

and forms more stable compounds with alkalies, ekatantalum pentoxide is a weak but decidedly basic oxide with no acidic properties.

All in all it may be said that the predictions made by the writer in 1926 as to the analytical properties of element 91 have thus far been proved by experiment; the expected characteristic properties of ekatantalum have been demonstrated. Once more Mendelejeff's law has proved its usefulness, and its application has rendered comparatively simple the solution of an apparently difficult task.

Summary

1. The unsuccessful attempts to isolate and obtain element 91—ekatantalum—were based on the assumption that it resembles its lower homolog—tantalum—just as radium resembles barium. The origin and development of this assumption and its contradiction to conclusions derived from the periodic law are discussed.

2. A study of the analytical chemistry of ekatantalum has been made with adequate amounts of its pure oxide. Its difference from tantalum predicted by the author on the basis of Mendelejeff's law, has been proved experimentally.

3. The behavior of ekatantalum toward different reagents is, in most of the cases observed, opposite to that of tantalum.

4. Ekatanalium pentoxide (Et_2O_5) is a distinctly, though feebly, basic oxide without acidic properties, whereas tantalum pentoxide (Ta_2O_5) is an acid anhydride and readily forms stable salts with basic oxides.

FUERSTERSTRASSE 10
BERLIN W 50, GERMANY

[CONTRIBUTION FROM THE CHEMICAL LABORATORIES OF THE UNIVERSITY OF UTAH]

THE BOILING POINTS OF CONSTANT BOILING HYDROCHLORIC ACIDS

BY WALTER D. BONNER AND ROLAND E. WALLACE

RECEIVED DECEMBER 5, 1929

PUBLISHED MAY 8, 1930

In connection with the work on the composition of constant boiling hydrochloric acid, recently published from this Laboratory,¹ we have made a precise determination of the boiling points of these acids over the same pressure range. The samples used were those whose analyses are given in the previous paper.

Foult and Hollingsworth have shown² that the variation in boiling point of the acid which distils unchanged in composition at 750 ± 0.2 -mm. pressure is not more than 0.003° , though they were not able to fix the temperature of boiling. In the work reported here a precision of 0.1 mm. was

¹ Bonner and Titus, *THIS JOURNAL*, **52**, 633 (1930).

² Foult and Hollingsworth, *ibid.*, **45**, 1224 (1923).

attained in the pressure control. This corresponds to a precision of temperature measurement of 0.021° at the lowest pressures, 50 to 150 mm. Above this pressure the boiling point curve rapidly flattens out, so that at 370-mm. pressure the temperature measurement has a precision of 0.007° , and at 760-mm. pressure one of 0.004° .

This is not, of course, so great a precision as that with which the vapor pressure of water is known. On the other hand, it is interesting to observe that with the apparatus we used it is much easier to obtain reproducible measurements with these acids than with either water or benzene. We suggest that this may be due to the binary solution overheating less readily than does a pure substance. Whatever the reason, it is an experimental fact that these acids yield more convenient fixed thermometric points than does water.

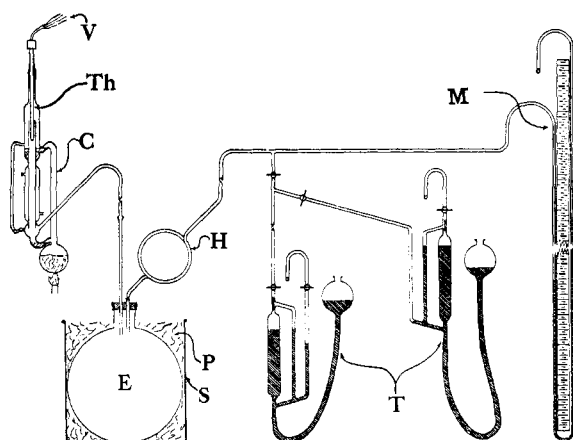


Fig. 1.—Apparatus. V, to bridge; Th, thermometer; C, boiling flask; H, glass spiral; P, paper packing; S, stone crock; E, 5-liter equilibrium flask; T, Toepler pumps; M, manometer.

Experimental

The apparatus, shown diagrammatically in Fig. 1, consisted of a pressure control device, a boiling flask and a platinum resistance thermometer. The pressure control device was essentially two Toepler pumps, the one to force air into the apparatus, the other to pump it out. The entire apparatus was of pyrex. All joints were either sealed or ground, with the exception of the entrance of the resistance thermometer, which was made tight with wrappings of rubber dam. The boiling flask was very kindly supplied by Professor C. A. Kraus, of Brown University. It was designed to minimize loss of distillate and to prevent superheating. The resistance thermometer was calibrated by the United States Bureau of Standards.

It was checked by us at the steam point and found to be exact. It was calibrated and used as a four-lead potential terminal resistor, with current reversing commutator. For this latter purpose we used a very heavy three-pole double-throw switch. Care was taken to have the leads from the commutator to the bridge heavy and of the same length. A radiation shield was not used, but the boiling point apparatus was carefully wrapped with heavy asbestos cord over the part containing the thermometer. Resistances were determined to 10^{-5} ohms by means of a calibrated Leeds and Northrup temperature bridge, of the Mueller type. The check on the steam point of the resistance thermometer was taken to be also a check on the calibration of the bridge. A high sensitivity D'Arsonval galvanometer,

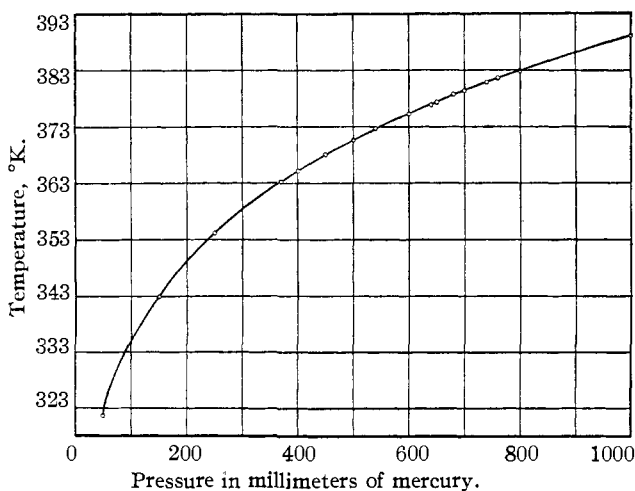


Fig. 2.

with lamp and scale at a distance of four feet, was used as the null instrument. The manometer in the pressure control device was very carefully made and calibrated. The mercury was twice distilled before being put into the apparatus. The two meter scale, of flat grained maple, was compared over its entire length with a standard meter. In making our calculations we have taken into account all essential corrections. The barometer used was of the Fortin type, carefully calibrated, and compared with the United States Weather Bureau instrument in Salt Lake City.

Each sample of hydrochloric acid was introduced into the clean, dry boiling flask. After sealing the apparatus, the pressure was roughly adjusted to within 5 mm. of the pressure at which the sample had been prepared. When the sample had boiled for ten to fifteen minutes, the pressure was very carefully adjusted to within 0.1 mm., all essential calibrations and corrections being considered. The sample was then allowed to boil for an hour before any readings were attempted. In that time the thermometer

had assumed the temperature of the condensing acid vapors, and its resistance was therefore constant. From the resistance of the thermometer the temperature was calculated, in accordance with the constants of the instrument and Callendar's equation. The boiling points so obtained are given in Table I and in the graph, Fig. 2.

TABLE I
BOILING POINT DATA

Press., mm.	Boiling point, °C.	Density, 25°	HCl, %	Press., mm.	Boiling point, °C.	Density, 25°	HCl, %
50	48.724	1.1118	23.42	640	103.967	1.0973	20.507
150	69.956	1.1073	22.520 ^a	680	105.564	1.0968 ^a	20.413
250	81.205	1.1042	21.883	700	106.424	1.0966	20.360 ^a
370	90.237	1.1016 ^a	21.365 ^a	740	107.859	1.0962 ^a	20.268
400	92.080	1.1010	21.235 ^a	760	108.584	1.0959	20.222
450	95.029	1.1002	21.075 ^a	800	110.007	1.0955	20.155
500	97.578	1.0993	20.916	1000	116.185	1.0933	19.734
540	99.653	1.0987	20.777 ^a	1220	122.98 ^a	1.0915	19.358
600	102.209	1.0980	20.638 ^a				

^a Interpolated.

Acknowledgment.—This work was assisted by a grant from the Warren Fund of the American Academy of Arts and Sciences, which we gratefully acknowledge.

Résumé

We have determined the boiling points of constant boiling hydrochloric acid at pressures between 50 mm. and 1220 mm. Pressure was maintained constant to 0.1 mm. and resistances were determined to 10^{-5} ohms. The temperatures given are precise to 0.02° for the lowest pressures and to 0.004° for pressures in the neighborhood of 760 mm.

SALT LAKE CITY, UTAH