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HIGH TEMPERATURE MASS SPECTROMETER OVEN

By

J. R. Walton

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HIGH TEMPERATURE MASS SPECTROMETER OVEN

J R Walton

January 8, 1953

MASS SPECTROMETRY LABORATORY
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ABSTRACT

A high temperature mass spectrometer oven has been developed to use with the Nier type ion source. This oven is fabricated from either tantalum or platinum. Controllable temperatures that approach the melting points of these metals (tantalum 2850°C and platinum 1755°C) have been attained. This type of oven has also afforded an opportunity to investigate the thermal ions of a number of elements

HIGH TEMPERATURE MASS SPECTROMETER OVEN

J R. Walton

A number of methods are known by which ions can be produced in sufficient quantities for study in the mass spectrometer. If the substance to be analyzed is in the vapor form, it is a simple matter to produce ions by collisions with slow electrons For the production of vapors of substances not available in a gaseous or high vapor pressure form, several heating methods are available. The best controlled method of heating a material is by the use of an indirectly heated oven. Our experience has shown[1] that the limit of the oven temperature is too low to vaporize some of the metal samples and many of the oxides. The large mass of the thermal insulating material also resulted in prolonged outgassing periods Bleakney, Blewett, Sherr, and Smoluchowski[2] used a new type of furnace which consisted of a cylindrical spiral made from a strip of 1 or 2 mil tantalum. A 1-KW 120/20 volt transformer supplied current (between 15 and 25 amperes) which passes directly through the tantalum strip. Ions were produced by electron impact in the molecular beam coming from the hot oven Sampson and Bleakney[3] analyzed substances vaporized from a furnace made of 1 mil tungsten or tantalum foil, folded into a V shape The ends of the foil were spot welded to heavy nickel leads The furnace was heated by a suitable insulated low voltage transformer Using this type of ion source, the abundances of the isotopes in manganese, niobium, palladium, platinum, iridium, rhodium, and cobalt were investigated. Palmer and Aitken[4] designed an ion source to analyze low vapor pressure solids using a minimum quantity of material As little as 1/10 mg of lead and zinc has been analyzed successfully. The material is held in a small crucible consisting of a short length of tantalum or tungsten rod. A small cap with a 1/2 mm diameter hole in the center is pushed over the end of the crucible in order to retain the sample. The crucible is heated by bombardment with electrons emitted from a tungsten filament.

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1. Baldock, Russell, and John R Sites, "A Versatile All-Metal 60° Mass Spectrometer," Y-755.
2. Bleakney, W., J. P Blewett, R. Sherr, and R. Smoluchowski, Phys. Rev 50, p 545, 1936.
3. Sampson, M. B., and W. Bleakney, Phys. Rev. 50, p 732, 1936.
4. Palmer, G. H., and K. L. Aitken, A.E.R.E. -GR-953.

Many low vapor pressure materials have been successfully analyzed in an all metal 60° mass spectrometer[1] utilizing a Nier type source and a cylindrical directly heated oven (Figure 1). These cylindrical ovens are constructed from both tantalum and platinum which makes it possible to heat samples to temperatures that approach the melting point of these metals (tantalum 2850°C, platinum 1755°C). The oven is small and contains no thermal insulating materials thus reducing the outgassing period

The oven sub-assembly is constructed from thin tantalum or platinum sheet stock (2 mil) which is cut into narrow strips approximately 60 mils wide These strips are drawn through successive smaller holes in a wire draw plate until a long cylindrical tube is formed The cylinder is not completely closed leaving a longitudnal slit through which, during use, the vaporized sample may escape. These open tubes are cut into 1/2 inch lengths Twenty mil wire (tantalum or platinum) is pushed a short distance into the ends of these short cylinders and spot welded in position The wire plugs retain the sample materials in the center of the oven. They add strength to the oven and serve as a structural member to be welded to stainless steel support legs. The support legs locate the oven in the proximity of the ionizing electron beam (Figure 2).

A six volt heavy duty battery, variable resistors, and an ammeter were used as the power supply Temperature and current characteristics for an empty oven (Figures 3 and 4) were determined in an auxillary vacuum system with the oven located directly under a glass window This permitted the use of an optical pyrometer to determine the temperatures The visible changes could also be watched during the heating cycle. A chromel-alumel thermocouple was spot welded to the bottom center portion of the tantalum oven Thermocouples, however, were usable only in the low temperature range. A number of investigations were made before the high temperature oven was used in the mass spectrometer A small piece of 347 stainless steel was inserted into the oven and observations were made as the oven was heated. The first evidence of the stainless steel becoming red was observed at approximately 800°C At 1166°C, the stainless steel showed evidence of being hotter than the oven. This could reasonably be due to the black body effect. The shape of the oven makes for excellent black body conditions. At 1530°C, the stainless steel shortened in length and looked as though part of it had flowed to one side of the oven

Chromic oxide (Cr_2O_3) was heated in a tantalum oven At 907°C, the oven was a dull red but the compound was dark At 1190°C, bright spots appeared in the Cr_2O_3. At 1385°C, the Cr_2O_3 color merged with that of the oven. The current dropped and was reset four times during a five minute period at 1850°C. A rise in pressure in the vacuum system was also noted This would suggest that the Cr_2O_3 had disproportionated This was later verified by an analysis in the mass spectrometer. The final temperature reached with this material was over 2700°C

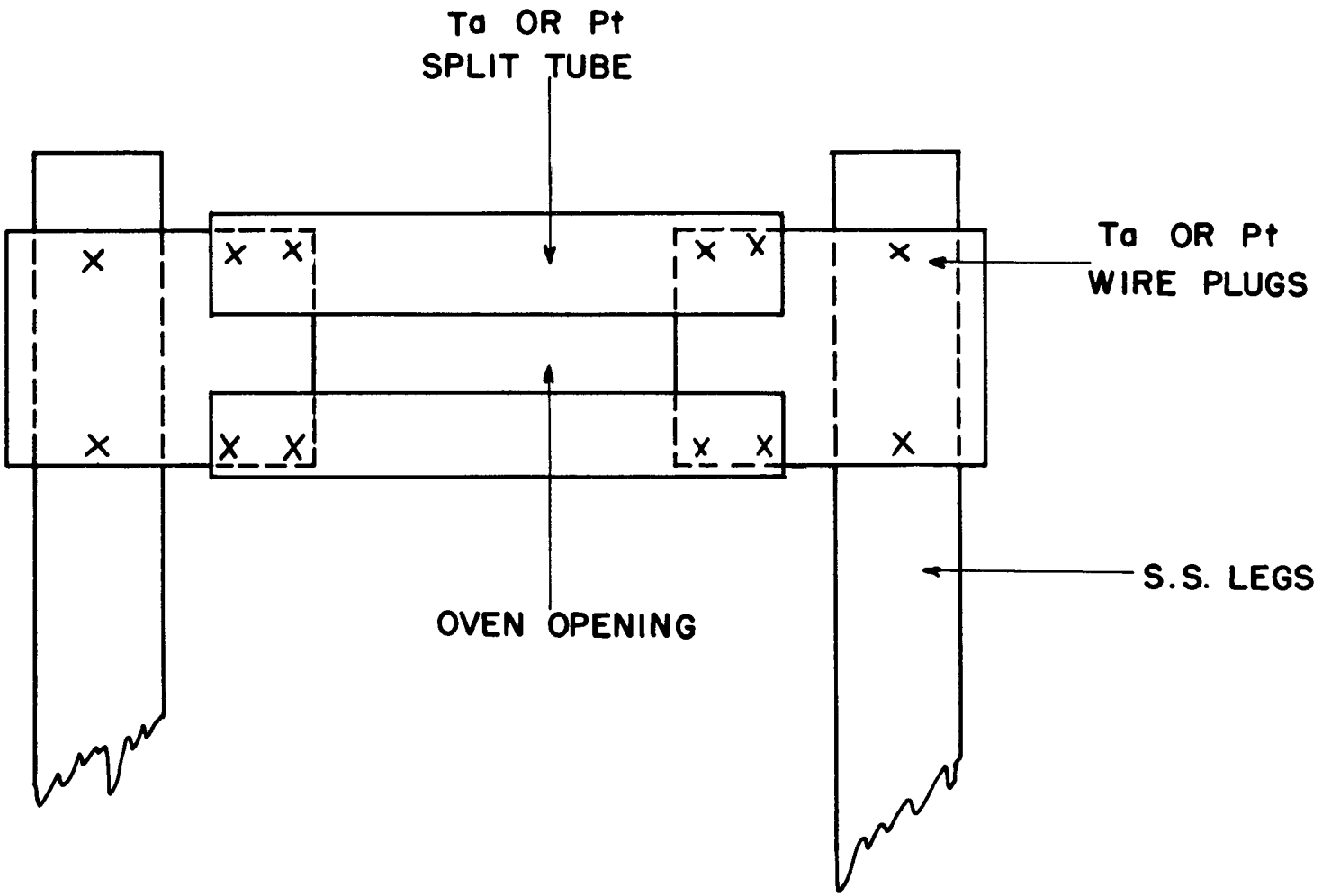

FIGURE I. MASS SPECTROMETER OVEN

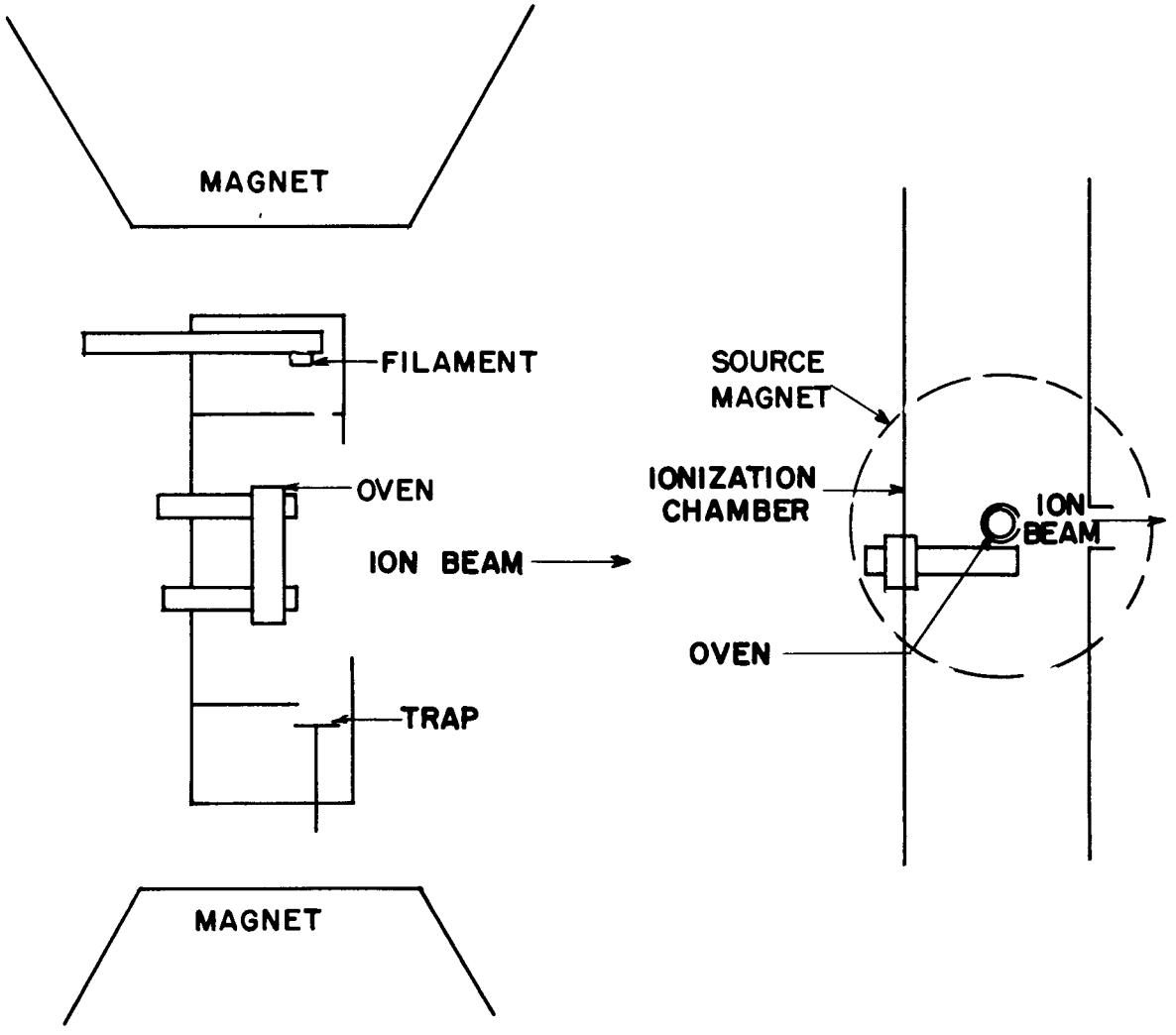

FIGURE 2. HIGH TEMPERATURE MASS SPECTROMETER OVEN

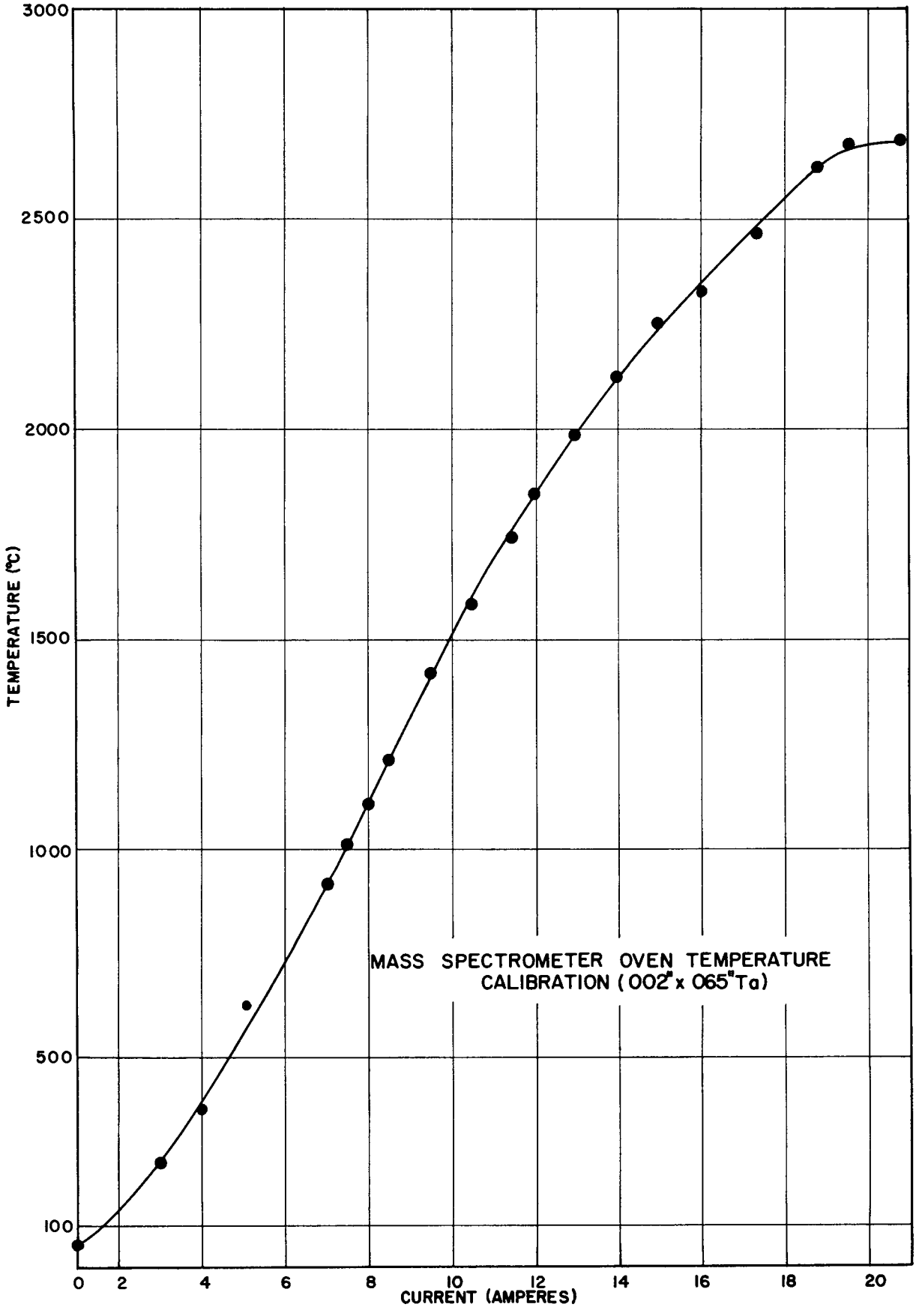
3000
2500
2000
1500
1000
500
100
TEMPERATURE (°C)
0
2
4
6
8
10
12
14
16
18
20
CURRENT (AMPERES)
MASS SPECTROMETER OVEN TEMPERATURE
CALIBRATION (.002" x .065" Ta)

FIGURE 3

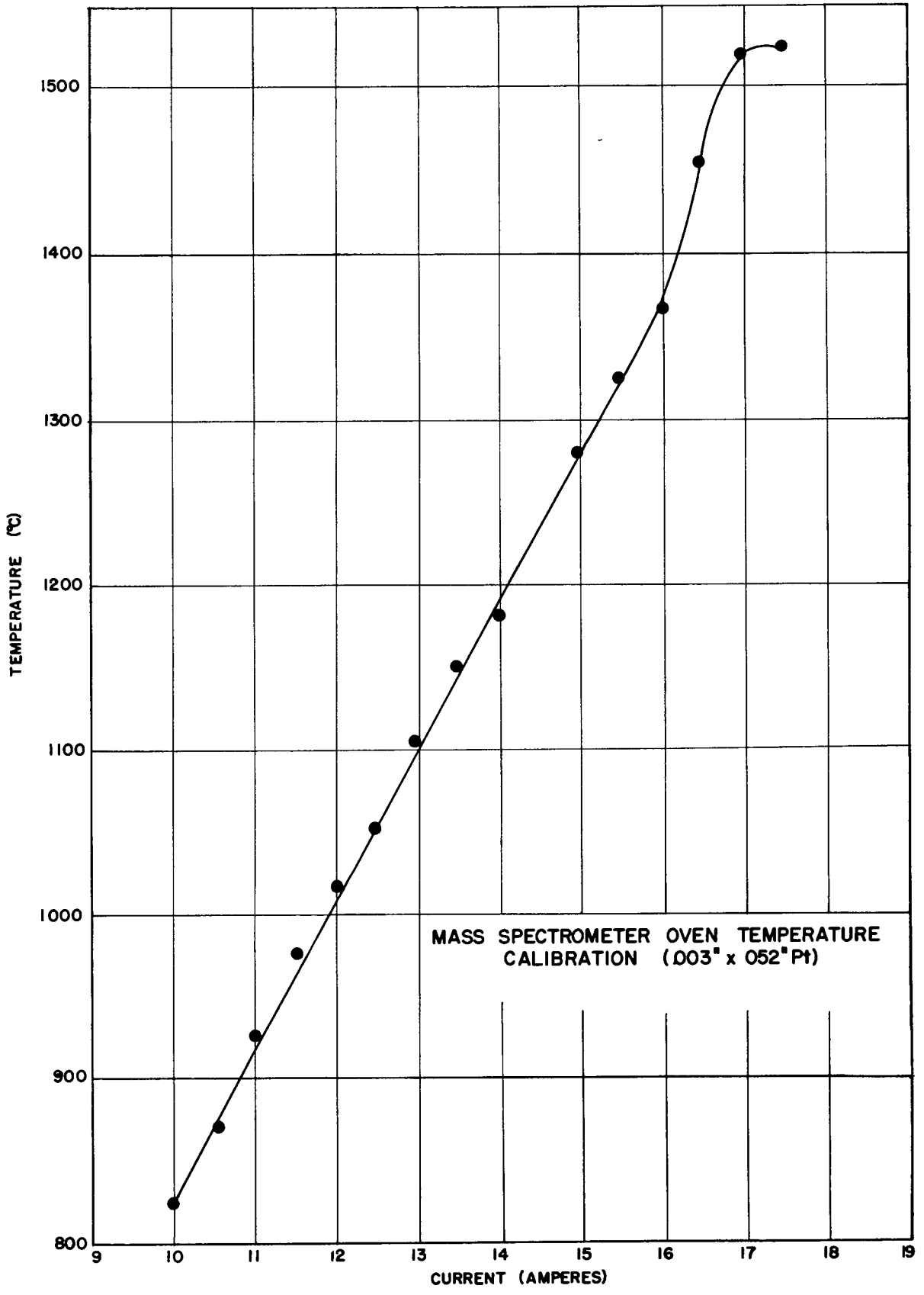
1500
1400
1300
1200
1100
1000
900
800
TEMPERATURE (°C)
9
10
11
12
13
14
15
16
17
18
19
CURRENT (AMPERES)
MASS SPECTROMETER OVEN TEMPERATURE
CALIBRATION (.003" x .052" Pt)

FIGURE 4.

The high temperature oven when placed in the mass spectrometer was found to work very well for nearly all the elements tried, giving a reasonably steady source of ions for sometime, depending on the rate of vaporization. In the early investigations, it was believed that because of the small capacity of these ovens, only short studies could be made, however, of 42 investigations made, only 3 have carried to sample exhaustion One and a half milligrams of uranium tetrafluoride (UF_4) was studied at 800°C for 30 hours without exhaustion. A sample (0 82 mg) of uranyl sulphate (UO_2SO_4) was operated for 10 hours at 1500°-2000°C after which upon inspection of the oven, there was visible material remaining.

Tantalum ovens proved satisfactory except when investigating a uranium compound (UO_2F_2) Mass interference at mass position $U235^+$, $U238^+$, UF^+ (Mass 257), and UF_2^+ (Mass 276) were due to $TaOF_2^+$ (Mass 235), TaF_3^+ (Mass 238), TaF_4^+ (Mass 257), and TaF_5^+ (Mass 276). The gettering by the tantalum gives misleading results when studying the dissociation products of the fluorides and oxides Oxygen did not come completely out of the tantalum at 2100°C

Platinum has shown no reactions with those sample materials thus far tried and the only time that platinum peaks were evident was near the melting point (1755°C) of platinum. The mass spectrometer operation techniques with the platinum oven are the same as with tantalum.

The ionization efficiency of the ion source has been increased by placing the oven nearer to the ion exit slit.

The fact that positive ions are emitted directly by hot bodies has been known for many years [5] Recently, attempts have been made to utilize this effect to produce a source of positive ions for mass spectroscopy. Blewett and Jones[6] coated the substance to be studied on filaments which took the form either of platinum strips or mesh, or tungsten spirals Ions of magnesium, calcium, strontium, barium, aluminum, gallium, indium, titanium, vanadium, manganese, yttrium, and cerium were emitted when the oxide of the metal in question is heated to a white heat on a tungsten filament A mass-spectrograph study of barium, chromium, indium, gallium, lithium, and sodium was made by Sampson and Bleakney[7] using the ions obtained from coated filaments

5. Richardson, O. W., "Emission of Electricity from Hot Bodies," Longmans, Green and Co., 1921.
6 Blewett, J P., and E J. Jones, Phys Rev 50, p 464, 1936.
7. Sampson, M B., and W. Bleakney, Phys. Rev. 50, p 456, 1936.

The total yield of positive ions from thermal filament sources is quite small. In order to increase the intensity and total yield, A. E. Shaw[8] designed a source in which a cylindrical crucible was used to hold the substance to be ionized The crucible was heated by electron bombardment. Nier[9] succeeded in obtaining singly charged positive ions of chromium by heating chromium in a tantalum furnace to 1200°C.

A mass analysis was made for a number of elements utilizing the thermal ions obtained from the cylindrical high temperature oven. It was only necessary to remove the ionizing electron stream, the oven remaining heated. Thermal ions of lithium, potassium, calcium, rubidium, strontium, caesium, barium, and barium fluoride were found. Some of the rare earth thermal ions observed are samarium, samarium oxide, samarium fluoride, and europium. Other thermal ions found were chromium and cobalt.

The success we have had with this source leads us to hope that it will make available analyses of materials that have been heretofore difficult to handle by other means.

The writer wishes to express his appreciation for the assistance he received from Russell Baldock under whose direction this work was carried out, to John Sites and George Wells for their aid in securing the data presented in this report.

8. Shaw, A. E., "An Intense Positive Ion Source for Solids," MDDC-308.
9. Nier, A. O., Phys. Rev. 55, p 1143 A, 1939.