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On the Detection of Chromium in Presence of Iron,
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(Concluded from page 266.)

SEVERAL experiments have also been made in order to ascertain whether the presence of peroxide of iron would interfere with the oxidation of the sesquioxide of chromium. Weighed portions of protosulphate of iron were boiled with nitric acid in order to oxidise the iron, and mixed with weighed portions of chrome alum; the solutions were then treated with a slight excess of caustic ammonia and boiled. After washing the precipitates formed, they were subjected to the action of oxidising agents.

I have operated upon mixtures composed of—

Grammes.	No. 1.	Grammes.	Per cent.
3.00 FeOSO ₃ + 7aq	}	FeO ₁₃ 0.8633	or 84.81
+ 1.00 KOSO ₃ ; Cr ₂ O ₃ . 3SO ₃ + 24aq		Cr ₂ O ₃ 0.1556	or 15.19
3.00 FeOSO ₃ + 7aq	}	FeO ₁₃ 0.8633	or 91.78
+ 0.50 KOSO ₃ ; Cr ₂ O ₃ . 3SO ₃ + 24aq		Cr ₂ O ₃ 0.0773	or 8.22
5.00 FeOSO ₃ + 7aq	}	FeO ₁₃ 1.4389	or 97.38
+ 0.25 KOSO ₃ ; Cr ₂ O ₃ . 3SO ₃ + 24aq		Cr ₂ O ₃ 0.0387	or 2.62
10.00 FeOSO ₃ + 7aq	}	FeO ₁₃ 2.8777	or 98.73
+ 0.25 KOSO ₃ ; Cr ₂ O ₃ . 3SO ₃ + 24aq		Cr ₂ O ₃ 0.0387	or 1.27
20.00 FeOSO ₃ + 7aq	}	FeO ₁₃ 5.7554	or 99.33
+ 0.25 KOSO ₃ ; Cr ₂ O ₃ . 3SO ₃ + 24aq		Cr ₂ O ₃ 0.0387	or 0.67

Portions of the precipitate produced by ammonia in each of these solutions were dissolved in chlorhydric acid, and a part of this solution was treated with an excess of cold potash lye of sp. gr. = 1.305; an excess of caustic soda of sp. gr. = 1.07 being added to another portion. After standing in the cold, out of contact with the air, during eighteen hours, the alkaline mixtures were filtered, and the filtrates thoroughly boiled. No precipitate of sesquioxide of chromium was produced in any of them, nor did these filtrates afford any chromic acid when boiled with peroxide of lead.

Other portions of the moist original precipitates were dissolved in chlorhydric acid, and these solutions treated with a slight excess of dilute caustic soda. A small quantity of peroxide of lead was now added to the mixture, and the whole thoroughly boiled during two or three minutes. On filtering, yellow-coloured solutions were obtained in every instance, and on testing these with peroxide of hydrogen the characteristic reaction of chromic acid was very distinct in each.

The oxide of chromium in other portions of all of the original precipitates was also readily oxidised by boiling them with bromine in presence of free alkali, as well as by dissolving them in concentrated nitric acid, and boiling this solution with chlorate of potash. The presence of chromium was, moreover, readily detected in Nos. 1 and 2 by boiling a mixture of precipitate and alkali with peroxide of manganese, or with permanganate of potash; but as these substances are evidently less conveniently applied than the others which I have mentioned, no further experiments were made with them.

It should perhaps be stated that the experiments upon the precipitate from mixture No 5, were made upon portions of it weighing two or three grammes, the entire weight of the moist precipitate being something more than a hundred grammes.

Among the various agents capable of oxidising oxide of chromium when in presence of sesquioxide of iron, it is not at first sight easy to decide which one should be preferred to the others for common use. Bromine in presence of the alkalis appears to be the most powerful of them, but since the acid solution of peroxide of hydrogen, which is used in testing, would react upon any bromide which might have been formed in the alkaline solution, bromine would be liberated, and, by imparting its colour to the ether, would obscure the reaction. A similar objection applies of course to iodine. This difficulty is easily overcome by neutralising the alkali with nitric acid, and boiling for a few minutes to expel the bromine: an objection to this process, however, is the fact that the solution is considerably diluted thereby.

Chlorate of potash, with concentrated nitric acid, is in some respects a very convenient agent: objections to it are, that some of the products of the decomposition of the chlorate which remain in the solution, appear to interfere with the formation of perchromic acid; moreover, if any nitrate of chromium remain unoxidised in the solution, it will impart a bluish purple tint to the aqueous solution of peroxide of hydrogen, which, though insoluble in ether, often interferes very materially with the detection of the colour of perchromic acid, if only traces of the latter are present. This remark applies to any process of oxidation in which nitric acid is employed. Both of these difficulties can generally be avoided by diluting the nitric acid solution with water, and using a quantity of ether somewhat larger than is usually necessary. In any case where hyponitric acid is generated, it dissolves in the ether, and may conceal the blue colour of perchromic acid.

Black oxide of manganese is not only a less energetic oxidising agent than peroxide of lead, but the chromate formed in its presence does not colour the solution so strongly. The latter remark applies also to permanganate of potash, another objection to which is the necessity of destroying its own colour whenever it has been used in excess, before testing for chromic acid.

On the other hand, the reagent of Chancel, peroxide of lead, is capable, as I have shown, of oxidising sesqui-