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Use of a Tungsten Filament Lamp as a Pirani Gauge for Continuous Gas Analysis

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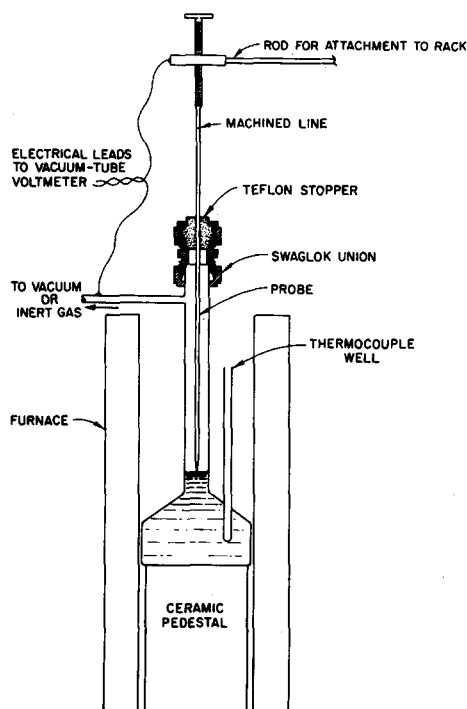


FIG. 1. Apparatus for measuring volume of volatile liquids at elevated temperature. The part of the probe that is above the Teflon stopper is longer, in scale, than indicated in the figure.

liquid surface. It is convenient to construct the neck of the vessel from high quality tubing of constant internal diameter.

The vessel is loaded, at room temperature, with a weighed sample which, when liquid, fills the lower portion of the neck. The sidearm welded to the neck (see Fig. 1) permits either evacuation of the vessel or pressurizing with inert gas. For the measurements on NaNO_3 outlined below, argon pressures of 5–6 atm were maintained; this served to decrease the size of any bubbles in the liquid and, also, to prevent in-leakage of air through the annular space between the probe and the hole in the Teflon stopper. As is done in the calibration with water, the volume of the test liquid is determined from the probe's vertical displacement, relative to the bottom of the vessel, when the liquid surface is contacted. Corrections are, of course, made for the thermal expansion of the vessel and the probe.

After measurements at elevated temperatures, the vessel and its contents are reweighed to check for possible losses of sample. The vessel is then recalibrated to be sure that thermal cycling and/or reaction with the liquid had not altered the volume of the vessel.

To test the reliability of this apparatus, measurements were performed with molten NaNO_3 which, at higher temperatures dissociates into $\text{NaNO}_2(\text{l})$ and $\text{O}_2(\text{g})$. Since the vessel was sealed by the Teflon stopper, the O_2 did

not escape from the vessel; the dissociation reaction was therefore limited. The vessel and probe were both constructed of nickel; the combined internal volume of the cup and conical top was 66 cm^3 ; and the neck had an internal diameter of 0.94 cm. The measurements were taken in the range $313\text{--}425^\circ\text{C}$ with the results given by the equation, $\rho (\text{g/cm}^3) = 2.131 - 7.08 \times 10^{-4}t (^\circ\text{C})$; the standard deviation in ρ was 1×10^{-3} . These results agree very closely with the density equation for NaNO_3 as compiled by Janz,² $\rho = 2.125 - 7.15 \times 10^{-4}t$.

The dilatometer has also been applied to molten alkali fluoroborates from which gaseous BF_3 dissociates on heating. The volume measurements, in apparatus of the dimensions given above, were reproducible to approximately $\pm 0.1\%$. The reproducibility can, no doubt, be improved by using tubing of smaller inner diameter for the neck and increasing the cup volume of the vessel.

* Research sponsored by U. S. Atomic Energy Commission under contract with the Union Carbide Corporation.

¹ J. L. White, "Liquid Densitometry" in *Physicochemical Measurements at High Temperatures*, J. O'M Bockris, J. L. White, J. D. Mackenzie, Eds. (Butterworths Scientific Publications, London, 1959).

² G. J. Janz, *Molten Salts Handbook* (Academic Press Inc., New York, 1967), p. 42.

Use of a Tungsten Filament Lamp as a Pirani Gauge for Continuous Gas Analysis*

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REPORTED herein is a thermal conductivity technique by which continuous and quantitative data have been obtained in a cataphoretic system without sample withdrawal. Spectroscopic techniques were the earliest ones used to measure cataphoresis.^{1–5} When endbulbs are present in the discharge system, Mittelstadt and Oskam⁶ have reported a clever way to periodically determine if the gas within an endbulb is spectroscopically pure. Mass spectrometers have been successfully used to obtain cataphoretic data, but samples must be removed from the discharge system.^{5, 7–9} Recent studies have been reported in which a thermal conductivity probe was utilized for measuring cataphoresis.^{10, 11} For this conductivity method, Flinn¹⁰ stated "The removal of a sample from the discharge system tended to disturb the system somewhat, but the 7 minutes average time between samples was adequate to restore the steady-state condition."

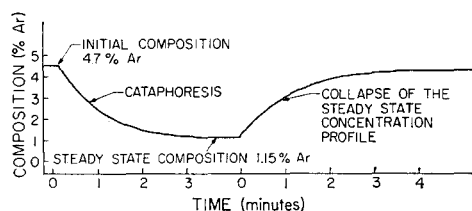


FIG. 1. Argon composition at the anode in a He+Ar mixture vs time for cataphoresis and for back diffusion after the discharge is turned off. Current—100 mA; pressure—3.2 mm.

The disturbance, associated with sample withdrawal in the above technique, may be eliminated by having each gas stream flow through a 60 W lamp. In the flow system the lamp filaments must be calibrated for both composition and flow rate. In the nonflow system involving end-bulbs the 60 W lamp is connected directly behind a porous molybdenum screen serving as the anode. The molybdenum screen is rolled tightly inside a 9.5 mm Kovar glass-to-metal seal which is grounded. The gas discharge system is cooled by a constant temperature water bath. For typical operating conditions in nonflow cataphoretic systems, the product of the Grashof and Prandtl numbers is $\sim 10^{-8}$, thereby indicating that heat transfer from the filament by free convection is negligible.^{12,13} Although the 60 W lamp filament is in a close helix-in-helix winding, a

simple conduction analysis of a straight thin wire predicts the calibration curves quite accurately. Binary mixtures with helium or hydrogen are ideally suited to this technique because of their relatively high thermal conductivities. The lamp filament provides one leg in a Wheatstone bridge. The imbalance of the bridge circuit is amplified and recorded on a strip chart. Typical results, shown in Fig. 1, are in agreement with theory.¹⁴

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¹³ William H. McAdams, *Heat Transmission* (McGraw-Hill Book Co., New York, 1954), 3rd ed., Chap. 7, p. 176.

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Letter to the Editor

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Comments on "Plasma Phasography"

[Herman M. Presby and David Finkelstein,
Rev. Sci. Instrum. **38**, 1563 (1967)]

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THE phase contrast microscope and the theory developed for the plasma phasography¹ are based mainly on the assumptions that one is dealing with microscopic inhomogeneities with characteristic dimensions comparable to the wavelength of the light. However, this is not the case in the experiments performed by the authors. They actually dealt with inhomogeneities, whose typical dimensions normal to the direction of propagation of the light are several orders of magnitude larger than the wavelength of the light. This should mean that the theory developed in the article is not applicable to the experiments.

The "phasograms" shown in the article can be explained as follows: The light beam used is diffracted just as in the usual schlieren technique and no part of it passes through the inhomogeneity as an undeviated wave. The diffraction will take place in every region where there is a gradient of the integrated electron density across the depth of the field (which must be larger than the plasma dimensions in that direction). When the deviation at the mirror is larger than the radius of the conjugate area, the beam is reflected from the mirror and reaches the image plane. When the deviation is smaller, the beam hits the conjugate area, from where only a small fraction is reflected to the image plane. According to this explanation one should expect an even better contrast at the image plane by using a mirror with a hole at the position of the conjugate area. A name such as "mirror schlieren" would perhaps cover the technique better than the name "phasograph."

Herman M. Presby and David Finkelstein, *Rev. Sci. Instrum.* **38**, 1563 (1967).