The Temperature Scales of Columbium, Thorium, Rhodium and Molybdenum at 0.667μ

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To establish temperature scales for columbium, thorium, rhodium and molybdenum spectral emissivities were measured for these metals at $\lambda = 0.667\mu$. The Mendenhall V wedge was used for columbium and rolled cylinders were used for the other metals. Rolled cylinders were more satisfactory for measuring emissivities than specimens in other forms. Rigorous heat treatment was found necessary for accurate results. Emissivities for all four metals were constant with temperatures. Results are:

	Emissivity $\lambda = 0.667\mu$	*Temperature range covered*
Columbium	0.374	1300°–2200°K
Thorium	.380	1300°–1700°
Rhodium	.242	1300°–2000°
Molybdenum	.382	1300°–2100°

A CONVENIENT method of establishing the temperature scale for a substance at incandescent temperatures is to measure its spectral emissivity as a function of the apparent temperature. By Wien's law:

$$1/T = 1/T_s + (\lambda/C_2)\log_e \epsilon_\lambda.$$

Hence if we measure the brightness temperature T_s by means of an optical pyrometer, and know the spectral emissivity, ϵ_λ, as a function of the brightness temperature, we can calculate the true temperature.

Previous Methods of Measuring Spectral Emissivity

For emissivity measurements it is necessary to have a black body and the surface of the material being studied at the same temperature. Readings of each can then be taken. For a molten metal one of the earliest ways of doing this was to sight an optical pyrometer directly on the liquid surface to get the brightness temperature and to sight down a small cavity in a carbon rod partly immersed in the metal for the true temperature. Another early method was to insert a small specimen of solid metal into a cavity in a carbon rod allowing it to project out from the surface of the rod. When heated, apparent temperatures were read from the metal surface and true temperatures from a nearby cavity in the carbon.

Dr. C. E. Mendenhall was the first to use the metal being studied as its own black body.[1] He took a thin strip of metal, bent it into a V wedge 3 cm or more long by 4 to 8 mm wide on a side, and measured the apparent temperatures from the outer surface and the true temperatures from the wedge cavity. For a metal with a spectral emitting surface having an angular opening of ten degrees or less, he showed that the radiation coming from the wedge was practically black body. With thin metal specimens there is no problem of temperature difference between inner and outer surfaces of the wedge. However, unless mounted so as to allow for expansion and contraction, there is some tendency for the wedge to warp out of shape upon heating and so make a poorer black body. Two separate pieces of tungsten were used by Mendenhall to form a wedge and there was some trouble with their separating along the line of contact. However, if the wedge does maintain its shape, results are quite reliable. Many experimenters have used the V wedge among them being Forsythe, Edgerton and Milford, Spence and others.

Burgess and Waltenburg used a secondary standard method of measuring emissivities.[2] They took a minute speck of metal, placed it on a strip filament of platinum or iridium, and measured the radiation from both the speck and the filament. The metals were heated to their melting points and readings taken above and below these temperatures. Three objections can be cited to this method. First, there is the danger of the specimen alloying with the filament thus invalidating results. Next, most of this work was done in an atmosphere of hydrogen. Work done with rhodium by the writer indicates that such results might be questionable if applied to metals being

[1] C. E. Mendenhall, Astrophys. J. **33**, 91 (1911).

[2] Burgess and Waltenburg, Bur. Stan. Bull. **11**, 591 (1914).

worked in a high vacuum. In any case, emissivity values should be obtained under conditions similar to those for which they are to be applied. Lastly, the method depends on the emissivity of platinum which was taken as 0.33 at 0.650μ. Judging by later work, this value is somewhat high. Worthing gives 0.30 for the emissivity of platinum at 1425°K and 0.31 at 1800°K.

A. G. Worthing used two types of tubular filaments for measuring emissivities.[3, 4] For tungsten he made up a squirted tube about 1.5 mm O.D. and 0.8 mm I.D. Fine holes were punctured through the sides of the tube for measuring true temperatures. The thickness of the walls made it necessary to correct for temperature differences through the walls. For molybdenum he used tubular filaments formed by winding molybdenum ribbon into closed helices. There was a tendency for hot spots to develop where windings touched and readings had to be taken at a distance from such places. He also used two other methods of measuring molybdenum emissivities. At room temperatures and at 1080°K he used reflectivity data. At 2000°K he took readings on a compound molybdenum-tungsten filament. This was formed by mounting a tungsten filament and a molybdenum filament end to end and heating carefully until the molybdenum just melted. Readings were taken above and below the junction. However, neither of these latter methods were as satisfactory as the tubular filament method. In the reflectivity work the data taken at 1080°K were obtained under unfavorable conditions, and with the compound filament the spread of alloying action at high temperatures prevented their long use.

Present Methods Used in Emissivity Measurements

In the present work the Mendenhall V wedge was used for columbium and rolled cylinders described below were used for thorium, rhodium and molybdenum. The V wedge was about 5.5 cm long and 4 mm wide. The opening was about ten degrees and good black-body conditions were obtained. The method of mounting was such as to minimize strain and distortion of the wedge, the method being similar to that used for the rolled cylinders.

The rolled cylinders were about 5.5 cm long and $2\frac{3}{4}$ mm in diameter. For soft metals these were easy to make. After taking a strip of the metal to be measured and giving it a slight lengthwise curl around a small metal rod, all that was necessary was to run it through smaller and smaller holes in an ordinary drill gauge. However, for more brittle metals such as thorium, cracks would develop in the edges of the specimen and ruin the cylinder. This difficulty was eventually met in a simple way. A thin flexible sheet of copper was soldered to a small diameter rod in such a way as to allow winding the copper sheet around the rod. Before winding, the specimen to be measured was laid on the copper sheet and rolled with it to practically a butt joint. The rolled copper was then loosened and the cylinder slipped out. Another rolling was sometimes needed to make the butt joint more perfect. A hole drilled in the side of the cylinder was used for measuring the true temperatures. Metals too brittle to fold into a wedge can be made into such cylinders.

Such rolled cylinders have many advantages for emissivity work. They are easy to make, and, because of their shape, are stronger than any other form. When mounted carefully there is no tendency for the butt joint to open up. They can be made quite long (6 cm or more) thus giving a region of uniform temperature at the center. It was not uncommon to have a region 2 cm in length along which temperature differences could not be detected. An advantage over the V wedge is that only one pyrometer is needed for readings instead of two. There is no need to correct for temperature difference through the walls as they are but 0.04 mm thick. The large inside diameter makes for excellent black-body conditions. In these respects the rolled cylinder is more satisfactory than the squirted tube with its thick walls and small inside diameter. The cylinders were mounted vertically, the return lead being a molybdenum strip rolled thin enough to expand about the same amount as the cylinder upon heating. Thus there was very little strain on the cylinder at any time.

[3] A. G. Worthing, Phys. Rev. **10**, 377 (1917).
[4] A. G. Worthing, Phys. Rev. **25**, 846 (1925).

Pyrometers and Their Calibration

All readings were taken with Holborn-Kurlbaum pyrometers with entrance and exit aperatures arranged as described by Fairchild and Hoover.[5] Readings were taken through flat Pyrex windows sealed to the experimental tube. The pyrometers were calibrated through these windows.

In the course of the work three pyrometers were used. The gold point of each, 1063°K, was determined in the usual way by matching with a black-body furnace in which gold had been melted. By the use of sectors a calibration curve for each pyrometer was then obtained for temperatures from 1150°K to 1850°K. As a check on the accuracy of the calibrations, all three pyrometers were sighted simultaneously on a common source and compared throughout this range. Finally, palladium was melted in the black-body furnace and readings taken. It was found that the palladium point, as determined by calibration from the gold point, was within a half of a degree of the point as determined by the actual melt for all three pyrometers. This fact was not only good evidence of the accuracy of calibration of the pyrometers, but also constituted a convincing check on the palladium point itself, 1828°K, as determined by Roeser, Caldwell and Wenzel.[6]

Results

The emissivities of all four metals were found to be constant over the temperatures measured. (See Fig. 1.) Whether there is any significance to this result or not, the writer is not prepared to say. However, it does make one feel that if measurements of emissivity change with temperature, one should take special precautions to make sure the effect is genuine and not due to changing surface conditions.

Columbium

Between the temperatures 1300°K and 2200°K an emissivity of 0.374 was found. The specimen was heat treated for over 500 hours and measurements made under pressure conditions of 10^{-6} mm or better as indicated by a McLeod gauge. This value is much lower than Burgess and Waltenburg's, which is 0.49.

Thorium

Readings on thorium were made after 600 hours of heat treatment and an emissivity of 0.38 was found over the temperature range 1300°K to 1700°K. The cylinder was somewhat shorter than those used for rhodium and molybdenum being about 3.5 cm long instead of 5.5 cm but conditions inside the cylinder appeared uniform. Burgess and Waltenburg's value for thorium is 0.36.

Rhodium

Work with rhodium showed that reliable results could not be obtained unless heat treatment were thorough. Preliminary work on this metal was done in a water-cooled brass chamber with a vacuum of $1–2\times10^{-6}$ mm. For temperatures between 1700°K and 2200°K emissivities increased from 0.24 to 0.29. Below 1700°K there was a sudden change of surface conditions which led to emissivities in the neighborhood of 0.60. Later work in an all Pyrex system showed that these results were completely untrustworthy. It was found that the temperature at which this sudden change of surface conditions took place was a function of the partial oxygen pressure. Vacuum conditions inside the brass container apparently were never satisfactory.

With the all Pyrex system (similar to that used for all the other metals) heat treatment was continued for over 700 hours and pressures as measured with an ion gauge were better than 4×10^{-8} mm. All traces of sudden surface changes disappeared as far as the eye could tell. However, in the early stages of heat treatment, values of 0.24 were found for lower temperatures and 0.26 to 0.28 for higher temperatures. Eventually after the 700-hour heat treatment, the constant value of 0.242 was found as the average over the entire temperature range. This value is low compared to the Burgess and Waltenburg value of 0.29.

Molybdenum

After over 100 hours of baking and 400 hours of outgassing an average value of 0.382 for emissivity was found in the entire temperature range 1300°K to 2100°K. As with rhodium, the pressures were about 4×10^{-8} mm. Although this

[5] Fairchild and Hoover, J. Opt. Soc. Am. **7**, 543 (1923).

[6] Roeser, Caldwell and Wenzel, Bur. Standards J. Research **6**, 1119 (1931).

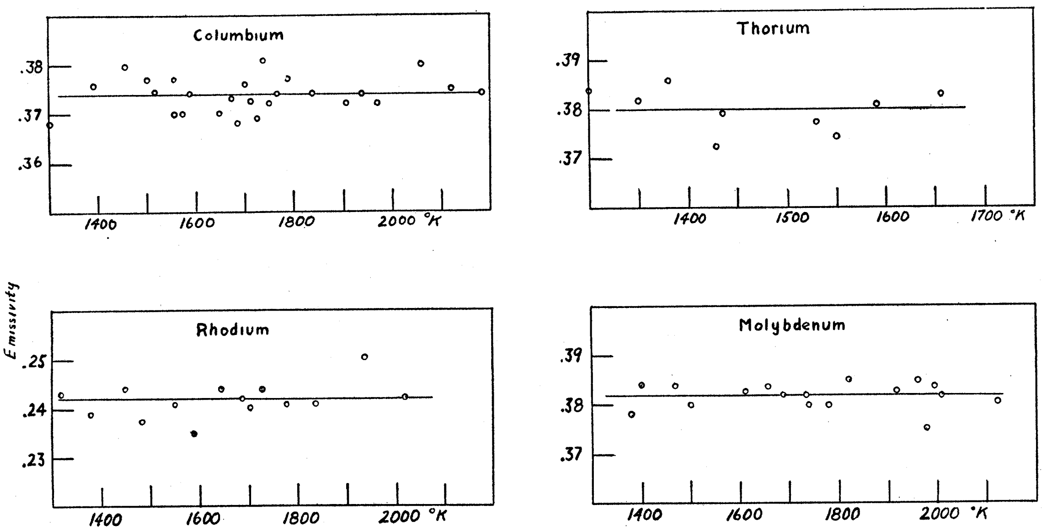

FIG. 1. Spectral emissivities at $\lambda = 0.667\mu$.

value is lower than Burgess and Waltenburg's who give 0.43, and lower than Mendenhall's who gives 0.44 to 0.39 over the range, it is definitely higher than Worthing's value at the high temperatures. Worthing obtained 0.354 at 2000°K. While it is true that almost every error one can make tends to give too high an emissivity, the following experimental evidence warns against assuming such a conclusion for all cases.

For several days readings were taken between 1300°K and 1800°K after heat treating at about 1950°K. Then the temperature was raised to over 2100°K and readings taken. The emissivity immediately dropped from the constant value 0.382, characteristic of the lower temperatures, to 0.369. The writer first believed this to be due to a cleaning up of the metal and expected the lower temperature points to fall in line after further heat treatment. This did not take place. Instead, the emissivity at the high temperatures gradually climbed to 0.382. It seemed that conditions were unsteady immediately after going to a high temperature and that the low emissivity was really characteristic of this unsteady condition.

The molybdenum used was the best grade obtainable from the Fansteel Company. To eliminate the possibility of differences due to different sources, a rolled cylinder was made up from molybdenum kindly furnished by Professor Wahlin by Dr. Forsythe. This molybdenum was similar to that used in A. G. Worthing's work. Measurements made on this sample checked the value 0.382.

This work was done under the direction of Dr. Mendenhall and Dr. Wahlin. The writer wishes to express his indebtedness to them for assistance in the course of the work and also to Mr. John Reynolds who aided in taking and checking many of the readings.