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HIGH-TEMPERATURE KNUDSEN EFFUSION CELL ASSEMBLY

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ARGONNE NATIONAL LABORATORY
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Chemistry Division

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To be described here is a Knudsen effusion cell assembly which was designed for use with a Bendix time-of-flight mass spectrometer for the study of vaporization processes at high temperatures. The cell is heated by electron bombardment, and temperatures up to 3100°K have been achieved. All of the original parts, except the effusion cell and the filament, are still in use after several thousand hours at temperature. Parts are easily replaced and aligned, and a variety of experimental arrangements is possible.

Most of the assembly details are evident in Figures 1, 2, and 3.

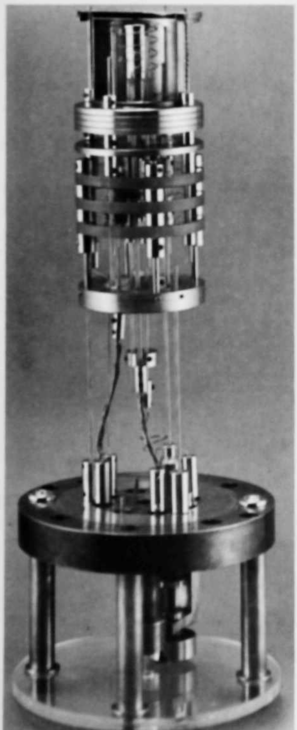


Figure 1

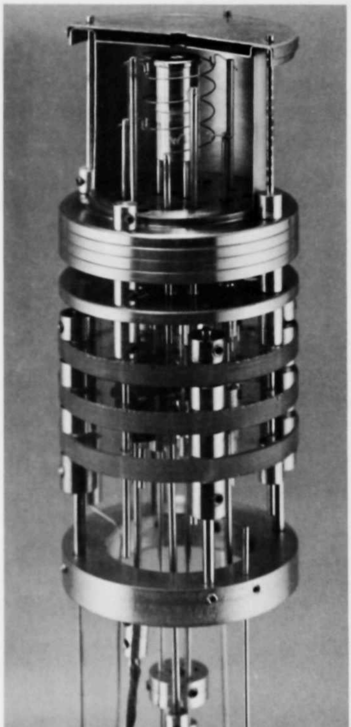
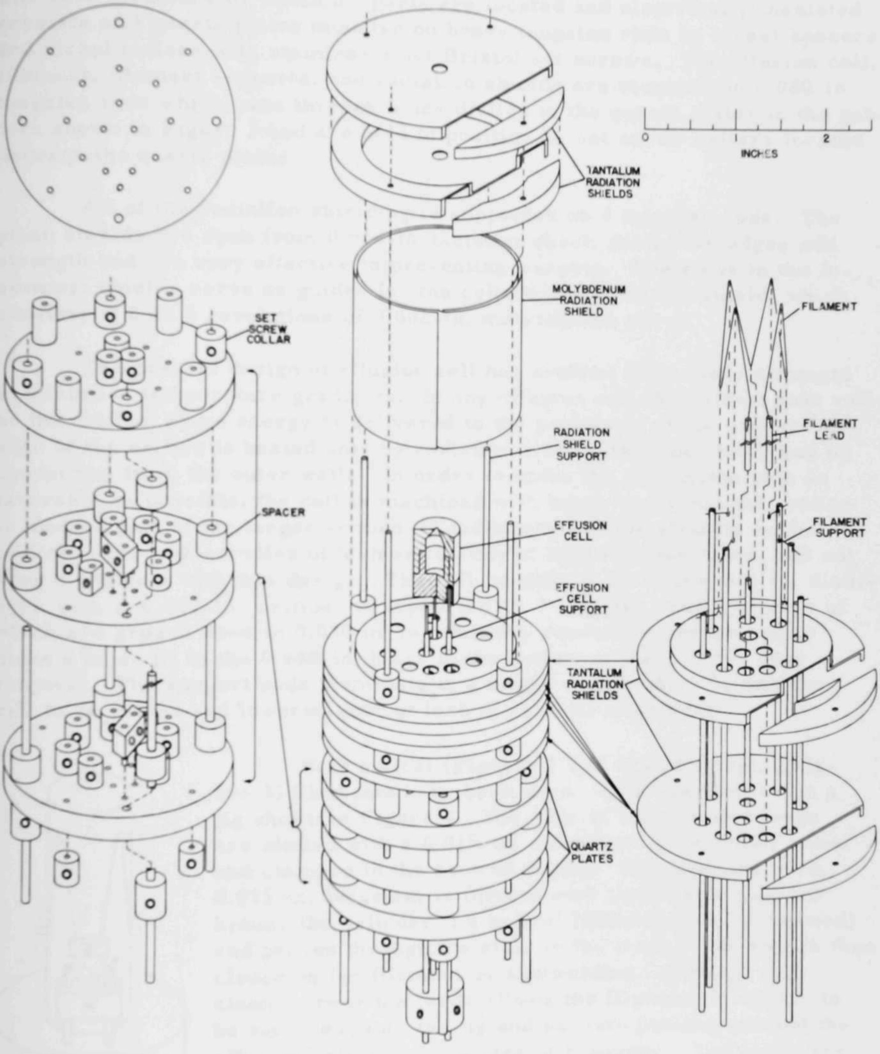


Figure 2



0
1
2
INCHES
TANTALUM RADIATION SHIELDS
MOLYBDENUM RADIATION SHIELD
FILAMENT
SET SCREW COLLAR
RADIATION SHIELD SUPPORT
FILAMENT LEAD
SPACER
EFFUSION CELL
FILAMENT SUPPORT
EFFUSION CELL SUPPORT
TANTALUM RADIATION SHIELDS
QUARTZ PLATES

Figure 3

The base structure by which all parts are located and electrically insulated consists of 3 quartz plates mounted on heavy tungsten rods by nickel spacers and nickel collars with stainless steel Bristol set screws. The effusion cell, filament, filament supports, and radiation shields are mounted on 0.080-in. tungsten rods which pass through holes drilled in the quartz plates in the pattern shown in Figure 3 and are held in position by set screw collars located between the quartz plates.

All of the radiation shielding is supported on 4 tungsten rods. The plane shields are spun from 0.020-in. tantalum sheet; the rolled edges add strength and are very effective in preventing warping. The steps in the innermost shields serve as guides for the cylindrical radiation shield, which consists of 2 or 3 revolutions of 0.002-in. molybdenum sheet.

The present design of effusion cell has evolved from many attempts to minimize temperature gradients. In any effusion cell the coldest part will be the orifice, since energy is delivered to the periphery of the cell and the edge of the orifice is heated only by radiation from within the cavity and by conduction from the outer walls. In order to make the conduction path as favorable as possible, the cell is machined with heavy walls and the orifice is made a part of the larger section. Condensation of the effusate in the orifice, even with samples of high emissivity at high effusion rates, has not been a problem with this design. The cell, usually $\frac{3}{4}$ in. long and $\frac{1}{2}$ in. diameter with a 0.040-in. orifice, is supported on 3 tungsten rods, the ends of which are ground down to 0.030 in. to minimize conduction losses and to make a loose fit in the 0.040-in. holes in the bottom of the cell for easy removal. The support rods terminate in a single collar which allows the cell to be raised and lowered without loss of vertical alignment.

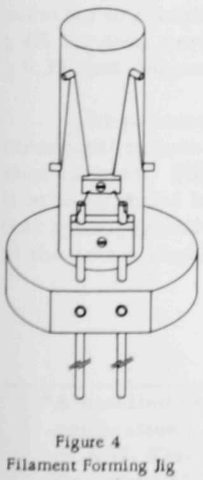


Figure 4
Filament Forming Jig

Both helical (Figures 1 and 2) and hairpin (Figure 3) filaments have been used. Both are formed on a jig shown in Figure 4. The ends of the filament leads are slotted with a 0.015-in. aluminum oxide cutoff wheel and clamped in the base of the jig. The filament wire, 0.015-in. tungsten, is formed over removable pins (or around the cylinder if a helical filament is to be formed) and passed through the slots in the leads. The slot is then closed on the filament by spotwelding. A small split clamp across the leads allows the filament assembly to be removed from the jig and put into position around the effusion cell without danger of breakage. The leads are prevented from rotating by double set screw clamps with locking pins, as shown in Figure 3. Seven filament supports, small hooks of 0.015-in. tungsten wire fastened to 0.080-in. tungsten rods as described above, are provided to prevent sagging of the filament. The positioning of the supports for both the helical and hairpin filaments is shown in the figures.

The base structure by which all parts are located and electrically insulated consists of 3 quartz plates mounted on heavy tungsten rods by nickel spacers and [illegible] collars with stainless steel Bristol set screws. The effusion cell filament [illegible] supports, and radiation shield [illegible] mounted on 0.060-in. tungsten [illegible] pass through holes drilled in the quartz plates in the pattern [illegible] Figure 2 and are held in position [illegible] collars located below [illegible] plates.

[illegible] of the radiation shielding is supported on 4 tungsten rods. The radiation shields are made from 0.020-in. tantalum sheet; the rolled edges are [illegible] very effective in preventing warping. The rods in the radiation shields serve as guides for the cylindrical radiation shield, which consists of 2 or 3 revolutions of 0.002-in. molybdenum sheet.

[illegible] present design of effusion cell has evolved from many attempts [illegible] temperature gradients. In any effusion cell the coldest part will be [illegible] energy is delivered to the periphery of the cell and the [illegible] is heated only by radiation from within the cavity and by [illegible] the outer walls. In order to make the conduction path as [illegible] as possible, the cell is machined with heavy walls and the orifice [illegible] of the larger version. Condensation of the effusate in the orifice [illegible] samples of high emissivity at high effusion rates, has not been a problem with this design. The cell is usually $\frac{1}{2}$ in. long and $\frac{1}{2}$ in. diameter [illegible] orifice, is supported on 3 tungsten rods, the ends of which are [illegible] down [illegible] to minimize conduction losses and to [illegible] in the 0.040-in. holes in the bottom of the cell for easy [illegible]. [illegible] support rods terminate in a single collar which allows the [illegible] and lowered without loss of vertical alignment.

Figure 4

[illegible]

Both helical (Figures 1 and 2) and hairpin (Figure 3) filaments have been [illegible]. Both are formed on a jig shown in Figure 1. The ends of the filament leads are slotted with a 0.013-in. [illegible] cutoff wheel and clamped in the base of the jig. The filament wire, 0.015-in. tungsten, [illegible] removable pins (or around the cylinder if a helical filament is to be formed) and passed through the slot [illegible]. The slot is then [illegible] on the filament [illegible] small split clamp across the leads [illegible] filament assembly to be removed [illegible] around the effusion cell without [illegible]. The leads are prevented from rotating [illegible] clamps with locking pins, as shown [illegible] filament supports [illegible] fastened to 0.015-in. tungsten [illegible] are provided [illegible] magnet [illegible] The positioning of the [illegible] for both [illegible] helical filaments is shown in the [illegible]

The entire assembly is mounted on a stainless steel ring which, in turn, is mounted on the base plate by means of three 0.060-in. tungsten rods (see Figure 1). Two of these are fastened directly to the base plate and oriented with their line of centers parallel to the long axis of the rectangular collimating slits in the spectrometer. The third is fastened to the end of a screw adjustment operated from outside the vacuum. As the third support rod is raised and lowered, the orifice traverses the slit and can be centered either by maximizing the ion current or by sighting through a pyrometer focussed on the orifice from above through the ionization region and the collimators. Electrical contact to the cell, filament, and radiation shields is made through flexible leads connected to the support rods and feedthroughs in the base plate by sleeve couplings.

The power regulator* used with this assembly was designed to deliver 0-20, 30, 50 volts at 25-amp AC filament power and 0-1500 volts at 4-amp DC bombarding power. The bombarding power is regulated by means of a Siemens-Halske Hall Multiplier which senses both the voltage and emission current and computes an error signal proportional to the power. The output voltage is varied by the error signal to keep the power at a preset level. Although only 1600 watts is required at 3100°K, the maximum power of 6000 watts is necessary because of the changing load impedance, which may vary from 100 to 50,000 ohms. At lower temperatures, a filament heating current of approximately 11 amp is required to produce emission, and the bombarding power is of low amperage but high voltage. As the temperature of the cell is increased, the temperature of the filament is raised by radiation from the cell and the filament heating current must be reduced until at about 2400°K it will be cut off entirely. At this point, manual control is lost and the emission may rise to 3 amp. However, even over the wide range of currents and voltages encountered, control is such that it is possible to maintain ion currents in the spectrometer constant to within ± 2% for long periods of time. This corresponds to power regulation within ± 0.3% and temperature control within 2 degrees at the highest temperatures.

At present, the temperature has been limited to 3100°K, not by any structural failure of the assembly, but by severe oscillations in the emission current. The cause of the oscillations has not been determined, but if it is to be found in the power regulator and can be eliminated, it is estimated that a temperature of 3300°K may be attained before the maximum current of the power supply is exceeded.

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*A detailed description of the power regulator is to appear in another publication. It was designed and built by Mr. H. H. Cremer, formerly of ANL Electronics Division, and now with Jet Propulsion Laboratory, Pasadena, California.

The entire assembly is mounted on a stainless steel ring which, in turn, is mounted on the base plate by means of three 0.060-in. tungsten rods (see Fig. [illegible]). Two of these are fastened directly to the base plate and [illegible] line of centers parallel to the long axis of the rectangu-lar [illegible] of the spectrometer. The third is fastened to the end of [illegible] operated from outside the vacuum. As the third sup-port [illegible] raised and lowered, the orifice traverses the slit and can be [illegible] by maximizing the ion current or by sighting through a [illegible] focussed on the orifice from above through the ionization region and the [illegible]. Electrical contact to the cell, filament, and reflector is made through flexible leads connected to the support rods and feed-throughs [illegible] base plate by sleeve couplings.

The power [illegible] used with this assembly was designed to de-liver 0-20 [illegible] 50 watts [illegible] AC filament power and 0-1500 volts at 4-amp DC bombarding [illegible]. The bombarding power is regulated by means of a Siemens-Halske Hall [illegible] which senses both the voltage and emis-sion current and computes [illegible] signal proportional to the power. The output voltage is varied [illegible] signal to keep the power at a preset level. Although only [illegible] required at 1100°K, the maximum power of 1000 watts is necessary [illegible] the changing load impedance, which may vary from 500 to 50 [illegible] lower temperatures, a filament heat-ing current of approximately [illegible] required to produce emission, and the bombarding power [illegible] high voltage. As the tempera-ture of the cell [illegible] filament is raised by radiation from the cell [illegible] current must be reduced until at about 2400°K it wil[illegible]. At this point, manual con-trol is lost and the emis[illegible]. However, even over the wide range of currents a[illegible] control is such that it is possible [illegible] constant to within ± 1% for long periods of [illegible] regulation within ± 0.5% [illegible] temperature [illegible] the highest temperature.

At present, the sample [illegible] 2700°K, not by any shortcoming [illegible] of the assembly [illegible] in the emis-sion current. The [illegible] of [illegible] but if it is to be based on the power [illegible] estimated that a temperature of 3100°K [illegible] before the maximum power of the power supply is reached.

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*A detailed description of the [illegible] regulator is to appear in [illegible] and was designed [illegible] by Mr. H. M. Cramer, formerly of AMF Electronics Division, [illegible] with Jet Propulsion Laboratory, Pasadena, California.