

found many of the industries managed on a scale only to be dreamed of in the earlier years, we must find splendid inspiration for future work. It is perhaps fair to say that nowhere else in the world can be found so extensive operations in the production of animal fats and oils; in the complete utilization of the waste animal products; in the manufacture of crude fertilizers, glue, soap, and glycerol; in the manufacture of malt, spirits, and the products of brewing, glucose and refined sugar. These are some of the products of technical work common to the city and while such a list must of necessity be incomplete it is sufficient to amply illustrate the sympathy with our work to be found in our immediate surroundings.

And within easy reach as we are of the great exhibitions of the products of chemical manufactures, those signs of the world's progress resulting from the efforts in which most of us are engaged, we must easily find valued causes for earnest congratulation.

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### THE TECHNICAL ESTIMATION OF LEAD.

BY ALBERT H. LOW.

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THE ordinary fire assay for lead in ores is frequently so erroneous in its results as to cause great dissatisfaction. With some ores the results are considerably too low, and with others, notably oxidized ores containing iron or copper, the lead button may contain several per cent. of such impurities, and weigh more than is warranted by analysis. In Colorado there has been considerable agitation in favor of abandoning the fire assay for a wet method, but thus far no concerted action has resulted. One of the main objections to a wet method is the lack of a suitable one. A number of schemes, both gravimetric and volumetric, have been proposed, but they all appear to be either too lengthy and troublesome or too inaccurate. The writer has devoted much of his spare time to the subject, and feels that the ideal method has not yet been evolved. Alexander's molybdate method is the shortest scheme proposed, but, unfortunately, lime interferes, and lime is a common constituent of ores. The writer's ferrocyanide method, described below, might

be made equally short if the question of lime were neglected. To be sure, the small percentage of lime found in perhaps the majority of the ores might with safety be neglected, on account of the solubility of its sulphate and consequent removal, but it would appear dangerous to employ a method in which lime might seriously interfere, as almost any attempt to remove much lime would also remove some lead. In the two following schemes, devised by the writer, almost any ore may be treated with safety with results that are quite accurate enough for technical work. The first method is based upon the writer's gravimetric method and Hempel's titration of the oxalate. In the second method the titration with ferrocyanide is apparently new. Both methods are of course susceptible of slight modifications in special cases and appear to give equally accurate results. The ferrocyanide method is considerably the shorter, but on the other hand the titration with permanganate is more rapid and satisfactory than where the tests have to be made on a plate. The methods are given for what they are worth as an addition to the literature on the subject.

#### PERMANGANATE METHOD.

Treat one gram of ore in a 250 cc. flask with about 10 cc. of aqua regia. Boil gently until decomposition appears about complete and then add 10 cc. of strong sulphuric acid and heat over a small naked flame until the sulphuric acid fumes are freely evolved. Cool and add 50 cc. of water and heat to boiling. Filter through a small filter and wash with dilute sulphuric acid, (1 : 10). Have boiling in a wash bottle a solution of commercial ammonium chloride, made by diluting a cold saturated solution with an equal bulk of water. Spread the filter upon a watch-glass, and with the hot ammonium chloride solution, rinse the contents into a small beaker, using about 50 cc. of the chloride solution. Stir the mixture to dissolve as much of the sulphate of lead as possible and decant the solution into the original flask, retaining any heavy residue in the beaker. This residue may contain lead. Add to it a few cc. of a strong solution of caustic soda and heat to boiling. Any lead salts will quickly dissolve. To this solution add sufficient dilute sulphuric acid to cause a precipitation of the lead, or

most of it, avoiding an excess, and rinse the contents of the beaker into the flask with the hot chloride solution. A failure of the sulphate of lead to redissolve is of no consequence. Place in the flask three pieces of sheet aluminum, each about one-sixteenth of an inch thick by five-eighths of an inch wide and an inch and three-quarters long. Commercial aluminum will answer. Heat the mixture to boiling and boil for five minutes. If the bulk of the solution is not too great the lead will now be all precipitated. Now nearly fill the flask with cold tap water, allow the lead to settle and then decant into a large dish in order that any escaping particles of lead may be observed and recovered. Fill up and decant five times to remove the chlorides. Finally, to the lead and aluminum in the flask add 5 cc. of a mixture of one part strong nitric acid and two parts distilled water and warm gently. The lead easily dissolves. Pour the solution into a small beaker and shake the pieces of aluminum along with it. Rinse off merely the lip of the flask and then wash the solution back into it, retaining the aluminum in the beaker. To the solution add one or two drops of phenolphthalein solution and then a very slight excess of strong caustic soda solution. Now add 10 cc. of a cold saturated solution of oxalic acid and cool the mixture if warm. Filter and wash thoroughly with cold water. Place in the flask about 75 cc. of distilled water and add a few cc. of strong sulphuric acid. Have this heating while the oxalate of lead is being washed. Finally drop the filter and contents into the hot solution and titrate at once with permanganate solution of the same strength used for iron, *i. e.*, about one-tenth normal. The factor for iron multiplied by 1.888 gives the factor for lead. This figure is empirical and had best be determined by each operator for himself by dissolving 0.30 to 0.40 gram of pure lead, reduced from the acetate, in dilute nitric acid, (two water to one acid), and putting this through the entire process.

None of the ordinary constituents of the ores interfere with this method. The results are accurate within one or two-tenths of a per cent.

#### FERROCYANIDE METHOD.

Treat one gram of ore in a 250 cc. flask with about ten cc. of

aqua regia. Boil gently until decomposition appears complete and then add ten cc. of strong sulphuric acid and heat over a small naked flame until the sulphuric acid fumes are freely evolved. Cool and add fifty cc. of water and heat to boiling. Filter and wash with cold water. See that the filter is well creased so as to lie as flat as possible and prevent solid matter from getting under the fold. Place a funnel in the original flask and rinse the contents of the filter (without removing it from its funnel) into it as thoroughly as possible with cold water. Not more than fifty cc. of water need be used. Add ten cc. of a cold saturated solution of commercial carbonate of ammonium and heat to boiling,—the heating being necessary to ensure the complete conversion of any sulphate of calcium to carbonate. Cool to ordinary temperature, under the tap or otherwise, and filter through the original filter, washing flask and precipitate thoroughly with cold distilled water. Place in the flask some dilute acetic acid, equivalent to about five cc. of glacial acid and twenty-five cc. distilled water, and have it heating while the filter is being washed. The flask usually contains a small residue of lead carbonate which is thus recovered. Place the filter and contents in a large clean beaker and add the boiling hot acetic acid. Stir the mixture about until the carbonates are entirely dissolved, heating if necessary. Dilute with about 100 cc. of cold distilled water and the solution is ready for titration with standard potassium ferrocyanide. The end point is indicated by the usual brown tinge when a drop is tested on a porcelain plate with a drop of a saturated neutral solution of uranium acetate. Reserve a portion of the solution and titrate the main body rapidly until the end point is passed, then add most of the reserved portion and again titrate with more caution till the end is again passed. Finally rinse in the balance of the reserve and finish the titration two drops at a time, stirring well after each addition. When the final brown tinge is thus obtained allow a little time for the previous tests to develop and then deduct from the reading of the burette two more drops than are indicated by the tests, as a solution containing no lead shows the test only after the addition of the third drop of ferrocyanide. The standard ferrocyanide should

contain about ten grams to the liter. On the basis of one gram of ore taken for assay, one cc. of this solution will equal about one per cent. of lead. It is standardized on about 0.30 to 0.40 gram of pure lead, reduced from the acetate. This is dissolved in a little dilute nitric acid, (two water to one acid), then boiled with ten cc. of sulphuric acid and put through the entire process.

An ore may be assayed in thirty minutes by the above method.

While the above method answers in ordinary cases, to provide against bismuth and antimony (of which the basic sulphates might cause trouble), the following modification may be adopted: To the sufficiently cool residue of the sulphuric acid evaporation add ten cc. of dilute sulphuric acid, (1 : 10), and about two grams of Rochelle salt. When this is dissolved add forty cc. of water, heat to boiling and proceed as usual.

Lime interferes with this method only by a solvent action exercised by the acetate on the brown uranium ferrocyanide of the test. Thirty per cent. of lime in an ore, however, occasions no serious interference. Calcium sulphate must not be allowed in the acetate solution as it would cause a loss of lead as sulphate.

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## THE GELATINE EXPLOSIVES.

BY P. GERALD SANFORD, F. I. C., F. C. S.

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**A**MONG the various forms of explosives the material known as gelatine is one of the most important, and is rapidly displacing the older forms of dynamite. The gelatine explosives chiefly in use are known under the names of blasting gelatine, gelatine-dynamite, and gelignite. They all consist of the variety of nitro-cellulose known as collodion-cotton (*i. e.*, the penta and tetra-nitrates, the hexa-nitro-cellulose being known as gun-cotton) dissolved in nitroglycerol, and made up with various proportions of wood pulp, and some nitrate, or other material of a similar nature. Blasting gelatine consists of collodion-cotton and nitroglycerol without any other substance and was patented by Nobel in 1875. It is a clear, semi-transparent, jelly-like substance, of a specific gravity of 1.5 to 1.55,