20 c.c. of nitric acid of 36°. (If the ore contains much lead, the lead sulphate of the residue is extracted with sodium hydroxide of 12° Beaumé.) The insoluble matter is filtered off, washed, and dissolved in potassium cyanide. The solution is diluted to 250 c.c.—it should contain 2 per cent. of cyanide—and electrolyzed for a few hours with a current of 0·05 ampère. The silver is taken up in 50 c.c. of nitric acid and 50 c.c. of water, and determined by Volhard's method. F. H. L.

Volumetric Estimation of Mercuric Chloride in Aqueous Solution. A. Archetti. (*Boll. Chim. Farm.*, 1900, xxxix., 765; through *Chem. Zeit. Rep.*, 1901, 11.)—This process depends on the formation of "white precipitate" when mercuric chloride is treated with ammonia. A few drops of alcoholic phenolphthalein are added to the mercury solution, and decinormal ammonia is run in till the pink colour is permanent. Each c.c. of reagent corresponds to 0·271 gramme of corrosive sublimate. F. H. L.

A New Method of Separating Metals of the Platinum Group. Leidié. (*Journ. Pharm. Chim.*, 1901, xiii., 18-23.)—The industrial methods employed for the extraction of platinum and iridium from the ore leave residues which contain various rare metals. For the separation of these the author recommends the following method, based on the characteristics of their double alkali nitrites:

I. Elimination of Foreign Metals and Conversion of the Platinum Metals into Double Alkali Nitrites.—The residue is ignited in the air at a dull red heat, and reduced in hydrogen. It is next treated with water, then with hydrochloric acid, and again reduced in hydrogen. The metallic residue is mixed with twice its weight of sodium chloride, pulverized, and heated in a current of dry chlorine. The fixed and volatilized products are treated with a large excess of water acidulated with hydrochloric acid, in which process nearly the whole of the silver chloride is dissolved (by means of the sodium chloride present), whilst but little lead chloride is dissolved.

The solution is partially neutralized with sodium carbonate and brought nearly to the boiling-point. Sodium nitrite is now introduced little by little until the liquid becomes neutral to turmeric, at which point sodium carbonate is added until the precipitate no longer increases. The liquid is then boiled, left to cool, and filtered.

The sodium nitrite precipitates the iron as sesquioxide, and the gold in the metallic form. The sodium carbonate precipitates all such metals as lead, silver, zinc, tin, bismuth, copper, etc., originally present in the mineral or introduced in the treatment.

The metals of the platinum group remain in solution as double nitrites, having the following formulæ:

Nitrite of ruthenium and sodium	$Ru_2(NO_2)_6.4NaNO_2$.
,, of platinum ,, ...	$Pt(NO_2)_2.2NaNo_2$.
,, of palladium ,, ...	$Pd(NO_2)_2.2NaNO_2$.
,, of iridium	$Ir_2(NO_2)_6.6NaNO_2$.
,, of rhodium	$Rh_2(NO_2)_6.6NaNO_2$.

The osmium is present in the form of the double chloride, $OsCl_3.3NaCl$.

II. Separation of the Platinum Metals—Osmium, Ruthenium. — The nitrites mentioned above are not precipitated either by alkalies or alkali carbonates. Sodium hydroxide is added to their solution, and a current of chlorine passed through, whilst the volatile products are collected in water containing alcohol. The osmium and ruthenium salts are thus converted into the volatile peroxides OsO_4 and RuO_4. At the end of the operation the distillation flask is slightly heated to expel them completely with the steam. The alcohol in the receiver reduces them to the metallic state, and they are then separated from each other by Deville and Debray's method (*Ann. de Chim. et Phys.* [5], lvi., 476, 480).

Iridium, Rhodium.—The residual liquid is acidified with hydrochloric acid and boiled, and the metals reconverted into nitrites by the addition of sodium nitrite until the reaction is neutral. It is then cooled and saturated with ammonium chloride. This yields a precipitate of the double nitrites of iridium and rhodium with ammonium, which are insoluble in solutions of ammonium chloride. The precipitate is collected after twenty-four hours, and repeatedly treated with hot *aqua regia* to convert the nitrites into the respective chlorides $IrCl_4$ and Rh_2Cl_6. The *aqua regia* is expelled by evaporation, the residue taken up with cold chlorine-water, and the solution saturated with ammonium chloride.

The iridium is precipitated as the double chloride of ammonium and iridium. This is collected, dried, mixed with its own weight of sodium chloride, and heated in a current of chlorine at 440° to 450° C. By this means iridium is left as sodium iridium chloride, which is soluble in water, whilst any rhodium present is left as the anhydrous sesquichloride, which is insoluble in water.

The sodium iridium chloride is again converted into the ammonium salt, and the latter, when reduced by hydrogen, yields metallic iridium.

The filtrate from the iridium precipitate is evaporated to incipient crystallization. The crystals which then separate consist of a mixture of ammonium rhodium chloride ($Rh_2Cl_6.6NH_4Cl$) and ammonium chloride. The rhodium salt is converted into the double nitrite of sodium and rhodium, and this is precipitated as the ammonium salt. (If iridium were present, its corresponding salt, being a little more soluble, would be left in the mother liquid.) The ammonium rhodium nitrite is reconverted by hydrochloric acid into ammonium rhodium chloride, and metallic rhodium is obtained from this by reduction in hydrogen.

Platinum, Palladium.—The solution, after the removal of iridium and palladium, still contains double nitrites of platinum and palladium. It is evaporated to dryness after the addition of hydrochloric acid to convert the nitrites into chlorides. As the amount of sodium chloride present would interfere with the precipitation of the platinum, the residue is reduced in hydrogen. The metallic platinum and palladium, with possibly traces of iridium, are washed with water, dissolved in *aqua regia*, the solution evaporated, and the residue taken up with water. The liquid is placed in a flask, which it nearly fills, the air expelled by means of a current of carbon dioxide, and a current of nitrogen dioxide passed in as a reducing agent, and finally expelled by carbon dioxide.

The solution is now saturated with ammonium chloride and left for twenty-four hours, after which the ammonium platinum chloride is collected, recrystallized once or twice, and reduced in hydrogen to metallic platinum.

To the filtrate is added mercuric cyanide, which precipitates the palladium as palladious cyanide, $Pd(CN)_2$. This is ignited, and the residue dissolved in nitric acid. The nitrate is converted into palladious chloride, and then into the nitrite of potassium and palladium, which is converted into the double chloride of potassium and palladium, and crystallized. This salt is reduced at a red heat in hydrogen, and the residue, when cooled in a current of carbon dioxide and washed with water to remove the potassium chloride, leaves metallic palladium.

In the course of this treatment any iridium is converted into the hydrated sesquichloride, and is not precipitated. C. A. M.

Titration of Free Alkali in Presence of Hypochlorites, Chlorates, and Chromates. H. von Huber. (*Zeits. Electroch.*, 1901, vii., 396; through *Chem. Zeit. Rep.*, 1901, 32.)—The hypochlorite is reduced with normal sodium sulphite or sodium thiosulphate, the chromate precipitated with barium chloride, and the free alkali is titrated with normal hydrochloric acid, using methyl orange as indicator. If large quantities of alkali metal chromates are present, the barium chromate precipitate should be filtered off, and an aliquot portion of the liquid titrated.

F. H. L.
