[CONTRIBUTION FROM THE T. JEFFERSON COOLIDGE, JR., CHEMICAL LABORATORY OF HARVARD COLLEGE.]

A REVISION OF THE ATOMIC WEIGHT OF TIN.[1] THE ELECTROLYTIC DETERMINATION OF TIN IN TIN TETRACHLORIDE.

BY GREGORY PAUL BAXTER AND HOWARD WARNER STARKWEATHER.

Received February 13, 1920.

A recent investigation upon the atomic weight of tin by Briscoe,[2] in which stannic chloride was compared with pure silver, yielded a very concordant series of results, with an average value 118.698 (Cl = 35.457). This value was adopted in 1916 by the International Committee on Atomic Weights in preference to the one found by Bongartz and Classen,[3] 119.0, which had been in general use for some time. Very recently Brauner and Krepelka[4] have completed the analysis of tin tetrabromide, with the result 118.699. Since the electro-deposition of cadmium and zinc in a weighed mercury cathode has been found to be a process capable of considerable accuracy,[5] and since tin amalgam promised to be unusually well adapted for quantitative handling, the electrolytic method has been applied to the analysis of stannic chloride also. In this way it is possible, in conjunction with Briscoe's work, to obtain a complete analysis of the salt, with the use of analytical processes differing widely in character.

Purification of Materials.

Water, and hydrochloric, nitric and sulfuric acids were purified by distillation, as previously described in papers from this laboratory. Tin condensers were used with water, quartz for nitric and hydrochloric acids and glass for sulfuric acid.

Alcohol was freed as far as possible from water by distillation from lime, and was once redistilled with a glass condenser. This alcohol left no weighable residue upon evaporation.

Mercury, which had been agitated with air under conc. sulfuric acid for a considerable period and sprayed through a dil. acid solution of mercurous nitrate many times, was thrice distilled in a vacuum.

Chlorine was prepared and purified exactly as described by Baxter and Moore,[6] by action on manganese dioxide with hydrochloric acid and drying the gas with conc. sulfuric acid and phosphorus pentoxide,

---

[1] A brief account of this investigation was published in *Proc. Nat. Acad. Sci.*, 2, 718 (1916).

[2] *J. Chem. Soc.*, **107**, 63 (1915).

[3] *Ber.*, **21**, 2900 (1888).

[4] Rozpravy České Akademievěd a umĕné, **1919**.

[5] Baxter and Hartmann, THIS JOURNAL, **37**, 113 (1915); Baxter, Grose and Hartmann, *ibid.*, **38**, 857, 868 (1916).

[6] THIS JOURNAL, **34**, 1645 (1912); *Z. anorg. Chem.*, **80**, 187 (1913).

in an all-glass apparatus. Although the generator and purifying train were thoroughly swept out with the chlorine current for some time before use, it is unlikely that the gas was wholly free from air and carbon dioxide. These impurities were less to be feared, however, than the hydrochloric acid and moisture which were removed in the purifying train.

Although the tin tetrachloride eventually was to be purified by fractional distillation, the metallic tin, from which the salt was prepared, was first freed from the greater portion of both basic and acidic impurities by electrolytically transporting the metal through an acid solution of stannous chloride. In this operation the cathode was a platinum wire, the anode a bar of block tin. The resulting bright crystals, after thorough washing, were dried and fused on an alundum boat in a current of pure dry hydrogen.[1] Before use the alundum boat had been digested for some time with constant boiling hydrochloric acid and ignited. During the fusion a white sublimate, presumably of tin chlorides, was observed. Electrodes for the production of spark spectra were now prepared by drawing some of the melted tin into hard glass capillary tubes thinly coated inside with soot, and by means of a Féry quartz spectrograph the spark spectra were photographed over the range $\lambda 2200$–$\lambda 5000$. The original tin contained lead and copper. Since a large part of both impurities seemed to have been removed by the electrolytic process, electrolysis was repeated in a similar fashion, using an electrolyte made from a portion of the once electrolyzed tin. Although the product still contained a very small proportion of both the copper and the lead, after being fused in hydrogen as above and cleansed with hydrochloric acid, it was used for preparing the stannic chloride without further treatment.

Preparation of Stannic Chloride.

The tin tetrachloride was prepared by the action of dry chlorine upon dry tin and was purified by fractional distillation. Two preparations were made, the methods differing only in minor details. Since the later experiment was naturally the more satisfactory, the description of the procedure will be confined to the second method.

The main portion of tin was sealed in the soft glass tube B (see Fig. 1), which was connected with the chlorine generator at A through a glass stopcock moistened with metaphosphoric acid. The tube B was given a slight tilt toward the bulb I so that the tetrachloride would flow out of B into I as fast as formed. Bulb I contained a few grams of pure tin cast into sticks about 1 to 2 mm. in diameter by drawing the melted metal into hard glass capillary tubes. Bulb I was connected with a Sprengel pump through the side tube F, with a small bulb, *a*, and with a special valve, G.

---

[1] Generated electrolytically and purified and dried by passing over fused potassium hydroxide. See Baxter and Grover, THIS JOURNAL, 37, 1035 (1915).

The valve G, designed by Briscoe and Little,[1] deserves particular attention because it immensely facilitated the carrying out of the fractionation. The sealed-in capillary tube was closed at the end. It could be broken by the sealed glass tube filled with mercury contained in the side tube. Until it was desired to break the capillary the glass-mercury hammer rested at the end of the side tube, but by inclining the apparatus it could be made to slide down the side tube and strike the capillary with enough force to break it. To make sure of the success of the latter operation the capillary was scratched at several points with a file before the sealed-in joint was made. In the course of the work a joint of this sort was used 4 times, successfully in every case.

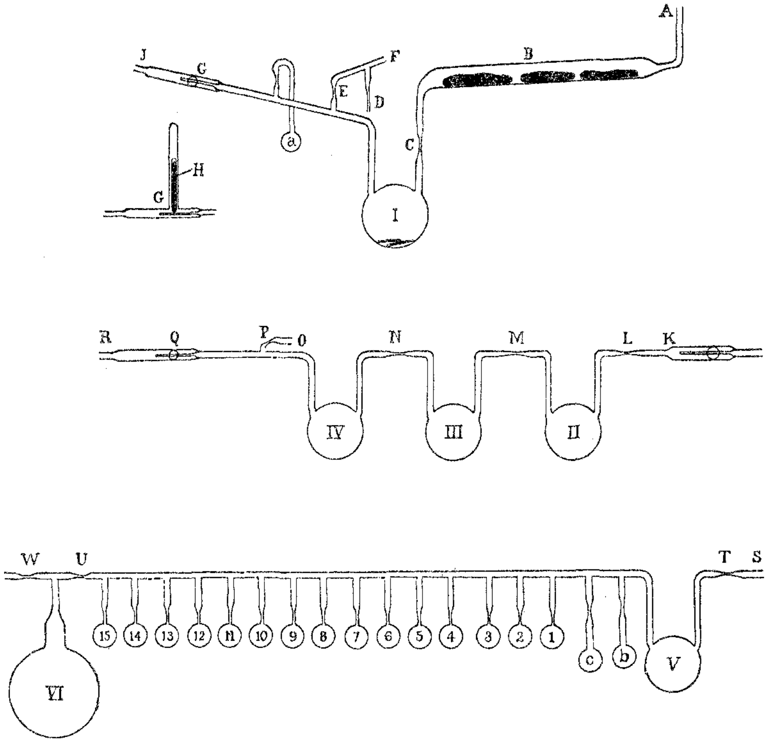

Fig. 1.

Before admitting chlorine, the bulb I was exhausted through F, D being sealed. As soon as the chlorine pressure in I became atmospheric, D was opened. The slight excess of the chlorine thus prevented air and moisture from entering the bulb I. When for any reason the current of chlorine was interrupted, D was always sealed.

The exothermic reaction between the chlorine and tin progressed so

---

[1] *J. Chem. Soc.*, 105, 1321 (1914).

rapidly that it was necessary to cool the tube B with a jet of air in order to prevent the melting of the tin. The liquid tetrachloride dissolved a part of the excess of chlorine and thus acquired a greenish yellow tint. After nearly all of the tin in B had been converted to chloride, the capillaries C and D were sealed, the stannic chloride in I was cooled with alcohol and solid carbon dioxide, and the bulb I was exhausted as far as possible (about 7 mm.) and maintained at this point for several hours. The residual pressure was probably due chiefly to chlorine dissolved in the liquid chloride. Then the capillary E was sealed.

The system was now allowed to stand, with occasional shaking, for about 3 weeks, in order to allow the tin to react with the excess of chlorine dissolved in the liquid, and also to reduce any lead tetrachloride which might be present. After a few days the color completely disappeared and no further change was apparent.

Next a small fraction of the tetrachloride was removed by cooling the bulb *a* with liquid air and when the bulb was nearly full, sealing it off at the capillary. This was done to eliminate so far as possible any hydrochloric acid which might have been formed by the action of moisture upon the tetrachloride.

The open end of the valve G was now sealed to the series of bulbs II, III and IV, which were attached at the other end to another similar valve Q, and the bulbs were exhausted as completely as possible by an efficient Töpler pump through the side tube O. Finally O was closed by sealing the capillary P. During the exhaustion the bulbs and connecting tubes were heated as hot as was safe, in order to expel moisture from the interior walls of the glass. Connection was then established between the bulb I and the rest of the apparatus by breaking the capillary in the valve G. The greater part of the stannic chloride could now be distilled into bulb II by cooling the latter with alcohol and solid carbon dioxide, the temperature being maintained just above the melting point of the chloride, —33°, lest the liquid freeze and break the bulb. During the distillation the stannic chloride in bulb I was kept at room temperature by a bath of water. About 20 g. of liquid residue was left in bulb I when it was separated from the others by sealing the capillary L. Two more fractionations, from II to III and from III to IV, followed, about the same quantity of residue being rejected in each case as in the first distillation, and the bulbs II and III being successively sealed off at the capillaries M and N as soon as each distillation was over.

The connecting tubes of bulb I were frosted with a white coating insoluble in stannic chloride and in time there were deposited upon the walls of the bulb from the residual stannic chloride transparent colorless crystals. No similar deposit could be seen in bulbs III and IV, which, with I, were preserved, for several years, sealed with the residual fraction of stannic chloride. We are inclined to the belief that the white coating was stannic acid formed by reaction with moisture. The colorless crystals may have been the hydrate of stannic chloride, $SnCl_4.3H_2O$, as suggested by Briscoe,[1] who observed a similar phenomenon.

The bulb IV was next sealed through the valve Q to the train including the bulb V, the small bulbs *b* and *c* and 1 to 15, and the large bulb VI. The train was exhausted to a pressure of 0.001 mm. by means of a Töpler pump, with thorough heating to expel adsorbed gases from the glass, and was sealed at W. Next the valve Q was broken. The large bulb VI was included so as to reduce the pressure of gases other than stannic chloride vapor in the system as far as possible. After the stannic chloride had been distilled from bulb IV to bulb V the two capillaries T and U were sealed. At this point we hoped that all gaseous impurities had been eliminated, and in fact we found no evidence that this was not the case, for the bulbs of stannic chloride, when subsequently broken under hydrochloric acid, showed no sign of containing any substance but stannic chloride liquid and vapor.

In order to remove any more volatile fraction the liquid might contain, the two bulbs *b* and *c* were successively chilled with liquid air and when half full were sealed off at the capillaries. These bulbs contained, even after warming to room temperature, a small amount of colorless, crystalline solid similar to that observed in bulb I. The remainder of the liquid was collected in the bulbs 1 to 12, each bulb being sealed off at the capillary when about $^2/_3$ full. All the material in bulb 1 and the greater portion of that in 2 was condensed with liquid air. Alcohol and solid carbon dioxide were used in filling the remainder. Bulb 12 contained all of the residue which could be condensed. Bulbs 1 and 2 contained small amounts of the crystalline solid found in *b* and *c*, but none could be found in bulbs 3 to 12. Bulb V, after condensation of all the liquid possible in bulb 12 by carbon dioxide and alcohol, contained a trace of white residue, obviously deposited by the solution during evaporation.

The chief difference between the 2 series of preparations is that in the first one, owing to the fact that some of the capillary tubes connecting the bulbs in which the stannic chloride was collected were too small, diffusion was very slow and the fractionation unsatisfactory. In order to hasten the operation several of the small bulbs in which the stannic chloride was collected were chilled at the same time.

In Series I, Fractions 1 and 2 were not analyzed or otherwise examined. Fraction 15, the final residue, was converted into electrodes for spectroscopic testing. Fraction 4 was the first one analyzed, and since tin was known to have been lost during evaporation of the solution the results are not recorded. Fractions 3, 6 and 10 were lost by accidents during

---

[1] *Loc. cit.*, p. 71.

the analyses. In Series II, Fraction 1 was not investigated. Fraction 12, the final residue, was converted into electrodes, and Fractions 5 and 6 were lost through accidents. All other fractions of both series appear in the tables of analytical results.

In order to determine whether any metallic impurities had accompanied the stannic chloride through the fractional distillation, different specimens were dissolved in hydrochloric acid solution and the metal was deposited electrolytically as completely as possible with platinum wire electrodes. The amalgams of analyses 13, 14 and 15 were combined and made the anode under dil. hydrochloric acid, the metallic tin being deposited upon a platinum wire cathode.

The specimens examined and the impurities found are as follows:

Series I	II (residue)	Pb, Cu
	15	Pb, Cu
Series II	III (residue)	Pb, Cu
	b, c,	Pb, Cu
	3, 4, 11	None
	12 (residue)	None

The proportions of lead and copper found were extremely minute. By comparison with spectra of alloys containing known proportions of these elements, we estimate the proportion of each impurity to be less than 0.001%. While the fact that lead seems to accompany the stannic chloride during the distillation was unexpected, although lead tetrachloride is a volatile liquid, the persistence of the copper is even more remarkable. At all events the proportion of neither is enough to affect the result by an important amount.

Method of Analysis.

After being weighed, the bulbs of stannic chloride were broken under dil. hydrochloric acid and the glass was collected and weighed. Then the solution was electrolyzed in a weighed glass cell in which was sealed a platinum cathode and anode, the cathode being covered with a considerable quantity of mercury. The electrolytic cells resembled those used by Baxter and Hartmann, and Baxter, Grose and Hartmann,[1] but for reasons to be explained later had a far larger capacity for the electrolyte.

The bulb containing the stannic chloride was thoroughly cleansed outside with cleaning solution, washed, wiped with a clean cloth and dried in a vacuum desiccator over fused sodium hydroxide for at least 24 hours. Then it was weighed in air by substituting weights for the bulb, the temperature and humidity in the balance case and the atmospheric pressure being noted. The weight of the bulb under water was next found, and

---

[1] *Loc. cit.*

the volume calculated from the loss in weight and the temperature of the water. The vacuum correction of the bulb could now be computed from the volume, pressure, temperature and humidity of the air displaced.

In a preliminary experiment the bulb was broken under 0.3 *N* hydrochloric acid, the glass was collected on a filter and the filtrate and washings were evaporated to about 30 cc. before electrolysis.

In this experiment the quantity of tin obtained was considerably less than was expected. As a loss of tin tetrachloride during the evaporation was feared, this point was tested by distilling a solution of 10 g. of stannic chloride in 250 cc. of 0.3 *N* hydrochloric acid and testing the distillate for tin with hydrogen sulfide. No tin was found until more then $^4/_5$ of the liquid had been distilled. In a repetition of the experiment with a solution of 10 g. of stannic chloride in 100 cc. of 6 *N* hydrochloric acid tin was found in increasing amounts in the successive portions of distillate.

On the whole, the safest method of avoiding the difficulty seemed to be to electrolyze the solution of stannic chloride directly without evaporation. Electrolytic cells with a capacity for about 125 cc. of electrolyte were, therefore, constructed. The weighed bulb containing the stannic chloride was broken under 70–100 cc. of 0.3 *N* hydrochloric acid in a large, thick-walled test-tube. Although much heat was evolved, the reaction was not violent enough to cause loss by spattering, and no bubbles of gas ever reached the surface of the liquid. After being stirred the solution was filtered through a filter paper in a quartz funnel into the cell, which had been weighed with a charge of mercury. The glass and test-tube were rinsed with cold 0.3 *N* hydrochloric acid into the cell until it was nearly full and then into a large quartz dish. In all about 800 cc. of acid was used in washing the glass. The filter with the glass was ignited gently in a platinum crucible and the residue was weighed. The glass, after ignition, showed no evidence of containing tin. The ash of the filter was only 0.11 mg.

In order to show that this method of determining the weight of the glass involved no gain or loss, in several experiments an unsealed bulb similar to those used in collecting the stannic chloride, after being weighed in a platinum crucible, was broken under an acid solution of stannic chloride, and the fragments were washed and collected upon a filter in exactly the same way as in an analysis. The filter was burned in the same crucible, and the change in weight, corrected for the ash of the filter, determined. As the following table shows, a very slight average loss is indicated, owing probably to solution of the glass in the acid. This loss is so small in comparison with the weights of material used, that no attempt has been made to apply a correction.

Excess in weight of crucible + bulb over counterpoise. G.	Excess in weight of crucible + glass over counterpoise. G.	Loss. G.
2.74104	2.74097	0.00007
2.78502	2.78494	0.00008
2.69270	2.69267	0.00003
2.75258	2.75257	0.00001
2.59472	2.59462	0.00010
	Average,	0.00006

The dilute washings in the quartz dish were evaporated with a small amount of sulfuric and about 10 cc. of conc. nitric acid, over an electric stove. In this way the hydrochloric acid was oxidized before the solution became sufficiently concentrated to introduce danger of losing stannic chloride by volatilization.

Before commencing the electrolysis the column of bulbs ground into the neck of the cell to catch spray was rinsed with constant boiling hydrochloric acid in order to prevent hydrolysis of any tin salt which might be projected into the bulbs. A current of 1 to 2 amperes was then passed through the cell, immersed in pure water, until chlorine ceased to be evolved. Usually this required 18 to 24 hours. At this point nearly all the tin had been deposited in the mercury cathode, but a considerable residual conductivity of the electrolyte was due to perchloric acid formed during the electrolysis. The column of bulbs was now rinsed into the cell and the aqueous contents of the cell were removed and placed in the quartz dish containing the evaporated rinsings of the glass of the bulb. The amalgam was rinsed a few times with water and the washings added to the contents of the dish. Then the solution in the dish was evaporated over an electric stove until all volatile material except the sulfuric acid and stannic sulfate had been expelled. This residue was dissolved in water, the solution was returned to the cell and electrolysis was continued for several hours.

Next the electrolyte was siphoned from a point near the surface of the mercury while the purest water saturated with electrolytic hydrogen was added at the top of the cell with the full potential of the battery, 16 volts, applied at the electrodes, care being taken not to break the electrical circuit. As soon as the reading of the ammeter in the circuit became zero, all the remaining aqueous liquid was removed and the amalgam was further rinsed, first with water, finally twice with alcohol. During the washing the cell was inclined and slowly rotated, so that liquid trapped between amalgam and cell wall might be removed. In order to prepare the cell for weighing, the outside was rinsed with water and wiped with a clean, lintless cloth. The special stopper was inserted and the cell was placed in a tubular vacuum desiccator containing fused sodium hydroxide, which was exhausted by means of a Geryk pump. Care was taken to reduce the pressure slowly at the end so as to avoid spurting of the amalgam. As soon as evaporation of the alcohol was complete, the cell was transferred to a second vacuum desiccator, which was immediately exhausted. After several hours the cell was weighed by substitution for a similar counterpoise containing mercury, which also was kept in an exhausted desiccator between weighings.

In order that the cell and mercury might be subjected to the same influences before and after use in an analysis, it originally was partly filled with dil. sulfuric acid, which was electrolyzed for from 2 to 12 hours with 1 ampere. It was then treated as described above, previous to the initial weighing.

The combined electrolyte and washings were evaporated nearly to dryness in a quartz dish over an electric stove, and transferred to a weighed platinum crucible. A small amount of nitric acid was added to insure complete oxidation of the tin to the stannic condition, and the solution was again evaporated, first on the electric stove, then over a flame, until the crucible was barely red for an instant.

In computing the correction for this residue the assumption is made that the tin was all in the form of stannic sulfate. But although tin was actually found to be present, it is probable that part of the residue consisted of sulfates of alkalies extracted from the glass of the cell. However, since the percentage of tin in stannic sulfate and the percentages of sodium and calcium oxides in the sulfates are not very different, 38, 43 and 41, respectively, and since the weight of the residue usually did not exceed 3 mg. and in only one case exceeded 4 mg., the uncertainty from this source cannot be large.

When the cell and the residue had both been weighed, the residue was dissolved in very dil. sulfuric acid[1] and returned to the cell, where it was electrolyzed for several hours, frequently overnight. The cell and electrolyte were then treated as before, and if a change in the total weight of tin was observed the process was repeated until the total remained constant within 0.1 mg. Occasionally only 2 electrolyses were necessary, but frequently a perceptible diminution in weight was observed in the second electrolysis. In this case usually the second and third weights agreed.

We are inclined to impute the slight loss to liquid trapped either under or in the amalgam. The fact that considerable solid amalgam always was present may account for this. In order to eliminate this error so far as possible, in the later analyses, where the quantity of stannic chloride was larger and the danger greater, the amalgam was liquefied by raising the temperature with a heavier current, and then cooling the cell before drawing off the electrolyte.

In the second series of fractions some of the bulbs were so large that it

---

[1] This process was omitted in Analyses 1, 3 and 5.

was necessary, on account of the violence of the reaction, to increase the volume of acid under which the bulb was broken. Here the original liquid, about 125 cc., was immediately electrolyzed in the cell. The residual electrolyte was taken out and preserved, and the first 125 cc. of washings was electrolyzed. Then the first and second electrolytes, together with the dilute washings, were combined, evaporated and treated as before.

In several analyses a slight black deposit was formed on the inner wall of the glass cell, insoluble in acid but soluble in chlorine. It was suspected that the deposit was platinum, dissolved from the anode by the chlorine evolved during electrolysis, but reduced by the stannous chloride. In one case the deposit was dissolved in aqua regia, great care being taken not to allow the solvent to touch either anode or cathode. When the solution had been evaporated to small bulk, a drop of cesium chloride gave an immediate yellow precipitate. It is obvious that the formation of this deposit involves no gain or loss in weight of the cell.

In Analyses 1 to 10, about 150 g. of mercury was used in each experiment; in Analyses 12, 13 and 15, about 200 g., and in Analyses 11, 14 and 16, 250 g.

Because of the large weight of the loaded cell, it could not safely be weighed on the No. 10 Troemmer balance used in weighing the bulbs and crucibles. Instead, in Analyses 1 to 5, a large Sartorius balance, sensitive to 0.2 mg., was employed; for all others a No. 6 Troemmer was secured, sensitive to 0.05 mg. with a load of 500 g. Weights were standardized by the Richards method of substitution. The cells and crucibles were weighed by substitution for counterpoises of very nearly the same shape, size and volume.

The amalgams were uniformly bright and lustrous. In order to show that oxidation is negligible, a bright lump of tin which had been fused in hydrogen on an alundum boat was dropped into a cell containing mercury and was weighed at intervals.

	G.
Weight of cell + amalgam (initial)	C + 6.34944
Weight of cell + amalgam after 2 days	C + 6.34943
Weight of cell + amalgam after 3 days	C + 6.34942

Further evidence on this point, as well as on the accuracy of the electrolytic process in general, was obtained as follows: A button of tin which had been fused in hydrogen on an alundum boat was placed in a platinum gauze basket and supported upon the anode of a cell which had been prepared for weighing. After the system had been weighed, 2 *N* hydrochloric acid was added, the tin was electrolytically transported from anode to cathode, and the hydrochloric acid decomposed by electrolysis. The amalgam was washed and dried, and the electrolyte was evaporated, as previously described. When the amalgam and residue had been weighed, the residue was dissolved in acid, returned to the cell and electrolysis repeated.

		I. G.	II. G.
Weight of tin		1.98341	7.19
Weight of cell		4.01336	
Weight of cell + tin		5.99677	9.43879
Weight of cell + amalgam	1	5.99654	9.43859
Weight of residue	1	0.00057	0.00039
Corrected weight of cell + amalgam	1	5.99676	9.43873
Change in weight	1	—0.00001	—0.00006
Weight of cell + amalgam	2	5.99653	9.43843
Weight of residue	2	0.00069	0.00075
Corrected weight of cell + amalgam	2	5.99679	9.43872
Change in weight	2	+0.00002	—0.00007

The close agreement of the weight of the factors with that of the products is a sufficiently rigid test of this method of determining tin. Furthermore, some time after the last 2 experiments had been completed, Mr. C. H. Wilson obtained additional evidence that the weight of the tin and mercury together is equal to the weight of the amalgam corrected for the weight of the residue, although in the case of zinc and cadmium a very slight excess in the weight of the products seems to be characteristic. This point will be considered in detail in a later paper.

In computing the vacuum corrections for tin amalgam, account was taken of the fact that a saturated solution of tin in mercury contains only 0.97% of tin at 15° and 1.21% at 25°, so that a large proportion of the tin obtained in the analyses was weighed in the form of solid amalgam. By extrapolating the results of Richards and Wilson[1] the density of a 1% liquid amalgam seems to lie between 13.460 and 13.475 at 20°. Using the average value, 13.468, the vacuum correction for 1 g. of tin dissolved in mercury to form a 1% amalgam is —0.006 mg. On the other hand, the correction for tin in the form of solid amalgam is somewhat larger and positive in sign.

Substance.	Temp. ° C.	Density.	Vacuum correction for 1 g. Sn. Mg.
Hg_2Sn	11.3	11.456[a]	+0.016
Hg Sn	14.2	10.369[a]	+0.017
Hg_2Sn	26	11.382[b]	+0.018
Hg Sn	26	10.345[b]	+0.018
		Average,	+0.017

[a] Matthiesen, *Pogg. Ann.*, **110**, 21 (1860).
[b] Kupffer, *ibid.*, **112**, 445 (1861).

---

[1] *Carnegie Inst. Pub.*, No. **118** (1909).

From the quantity of mercury used in each experiment and the weight of tin obtained, the vacuum corrections for the tin in the form of liquid and solid amalgams were computed in each case.

THE ATOMIC WEIGHT OF TIN.

Cl = 35.457.

No. of analysis.	Fraction.	Weight of $SnCl_4$ in vacuum. G.	Weight of tin in amalgam in vacuum. G.	Weight of residue. G.	Total weight of tin in vacuum. G.	Sn:4Cl.	Atomic weight of tin.
			Series I.				
1.....	7	13.5943	6.1927	0.0021	6.1935	0.83687	118.691
2.....	13	10.7469	4.8956	0.0024	4.8965	0.83695	118.703
3.....	11	12.2869	5.5959	0.0062	5.5983	0.83699	118.709
4.....	8	10.0897	4.5962	0.0022	4.5971	0.83696	118.705
5.....	9	11.4319	5.2069	0.0028	5.2080	0.83677	118.678
6.....	12	12.20889	5.56205	0.00212	5.56286	0.837020	118.713
7.....	14	11.54233	5.25760	0.00226	5.25846	0.836818	118.684
8.....	5	11.64269	5.30452	0.00120	5.30498	0.837050	118.717
			Series II.				
9....	7	15.04889	6.85605	0.00235	6.85695	0.837037	118.715
10....	8	18.36074	8.36361	0.00381	8.36507	0.836869	118.692
11....	9	21.58929	9.83561	0.00302	9.83676	0.836991	118.709
12....	10	17.22210	7.84665	0.00138	7.84718	0.837040	118.716
13....	4	17.29151	7.87737	0.00297	7.87850	0.836980	118.707
14....	3	16.23310	7.39587	0.00202	7.39664	0.837059	118.718
15....	11	15.70516	7.15512	0.00201	7.15589	0.837018	118.713
16....	2	15.65437	7.13141	0.00148	7.13198	0.836852	118.689
Total.....		230.6487			105.0893	0.836969	118.706
			Average of Series I............			0.836929	118.700
			Average of Series II...........			0.836955	118.707
			Average of Series I and II.....				118.703

In Analysis 8 amalgam was lost through spattering after the first weight had been obtained. This weight is somewhat uncertain, since the appearance of the amalgam was not as lustrous as usual. In Analysis 10 a similar loss of mercury was suspected in the third electrolysis, so that although the result of the second electrolysis was considerably less than that of the first, the second weight is the one recorded. In Analysis 11 five electrolyses in all were completed, the extreme weights differing by 0.45 mg., the last 3 by 0.19 mg. The fourth weight, which was the lowest, is given in the table.

If the results for the atomic weight of tin are arranged in the order in which the fractions were distilled, only irregular variations are apparent. Hence there can be little question that the different portions were essentially identical in composition.

The final average, Sn = 118.703 (Cl = 35.457) is in very close agreement with the one obtained by Briscoe by comparing tin tetrachloride with silver, 118.698. The percentage of tin in stannic chloride found by us is 45.562, while Briscoe found 54.439% of chlorine. The sum is 100.001%, a highly satisfactory and convincing outcome. The more recent experiments of Brauner and Krepelka also are in entire accord with the above result.

It is a great pleasure to express our gratitude to the Carnegie Institution of Washington and to the Elizabeth Thompson Science Fund for very generous assistance in providing indispensable apparatus.

CAMBRIDGE, MASS.

[CONTRIBUTION FROM THE CHEMICAL INSTITUTE OF BOHEMIAN CHARLES UNIVERSITY OF PRAGUE.]

A REVISION OF THE ATOMIC WEIGHT OF TIN.[1] I.

BY BOHUSLAV BRAUNER AND HENRY KREPELKA.

Received February 13, 1920.

In his critical discussion on atomic weight determinations,[2] Brauner has given the following summary of all the earlier atomic weight determinations of tin:

	Ratio.	At. wt.
Gay-Lussac. 1811......	$Sn : SnO_2$	117.65
Berzelius. 1812..........	$Sn : SnO_2$	117.65–118.08
Mulder. 1849...........	$Sn : SnO_2$	117.00–116.30
Vlaanderen. 1858........	$Sn : SnO_2$	118.16
Dumas. 1859...........	*a.* $Sn : SnO_2$	118.06
	b. $SnCl_4 : 4\ Ag$	117.98
V. d. Plaats. 1885........	*a.* $Sn : SnO_2$ (oxidation)	118.09
	b. $SnO_2 : Sn$ (reduction)	118.07
Bongartz and Classen. 1888	*a.* $Sn : SnO_2$	119.34
	b. $(NH_4)_2SnCl_6 : Sn$	119.09*
	c. $K_2SnCl_6 : Sn$	119.07*
	d. $SnBr_4 : Sn$	118.97*
	e. $Sn(SnS_2) : BaSO_4$	119.05*
	The Mean of starred values......	119.05

While our work was being carried on, Briscoe[3] and Baxter and Starkweather[4] published the values 118.69 ($SnCl_4 : 4Ag$) and 118.703 ($Sn : 4Cl$), respectively. On account of the embargo placed by Austria on English publications during the war the present authors were entirely ignorant of both above-mentioned researches until some time after the armistice was signed.

Until the year 1888 the atomic weight of tin had been taken as 118;

---

[1] Presented to the Bohemian Academy of Sciences, Prague, 1917.

[2] Abegg's "Handbuch der anorg. Chemie," Group 4, 529.

[3] *J. Chem. Soc.*, 107, 76 (1915).

[4] *Proc. Nat. Acad. Sci.*, 2, 718 (1916).