LABORATORY AND PLANT

THE PYROMETER IN THE ASSAY MUFFLE¹

By FREDERIC P. DEWEY²

Standing alone, by itself, a pyrometer reading has absolutely no value as a control of assay operations in a muffle or as a guide to the assayer in carrying on such operations. The reasons for this are varied and complex. (1) The temperature that controls the success of the operation is that of the lead button undergoing oxidation. At present we have no means of learning this temperature under practical working conditions, so that some suitable place must be selected within the muffle for the location of a pyrometer. (2) Unfortunately, however, there is absolutely no approach even to a fixed relation between the pyrometer reading at any given point available and the temperature of the oxidizing button. The oxidation of the lead supplies much heat to the button, but its effect upon the pyrometer is negligible. One factor governing the amount of heat utilized by the button is the rate of oxidation of the lead, and this in turn is, within wide limits, largely influenced by the passage of the air over the button, so that to fully utilize and apply the pyrometer reading we must also know the height of the barometer and the effect of variations in the barometer readings upon the draft of the particular muffle under consideration. Further and most important, from a practical standpoint, is the freedom of entrance for the air to the muffle. In other words, by manipulating the door or the stopper of the muffle, widely varying differences between the button temperature and the pyrometer reading may be produced. The effect of the door conditions is twofold. It affects the supply of air to the button and also the actual temperature of the bottoms of the muffle on account of the varying amounts of air that have to be heated there in passing through the furnace. Finally the relation of the position of the button within the muffle to that of the pyrometer is vital. Therefore, to intelligently utilize any stated pyrometer reading it is essential to have exact information upon a variety of other conditions surrounding the operation.

Bradford³ pointed out the inconsistencies of various statements regarding pyrometer readings in assaying and well established facts, such for instance as advising a temperature of 700° to 750° for cupellation when it requires at least 906° to fuse litharge. In a series of tests he demonstrated the large amount of heat supplied by the oxidation of the lead and the higher temperature thereby attained by the button. His arrangement of apparatus was ingenious, but risky to the pyrometer couple and not applicable to routine work. He gives an excellent description of the conditions immediately surrounding a cupellation.

Fulton, Anderson, Goodner and Ossa⁴ determined

the difference in temperature between an empty cupel and the cupelling lead in an adjoining cupel as 145°, and also give various other temperature determinations, under the conditions employed by them.

For a long time I have been engaged upon an investigation into the conditions surrounding the assay of gold bullion as affecting the accuracy of the results obtained. Naturally the question of the temperature of cupellation early attracted attention, but there were so many other conditions to be investigated where our information was meagre, while the temperature question seemed to be under fairly good control by the eye of experienced cupellers, that the use of the pyrometer was not actively taken up until recently.

In the early days of the investigation various points regarding temperature were carefully considered and some of the problems were worked out. Some of the problems presented themselves with emphasis. In this connection a careful distinction should be drawn between the problem of ascertaining the effect of the various conditions of the cupellation upon the temperature of the cupelling bead and the problem of the regulation and adjustment of these conditions so as to produce the best possible conditions for cupelling, and the final problem of establishing a suitable indicator or guide to show that the proper conditions are being maintained, and especially an indicator which may be applied in different muffles and under varying conditions.

In a broad and general way the time required to work off a given weight of lead is a crude indication of the temperature of the cupellation. When carrying on uniform work in quantity the decreasing size of the button is a general guide for the temperature and a rough notation of the time will often be useful in explaining irregularities of the results. If the general conditions remain uniform, a prolonged cupellation indicates lack of heat, and a rapid one an excess of heat. In making time observations it is essential to adopt some fixed point in the operations to begin taking the time and another to stop. If all the other conditions could be rigidly controlled and the time be very carefully observed, it would furnish a good guide to the temperature, but it would be available only at the finish of the run and could not be used to change conditions during the run. Also, it could not be applied at other times or places or under different conditions. Again it furnishes no preliminary evidence that the furnace is in good condition before starting the work.

It is, however, hardly ever possible to control the other conditions and sometimes accidental variations creep in. On one occasion, when the conditions, including the temperature, appeared to be normal, it became evident that the lead was not oxidizing fast enough. An examination showed that in setting a new muffle the workman had not put the slit in the back of the muffle exactly opposite the chimney outlet. This choked off the draft and retarded the oxidation. The retardation of the work was, of course, excessive, but

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² Assayer, Bureau of the Mint.

³ *THIS JOURNAL*, 1, 181.

⁴ *West. Chem. Met.*, 4, 31.

this experience emphasizes the natural effect of changes in the barometer upon cupellation. A resetting of the muffle corrected the difficulty.

Another *post facto* temperature indicator is the amount of gold absorbed by the cupel in gold bullion assaying, high absorption under similar conditions, indicating high temperature. Here again controlling the other conditions is difficult and I have found this indicator to be of value largely in emphasizing the fact that variations in the other conditions may falsify the pyrometer reading. It makes a difference whether the pyrometer is rising or falling. If we could hold the pyrometer at the same point for a long time before making the cupellation this cause of difference would be minimized, but this is impracticable in every-day work. It makes a difference if the muffle be new and in good condition or old and nearly worn out, and it must not be forgotten that a new muffle may be poor and leak more than an old one, which was of good quality when new.

The only feasible place to put a pyrometer in an assay muffle is close to the top of the arch of the muffle and for convenience it must be inserted from the back. We all know, of course, that closing the muffle increases the temperature and that on moving toward the back of the muffle the temperature rises, as practical every-day working facts. In order to get a more exact idea as to the difference in the temperature in different parts of the muffle and the relations between these temperatures and the fixed pyrometer readings, a second portable pyrometer was placed on the bottom of the muffle in varying relations to the fixed pyrometer as follows: directly under the fixed pyrometer, at the right side and at the left side in the same cross-section as the fixed pyrometer, close to the front in the middle and on each side of the muffle. In some of the arrangements empty cupels were placed beside the bottom pyrometer.

There are three principal causes for differences in the two pyrometer readings: position within the muffle, freedom of entrance of air to the muffle, and condition of the burners on either side of the muffle. In general, the door conditions, governing the entrance of air to the furnace, exert a powerful influence upon the temperature within the muffle, and often cause wide differences in temperature in different parts of the muffle. Under the conditions of these tests, on opening the closed muffle, either pyrometer may fall 100° or more in ten minutes, and a further 10° or 20° before becoming steady. In the closed muffle the two pyrometers registered alike in only one instance, and differed 40° in one. In every instance the movable pyrometer, on the bottom of the muffle, fell more than the fixed pyrometer, at the top of the arch, on opening the closed muffle, and in one instance 40° more.

A point of grave concern is the stability of the pyrometer. In the above tests both of the pyrometers were practically new and may be depended upon, but the effect of long use, for continuous periods, in the litharge-laden atmosphere of the muffle upon the instrument is unknown. It is known that the hot litharge fume is destructive to the tube, and it is only a question of time when it will affect the enclosed couple. At present there are no ready means for testing the accuracy of the pyrometer from time to time, and

it is difficult for an assayer to judge when it is beginning to fail.

In conclusion, I would say that, notwithstanding the objections I have described, the pyrometer occupies a useful field as a general guide to the heat conditions in the assay muffle. In the old and established practice of assaying in the Mint service, in the large laboratories, one or two men do practically all of the cupelling and they grow to be very expert in judging the heat of the muffle and the condition of the cupelling bead by the eye, but the careful and proper use of a pyrometer would often help them, while the man who cupels only intermittently will find it a good general aid. But too much dependence must not be placed upon the pyrometer, and the man who depends upon it entirely will never be a good cupeller.

BUREAU OF THE MINT
TREASURY DEPARTMENT, WASHINGTON

APPROVED BUREAU OF MINES EXPERIMENT STATION AT PITTSBURGH

Plans for the proposed \$500,000 Experiment Station of the United States Bureau of Mines to be located in Pittsburgh, have been approved by the commission appointed by Congress for that purpose.

Congress, a year ago, in the Public Buildings Bill, authorized a new home for the Bureau of Mines to cost \$500,000. It is now expected that Congress, in its present session, will make a specific appropriation so that construction work may begin. It is hoped that contracts may be let by July 1st and the buildings completed in the fall of 1915.

The State of Pennsylvania has appropriated \$25,000 for coöperation in establishing this Experiment Station.

The group consists of three main buildings facing Forbes Street. The central building of the group, the Mining Building, will be three stories in height, flanked by two main buildings, one the Mechanical and the other the Chemical Building. In the rear of these and enclosing a court will be the Service Building. Beyond the Service Building and spanning what is known as Panther Hollow and thus connecting the Bureau of Mines Buildings with the Carnegie Schools, will be two buildings over the roofs of which will pass the roadway from Forbes Street to the Carnegie School Buildings and Schenley Park.

Between the main group and the power and fuel group will be the entrance to a series of mine shafts. One of these will be used as an elevator to carry heavy material and passengers from the lower level to the upper; another will be for tests of hoisting ropes and similar mining appliances; another will be an entrance to tunnels extending under the buildings and in which mining experiments, such as fighting mine fires, will be conducted.

The portion of Panther Hollow above the Power Buildings will be arranged as a Miners' Field, the slopes of the ravine being utilized as an amphitheatre which will accommodate 20,000 spectators who may assemble here to witness demonstrations and tests in mine rescue and first-aid.

The Mining Building will contain the administrative offices, and those of the mining force. In it will be