the methods as indicated. Special acknowledgment should be made to Mr. F. W. Gill for results on arsenic, chlorine and carborundum.

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THE COMPARATIVE OXIDIZING POWER OF SODIUM PEROXIDE AND ITS USE IN QUALITATIVE ANALYSIS.

BY D. F. CALHANE.

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The detection of chromium in qualitative analysis rests on its oxidation to the chromate and the precipitation of chromate of lead in acetic acid solution. The oxidation is usually effected by chlorate of potash on the solution of chromium hydroxide in strong nitric acid. If by any chance the nitric acid chosen is not strong enough or has become diluted, the oxidation will not occur and the test fails. It is necessary for success that the hydroxides possibly containing iron, aluminium and chromium be freed of water as much as it is possible by drying them quickly by heating. In this part of the procedure the student usually fails to work properly, not enough water is removed, and the subsequent oxidation does not occur. As I have found, in teaching classes in this subject, so much trouble in getting the student to properly observe precautions, it seemed of advantage to apply a test which would be effective and not so much dependent on a certain set of conditions.

Some experiments were accordingly made on the action of certain other oxidizing agents. Among these were chosen sodium peroxide and bromine water. The preference was for the former, as it is to be obtained in a convenient solid form, and possesses few of the disagreeable features of bromine water. A 0.1 N solution of chrome alum was prepared, each cubic centimeter holding 0.0332 g. of chrome alum, answering to 0.005 g. of Cr_2O_3. Next, the oxidation of portions of this solution with peroxide of sodium and bromine water was carried out to see what the comparative efficacy of these two reagents is on solutions containing known amounts of chromium. In this connection an interesting feature in the action of sodium peroxide on chromium solutions was met with, that at first led to the belief that bromine water was more efficient. The dilutions were carried to the point where the amount of chromic oxide present was only 0.000125 g., corresponding to 0.000085 g. of chromium. At this extreme dilution, a safe test was secured with bromine water, but the sodium peroxide apparently failed at a concentration answering to 0.00025 g. chromic oxide. The bromine water appeared to act equally well both hot and cold at all the different concentrations from 0.005 g. chromic oxide down to 0.000125 g. With the sodium peroxide there was no test with the presence of 0.005 g.

chromic oxide, if the solutions were treated cold. When the sodium peroxide was added to the hot solution a small amount of yellow precipitate was obtained. An explanation of this surprising fact was arrived at later.

The next procedure was to find out what the limit of delicacy for the test is in actual analysis. Normal solutions of aluminium nitrate and ferric chloride were prepared, one cubic centimeter of which answered to 0.071 g. aluminium nitrate and 0.054 g. ferric chloride, giving a precipitate of 0.026 g. aluminium hydroxide, 0.0356 g. ferric hydroxide, and 0.0033 g. chromic hydroxide from 1 cc. of the chrome alum. The analysis in the first instance was carried through with a mixture of 1 cc. of each of the three solutions. The aluminium and iron here exceeded the chromium in the ratios of 8 and 12 times, respectively. The solution of the mixed salts was treated with 10 cc. of barium carbonate emulsion and allowed to stand for 2 or 3 minutes. Next a filtration was made, and the filtrate tested for iron, chromium and aluminium. The tests proved complete precipitation of the metals. The barium carbonate residue was dissolved in hot hydrochloric acid. The hydroxides of the three metals reprecipitated with ammonia. These were dissolved in hydrochloric acid and the diluted solution divided into two equal parts. There could be present in each part the equivalent of 0.0025 g. chromic oxide, answering to 0.0016 g. chromium hydroxide. One part was added to a large excess of bromine water, after previously having added excess of sodium hydroxide. After filtration from the iron, no test for chromium appeared on adding acetic acid and lead acetate. In the other portion similarly treated, using sodium peroxide as the oxidizing agent, a good test was obtained. Next, the above procedure was repeated, using 2 cc. of the iron and aluminium solutions and 1 cc. of the chromium. The ratios here were 24 iron, 16 aluminium to 1 of chromium hydroxide. Here again the bromine water failed to act sufficiently for a test, while the sodium peroxide gave a good test. In the third experiment 5 cc. each of the iron and aluminium solutions were taken and 0.5 cc. of the chrome alum solution. After proceeding as before, the bromine water failed to give a test, while 0.5 g. sodium peroxide gave a reliable indication. The ratios here were 81 aluminium and 111 iron to 1 of chromium. The mixed precipitate contained 0.130 g. aluminium hydroxide, 0.178 g. ferric hydroxide, and 0.0016 g. chromium hydroxide. In each of the last two tests the maximum amount of chromic oxide present for oxidation was 0.0008 g., corresponding to 0.0005 g. chromium.

In the next experiment it was desired to contrast the delicacy of the usual method with potassium chlorate and nitric acid with the peroxide procedure. Amounts taken were 5 cc. aluminium nitrate, 5 cc. ferric chloride, and 0.5 cc. chrome alum. The analysis was carried through in the usual way and the precipitate of iron, chromium and aluminium hydroxides given by ammonia was dissolved in strong nitric acid and boiled with potassium chlorate. The solution was divided into two equal portions and one tested with sodium hydroxide acetic acid and acetate of lead. The result was negative for chromium. Here, as in the previous cases, 0.0008 g. of chromium hydroxide was present. The result shows that the limit of successful oxidation by this method has been exceeded. The time required to carry this out is longer and the test is much less delicate than with the peroxide method.

Another test was made to compare the fusion method of oxidation with that using bromine water, potassium chlorate and nitric acid and sodium peroxide. The usual method of fusing the hydroxides of iron, chromium and aluminium mixed with dry potassium nitrate and sodium carbonate on platinum was followed, having present the same amount of chromium, iron and aluminium as in the previous cases. The result here for chromium by the usual test as lead chromate after oxidation, was surprisingly convincing. Five cubic centimeters of the ferric chloride aluminium nitrate and 0.5 cc. of the 0.1 *N* chrome alum solution were precipitated by ammonia. The precipitate was filtered, washed and fused in a platinum crucible with potassium nitrate and sodium carbonate mixture for a few minutes. The cooled mass was lixiviated with hot water. The iron was filtered off and one-half of the clear filtrate, which was decidedly yellow, was made acid with acetic acid and lead acetate added. A good yellow color was produced due to the formation of lead chromate. This test appears to be equally delicate with the sodium peroxide method. There was present in this fusion test 0.0008 g. chromic oxide, or 0.0005 g. chromium.

In the experiments having to do with the oxidation of the pure chrome alum solution with bromine water and peroxide, it was surprising that the peroxide apparently failed to oxidize the chromium at a concentration where bromine water gave excellent results. It was found, however, that if the solution was hot during the test a very slight precipitate of lead chromate was obtained with the sodium peroxide procedure. Bromine water acted equally well in hot or cold solution. It has been previously shown that in actual analysis sodium peroxide is by far the more powerful oxidizing agent.

It was found on further experimentation that whereas a solution of bichromate was readily oxidized to perchromate by barium peroxide in acid solution, a chromic salt gave no indication of oxidation to the perchromate by the above agent. The chromic salt was only slightly oxidized to the chromate.

Sodium peroxide acting on a solution of the chromic salt gave a yellow color. On acidification a violet color appeared, which concentrated in ether indicated perchromic acid. In acid solution sodium peroxide gave no such oxidation. If a chromic salt were oxidized by sodium peroxide as above, the solution acidified with acetic acid and lead acetate added, in dilute solutions such as were used in this work, no precipitate appeared. On allowing the solution to stand, or more quickly, on warming, the customary yellow precipitate of lead chromate appeared and oxygen continued to be evolved for some time.

These results show the reason of the failure to obtain the tests for chromate of lead in the earlier part of the work where dilute solutions of chrome alum were severally treated with sodium peroxide and bromine water. The sodium peroxide forms an alkaline solution with the evolution of oxygen. The chromic salt is oxidized to the perchromate, the sodium salt being formed in the alkaline solution. This substance is stable and gives a yellow color to the solution. On acidification with acetic acid, lead acetate produces no precipitate, as the lead perchromate is soluble in this medium. On standing or warming, oxygen is given off and the lead perchromate breaks down to the chromate, giving the customary yellow precipitate.

Oxone, the fused form of sodium peroxide, acts the same as the unfused variety, as would be expected. For analytical work it is less desirable, owing to the impurities it contains. In addition to the silica found in the unfused variety, it carries about 1.5 per cent. of copper as the result of several analyses showed. In addition, small amounts of iron were found to be present. The results obtained in this investigation show that sodium peroxide is the best oxidizing agent for chromium in solution. Sodium perchromate is formed by this agent in alkaline solution. This solution is stable, and, on acidification, oxygen is rapidly and continuously evolved. The oxygen can be liberated as fast as desired, thus giving a powerful oxidizing source simply controlled.

The properties of this alkaline perchromate solution will be further investigated.

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THE DETERMINATION OF CARBON IN STEEL, FERRO-ALLOYS, AND PLUMBAGO BY MEANS OF AN ELECTRIC COMBUSTION FURNACE.

BY C. M. JOHNSON.

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Several months ago it occurred to the writer that the Hoskins resistance wire could be applied to the heating of combustion tubes. A drawing was prepared for a furnace of a muffle type to heat four tubes lying in the same plane and parallel.