

THE JOURNAL  
OF THE  
American Chemical Society

with which has been incorporated the

American Chemical Journal  
(Founded by Ira Remsen)

---

TWENTY-FIFTH ANNUAL REPORT OF COMMITTEE ON  
ATOMIC WEIGHTS.

DETERMINATIONS PUBLISHED DURING 1917.

BY GREGORY PAUL BAXTER.

Received May 18, 1918.

Only in the case of zirconium has evidence been presented during the past year which seems to demand an important alteration in the previously accepted atomic weight value.

**Hydrogen.**—Guye<sup>1</sup> discusses critically the results of Burt and Edgar upon the combining ratios by volume of hydrogen and oxygen, and the atomic weight of hydrogen in general, advocating finally the value 1.0077.

**Helium.**—Using a quartz microbalance, Taylor<sup>2</sup> determined the density of helium by comparison with both hydrogen and oxygen. The pressures in millimeters calculated to 0° at which equilibrium of the balance was reached were as below.

Using the densities 1.42900 for oxygen and 0.089873 for hydrogen, that of helium is calculated to be 0.17850 and 0.17848. By the method of limiting densities, the atomic weight of helium is found to be 4.0008. In the latter calculation, the coefficients of deviation from Boyle's law are assumed to be 0.00097, — 0.00077 and 0.00000 for oxygen, hydrogen, and helium.

<sup>1</sup> *J. chim. phys.*, **15**, 208 (1917).

<sup>2</sup> *Phys. Rev.*, **10**, 653 (1917).

Oxygen.	Hydrogen.	Helium.
101.91	1620.2	815.54
101.89	1620.0	815.70
101.82	1619.5	815.58
101.86	1620.5	815.83
101.91	1620.1	815.77
101.89	1619.6	815.45
101.84	1619.5	815.68
101.96	1619.2	915.65
101.86	1619.9	815.49
101.90	-----	815.61
101.86	Av., 1619.82	815.57
101.84		815.65
101.88		-----
101.84		Av., 815.62
101.93		
101.83		
-----		

Av., 101.88

**Carbon and Sulfur.**—Moles<sup>1</sup> points out that the values for carbon and sulfur are calculated by Richards and Hoover from the molecular weights of sodium carbonate and sulfate, respectively, with the use of the atomic weight 22.995 for sodium, whereas according to the work of Richards and Wells the number 22.997 should have been used ( $A_g = 107.880$ ). If the change is made, Richards' and Hoover's values for carbon and sulfur become 12.001 and 32.056, respectively.

Guye<sup>2</sup> criticizes the International Committee upon Atomic Weights for accepting the values of carbon and sulfur as calculated by Richards and Hoover.

**Bromine.**—Two investigations upon the weight of the normal liter of hydrobromic acid gas have been published. Reiman<sup>3</sup> prepared the gas (1) by synthesis from the elements and, after solution in water and distillation of the solution, regeneration of the gas by dropping the solution upon phosphorus pentoxide; and (2) by synthesis from the elements; and (3) by the action of phosphoric acid upon potassium bromide. In every case the product was fractionally distilled at low temperatures. The gas was weighed in globes, filled at 0° and about one atmosphere.

In two series of experiments the weight of a liter of gas at 0° and 506.67 mm. was found to be 2.42201 grams and that at 0° and 253.33 mm. to be 1.20726 grams. By a method of limiting densities, using oxygen as the standard, the molecular weight of hydrobromic acid is calculated to be 80.932, and the atomic weight of bromine, 79.924.

<sup>1</sup> *J. chim. phys.*, 15, 51 (1917).

<sup>2</sup> *Ibid.*, 15, 60 (1917).

<sup>3</sup> *Ibid.*, 15, 293 (1917); *Compt. rend.*, 164, 44, 180.

Method of preparation.	Globe I. 615.18 cc.	Globe II. 565.19 cc.	Globe III. 479.46 cc.
I.....	3.64400	3.64391	3.64290
I.....	3.64506	3.64325	....
I.....	3.64502	3.64331	....
I.....	3.64694	3.64525	....
I.....	3.64453	3.64652	3.64623
I.....	3.64211	3.64425	3.64416
I.....	3.64244	3.64483	3.63344
I.....	3.64199	3.64488	3.64299
2.....	3.64424	3.64483	3.64449
2.....	3.64420	3.64379	3.64460
3.....	3.64417	3.64366	....
3.....	3.64361	3.64435	....
Av.....	3.64403	3.64440	3.64411
Average of all determinations.....			3.644192
Average of Method 1.....			3.64427
Average of Method 2.....			3.64436
Average of Method 3.....			3.64395

Murray<sup>1</sup> prepared hydrobromic acid (1) by hydrolysis of aluminum bromide, and (2) by the action of phosphoric acid upon potassium bromide, the final product being fractionally distilled at low temperatures. The weight of the normal liter was determined in the same way as by Reiman.

Method of preparation.	Globe 2. 596.59 cc.	Globe 3. 455.73 cc.	Globe 5. 735.58 cc.	Globe 12. 610.25 cc.
I.....	3.6497	3.6410	....	3.6422
I.....	3.6415	3.6401	....	3.6453
I.....	3.6402	3.6482	....	3.6463
I.....	3.6408	3.6439	....	3.6494
I.....	3.6467	....	....	3.6430
I.....	3.6432	3.6422	....	3.6451
2.....	3.6438	3.6421	3.6414	....
Av.....	3.64370	3.64291	3.6414	3.64522
Average of all determinations.....				3.643805

As gas prepared by Method 1 is considered purer than that prepared by Method 2, the average of determinations made with the former material is preferred, 3.64405 grams.

This value is somewhat lower than the one found by Reiman, while Moles<sup>2</sup> obtained 3.64442 grams. The average of the three series leads to the value for the atomic weight of bromine given above. In view of the fact that the calculation by the method of limiting densities involves extrapolation over a range of one-third of an atmosphere, it is reassuring that the density method gives a result in as close agreement as it does with the value obtained by comparison with silver.

<sup>1</sup> *J. chim. phys.*, 15, 334 (1917); *Compt. rend.*, 164, 182.

<sup>2</sup> See 24th Annual Report.

**Zirconium.**—Venable and Bell<sup>1</sup> purified zirconium oxychloride by crystallization from hydrochloric acid, and then prepared the tetrachloride from the oxide by heating in a current of chlorine saturated with carbon tetrachloride. The product was finally distilled into glass bulbs which were sealed and weighed. Preliminary experiments in which the tetrachloride was hydrolyzed to the dioxide in a quartz flask yielded results slightly above 92 for the atomic weight of zirconium. In a final series of experiments the tetrachloride was dissolved and compared with silver nephelometrically, the fragments of the glass bulb being determined separately. The weights are corrected to vacuum. Ag = 107.880; Cl = 35.458.

Wt. of ZrCl <sub>4</sub> .	Wt. of Ag.	At. wt. Zr.
5.55372	10.26178	91.71
3.78445	6.99198	91.73
2.97546	5.49645	91.77
7.22480	13.34222	91.84
6.35532	11.73094	91.95
6.78989	12.64259	91.77
5.65352	10.44718	91.68
2.76975	5.11980	91.62
4.30337	7.95180	91.70
4.31934	7.98115	91.70
2.42721	4.48516	91.69
4.80724	8.87607	91.88
2.35598	4.35052	91.85
Average of all.....		91.76

This value is over a unit higher than that given by the International Committee upon Atomic Weights, 90.6. Further work is promised by the authors.

**Tellurium.**—Stähler and Tesch<sup>2</sup> purified tellurium by distillation in vacuum, then converted the element to tetrachloride which was fractionated in a current of chlorine. After reduction, the metal was again redistilled in vacuum. Weighed quantities were converted to dioxide by solution in nitric acid, evaporation, and final fusion of the residue. An average value of 127.51 for the atomic weight of tellurium was obtained.

**Samarium.**—Stewart and James<sup>3</sup> prepared pure samarium material by prolonged systematic fractional crystallization of the double magnesium nitrate, until the least soluble fraction was free from neodymium and the most soluble from europium. Ten consecutive fractions were combined in four as indicated in the table, and converted to chloride, and the chloride was dehydrated and fused in an atmosphere of dry hydrochloric acid

<sup>1</sup> THIS JOURNAL, 39, 1598 (1917).

<sup>2</sup> Z. anorg. allgem. Chem., 98, 1 (1916); Chem. Abs., 11, 551 (1917).

<sup>3</sup> THIS JOURNAL, 39, 2605 (1917).

gas. After being weighed the chloride was compared with pure silver, by precipitation in very dilute solution and testing the residual solution in a nephelometer for excess of silver or chloride. Vacuum corrections are applied.  $\text{Ag} = 107.880$ ;  $\text{Cl} = 35.457$ .

Fractions.	Wt. of $\text{SaCl}_2$ .	Wt. of Ag.	At. wt. of Sa.
Preliminary Series.			
6 + 7	4.05609	5.10860	150.59
6 + 7	3.00065	3.78044	150.51
6 + 7	5.28891	6.66424	150.48
8 + 9 + 10	3.75782	4.73748	150.34
8 + 9 + 10	4.76695	6.00873	150.39
11 + 12 + 13	2.97764	3.75234	150.45
11 + 12 + 13	3.82407	4.81761	150.53
14 + 15	4.47775	5.64365	150.41
14 + 15	3.33781	4.20712	150.40
Average.....			150.45
Final Series.			
6 + 7	4.65174	5.86128	150.48
6 + 7	5.13023	6.46577	150.42
8 + 9 + 10	4.52826	5.70727	150.41
8 + 9 + 10	6.65425	8.38628	150.43
11 + 12 + 13	6.02908	7.59669	150.49
11 + 12 + 13	5.09858	6.42520	150.45
14 + 15	4.68220	5.90111	150.42
14 + 15	4.32763	5.45551	150.36
Average.....			150.43
Average of both series.....			150.44

This result supports the value selected by the International Committee on Atomic Weights, 150.4.

Bovard and Hulett<sup>1</sup> have obtained an agreement between cathode gain and anode loss in the silver voltameter of 0.007% and find the value of the inclusion in the standard platinum cup voltameter to be 0.0067% of the deposit.

Oblata<sup>2</sup> also has studied the silver voltameter, and finds the value of the inclusion to be 0.0079% with the Smith type of cell.

de Gramont<sup>3</sup> emphasizes the desirability of spectroscopic control of pure materials, especially those used for determinations of atomic weights.

Guye<sup>4</sup> and Renard<sup>5</sup> criticize premature changes made in the past in the International table of atomic weights.

<sup>1</sup> THIS JOURNAL, 39, 1077 (1917).

<sup>2</sup> Proc. Tokyo Math. Phys. Soc., [2] 8, 437 (1916); 9, 129 (1917).

<sup>3</sup> J. chim. phys., 14, 336 (1916).

<sup>4</sup> Ibid., 14, 449 (1916).

<sup>5</sup> Ibid., 15, 541 (1917).

Guye and Moles<sup>1</sup> discuss surface condensation of moisture and other gases, and its effect upon the weights of substances weighed in the air, while Guye<sup>2</sup> emphasizes the bearing of this effect and of occluded gases upon the atomic weights of silver and other elements whose atomic weights have been found by reference to silver.

Moles<sup>3</sup> discusses critically the determinations of atomic weights published in 1916.

Sears<sup>4</sup> finds tantalum pentachloride unsuited for the very exact quantitative manipulation required for atomic weight experimentation.

CAMBRIDGE, MASS.

[CONTRIBUTION FROM THE SEVERANCE CHEMICAL LABORATORY OF OBERLIN COLLEGE.]

## THE PEPTIZATION OF FERRIC ARSENATE AND PHOSPHATE AND THE FORMATION OF THEIR GELS.

BY HARRY N. HOLMES AND ROSSLEENE ARNOLD.

Received December 22, 1917.

The formation of colloids by peptization and the development of gel structure are two phenomena in need of further elucidation. Previous work with R. E. Rindfusz<sup>5</sup> has shown that ferric arsenate and phosphate have properties which make them unusually suitable for such a study.

In the present paper two main topics are presented: First, peptization by sodium hydroxide and by ferric chloride as influenced by concentration changes, rate of adsorption of the peptizing agent and the presence of certain salts and sugars; second, variations in the structure and time of formation of gels from dialysis of the various ferric chloride peptization products.

### I. Peptization by Sodium Hydroxide.

In our earlier work we reported that it was necessary to use very dilute base to prevent coagulation of the colloid formed, but later we found that this was true only of thoroughly washed precipitates of ferric arsenate or phosphate. Unwashed precipitates are stabilized by any soluble arsenates or phosphates that may be present. Therefore, much more concentrated hydroxide may be used as peptizing agent, and added more rapidly and in greater excess. In fact, the total amount of base required is greater. We found *N* sodium hydroxide suitable for addition to the wet precipitate. Colloidal arsenates or phosphates that otherwise coagulate in a short time are stabilized by dilution after peptization. Potassium hydroxide shows a greater peptizing power than sodium hydroxide, a fact of great

<sup>1</sup> *J. chim. phys.*, **15**, 360, 405 (1917).

<sup>2</sup> *Ibid.*, **15**, 549 (1917).

<sup>3</sup> *Ibid.*, **15**, 433 (1917).

<sup>4</sup> *THIS JOURNAL*, **39**, 1582 (1917).

<sup>5</sup> *Ibid.*, **38**, 1970 (1916).