[CONTRIBUTION FROM THE GEOPHYSICAL LABORATORY, CARNEGIE INSTITUTION OF WASHINGTON]

The Melting Point of Tellurium

BY F. C. KRACEK

In the course of an investigation on the ternary system gold–silver–tellurium, the details of which are soon to be published, we had occasion to purify some tellurium metal, and to determine its melting point. Inasmuch as the values reported in the literature are highly discordant,[1] it was thought advisable to publish a separate note on the subject.

The tellurium used in this determination was purified by vacuum distillation in Pyrex glass at about 575°C., starting with electrolytic tellurium which contained traces of selenium and of heavy metals, such as copper and iron, and a little oxide. This electrolytic tellurium was first melted in a vacuum, to remove volatile impurities, and was then distilled. The metal collected in well-formed shiny crystals, many being more than a centimeter long. Tests showed selenium to be absent. No analysis was performed for the heavy metals, as it is very unlikely that any of these would be carried over into the distillate. Only the best-formed crystals were used for the melting point determinations.

The distilled metal was sealed up in evacuated Pyrex melting tubes, provided with a re-entrant thermocouple well,[2] to guard it against oxidation during the heating. The melting tubes were placed inside a hollow monel metal block and heated in a tubular nichrome-wound furnace. The rate of temperature rise or fall was controlled by a regulating rheostat, and kept uniform in each experiment, at about 1° per minute.

The melting and freezing point determinations were made by differential thermal analysis, the reading thermocouple being imbedded in the thermocouple well of the melting tube, and the reference couple in the monel block (but electrically insulated from it). A shielded Wolff–Feussner potentiometer system with a galvanometer sensitivity of 1 microvolt per scale division was used. The thermocouples were platinum *vs.* platinum–10% rhodium, calibrated as outlined below. The thermocouple readings were converted to centigrade temperatures in terms of the standard reference tables of Adams.[3]

The thermocouples were calibrated with the National Bureau of Standards' "standard temperature samples" of tin and zinc in sealed melting tubes as used for tellurium. The corrections to be added (with reference to Adams' tables) were 2 microvolts for tin (231.9°C.) and 3 microvolts for zinc (419.4°C.). For completeness, calibrations were also made by another method with sodium chloride (800.4°C.) and gold (1062.6°C.) at which points the corrections were 5 and 6 microvolts, respectively. A straight line through these points, plotting the correction *vs.* the temperature, cuts the axis less than 1 microvolt above the origin and yields a correction of 3.3 microvolts at 450°C. We have neglected fractions, and added 3 microvolts to the thermocouple readings in the neighborhood of 450°C.

The melting point was determined on four different lots of distilled tellurium. The individual values obtained were 3718, 3720, 3719 and 3720 microvolts, corresponding to 449.6, 449.8, 449.7 and 449.8°C., corrected. The melting range for the samples melting at 3720 microvolts was exceedingly narrow, about 90% of the melting taking place in a range of 0.5 microvolt. In freezing, after a preliminary undercooling, the thermocouple e. m. f. rose to just over 3719 microvolts for both these samples, that is, to over 449.7°C., corrected. The two samples melting at 3718 and 3719 microvolts had a slightly greater melting range, and were probably not as pure as the other two. Accordingly, we take 449.8 ± 0.2°C. as the melting point of tellurium distilled in Pyrex glass, and tested to be free of selenium.

The character of the melting is illustrated in Fig. 1 for one of the lots of electrolytic tellurium, before and after distillation.

Šimek and Stehlik[4] measured the melting point of spectroscopically pure tellurium, and obtained on five samples 3704.5 ± 0.5 microvolts with a platinum *vs.* platinum—10% rhodium thermocouple for which they found the following corrections: 8 microvolts at the zinc point, 22 microvolts at the antimony point, and 32 microvolts at

---

(1) The literature to 1931 is summarized in "Mellor's Comprehensive Treatise on Inorganic and Theoretical Chemistry," Vol. XI, London, 1931, p. 15. The National Bureau of Standards in its compilation of the melting points of elements, publication M126 issued in 1937, places the melting point of tellurium at 450 ± 10°.

(2) F. C. Kracek, *J. Phys. Chem.*, **34**, 225 (1930); crystalline alumina was used in the thermocouple well to provide heat transfer, in place of the oil mentioned in the article.

(3) "International Critical Tables," Vol. I, 1925, p. 57. The new tables of Roeser and Wensel, *Bur. Standards J. Research*, **10**, 275 (1933), yield the same temperatures to within 0.1°C. in the region we are concerned with, when account is taken of the fact that their reference thermocouple table assigns a higher e. m. f. than the couple on which the tables of Adams are based. Thus, they give 3436 microvolts, as compared with Adams' 3430 at the melting point of zinc. Added from the comment of the referee: "because the melting point of tellurium is not far from that of the standard sample of zinc whose melting point was measured at the National Bureau of Standards with a platinum resistance thermometer on the International Temperature Scale, the melting point of tellurium as here determined is substantially given on the International Temperature Scale."

(4) A. Šimek and B. Stehlik, *Collection Czechoslovak Chem. Commun.*, **2**, 304 (1930).

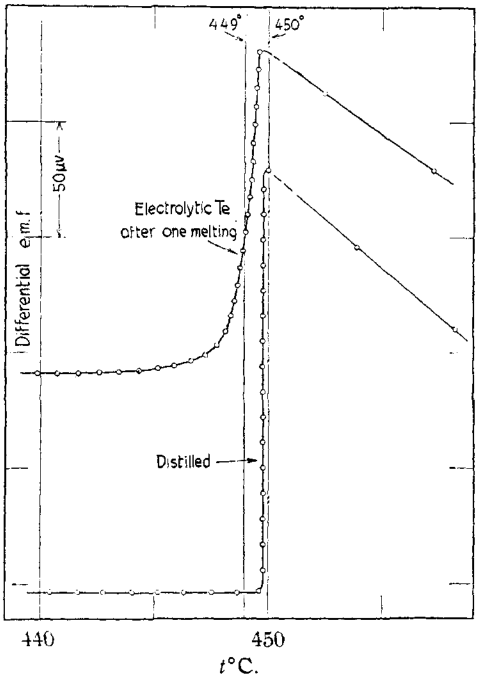

Fig. 1.—Typical differential heating curve characteristics of melting electrolytic tellurium before and after distillation in Pyrex glass.

the boiling point of sulfur. From these they derive a correction of 40 microvolts at 450°C., and state the melting point of tellurium to be 452°C. Now, their sulfur point seems obviously unreliable. If we neglect it, we may derive a correction of approximately 15 microvolts from their other two points, which would make their value 449.5°C., within 0.3°C. of the value determined in this work.

It is often mentioned that tellurium melted in a glass vessel causes a shattering of the glass on freezing, owing to an apparent expansion. This does not happen with pure tellurium, in our experience. Impure metal often carries considerable quantities of oxide as impurity, and we believe the shattering to be due to a reaction of the impurities with the glass and subsequent adhesion of the ingot to the glass, rather than to some peculiarity of tellurium itself. In our distillation experiments we have observed a shattering even with vitreous silica.

Acknowledgment.—We are indebted to S. Skowronski and the International Smelting and Refining Company for supplying some of the electrolytic tellurium used in this work.

Summary

The melting point of tellurium purified by vacuum distillation in Pyrex glass, and tested to be free of selenium, was determined to be 449.8° ± 0.2°C.

WASHINGTON, D. C. RECEIVED MAY 7, 1941

[CONTRIBUTION FROM THE RESEARCH LABORATORY OF THE RADIO CORPORATION OF AMERICA AND THE DEPARTMENT OF CHEMISTRY, STANFORD UNIVERSITY]

An Electron Microscope Study of Curd Fibers of Sodium Laurate

BY L. MARTON, J. W. MCBAIN AND R. D. VOLD

The electron microscope has been used to photograph curd fibers of sodium laurate with direct magnification up to 19,000 diameters. The image so obtained is sufficiently sharp to relate the physical and mechanical properties of soap curd more closely to its ultimate fine structure. The results afford a striking confirmation of previous conclusions based on less direct evidence, and add greatly to the detail with which the general picture can now be presented.

Experimental

The sodium laurate used in this work has already been described.[1] Samples were prepared for the electron microscope by dipping a 400-mesh screen into a 5.6 wt. % solution of this soap in water, which contained 0.02 wt. % excess sodium hydroxide to prevent hydrolysis. The screen was immediately dried in vacuum and introduced into the object chamber of the electron microscope.[2] The whole process of fiber formation occurs in this instance in only a few minutes so there is little opportunity for the formation of regular, highly oriented structure of macroscopic size. As the fibers are formed on cooling and desiccation, they shrink and pull apart along their length. Almost all the "free" ends of fibers occur in pairs and were originally joined together.

From the many photographs taken, Plates 1, 2 and 3 showing curd fibers of sodium laurate at a direct magnification of 11,000 to 18,000 diameters, were selected.

Discussion

The most obvious conclusion from the photographs is that the sodium laurate curd consists of

---

[1] R. D. Vold and M. J. Vold, THIS JOURNAL, **61**, 37 (1939).

[2] L. Marton, *Phys. Rev.*, **58**, 57 (1940); *J. Bact.*, **41**, 397 (1941).