

- (3) Irradiating the charged capsule in CP-5 reactor.
- (4) Cooling the capsule the necessary length of time.
- (5) Charging the capsule to the work piece.
- (6) Charging the workpiece to the furnace assembly.
- (7) Evacuating the vacuum tank.
- (8) Heating the furnace assembly to a predetermined steady state temperature.
- (9) Recording temperatures; recording vapor and liquid activity by counting on a single channel analyzer.
- (10) Adjusting induction heating controls to obtain a new higher steady state temperature and then repeating step (9).
- (11) Cooling the furnace to room temperature.
- (12) Removing the furnace assembly from the vacuum tank for disassembly and recharging.

In runs which were made to determine the density of alkali metals, charging the capsule involved the use of a high purity inert atmosphere (helium containing <10 ppm total H_2O+O_2) glovebox and the capsule charging assembly shown in Fig. 2. The desired amount of alkali metal was charged to the capsule body using a 1 cc hypodermic syringe for cesium (which melts at 28°C) and using a scalpel for rubidium, sodium, and potassium (solids at room temperature). The amount of material charged was determined by weighing on an Ainsworth analytical balance contained within the glovebox. After the alkali metal was charged to the capsule body, the capsule body, gasket, and plug (Fig. 1) were placed in the capsule charging assembly and the capsule was sealed under a vacuum of 20 μ . Final closure was made by Heliarc welding of the capsule as shown in Fig. 1.

The capsule was tested for integrity by heating to 1275°C in the inductively heated furnace assembly contained in the vacuum tank. The capsule was reweighed after heating to ascertain if any alkali metal had escaped. If the capsule were found to be sound, it was submitted to the CP-5 research reactor for irradiation to a calculated level of activity. The radioactive isotopes used in determining the critical constants of alkali metals are shown in Table I. This reactor can perform irradiations at thermal neutron fluxes up to 5×10^{13} n/cm² sec. In order to obtain a reading of about 100 000 counts/min at the counter, it was necessary to irradiate the alkali metal to a level of about 50 mCi of activity (2×10^9 disintegrations/sec).

After the irradiated capsule was removed from the reactor in cases where cesium and rubidium were irradiated, the capsule was cooled for about 20 days to permit decay of the 1 day ^{187}W and the 2.75 day ^{99}Mo produced by irradiation of the Mo-30 wt.% W capsule containing the alkali metal. Longer cooling times would be allowable for cesium (2.1 year half-life) and some other materials with long half-lives. Much shorter times were required for sodium (15 h half-life) and potassium (12.5 h half-life).

TABLE I. Radioactive isotopes used in determining critical constants of alkali metals.

Isotope	Half life	Gamma energy Mev	Precursor (n, γ) reaction	Abundance of precursor %	Thermal neutron cross section b
^{86}Rb	18.7 days	1.08	^{85}Rb	72.5	1
^{134}Cs	2.1 years	0.605	^{133}Cs	100	28
^{42}K	12.5 h	1.53	^{41}K	6.88	1.1
^{24}Na	15.0 h	2.75	^{23}Na	100	0.53

Sodium and potassium can be run after 1 day cooling of the capsule because their high energy gamma activities (>1.5 MeV) can be counted without interference from ^{187}W (0.7 MeV) and ^{99}Mo (0.74 MeV). When the capsule was sufficiently cooled, it was charged to the work piece. When the capsule was placed inside this work piece the radioactivity measured at the surface of the work piece was reduced by a factor of 10 from the activity measured at the surface of the capsule. When the capsule was placed inside the split design work piece (Fig. 3), the radioactivity was reduced by a factor of 15. The work piece was then charged to the furnace assembly which consists of the silicon carbide crucible and tungsten carbide insulation held in place by a Fiberfrax lid on the crucible. The crucible was supported by a lava stand adjustable in the vertical dimension. The assembly was heated by a 5 turn water cooled copper induction coil made of flattened 1.27 cm diam cooper tubing. Power was supplied by either a 30 kW or a 15 kW high frequency (10 kc) motor generator.

Prior to heating, the vacuum tank was evacuated to a pressure of less than 1 μ using the Stokes Microvac roughing pump and diffusion pump. When a suitable vacuum was obtained, the motor generator was started and the high frequency power turned on at such a value of power input that the rate of degassing of the insulation would not cause excessive pressure rise. The furnace assembly was heated to the desired temperature and when a constant tempera-

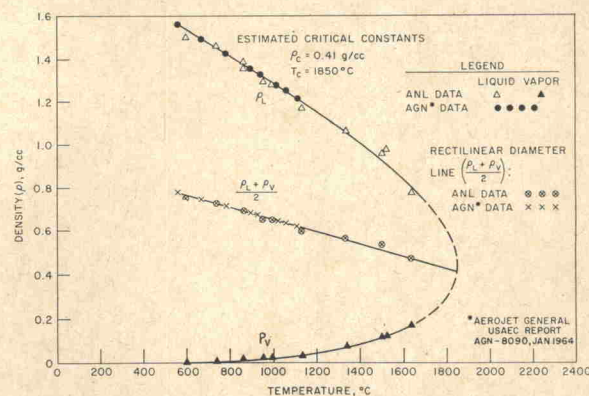


Fig. 5. Rectilinear diameter plot of vapor and liquid densities for cesium.

ture was attained, readings were taken every 5 min of (1) activity emanating from the vapor phase, (2) activity emanating from the liquid phase, and (3) temperature as measured by the tungsten-5% rhenium/tungsten-26% rhenium thermocouples. At least four separate 5 min counts were taken of the activity from each phase.

When sufficient data had been taken at any one temperature, the power input was increased and the data taking procedure was repeated at the new higher temperature. As soon as the run was completed, the power was turned off and the unit cooled to room temperature. The furnace assembly was then removed from the vacuum tank, disassembled and a new charge was made. Typical density data obtained by this method are shown in Table II and

TABLE II. Density data for cesium obtained by a radioactive counting technique.

Temperature °C	Vapor density g/cc	This work	Liquid density g/cc	
			Data of Hochman and Bonilla ^a (Electrical conductivity method)	Smoothed data of Hochman and Bonilla ^a
1220	0.043	1.142	1.208	1.136
1325	0.065	1.080	1.084	1.063
1437	0.090	0.990	1.022	0.980
1551	0.120	0.895	0.962	0.899
1640	0.170	0.785	0.830	0.829

^a See Ref. 3.

Fig. 5. Figure 5 is a rectilinear diameter plot of the type developed by Cailletet and Mathias.¹² The density data of Aerojet General (AGN) shown in Fig. 5 were obtained by a dilatometer technique.¹³ Comparison of AGN data

¹² L. Cailletet and L. Mathias, *Compt. Rend.* **102**, 1202 (1886).

¹³ P. Y. Achener, "The Determination of the Latent Heat of Vaporization, Vapor Pressure, Enthalpy, Specific Heat, and Density of Liquid Rubidium and Cesium up to 1800°F," USAEC Rept. No. AGN-8090 (January 1964).

with the lower temperature data of this study shows good agreement and indicates the high degree of accuracy of the radioactive counting technique.

DISCUSSION

The radiation counting method outlined in this paper has been demonstrated by obtaining vapor and liquid densities of alkali metals from room temperature up to near the critical point using the high temperature-high pressure cells previously described. The cells performed well their function of high temperature containment and shielding. This radiation counting method of obtaining densities is general in nature and applicable to determination of vapor and liquid densities for many substances. Data obtained by this method agree well with the only available high temperature (>1250°C) measurements of other investigators, namely Hochman and Bonilla's electrical conductivity measurements on cesium.³ Hochman and Bonilla estimate their accuracy as $\pm 5\%$. If the substance were not activated by neutron irradiation, an attenuation method might be used. This would involve directing a collimated beam of radiation from a radioactive source through the material.

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