

M aqueous solution and the results are shown in the accompanying figure. The *P_H* of the first two solutions was brought to 11.7 and that of the third to 11.8 by the addition of sodium hydroxide, the *P_H* values being ascertained by means of the hydrogen electrode. In this range of alkalinity the blue dye ion predominates and there is no noticeable change in the absorption maximum when higher alkalinities are used. Shifting the wave length 2 *mμ* to one side or the other of the absorption maximum gave reproducible decreases in the absorption index. A Bausch and Lomb instrument was used. The solutions in all cases were freshly prepared from W. M. Clark's dyes and it was found that the absorption maxima did not alter during two days' standing. The absorption maximum of the first dye was at 625 *mμ*, and that of the second and third at 610 *mμ*. It was also found that Lambert and Beer's law held for solutions of twice the concentration.

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The Acid Soluble Oxidizing Material from Charcoal

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In the course of an investigation of the mechanism of combustion of carbon, it seemed advisable to check the observation of Lamb and Elder¹ that a peroxide-like substance is dissolved out of charcoal by dilute sulfuric acid, which will then liberate iodine from potassium iodide. We applied this test to several commercial charcoals, coal, and ash-free lamp-black, and found that an extract capable of liberating iodine from potassium iodide was obtained only from those materials that contained iron in their ash. Measurements on one of the extracts, which liberated about a tenth as much iodine as those of Lamb and Elder, showed that the iodine liberated was equivalent to the ferric ion extracted. These experiments throw some doubt on the existence of the acid soluble peroxide-like material reported to have been extracted from charcoals.

Several commercial charcoals and Edenborn coal² were washed with 1.0 *M* sulfuric acid, at which concentration Lamb and Elder found the greatest activity. The filtrates liberated iodine from 10% potassium iodide solution, and decolorized a few drops of potassium permanganate, but did not give the characteristic test for hydrogen peroxide with chromic acid, nor that for ozone with silver.³ No gas appeared when an inverted test-tube

(1) Lamb and Elder, *THIS JOURNAL*, **53**, 157 (1931); *cf.* also I. M. Kolthoff, *ibid.*, **54**, 4478 (1932).

(2) U. S. Bureau of Mines, Tech. Paper No. 525 (1932).

(3) Only the first of these tests was considered significant. The reduction of potassium permanganate might have been caused by minute amounts of organic material or by ferrous iron. The peroxide and ozone tests may not have been sufficiently delicate.

filled with solution was exposed to diffuse sunlight for several weeks. When the solution was concentrated, either by boiling or by evacuation at room temperature, there was no loss of activity, but a fine black precipitate separated out when the acid became concentrated. When the solution was evaporated to dryness and the precipitate washed with acid, the reaction with potassium iodide could still be obtained. A trace of iron was indicated by potassium thiocyanate.

Since ferric ion is able to oxidize I^- , it was necessary to use iron-free reagents to show conclusively the presence of a peroxide. Charcoal made from recrystallized sugar could not be obtained iron-free, but a lampblack prepared from natural gas was ashless.⁴ Iron-free sulfuric acid was prepared by dissolving pure sulfur trioxide in distilled water. When the experiment was repeated using these pure reagents, no liberation of iodine could be detected.

To identify further the action of ferric ion with that of the active material from charcoal, a solution of ferric chloride was added to a 1.0 *M* sulfuric acid solution to which an excess of thiocyanate had been added until its color matched that of a portion of the acid with which one of the commercial charcoals had been washed. Calculation showed that about 6 mg. of iron had been washed out of each hundred grams of charcoal. Equal amounts of both the iron and the charcoal wash solutions were tested with 10% potassium iodide solution, and titrated with sodium thiosulfate solution (one ml. = 0.383 mg. of iodine), using starch as indicator. The titration showed that the iron solution, which had been made up to contain 0.02 mg. of iron per ml., contained 0.017 mg./ml., while the charcoal wash solution contained 0.016 mg./ml. Since both solutions contained the same concentration of ferric ion, as shown by the thiocyanate color test, and since they both liberate the same amount of iodine, the oxidizing action of the charcoal wash solution must be due entirely to its ferric-ion concentration and not, in part, to the presence of a peroxide.

These results show that evidence previously adduced to show the existence of an acid soluble peroxide of carbon is not conclusive. The oxidation of potassium iodide is shown to be an insufficient test for peroxide in the presence of inorganic ions.

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(4) This lampblack was evacuated to a pressure of 10^{-3} for four hours between 350 and 400°. Howard and Hulett [*J. Phys. Chem.*, **28**, 1082 (1924)] found that such a black had nearly 60% the adsorptive capacity of an activated coconut charcoal.