The average of the twelve analyses is slightly higher than that found by Baxter and Thomas. This may be in part due to removal of rubidium and potassium from the material used by them.

With the conversion factor 1.000275 our new value for cesium yields -3.79×10^{-4} for the packing fraction of cesium. Interpolation from Dempster's[4] packing fraction curve gives -3.8×10^{-4}, while from the curve of Hahn, Flügge and Mattauch[5] the value for cesium is -4.0×10^{-4}. Aston[6] found the packing fraction of cesium to be 0.3×10^{-4} smaller than that of xenon. Using the recent value for xenon, -4.1×10^{-4}, as calculated by Hahn, Flügge and Mattauch,[5] and Aston's difference, the packing fraction of cesium is -3.8×10^{-4}. This close agreement together with similar agreement in the case of iodine[7] is reassuring in its indication that in this region the chemical and physical scales are not far apart. It should be pointed out, however, that no recent direct determination of the packing fraction of cesium has been made and that the atomic weight of silver, to which cesium is referred chemically, is subject to slight uncertainty.

Summary

1. By comparison of cesium chloride with silver the atomic weight of cesium is found to be 132.913 (Ag = 107.880).

2. The packing fraction of cesium is calculated to be -3.8×10^{-4}, in close agreement with recent values.

CAMBRIDGE, MASS. RECEIVED MAY 13, 1940

[4] Dempster, *Phys. Rev.*, **53**, 870 (1939).

[5] Hahn, Flügge and Mattauch, *Ber.*, **73A**, 1 (1940).

[6] Aston, *Proc. Roy. Soc.* (London), **A134**, 573 (1932).

[7] Baxter and Lundstedt, THIS JOURNAL, **62**, 1829 (1940).

[CONTRIBUTION FROM THE T. JEFFERSON COOLIDGE, JR., MEMORIAL LABORATORY OF HARVARD UNIVERSITY]

A Revision of the Atomic Weight of Potassium

BY GREGORY P. BAXTER AND CHARLES D. HARRINGTON

As a check on the analytical procedure of the analysis of cesium chloride described in the preceding paper a few analyses of potassium chloride were made. The results fall between those of Richards and Staehler,[1] Richards and Mueller,[2] Baxter and MacNevin,[3] Baxter and Alter,[4] and Hönigschmid and Sachtleben,[5] and that of Johnson.[6]

Potassium chlorate which gave no evidence spectroscopically of containing cesium or rubidium was crystallized three times from aqueous solution. The chlorate was converted to perchlorate by heating barely to fusion in platinum, and the perchlorate was separated from the chloride by crystallization. After two more crystallizations of the perchlorate it was converted to chloride in platinum. The chloride was dissolved, the solution filtered through platinum sponge and the salt recovered by crystallization from concentrated hydrochloric acid solution. Four more crystallizations from hydrochloric acid and one from water, all in platinum, followed (Sample A). No cesium or rubidium could be detected spectroscopically.

A second sample of chloride which had been similarly purified by Baxter and MacNevin was used in one analysis (Sample B).

The details of the method of preparing potassium chloride for analysis and of the analytical method are essentially like those described for cesium chloride in the preceding paper. In analyses 1, 2 and 5 the fusion atmosphere was hydrogen plus a few per cent. of hydrogen chloride. In analysis 3 the salt was fused first as above, then in hydrogen alone, and in analysis 4 first in nitrogen, then in hydrogen plus hydrogen chloride.

The following densities were used in computing vacuum corrections: air (0°, 760 mm.), 1.293; KCl, 1.988; Ag, 10.49.

From Dempster's[7] curve the packing fraction of potassium is -7×10^{-4}, while Hahn, Flügge and Mattauch[8] give -6×10^{-4}. Brewer[9] finds the

[1] Richards and Staehler, THIS JOURNAL, **29**, 623 (1907), 39.096.

[2] Richards and Mueller, *ibid.*, **29**, 639 (1907), 39.096.

[3] Baxter and MacNevin, *ibid.*, **55**, 2185 (1933), 39.094.

[4] Baxter and Alter, *ibid.*, **55**, 3270 (1933), 39.096.

[5] Hönigschmid and Sachtleben, *Z. anorg. allgem. Chem.*, **213**, 365 (1933), 39.094, 39.097.

[6] Johnson, *J. Phys. Chem.*, **39**, 781 (1933), 39.100.

[7] Dempster, *Phys. Rev.*, **53**, 870 (1939).

[8] Hahn, Flügge and Mattauch, *Ber.*, **73A**, 1 (1940).

[9] Brewer, THIS JOURNAL, **58**, 365, 370 (1936).

TABLE I

THE ATOMIC WEIGHT OF POTASSIUM

Anal.	Sample of KCl	KCl in vacuum, g.	Ag in vacuum, g.	Ag added, g.	Ag in vacuum, corrected, g.	Ratio KCl:Ag	Atomic weight of K
1	A	4.88482	7.06844	0.00025	7.06819	0.691099	39.099
2	A	4.08265	5.90751	.00000	5.90751	.691094	39.098
3	A	8.52040	12.32907	.00000	12.32907	.691082	39.097
4	A	8.62997	12.48729	.00020	12.48749	.691089	39.098
5	B	8.77749	12.70116	.00020	12.70096	.691089	39.098
					Average	.691085	39.098

average K^{39}/K^{41} ratio in mineral sources to be 14.25. With this abundance ratio the two values of the packing fraction give 39.093 and 39.097 for the atomic weight of potassium (conversion factor = 1.000275).

Summary

By comparison of potassium chloride with silver the atomic weight of potassium is found to be 39.098.

CAMBRIDGE, MASSACHUSETTS RECEIVED MAY 13, 1940

[CONTRIBUTION FROM THE AVERY LABORATORY OF CHEMISTRY OF THE UNIVERSITY OF NEBRASKA]

The Transition Temperatures of the Liquid-Solid Phases of Trimethyl Phosphate

BY H. ARMIN PAGEL AND WALTER A. SCHROEDER

No reference to the melting or freezing point of trimethyl phosphate could be found in the literature. The work carried out in this Laboratory showed that two solid forms can be obtained, and that these forms are monotropic.

Experimental.—Commercial grade trimethyl phosphate, obtained from the Commercial Solvents Corporation, was purified by fractional distillation at two different pressures. Distillations were carried out in a 2-liter all-glass still, fitted with an "indented" type of distilling head (similar to the Vigreux), which was in turn attached to a vertical condenser. The ground glass joint necessary for filling was sealed against leakage under reduced pressure by means of external rubber housings. A mercury trap in series with the aspirator line prevented possible moisture contamination of the ester. Essentially constant reduced pressures were maintained by the use of a specially constructed vacuum valve.

About 3 liters of the ester were fractionally distilled in 1-l. lots at 380 ± 1 mm. The approximate volumes, densities, and refractive indices of the first, middle, and residual fractions (arbitrarily labelled in order: (1-1), (2-1), and (R-1) for later reference) were: (1-1), 10%, d^{20}_4 1.19701, n^{20}_D 1.3936; (2-1) 75%, d^{20}_4 1.21439 ± 0.00001, n^{20}_D 1.3967; (R-1) 15%, d^{20}_4 1.21499, n^{20}_D 1.3968. About one-half of the total (2-1) fraction was then further fractionated at 192 ± 3 mm., labelled in order (1-2) and (R-2), yielding: (1-2) 75%, d^{20}_4 1.21436 ± 0.00000, n^{20}_D 1.3967; (R-2), 25%, d^{20}_4 1.21442, n^{20}_D 1.3967.

Since the physical properties of fractions (2-1) and (1-2) appeared to be constant, these fractions were used. Further possible purification was sought by slowly fractionally freezing 100 ml. of fraction (1-2) until about 30% of liquid remained. The remaining liquid was siphoned off, and the remelted solid used (labelled R-3).

Low temperatures were obtained with a mixture of solid carbon dioxide and alcohol. The low temperature-bath consisted of a cylindrical metal can, 11 cm. in diameter and 17 cm. tall, surrounded by rock wool insulation, 4 cm. thick. Test-tubes for maintaining seed crystals, a pentane thermometer, and a 25 mm. by 200 mm. test-tube fitted into a slightly larger glass shield tube were introduced into the bath through carefully fitted holes in a wooden cover. The shield tube arrangement was found necessary to control the temperature gradient in order to obtain slow freezing. Since alcohol and carbon dioxide vapors are soluble in trimethyl phosphate, these were constantly removed from the surface of the bath through a suitable hole in the cover connected to a filter pump.

The large test-tube described above was fitted with a cork to accommodate a double junction thermocouple and a manually operated ring type glass stirrer. Since the ester is noticeably hygroscopic, purified air was continuously passed into the upper part of the test-tube to prevent possible entrance of moist air. Samples of approximately 35 ml. were used. All temperature observations were made at one-minute intervals. In the freezing point determinations, the observations were usually continued until the solidified portion prevented further effective stirring (estimated 40 to 50% solid) whereupon the container was quickly transferred to a Dewar flask and the melting temperature observed.

Temperature measurements were made with a Leeds and Northrup Student Type potentiometer using double junction copper-constantan thermocouples made of Leeds and Northrup standardized wire. The potentiometer was checked against a calibrated Type K potentiometer at the desired values, and the thermocouples in turn checked against the transition point of sodium sulfate decahydrate, the melting point of ice and the freezing point of reagent mercury, as fixed thermometric points. Within the pre-