[CONTRIBUTION FROM THE WOLCOTT GIBBS MEMORIAL LABORATORY, HARVARD UNIVERSITY]

THE ATOMIC WEIGHT OF COPPER FROM THE LAKE SUPERIOR REGION AND FROM CHILE[1]

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RECEIVED SEPTEMBER 18, 1928 PUBLISHED FEBRUARY 5, 1929

Examination of the atomic weights of elements from different geographical and geological sources is of importance because of the bearing on the genesis of the elements. None of the comparisons,[2] except for products of radioactive disintegration and possibly boron, has shown any segregation of isotopes. The results here presented confirm this conclusion for the case of copper. Furthermore, they contribute to the determination of the atomic weight of this element by the analysis of yet another compound. Most of the work on the atomic weight of copper for thirty years was from the point of view of the electrochemical equivalent, which Clarke in his excellent critical review[3] shows to be inferior to the chemical method.[4]

Copper was chosen for investigation because it could be obtained from different known geological ages and from regions widely separated, because it was known to have at least two isotopes and because its atomic

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[1] *Note by the Editor.*—This research was completed in 1923 under the direction of Professor Theodore W. Richards but the present account of it was prepared after the death of Professor Richards, by Dr. Lawrence P. Hall.

[2] For example. Richards, *Proc. Am. Acad. Arts Sci.*, **23**, 178 (1887); Richards and Cushman, *ibid.*, **33**, 102 (1897); Richards, THIS JOURNAL, **24**, 374 (1902); Richards and Wells, *Carnegie Inst. Pub.*, **28**, 13, 17 (1905); Baxter and Thorvaldson, THIS JOURNAL, **33**, 337 (1911); Baxter and Hoover, *ibid.*, **34**, 1657 (1912); Baxter and Grover, *ibid.*, **37**, 1027 (1915); Curie, *Compt. rend.*, **172**, 1025 (1921); Gleditsch and Samdahl, *ibid.*, **174**, 746 (1922); Brönsted and Hevesy, *Z. anorg. allgem. Chem.*, **124**, 22 (1922); Dorenfeld, THIS JOURNAL, **45**, 1577 (1923); Gleditsch, *J. chim. phys.*, **21**, 456 (1924); Harkins and Stone, *Proc. Nat. Acad. Sci.*, 11, 643 (1925); Briscoe and Robinson, *Nature*, 117, 381 (1926); E. Gleditsch and I. Gleditsch, *J. chim. phys.*, **24**, 238 (1927). Muzaffar [THIS JOURNAL, **45**, 2009 (1923)], in a preliminary paper, reported apparent differences in the atomic weight of antimony, depending upon the source. However, this work has been disproved by Krishnaswami [*J. Chem. Soc.*, 2534 (1927)]. Briscoe and Robinson, *J. Chem. Soc.*, **127**, 696 (1925), Briscoe, Robinson and Stephenson, *ibid.*, 70 (1926), Briscoe, Robinson and Smith, *ibid.*, 282 (1927), report variations in the atomic weight of boron from different sources. For examples of similarity of elements from terrestrial and meteoric origin, see Baxter and Parsons, THIS JOURNAL, **43**, 507 (1921); Baxter and Hilton, *ibid.*, **45**, 694 (1923); Baxter and Dorcas, *ibid.*, **46**, 357 (1924).

[3] F. W. Clarke, "The Constants of Nature," Part V, A Recalculation of the Atomic Weights, 4th ed., Washington, 1920, p. 125.

[4] Since the completion of the research here recorded, Ruer and Bode [*Z. anorg. allgem. Chem.*, **137**, 101 (1924)] employed the copper oxide method and found a value 63.546, which they have defended against the criticisms of the German Atomic Weight Commission [Ruer and Bode, *Ber.*, **58A**, 852 (1925); *ibid.*, **59B**, 1698 (1926)].

weight needed confirmation. One sample was obtained from the Lake Superior region and another from the Chile ore deposits.

The former, from the Calumet and Hecla Mines, contained equal parts from the upper and lower lodes. There is reason to believe that this deposit was formed not later than the Cambrian.[5] The deposits at Chuquicamata, Chile, from which the second sample came, are said to consist of a series of lodes in granitic rock, intrusive in Jurassic strata.[6]

Methods previously used for the determination of the atomic weight of copper were based on analyses of CuO, $CuSO_4$ or $CuBr_2$, and by comparison with silver, directly as well as electrolytically. For the purpose of confirmation of the atomic weight it was deemed advisable to analyze some other compound by means primarily chemical.

Tests were conducted on both cuprous and cupric chlorides to determine their suitability for analysis in the anhydrous condition. Samples of each chloride were fused in an atmosphere of chlorine in a bottling apparatus and nitrogen was used to displace the chlorine. Both cupric and cuprous chloride yielded bluish black crystals, which were partially soluble in water, giving a blue solution and a white precipitate. Under available conditions the equilibrium between cuprous chloride, cupric chloride and chlorine did not give a suitable salt.[7]

A simpler method was adopted based upon work done with cupric bromide.[8] This method, in brief, consisted in determining the copper electrolytically and the chloride content by comparison with silver in a solution of recrystallized hydrated cupric chloride.

Purification of Materials

Water, nitric acid and sulfuric acid were carefully purified and before use all were proved to be free from chloride by nephelometric tests. All alcohol and hydrochloric acid used were carefully distilled. All other reagents were also purified before use.

Pure silver was prepared from residues of silver bromide resulting from previous atomic weight work. The bromide was reduced with pure zinc, the resulting silver being washed with sulfuric acid and water before solution in nitric acid. After three crystallizations of the nitrate the silver was reduced with freshly prepared ammonium formate. The silver was thoroughly washed, fused into buttons and electrolyzed, and the crystals were again fused on lime in a hydrogen atmosphere. The resulting buttons were etched with nitric acid, washed twenty times and dried in a vacuum

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[5] *Cf.* Lindgren, "Mineral Deposits," McGraw-Hill Book Co., New York, 1928, p. 509.

[6] Ref. 5, p. 958.

[7] Rose [*Pogg. Ann.*, **38**, 121 (1836)] had noticed that when cupric chloride is heated free chlorine is found in the vapors and cuprous chloride results.

[8] Richards, *Proc. Amer. Acad. Arts Sci.*, **25**, 195 (1890).

at 400°. A few of the buttons were electrolyzed again to provide small crystals used in adjusting weights of silver required in analysis. The silver was kept in a desiccator over fused potassium hydroxide.

Purification of cupric chloride was accomplished by repeated crystallization as $CuCl_2 \cdot 2H_2O$. The copper from Calumet and Hecla, Sample A, was a block of pure copper weighing about 1500 g. The center of this block was turned out on a lathe. For the first series of crystallizations 500 g. of shavings was taken; for the second series (A II) 300 g. A third series (A III) was obtained by recrystallization of the mother liquor from the first two series.

The copper turnings, washed with alcohol to remove grease, were dissolved in hydrochloric acid to which had been added a small amount of nitric acid. The cupric chloride was recrystallized four times in pyrex dishes with evaporation over the steam-bath and with centrifugal drainage. Any insoluble matter was removed by filtration after the first crystallization. Tests for iron were negative after the first crystallization. Two more crystallizations of the cupric chloride were performed in quartz dishes, evaporation in these cases taking place in vacuum desiccators over sulfuric acid. The solution was filtered after the fifth crystallization. Care was taken not to reach a high vacuum which would cause bumping and splashing. As an emergency precaution to prevent formation of hydrogen chloride by splashing solution into the concentrated sulfuric acid, watch glasses were placed beneath the quartz dishes. Since usually the crystals, forming on the surface, made a crust, the desiccator was opened from time to time and the crystals were pushed down with a glass rake. In this way was avoided the appearance of any brownish color due to the presence of anhydrous cupric chloride.

At each stage of crystallization tests were made for free hydrochloric acid. Such tests were at the best comparative. However, since each sample was purified until the same acidity was reached, the atomic weights of the different samples should be entirely comparable.

The method used in these tests was similar to that used in the analysis of cupric bromide.[8] One cubic centimeter of a concentrated cupric chloride solution was diluted to 10 cc. and one drop of methyl orange solution was added. The resulting color was purple if the solution was acidic and yellowish-green if basic. The turning point in color was marked. In order to obtain a standard for comparison the change in acidity was carefully followed in a series of nine crystallizations. These experiments showed that during the first three crystallizations all excess acid was eliminated. During the next five crystallizations the shade of purple was almost constant. The standard chosen was the purple thus produced. After the ninth crystallization the turn in color was observed. The standard purple color was restored by the addition of less than four milligrams of hydrochloric acid to a solution containing 60 g. of cupric chloride. In the crystallization of Sample A there was a slight loss of color during the first three crystallizations, but during the next three the color remained practically constant.

The Chile copper, Sample B, was received in the form of small ingots of about 200 g. each. An ingot and a half were taken for the first series of crystallizations (B I and B II). The same method of purification was followed for Sample B as for Sample A except that three crystallizations over sulfuric acid were necessary to give the normal acid test. A second set of crystallizations was performed with the mother liquors from the last four crystallizations of the first series (B III).

Analysis of Solutions

About 40 g. of the cupric chloride crystals was dissolved in 1100 cc. of best water in a "Non-Sol" bottle. Samples of 100 cc. each were withdrawn with a pipet using a suction pump and these samples were placed in weighed Florence flasks, which were provided with stoppers consisting of short sections of glass tubing, fitting into the neck of the flasks and supported by a small section of rubber tubing resting on the lip of the flask. The solutions were then weighed to one milligram. In half the samples the copper content was determined and in the other half the chlorine was precipitated with silver. In certain cases it was found necessary to apply a correction for the formation of atacamite. This correction will be discussed below in detail.

Determination of Copper.—The weighed sample was carefully transferred from the flask to a quartz evaporating dish and the flask rinsed many times. The solution was then evaporated on the steam-bath to dryness. The residue was taken up in a small amount of water and sulfuric acid added to convert the chloride to sulfate. The solution was again evaporated, treated with water, and evaporated to make certain of expelling all of the hydrochloric acid. The copper sulfate was dissolved in 10 cc. of water and transferred to a scrupulously clean, weighed platinum crucible. The evaporating dish was washed repeatedly with small portions of water and the washings were stirred into the main solution. The crucible was then made the cathode in an electrolytic stand. All the usual precautions were taken in the electrolysis. The washings were collected and tested for copper. The crucible was dried at 85° before the first weighing and at 120° before reweighing. In every analysis the deposit of copper was smooth, firm, even, coherent and of bright, uniform color. No flakes and no crystalline character were observed.

The washings were evaporated to about 10 cc., made alkaline with ammonium hydroxide, evaporated and heated gently to drive off ammonium sulfate. The residue was taken up in 5 cc. of water and tested for copper with potassium ferrocyanide. In every case a faint coloration resulted. The amount of copper which would cause this coloration was ascertained by comparison with known amounts of copper. The amount of undeposited copper was quite uniform in the washings from different analyses and amounted to 0.00006 g. This correction was applied to all analyses.

Determination of Chlorine.—A weighed sample of cupric chloride solution was transferred to a 3-liter precipitation flask. With fifteen or more rinsings of the Florence flask the solution was diluted to 500 cc. From the previous copper determination the equivalent amount of silver was calculated (assuming 63.565 as the atomic weight of copper) and weighed out with proper allowance for weight corrections and for the buoyancy of air. The silver was dissolved in nitric acid in a flask with a bulb tower and the resulting solution diluted to 500 cc. The silver solution was then added to the cupric chloride solution in a dark room, in which all further work with the solution was conducted. By the usual nephelometric tests over a period of several months the exact end-point was measured.

Correction for Atacamite

After solution A I had stood for a few days a slight precipitate was observed at the bottom of the container. Upon examination this precipitate was found to consist of small clusters of fine, light-green rhombic crystals which had grown from the solution. Their form and color indicated that they might be cupric oxychloride or atacamite, sometimes written $CuCl_2 \cdot 3CuO \cdot 4H_2O$. In the early work on cupric bromide a similar precipitate had been found and made the subject of a short paper.[9]

Solution A I was filtered and kept for six months. No further growth of crystals was observed. The second solution A II was carefully watched. Seven days after the recrystallized cupric chloride had been dissolved crystals were observed. This solution was left for four months before being filtered. The crystals were then analyzed and the solution was used for atomic weight determinations. In the meantime solution A III was prepared, but with the addition of one cubic centimeter of nitric acid (40%) to a concentrated solution of the crystals before dilution. As no crystals grew in solution A III in forty days, the solution was analyzed.

Solution B I yielded crystals in five days. After fifteen days the crystals were collected on a Gooch-Munroe crucible. This solution stood for four months without any more crystals appearing. Solution B II was prepared from the same crystals as solution B I. However, in this case one cubic centimeter of nitric acid was added to the first concentrated solution before final dilution. In the case of solution B III crystals were found on

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[9] Richards, *Proc. Am. Acad. Arts Sci.*, **25**, 215 (1890).

the fourth day. This solution was filtered after twenty-one days, the crystals were investigated and the solution was analyzed.

Apparently the growth of crystals was complete in a week. It is significant that the weights of the crystals were practically the same in solutions B III and A II.

The precipitates from these two solutions were washed with distilled water and dried at 120° to constant weight. They were then dissolved in nitric acid and the chlorine was determined as silver chloride. Excess silver was removed with hydrobromic acid, and after conversion to the sulfate the copper was determined electrolytically. The results of the analyses are given in Table I.

TABLE I

ANALYSIS OF PRECIPITATES

Soln.	Precipitate, g.	AgCl, g.	Chlorine, g.	Copper, g.	Copper, %	Chlorine, %
A II	0.02262	0.01572	0.00389	0.01294	57.2	17.2
B III	.02277	.01540	.00381	.01305	57.3	16.7

The copper content of atacamite should be 57% according to the formula usually assigned it. The chlorine content was likewise slightly higher than theoretical, but since the ratio of chlorine to copper was similar to that for atacamite, it was concluded that the crystals consisted of this compound.

It was necessary to investigate the method of formation of this precipitate, because in it the weight of chlorine was much smaller on the basis of the copper present than in pure cupric chloride. If this difference of chlorine had been retained in solution in the formation of the atacamite, there would be an excess of chlorine in the solutions from which the precipitate had been removed. On the other hand, if in some way the chlorine had escaped from solution, no correction for excess chlorine would be necessary in the case of removal of the precipitate. But in the cases where the atacamite was held in solution with nitric acid, a loss of chlorine would mean an excess of copper or a deficiency of chlorine, and a correction need be added to the chlorine found in the analysis of the solution. For example, in the atacamite from solution A II the weight of chlorine found was 0.00389 g., whereas the weight of chlorine required for an equivalent amount of $CuCl_2$ is 0.01443 g. This difference of 0.01054 g. of chlorine corresponds in the chloride analysis to 0.03207 g. of silver for the whole solution or 0.00291 g. of silver for each 100-cc. sample analyzed. Similarly for solution B III the difference in silver would be 0.00297 g. for each 100-cc. sample.

It became important then, to find out whether the chlorine left the solution or not. This problem was attacked in several ways. The acidity of the solutions was carefully followed before and after precipitation of the oxychloride. Since acidity was no greater after precipitation of atacamite than before, it appeared that the atacamite was not formed by hydrolysis.

If cuprous chloride had been present in the crystals of cupric chloride, oxidation of the cuprous chloride to cupric would have formed the oxychloride.[10] Both cuprous chloride and atacamite are soluble in concentrated cupric chloride. Growth of crystals from solution would require some time. On this conjecture, to test for loss of chlorine, a solution of purest cupric chloride was placed in a vacuum desiccator for evaporation over chloride-free sulfuric acid. Ten cubic centimeters of saturated potassium hydroxide solution was placed within the desiccator. After one month the potassium hydroxide was tested. Compared with a blank test with a similar unexposed solution of potassium hydroxide, an increase in chloride content of the hydroxide was found.

A second test was conducted in a similar manner except that the cupric chloride solution was treated with a little potassium hydroxide. This solution could not have contained excess acid or chlorine. Again the potassium hydroxide gained chlorine.

In order to determine whether this loss during crystallization was chlorine or hydrochloric acid, pure cupric chloride crystals, moistened with water, were placed in a desiccator over chloride-free sulfuric acid with all the precautions previously employed. A strip of starch-iodide paper was suspended within the desiccator. Although the strip was moistened when introduced it quickly became dry. In two months the paper was distinctly purple, whereas in a blank test the paper did not change in color.

Since this test with dry starch-iodide paper was not entirely convincing, a more delicate method was tried. In accordance with a method proposed by Ganassini[11] a strip of chlorine-free filter paper was moistened with a 10% solution of potassium bromide, dried, then dipped into a 0.04% ammoniacal alcoholic solution of fluorescein and again dried. When this paper was left in a vacuum desiccator with moist cupric chloride crystals, it gradually became pink, due to conversion of fluorescein into eosin. The first coloration was observed in seven days. The color developed for about one month, after which no increase in intensity was observed. Blank tests with fluorescein-bromide paper showed no change in color.

It appeared, then, that chlorine was lost in the crystallization of the cupric chloride, undoubtedly from formation of cuprous chloride. The atacamite resulted, not from hydrolysis, but most probably from oxidation of cuprous chloride and crystallization of this rather insoluble complex on dilution and long standing. It seems probable from Henry's Law that all of the free chlorine left the solution. The chlorine lost was undoubtedly represented by the precipitate on account of the insolubility of the atacamite. Agreement of analyses with and without atacamite in

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[10] *Cf.* Gröger, *Z. anorg. Chem.*, **28**, 154 (1901).

[11] Ganassini, *Chem. Zentr.*, I, 1172 (1904).

the solution further corroborates the hypothesis. The experience with cupric chloride is quite comparable with that with the bromide.[12]

In the analyses, therefore, a correction was added of silver equivalent to the chlorine lost in the cases of solutions A III and B II where the precipitate was dissolved in nitric acid. The correction was based upon the analysis of atacamite from the other solutions and could be legitimately applied because of the similarity of treatment accorded the different series of crystallizations, because all samples gave identical tests for acidity and because the weights of precipitates in the other solution were similar. No correction was applied for solutions A II and B III where the precipitate was removed from the solution.

In Tables II to V are recorded the results of all the successful analyses with the exception of several preliminary ones performed only for practice. Solutions A I and B I were used in the investigation of atacamite.

All values given have been corrected to vacuum. In calculating the necessary corrections the following densities were used: weights, 8.4; $CuCl_2$ solutions, 1.02; silver, 10.49. The atomic weight of copper is referred through chlorine directly to the atomic weight of silver, which was taken as 107.880.

The atomic weights of the samples of copper from Calumet and Hecla and from Chile are identical within the limit of accuracy of the present methods. Neither the correction for atacamite nor the question of acidity influences this comparison. It is a striking case of identity of isotopic constitution of a chemical element taken from sources differing greatly in geological age and geographical distribution.

TABLE II

ANALYSIS OF CUPRIC CHLORIDE FROM LAKE SUPERIOR COPPER (SOLUTION A II)

Copper Analyses

Sample	Corr. wt. of sample, g.	Corr. wt. of copper found, g.	Copper correction	Corr. copper per 100 g. of soln., g.
4	101.833	1.20672	+0.00006	1.18505
5	101.821	1.20660	+ .00006	1.18508
6	101.879	1.20730	+ .00006	1.18509
			Average	1.18507

Chlorine Analyses

Sample	Corr. wt. of sample, g.	Corr. silver weighed out, g.	Nephelometer correction	Corr. silver equiv. for sample, g.	Corr. silver equiv. per 100 g. of soln., g.
1	101.740	4.09307	+0.00010	4.09317	4.02317
2	101.836	4.09737	− .00079	4.09658	4.02272
3	101.788	4.09475	+ .00026	4.09501	4.02308
				Average	4.02299

From 4.02299:1.18507 = 215.760:X, X = 63.557, the atomic weight of copper.

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[12] Ref. 8, p. 200.

TABLE III

ANALYSIS OF CUPRIC CHLORIDE FROM LAKE SUPERIOR COPPER (SOLUTION A III)

Copper Analyses

Sample	Corr. wt. of sample, g.	Corr. wt. of copper found, g.	Copper correction	Corr. copper per 100 g. of soln., g.
6	102.333	1.29116	+0.00006	1.26182
7	102.279	1.29064	+ .00006	1.26194
8	102.408	1.29229	+ .00006	1.26196
9	102.424	1.29239	+ .00006	1.26186
10	102.462	1.29298	+ .00006	1.26197
			Average	1.26191

Chlorine Analyses

Sample	Corr. wt. of sample, g.	Corr. silver weighed out, g.	Nephelometer correction	Corr. for loss of chlorine	Corr. silver equiv. for sample, g.	Corr. silver equiv. per 100 g. of soln., g.
1	102.533	4.39201	−0.00282	+0.00291	4.39210	4.28360
2	102.399	4.38642	− .00255	+ .00291	4.38678	4.28400
3	102.411	4.38689	− .00248	+ .00291	4.38732	4.28400
4	102.351	4.38441	− .00262	+ .00291	4.38470	4.28395
5	102.338	4.38386	− .00272	+ .00291	4.38405	4.28390
					Average	4.28389

From 4.28389:1.26191 = 215.760:X, X = 63.557, the atomic weight of copper.

TABLE IV

ANALYSIS OF CUPRIC CHLORIDE FROM CHILE COPPER (SOLUTION B II)

Copper Analyses

Sample	Corr. wt. of sample, g.	Corr. wt. of copper found, g.	Copper correction	Corr. copper per 100 g. of soln., g.
6	102.355	1.28032	+0.00006	1.25092
7	102.398	1.28075	+ .00006	1.25082
8	102.370	1.28045	+ .00006	1.25087
9	102.366	1.28041	+ .00006	1.25088
10	102.357	1.28030	+ .00006	1.25088
			Average	1.25088

Chlorine Analyses

Sample	Corr. of sample, g.	Corr. silver weighed out, g.	Nephelometer correction	Corr. for loss of chlorine	Corr. silver equiv. for sample, g.	Corr. silver equiv. per 100 g. of soln., g.
1	102.308	4.34115	+0.00054	+0.00291	4.34460	4.24660
2	102.295	4.34085	+ .00007	+ .00291	4.34383	4.24637
3	102.370	4.34399	+ .00005	+ .00291	4.34695	4.24621
					Average	4.24639

From 4.24639:1.25088 = 215.760:X, X = 63.557 the atomic weight of copper.

TABLE V

ANALYSIS OF CUPRIC CHLORIDE FROM CHILE COPPER (SOLUTION B III)

Copper Analyses

Sample	Corr. wt. of sample, g.	Corr. wt. of copper found, g.	Copper correction	Corr. copper per 100 g. of soln., g.
4	101.881	1.28720	+0.00006	1.26350
5	101.941	1.28794	+ .00006	1.26348
6	101.927	1.28748	+ .00006	[1.26321]*
			Average	1.26349

Chlorine Analyses

Sample	Corr. wt. of sample, g.	Corr. silver weighed out, g.	Nephelometer correction	Corr. silver equiv. for sample, g.	Corr. silver equiv. per 100 g. of soln., g.
1	101.897	4.37043	+0.00021	4.37064	4.28926
2	101.974	4.37377	− .00005	4.37372	4.28905
3	101.929	4.37153	+ .00052	4.37205	4.28931
				Average	4.28921

From 4.28921:1.26349 = 215.760:X, X = 63.557, the atomic weight of copper.

* Since the third copper analysis is widely divergent from the other two it is omitted in computing the average. If included the atomic weight of copper found from solution B III becomes 63.554.

The value found by the analysis of cupric chloride adds valuable evidence to the atomic weight of copper. The condensed results of all accepted analyses are listed in Table VI.

As a result of fifteen copper determinations and fourteen chlorine determinations the atomic weight of copper is found to be 63.557.

This value does not seem to be influenced perceptibly by the formation of atacamite, since identical values are obtained whether the atacamite is removed or dissolved and due allowance made for it. The relative acidity, while not affecting the comparison of the samples, since all gave the same indicator test, might have caused a slight deviation from a true value. From experimental evidence the total change from a distinctly acidic to a distinctly basic reaction would correspond to 0.006 unit in atomic weight. If it is assumed that the true acidity for the cupric chloride solutions was midway between these conditions, the atomic weight found would be about 0.003 unit low.

TABLE VI

ATOMIC WEIGHT OF COPPER

From solution A II	63.557
From solution A III	63.557
From solution B II	63.557
From solution B III	63.557
Grand average	63.557

Acknowledgment for generous financial assistance in this research is made to the Carnegie Institution of Washington and to an anonymous benefactor of this Laboratory.

Summary

No difference was found in the atomic weights of specimens of copper from the Calumet and Hecla mines in the Lake Superior region in Michigan and from Chuquicamata, Chile.

The ratio of the atomic weight of copper to that of silver was determined by analysis of pure, recrystallized cupric chloride. The copper was determined electrolytically and the chlorine by nephelometric titration with pure silver. On the basis of Ag = 107.880 the atomic weight of copper was found to be 63.557.

CAMBRIDGE, MASSACHUSETTS

[CONTRIBUTION FROM THE SCHOOL OF CHEMISTRY, UNIVERSITY OF MINNESOTA]

THE SURFACE TENSION OF LIQUID SULFUR DIOXIDE

BY VERNON M. STOWE

RECEIVED OCTOBER 1, 1928 PUBLISHED FEBRUARY 5, 1929

The surface tension of liquid sulfur dioxide has been determined once only and that at a single temperature. At −25° Grunmach[1] found the surface tension of the liquid to be 33.5 dynes per cm. The author has been interested for several years in the work of Dr. Bond[2] of the University of Iowa on solubility. Now Hildebrand[3] recounts the relation between surface tension and solubility. Therefore, it seemed very desirable to determine the surface tension of liquid sulfur dioxide at several temperatures in order that comparisons might be made with other liquids. The problem of the solubility relations of liquid carbon dioxide has already been attacked in this same manner by Quinn.[4]

Experimental

The capillary tube method was used. A large number of capillary tubes were drawn from a variety of soft glass tubes and of these six were chosen for uniformity of bore, determined by means of a micrometer. They were washed with a mixture of potassium dichromate and sulfuric acid for over twenty-four hours. They were thoroughly rinsed with tap water, then with distilled water and finally with conductivity water. They were dried by drawing air through them briefly and were placed in a desiccator over phosphorus pentoxide for more than a week. From the time they were removed from the cleaning solution they were handled with carefully cleaned hands and only at one end. This end was broken off immediately to prevent impurities from

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[1] Grunmach, *Ann. Physik*, [4] **4**, 367 (1901).

[2] Bond and Beach, THIS JOURNAL, **48**, 348–356 (1926); various University of Iowa theses.

[3] Hildebrand, "Solubility," The Chemical Catalog Co., Inc., New York, **1924**.

[4] Quinn, THIS JOURNAL, **49**, 2704 (1927).