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George Burnet and William Buchanan*

Abstract--A condenser, suitable for use in the distillation of metals was designed. The temperature of the condensing surface was established by controlling the pressure over boiling NaK-78 contained within the condenser. Performance was evaluated in test units in which pure bismuth was distilled as the test metal.

The use of liquid metals in liquid metal fueled nuclear reactors has created the problem of separating fission products from the fuel and carrier metal mixture. One possible method is distillation. In addition to being one of the oldest and best developed methods of separating materials which exhibit a difference in volatility, distillation has the advantage of offering a possible method of purification which does not require any chemical change in the materials being processed.

Since distillation is one of the most highly developed unit operations, problems accompanying its application to liquid metals are not in the development of the general principles, but rather in the areas of design and materials of construction.

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The theory of vacuum distillation of liquid metal mixtures has been presented by Vivian.[3, 4] Beginning with the basic concepts of vacuum distillation, he has derived general relations to express vapor-liquid equilibria and average production rates for metal systems where the relative volatilities are generally large. He cautions that the results require experimental verification.

In any distillation apparatus, three major components must be present: a reboiler, a "column" containing some type of vapor-liquid contacting device, and a condenser, plus, where required, some provision for separating the condensate into reflux and product. A satisfactory reboiler and condenser must be developed before contacting devices can be evaluated. This investigation was concerned with the development of a suitable condenser for liquid metal service.

Previous Work

A comprehensive experimental investigation of the separation of polonium from bismuth was carried out by Endebrock and Engle.[1] Using simple batch-distillation (without rectification) they condensed the distilled metal in the cold section of their column as a solid. Martin and Brown[2] investigated the distillation of bismuth from mixtures containing magnesium, zirconium, uranium, and fission products. Their equipment consisted of a graphite still pot and a mild steel condenser. The latter was cone-shaped with the outer edge extending over the sides of the pot so that the liquid metal condensing on the under side of the cone would collect in an annular reservoir around

the pot. Heat was removed from the condenser by conduction and radiation. The still was operated at 900°C with the condenser at about 600°C.

Condenser Design

In conventional distillation, whether batch or continuous, the normal procedure is to totally condense the overhead vapors and collect the condensate in a receiver. From this vessel a portion is returned to the column as reflux and the remainder withdrawn as product. The difficulty in so handling liquid metals at high temperature and very low pressures would make an "in-column" partial condenser or dephlegmator appear to be a more satisfactory arrangement. The condenser would be so positioned in the distillation apparatus that that portion of the vapors condensing on its surface would fall back into the boiling liquid as reflux (if required) while the remainder would be drawn off and condensed separately as product. The temperature of the condensing surface would determine the fraction of the vapors condensed, or the reflux ratio.

The condenser design selected is based upon the principle that a liquid of known composition will boil at a specific temperature which is a function of the pressure maintained over the liquid. Thus the temperature of the outer surface of any vessel containing the boiling liquid could be readily controlled by simply maintaining the necessary pressure within the vessel. Heat transfered to the vessel by condensing vapors, conduction or radiation, would be removed as heat of vaporization. The vessel could have any configuration required to provide a condensing surface with the geometry required.

Test Unit

Because of its use as a fuel carrier and heat transfer medium in nuclear reactors, bismuth was selected as the metal that would be used to test the condenser. This choice served to fix the temperature range in which the condenser would operate. The sodium-potassium (NaK) eutectic, containing 78 percent potassium by weight, was chosen as the liquid to be vaporized in the condenser. Figure 1 shows the vapor pressure-temperature relationship for this alloy. From this plot it is seen that the alloy will boil at temperatures from 350 to 750°C, depending upon the absolute pressure. The minimum temperature at which such a condenser would give a liquid condensate from Bi vapors would be 271°C, the melting point for Bi.

Details of the condenser design are shown in Fig. 2. The primary condenser section was 20 inches long with a 3-inch length of $\frac{1}{2}$-inch pipe protruding from the bottom as a cold finger. This section contained the boiling NaK-78. A secondary oil-cooled condenser extended down 8 inches into the top of the primary condenser and served to condense the rising NaK-78 vapors and return the condensate to the cold finger. A spiral baffle of stainless steel rod around the secondary condenser assured adequate contact between the vapors and the cooling surface.

The temperature of the oil in the secondary condenser was controlled by wrapping the portion of the condenser above the primary condenser with cooling coils through which cooling water flowed. A length of 1/8-inch stainless steel pipe served as a well for the thermocouple used to measure the temperature of the NaK.

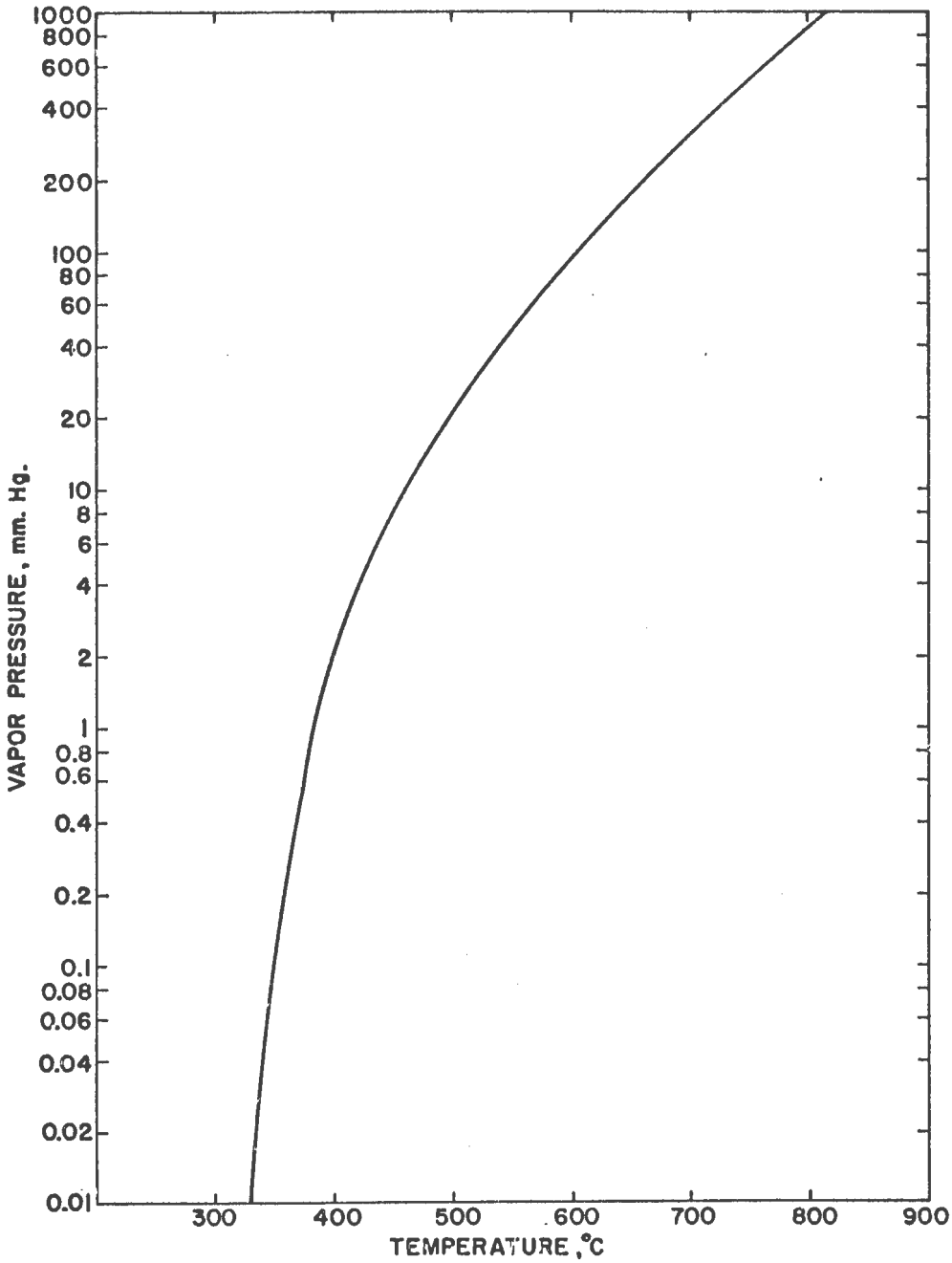

Figure 1. Variation of Vapor Pressure with Temperature for NaK-78.

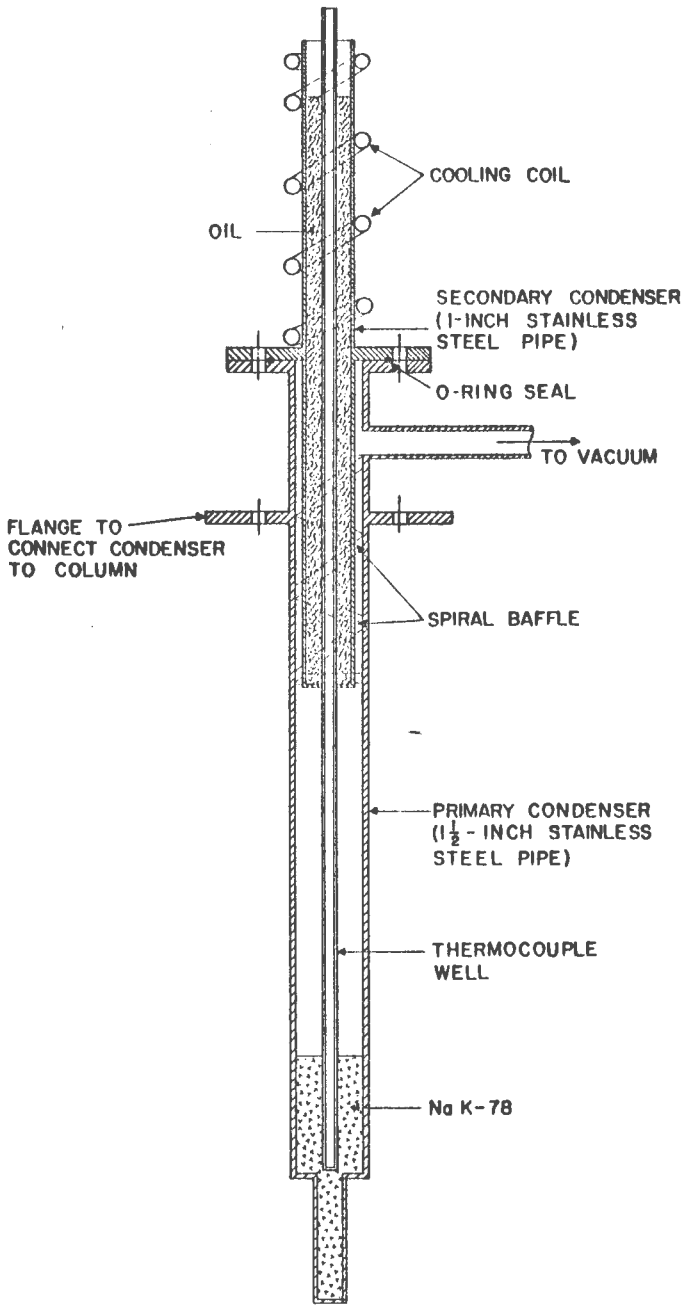

Figure 2. Sectioned View of the Liquid Metal Condenser.

Just below the top of the primary condenser a $\frac{1}{2}$-inch vacuum connection provided a means for controlling the pressure over the boiling NaK-78. Valving in the vacuum system provided a means for isolating the vacuum pump and introducing inert gas into the system. A cold trap prevented any metal vapors escaping from the condenser from reaching the vacuum system.

Simulated Column Tests

In order to determine the accuracy with which the temperature of the cold finger could be predicted, the condenser was assembled and tested by inserting the cold finger into a cylindrical resistance furnace. The results of several tests are shown in Table I where good agreement is evident between the temperature of the boiling NaK-78 as measured by the thermocouple immersed in the NaK-78 and that predicted from the inert gas pressure over the liquid.

Table I. Results of preliminary tests made by heating the condenser in a resistance furnace.

Inert gas pressure, microns	Measured temperature of NaK-78 in the cold finger, °C	Theoretical temperature, from inert gas pressure, °C
30	340	345
100	360	365
14,000	485	475
20,000	505	495

In-Column Tests

Figure 3 is one of the distillation units in which the condenser was tested. The condenser was flanged to the top of the column and so positioned that those vapors passing the cold finger were drawn off into a separate condensing section as product. Vapors condensed on the cold finger fell back into the pot as "reflux". Except for the condenser, construction was entirely of mild carbon steel.

Temperatures were maintained by enclosing the column and condensing section in jackets which were filled with the lead-bismuth eutectic (55.5 percent Bi; melting point 125°C). The jackets were made the hot and cold legs respectively of a thermal convection loop. Heat was supplied to the column jacket by encasing the column in a cylindrical electric resistance furnace. Heat was removed in the condensing section in an outer cooling jacket through which air or water could be passed as the cooling medium. Several hundred hours of successful operation with this unit were experienced before a leak developed in the jacket on the hot leg due to atmospheric oxidation. The column was maintained at approximately 600°C.

Figure 4 is a photograph of the condenser installed in a second unit made of type 347 stainless steel. This unit consisted simply of (1) a reboiler (resting on firebrick), (2) a short, vertical, furnace-encased column on top of which the condenser was mounted, (3) a horizontal, furnace-encased vapor draw-off line and (4) an insulated

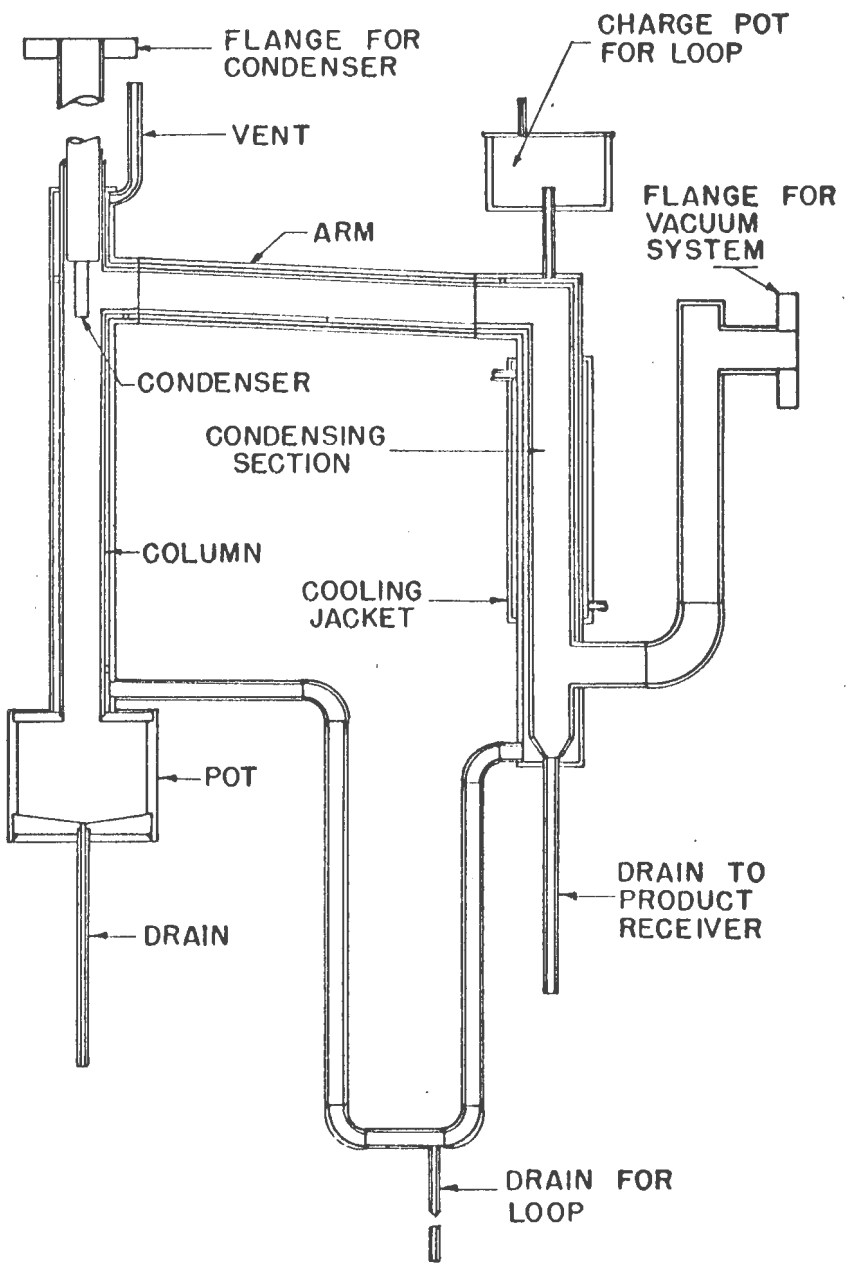

Figure 3. Mild Carbon Steel Distillation Unit with Condenser in Place.

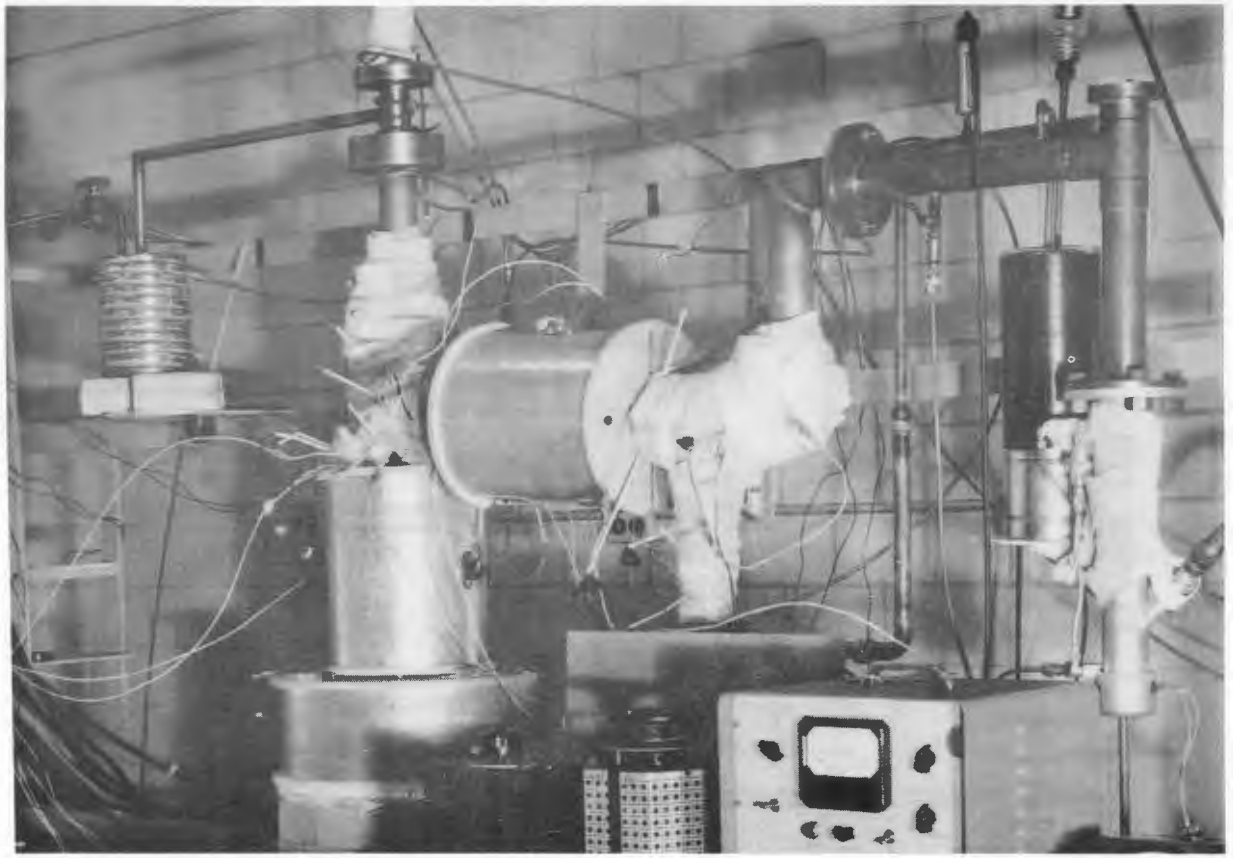

Figure 4. Stainless Steel Distillation Unit with Condenser in Place.

product condenser section. Heat was removed from the latter by natural losses to the environment. The product was collected in a water-cooled receiver at the base of the vertical condensing section.

Results of several runs carried out in this unit while distilling pure bismuth are shown in Table II. The only thing varied from run to run was the temperature of the condenser. As the temperature was increased, the reflux ratio would decrease and more "product" vapors would enter the product condensing section.

Table II. Results of tests made with the stainless steel unit while distilling pure bismuth.

Pressure in distillation unit, microns	Reboiler temp, °C	Condenser temp, °C	Product obtained, grams
0.08	660	482	2.3
0.10	671	548	4.3
0.08	671	571	6.7
0.10	671	593	7.1

Applications

This work has demonstrated the feasibility of using a boiling liquid metal-cooled condenser in the distillation of liquid metals. The design utilized is independent of the configuration of the condensing surface and hence could be used regardless of the type of distillation apparatus employed.

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