. Add 3 cc. of concentrated H₂SO₄ and boil until the HNO₃ is all expelled, and SO₃ fumes are coming off freely. Cool, add 50 cc. of water and 5 grams of sodium acetate; boil and filter off the gold (the gold residues are weighed as an approximate check and are saved for their commercial value only. Gold is best determined by fire assay). Cool the filtrate and add 5 grams of potassium iodide. Stir until dissolved. Titrate with decinormal hyposulphite of sodium: 1 cc. = 0.0063 gram Cu.

GOLD.

Scorify 0.5 gram of filings with 40 grams of test lead, 1 gram of borax glass and 1 gram of powdered silica. The borax and silica flux are placed on the mixture of alloy filings and test lead. If the lead button resulting from scorification is hard, repeat the scorification, adding 10 grams more test lead and 2 grams of borax and silica flux. Cupel carefully and weigh Au plus Ag.

The determinations previously made make it possible to calculate quite closely the amounts of Au and Ag, etc., in the alloy.

We make up a mixture containing the same metals in approximately the same proportions. 0.5 gram of this "control" mixture is then scorified and cupelled side by side with an equal weight of the alloy. After weighing the gold and silver buttons from the assay and the "control," they are alloyed separately with three parts of pure Ag.

The alloys must be thoroughly melted in order to insure homogeneity. Flatten the buttons by hammering or rolling.

Part with HNO₃ of graded strengths as is the usual assaying practice. Boil well with water, dry, ignite and weigh Au. Deduct Au from Au plus Ag. The difference is Ag. Increase both Au and Ag figures by the amounts of loss shown by the Au and Ag used in the "control" assay. The fire-assay for Au and Ag yields in most chemists' hands the highest and the most accurate results. The correction for fire loss (volatilization) is far higher for Ag than for Au. Gold alloys analyzed by us had the following limits of composition.

	Per cent.
Gold.....	48.0 to 99.50
Silver.....	0.5 to 26.00
Copper.....	0.0 to 18.00
Zinc.....	0.0 to 7.50
Tin.....	0.0 to 2.00

Those alloys highest in Ag were most difficult to dissolve in *aqua regia*.

We found the ratio 1 HNO₃ to 4 HCl most efficient. *Aqua regia* 1 to 3 was less efficient and 1 to 2 very unsatisfactory. Increasing the percentage of HNO₃ in the mixture failed to cause more rapid solution, but increase of HCl produced unexpectedly rapid and complete decomposition.

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CUPELLATION.

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In the last few years there have been so many kinds of cupels proposed and placed on the market that it seemed desirable to determine the relative value of some of the best known, regarding silver loss, conditions of surface, etc., after cupellation. These observations suggested the investigation of the properties of substances other than bone ash, for separating lead and silver in the process of cupellation.

In order to obtain uniform and comparative conditions an electrically heated muffle was used for all cupellations in this investigation. The temperature was determined by means of a Le Chatelier platinum iridium pyrometer. Two cupels were run side by side and the pyrometer placed between them about 1/4 inch above the tops of the cupels. The muffle used was just wide enough to take two cupels and allow room for moving them about. The back part of the furnace was always hot enough to "open" the buttons and to start them "driving," after which they were pulled forward near the door of the muffle.

After thorough drying, the cupels were heated in the muffle, at about 800° C. The lead button was dropped into the cupel and the muffle door closed until the lead had melted down and begun to oxidize. The cupels were then pulled to the front of the muffle where the pyrometer registered about 665° C. and continually watched. If any signs were discovered of more litharge being formed than was absorbed, the cupel was pushed back a little. It was found sufficient in many such cases to merely turn the cupel around, so that the colder side would be facing the hotter part of the furnace. When the button had become quite small, seemed to be revolving, and would "blik" in from 2 to 3 minutes, the cupel was pushed back into the furnace where the temperature was from 750° C. to 800° C. This was found necessary on account of a small amount of lead being retained in the silver button if the cupellation were completed at a lower temperature.

The bone ash obtainable for making cupels varies so much in size and purity that some standard was necessary, with which to compare the cupels tested. One was selected which gave the following screen test:

	Per cent.
On 60 mesh.....	0.007
On 80 mesh.....	7.83
On 90 mesh.....	33.22
On 100 mesh.....	8.58
On 120 mesh.....	43.65
Through 120 mesh.....	6.72