

The Crescent iron ore, put out by the Bureau of Standards, gave the following results:

Time, minutes.....	30	30	30	45	30
Acid, cc. H ₂ SO ₄	5	10	10	20	15
Anode.....	Pt wire	Amalg. Zn	Amalg. Zn	Amalg. Zn	Amalg. Zn
Cathode.....	Pt gauze	Pt gauze	Cu gauze	Cu gauze	Cu gauze
Current, amp.....	2.2	1.9	1.6	0.0	0.0
Blank, cc.....	0.0	0.13	0.13	0.13	0.13
% Fe.....	58.70	58.66	58.70	58.77	58.70

In the preliminary treatment of the ore, the recommendations of the Bureau were followed and the permanganate used in the titrations was standardized by sodium oxalate obtained from the Bureau of Standards. No correction has been applied for the small amount of titanium (0.07% TiO₂) present in the ore which may account for the values being higher than the value of 58.62% given by the Bureau of Standards.

Conclusions.

1. It is possible to get practically complete reduction of ferric iron, as indicated by permanganate consumed, by using a large platinum cathode and small platinum wire anode if the concentration of sulfate is small. No blank should be subtracted.

2. By using a soluble anode and platinum cathode, complete reduction is possible with wide range of acidity, but with low acidity some of the iron may be deposited on the cathode with high current density.

3. Chlorides must be absent with platinum anode, but with soluble anode may be present unless too great current density is employed.

4. By using a short-circuited cell with platinum gauze cathode and zinc anode, complete reduction may be obtained with wide range of acidity.

5. The most practical method of reduction in most cases is a short-circuited cell with copper gauze cathode and amalgamated zinc rod anode. The concentration of acid must be at least 10 cc. conc. H₂SO₄ per 175 cc. solution. Under these conditions 0.12 g. of iron may be reduced at room temperature in eight minutes with a zinc efficiency of 70%.

6. With short-circuited cell, chlorides do not interfere with the reduction.

7. Increase in temperature in every case increases the rate of reduction.

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NOTE.

*A Thermo-Regulator for Electrically Regulated Constant Temperature Chambers.*¹—The need for an adjustable thermo-regulator for electrically controlled constant temperature chambers of any description has led the writers to devise the form represented in the accompanying illustration, Fig. 1. Regulators of this type which have been in use for some time

¹ Published by permission of the Secretary of Agriculture.

have proved so satisfactory that it seems worth while to publish a brief description of the apparatus.

This regulator consists of a main "bulb" whose form may be varied according to requirements, and a capillary stem provided with a small auxiliary bulb¹ *g* connected with the stem by a capillary neck. Platinum wires are sealed in at the points *d* and *d'*. The wire at *d* should barely protrude into the capillary, and an enlargement of the bore at that point should be carefully avoided. The stem of the regulator is graduated in

millimeter units for a distance of one or two centimeters above and below the wire *d*. This graduation greatly facilitates the adjustment of the regulator. In the unsealed regulator the stem *i* should extend 15 to 20 cm. beyond the auxiliary bulb.

The apparatus is filled in the following manner: The capillary stem is connected with a vacuum pump by means of an intermediary three-way stopcock. When the air has been exhausted from the apparatus, mercury is allowed to run in until the bulb and part of the capillary stem have been filled. A little mercury is allowed to run into the auxiliary bulb also. The stopcock is then removed and the tube from the vacuum pump is attached directly to the stem of the regulator. The tube should be firmly wired on. The regulator is suspended by means of this tube while the air is exhausted with a high vacuum pump. While

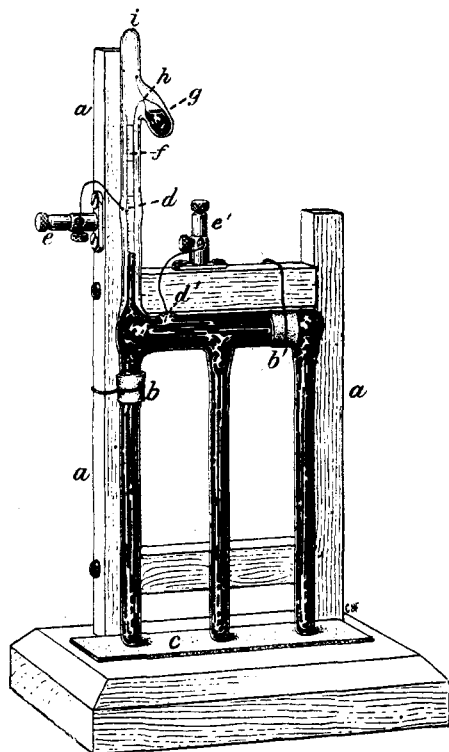


Fig. 1.

thus evacuated the parts of the regulator are carefully heated section by section until all the mercury has been boiled. Mercury is now boiled over from the auxiliary bulb into the stem until the stem is completely filled to the neck of this bulb. Finally, the mercury in the auxiliary bulb is kept boiling under the low pressure while the stem is sealed off at *i*. In this way the air is entirely removed from the apparatus and corrosion of the mercury surface, due to sparking at the point of contact with the wire *d*,

¹ A side-bulb resembling the one here described but connected with the stem by a wide neck has been described by Regaud and Fouilland for another form of regulator. *Ann. rev. chim. analyt.*, 14, 141-146 (1909).

is obviated. In order to avoid sparking, the regulator should be used with a current of very low amperage (0.01 amp. or less) which actuates a relay. The heating current can not be passed through the apparatus.

To adjust the regulator, a small quantity of mercury is shaken from the auxiliary bulb into the stem or from the stem into the bulb, according to whether adjustment for a lower or higher temperature is desired. The capillary connection *h* should be rather narrow, as otherwise it is not possible to transfer sufficiently small globules of mercury to effect an accurate adjustment. The adjustment is greatly facilitated by the graduation on the stem. When the regulator is brought to the desired temperature by means of a water bath, the number of graduations between the level of the mercury column and the wire *d* will indicate the quantity of mercury which must be transferred from the stem to the auxiliary bulb, or conversely. The transfer may be made after the regulator has returned to room temperature. The form of the main "bulb" of the regulator is, of course, immaterial and may be varied according to requirements. In the type illustrated, the bulb consists of a large tube 1 cm. in diameter with three small tubes each 10 cm. long. This form has been found very convenient where the regulator is placed directly in the chamber to be controlled. Where the temperature is indirectly controlled, as in the water jacketed incubators in common use in laboratories, an oblong bulb would be more convenient. In the regulator illustrated, the volume of the mercury up to point of contact *d* is about 20 cc. The increase in volume per degree would be 0.0036 cc. The diameter of the capillary *f* is about 0.16 cm. Its cross-section would, therefore, be 0.02 sq. cm. The linear expansion in the capillary per degree centigrade would be 0.18 cm. The length of the capillary is 5 cm., its volume 0.1 cc. This volume of mercury would correspond to the expansion of 20 cc. of mercury caused by a rise through 28°. The volume of the bulb *g* is about 0.5 cc., allowing a possible temperature range of over 100°.

The apparatus was constructed for the writers by E. Machlett & Son, 153 East 84th St., New York, N. Y.

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ON THE STEARATES AND PALMITATES OF THE HEAVY METALS WITH REMARKS CONCERNING INSTANTANEOUS PRECIPITATIONS IN INSULATING SOLUTIONS.

BY ALFRED E. KOENIG.
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Introduction.

The stearates and palmitates of the heavy metals have been known for a long time. Brief accounts of their preparation and analysis are found