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# FIRST REPORT OF THE COMMITTEE ON ATOMIC WEIGHTS OF THE INTERNATIONAL UNION OF CHEMISTRY

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In September, 1930, at the meeting of the International Union of Chemistry at Liege, the Committee on Elements which has functioned since 1923 was replaced by three committees, on Atomic Weights, Atoms, including isotopy and atomic structure, and **R**adioactive Constants. Hereafter the function of the first of these committees will be to prepare annually a table of atomic weights on the basis of the most recent evidence. National Committees on Atomic Weights are requested by the International Union to refrain from publishing tables of their own.

While it was impossible for the new committee to issue a report earlier this year, it is the intention of the committee normally to issue its report so far as possible in the first (January) number of current chemical and physical periodicals. These reports will cover the twelve months from October to September preceding.

Authors of papers bearing on the subject are requested to send copies to each of the five members of this committee at the earliest possible moment.<sup>1</sup>

Since the reports of the German and American committees<sup>2</sup> adequately cover the ground of progress during 1929, only investigations published since January 1, 1930, are reviewed in this report.

Batuecas, Schlatter and Maverick<sup>3</sup> have published new determinations

|                   | I       | II      |
|-------------------|---------|---------|
| $N_2$             | 1.0040  |         |
| $\mathbf{NH}_{3}$ | 1.01543 | 1.01515 |
| HCl               | 1.00787 | 1.00737 |
| CO                | 1.00048 |         |
| $H_2S$            | 1.01031 | 1.01035 |

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<sup>2</sup> Ber., 63B, 1 (1930); THIS JOURNAL, 52, 857 (1930).

<sup>8</sup> Batuecas, Schlatter and Maverick, J. chim. phys., 27, 36, 45 (1930).

of  $(PV)_0/(PV)_1$  by the expansion method. In Column I the assumption is made that the quantity varies lineally with the pressure; in Column II an equation of the second degree is used.

Nitrogen.—Moles and Batuecas<sup>4</sup> have redetermined the density of ammonia at various pressures. The gas was prepared (1) from ammonium oxalate and potassium hydroxide, (2) by synthesis (technical) from the elements, (3) by hydrolysis of magnesium nitride. After chemical purification the gas was fractionally distilled and in most cases was dried finally with phosphorus pentoxide. Correction for adsorption on the walls of the

| Method of preparation    | Globe N-3<br>773 ml. | Globe N-2<br>647 ml.<br>Preliminary | Globe G,<br>1007 ml. | Average |
|--------------------------|----------------------|-------------------------------------|----------------------|---------|
|                          |                      | 1 Atm                               | osphere              |         |
| 1                        | 0.77167              | 0.77196                             |                      | 0.7718  |
|                          | .77234               | .77184                              |                      | .7721   |
|                          | .77226               | .77207                              |                      | .7722   |
|                          | .77137               | .77219                              |                      | .7718   |
| Aver                     | age .77191           | .77202                              |                      | .7720   |
|                          | .77174               | .77130                              |                      | .77152  |
|                          | .77168               | .77190                              |                      | .77179  |
|                          | .77113               | .77158                              |                      | .77136  |
|                          | .77160               | .77168                              |                      | .77164  |
|                          |                      | .77166                              |                      | .77166  |
|                          | .77170               | .77149                              |                      | .77160  |
|                          | .77184               | .77212                              |                      | .77198  |
| Aver                     | age .77162           | .77168                              |                      | .77165  |
| 2                        | .77118               | .77185                              |                      | .77152  |
|                          | .77153               | .77207                              |                      | .77180  |
|                          | .77180               | .77119                              |                      | .77149  |
|                          | .77144               | .77188                              |                      | .77166  |
|                          | .77161               | .77193                              |                      | .77177  |
| Aver                     | age .77151           | .77178                              |                      | .77165  |
| 3                        |                      |                                     | 0.77169              | .77169  |
|                          |                      |                                     | .77165               | .77165  |
|                          | .77195               |                                     | .77193               | .77194  |
|                          | .77168               |                                     | .77170               | .77169  |
|                          | .77187               |                                     | .77206               | . 77197 |
| Avera                    | age .77183           |                                     | .77181               | .77179  |
| Average of               | all .77163           | .77172                              | .77181               | .77169  |
| Method of<br>preparation | Globe N3             | Globe N2                            | Globe G              | Average |
|                          |                      | ²/a Atmosph                         | ere                  |         |
| 1                        | 0.76758              |                                     | 0.76763              | 0.76761 |
|                          | .76734               |                                     | .76773               | .76754  |
|                          | .76839               |                                     | .76842               | .76841  |
|                          | .76803               |                                     | .76844               | .76824  |
| Avera                    | age .76784           |                                     | .76806               | .76795  |

<sup>4</sup> Moles and Batuecas. Anales soc. españ. fís. quím., 28, 871 (1930).

| May, 1931 REPORT                  | OF THE COMMITTEE ON A                  | TOMIC WEIGHTS |
|-----------------------------------|--|---------------|
| Method of<br>preparation Globe N3 | Globe N2 Globe                         | G Average     |
| 2 0.76754                         |  | 0.76762       |
| .76770                            |  | .76770        |
| .76752                            |  | .76761        |
| Average .76759                    | .76770                                 | .76764        |
| 3 .76743                          |  |               |
| .76737                            |  |               |
| Average .76740                    |  |               |
| 0 -                               |  |               |
| Average of all .76766             | .76770 .7678                           | 85 .76773     |
|                                   | <sup>1</sup> / <sub>2</sub> Atmosphere |               |
| 1 0.76511                         | 0.766                                  | 24 0.76568    |
| . 76623                           |  |               |
| . 76586                           |  |               |
| Average .76573                    | . 765                                  | 93 .76583     |
| 2 .76592                          | .766                                   | .76599        |
| .76582                            | .765                                   | 92 .76587     |
| Average .76587                    | .765                                   | 99 .76593     |
| 3                                 | 0.76539                                | .76539        |
| .76641                            |  | .76641        |
| .76577                            | .76561                                 | .76569        |
| .76610                            | .76593                                 | .76602        |
| .76605                            | .76541                                 | .76573        |
| Average .76608                    | .76559                                 | .76585        |
| Average of all .76592             | .76559 .7659                           | 95.76585      |
|                                   | <sup>1</sup> /; Atmosphere             |               |
| 1 0.76328                         | 0.763                                  | 0.76325       |
| .76378                            | .7634                                  | 41 .76360     |
| .76395                            | .7643                                  | .76415        |
| Average .76367                    | .7636                                  | .76366        |
| 2                                 | .763                                   | .76369        |
|                                   | .7634                                  | 48 .76348     |
| .76400                            | .7640                                  | .76402        |
| .76416                            | (.7626                                 | .76416        |
| .76444                            | .7638                                  | .76416        |
| . 76387                           | .7630                                  | .76374        |
| Average .76418                    | . 7636                                 | .76390        |
| 3 .76391                          |  |               |
| .76438                            |  |               |
| .76342                            |  |               |
| . 76350                           |  |               |
| .76405                            | .7642                                  | .76416        |
| Average .76385                    | .7642                                  |               |
| Average of all .76392             | .7637                                  | .76383        |

globes was made. The results are expressed in the weight of the liter at 0° and 760 mm.

From the densities at various pressures the limiting density of ammonia is calculated by the method of differences to be 0.75990. The corresponding molecular weight of ammonia is then 17.032, and the atomic weight of nitrogen 14.009.

Phosphorus.—Ritchie<sup>5</sup> has determined the density of phosphine at different pressures. The gas was prepared from phosphonium iodide by means of potassium hydroxide and was fractionated.

| P,<br>atmospheres | Globe I,<br>336 ml. | Globe II,<br>341 ml. | Average |
|-------------------|---------------------|----------------------|---------|
| 1                 | (1.5311)            | 1.5308               |         |
| 1                 |                     |                      | 1.5308  |
|                   | 1.5308              | 1.5307               | 1.5308  |
|                   | 1.5307              | 1.5305               | 1.5306  |
|                   | 1.5307              | 1.5307               | 1.5307  |
|                   | 1.5308              | 1.5308               | 1.5308  |
|                   | 1.5306              |                      | 1.5306  |
|                   | Average 1.5307      | 1.5307               | 1.5307  |
| 0.75              | 1.5274              | 1.5272               | 1.5273  |
|                   |                     | 1.5273               | 1.5273  |
|                   | 1.5271              | 1.5272               | 1.5272  |
|                   | Average 1.5273      | 1.5272               | 1.5272  |
| 0.50              | 1.5241              | 1.5237               | 1.5239  |
|                   | 1.5242              |                      | 1.5242  |
|                   | 1.5237              | 1.5236               | 1.5237  |
|                   | 1.5233              | 1.5238               | 1.5236  |
|                   | 1.5238              | 1.5238               | 1.5238  |
|                   | Average 1.5238      | 1.5237               | 1.5238  |
| 0.25              | 1.5204              | 1,5202               | 1.5203  |
|                   | 1.5202              | 1.5203               | 1.5203  |
|                   |                     | 1,5201               | 1.5201  |
|                   | 1.5205              | 1.5205               | 1.5205  |
|                   | Average 1.5204      | 1.5203               | 1.5203  |
|                   | -                   |                      |         |

Assuming a linear relation between PV and pressure,  $(PV)_0/(PV)_1$  is calculated to be 1.0091.

If the normal liter of oxygen weighs 1.4290 g, and the coefficient of deviation from Boyle's Law per atmosphere is -0.00096, then  $PH_3 = 34.000$  and P = 30.977. This value for phosphorus is appreciably lower than the chemical value.

Sulfur.—Hönigschmid and Sachtleben<sup>6</sup> have completed a synthesis of silver sulfide from its elements. The compound was found to be stable up to 300° but to lose sulfur by decomposition above this temperature. When reheated in sulfur vapor, partially decomposed sulfide takes up quantitatively the deficiency in sulfur. Excess sulfur is given up at 300°. To carry out a synthesis weighed quantities of the purest silver were heated in sulfur vapor until the reaction was complete and then the excess of sulfur was eliminated in a current of pure nitrogen at 280°. Constancy in weight of the sulfide was readily attained. The sulfur was prepared by precipita-

<sup>8</sup> Ritchie, Proc. Roy. Soc. (London), A128, 551 (1930).

<sup>6</sup> Hönigschmid and Sachtleben, Z. anorg. Chem., 195, 207 (1931).

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tion from thiosulfate and double distillation in vacuum. Weights are corrected to vacuum. In the twelfth analysis the materials of the eleventh were reweighed in exhausted receptacles.

#### THE ATOMIC WEIGHT OF SULFUR

| Wt. of Ag, g. | Wt. of Ag2S, g. | Ratio<br>Ag2S:2Ag | Atomic weight<br>of sulfur |
|---------------|-----------------|-------------------|----------------------------|
| 7.90291       | 9.07742         | 1.148617          | 32.066                     |
| 9.42181       | 10.82209        | 1.148621          | 32.066                     |
| 9.74522       | 11.19355        | 1.148620          | 32.066                     |
| 9.59836       | 11.02489        | 1.148622          | 32.067                     |
| 9.20378       | 10.57166        | 1.148622          | 32.067                     |
| 10.75224      | 12,35021        | 1.148617          | 32.066                     |
| 8.28317       | 9.51424         | 1.148623          | 32.067                     |
| 9.86327       | 11.32913        | 1.148618          | 32.066                     |
| 10.43748      | 11.98871        | 1.148621          | 32.066                     |
| 7.21091       | 8.28265         | 1.148627          | 32.068                     |
| 9.84440       | 11.30749        | 1.148621          | 32.067                     |
| 9.84439       | 11.30748        | 1.148622          | 32.067                     |
|               | Average         | 1.148621          | 32.066                     |

Since all recent determinations of the atomic weight of sulfur have yielded a value not far from 32.06, this value has been adopted for the table.

**Chlorine**.—Scott and Johnson,<sup>7</sup> call attention to an error in the solubility of silver chloride at  $0^{\circ}$  assumed by Hönigschmid and Chan<sup>8</sup> in their syntheses of silver chloride, which amounts to 0.002% in the weight of silver chloride.

**Calcium**.—Hönigschmid and Kempter<sup>9</sup> purified calcium nitrate from marble by ten recrystallizations, and converted the product to chloride by precipitation of the carbonate and solution of the latter in hydrochloric acid (Sample I). Sample II was prepared from commercial nitrate by fifteen crystallizations. After recrystallization of the chloride it was prepared for weighing by dehydration and fusion in hydrogen chloride, and allowed

#### ATOMIC WEIGHT OF CALCIUM

| Sample | Wt. of CaCl2, g. | Wt. of Ag, g. | Ratio<br>CaCl2:2Ag | Atomic weight<br>of calcium |
|--------|------------------|---------------|--------------------|-----------------------------|
| Ι      | 1.84526          | 3.58692       | 0.514441           | 40.082                      |
| Ι      | 1.62314          | 3.15509       | .514451            | 40.084                      |
| Ι      | 1.42216          | 2.76444       | .514447            | 40.083                      |
| Ι      | 2.21933          | 4.31400       | .514448            | 40.083                      |
| Ι      | 1.03950          | 2.02064       | .514441            | 40.082                      |
| Ι      | 1.45783          | 2.83364       | .514472            | 40.088                      |
| II     | 2.93786          | 5.71052       | .514464            | 40. <b>086</b>              |
| II     | 2.45368          | 4.76952       | .514451            | 40.084                      |
| II     | 2.11276          | 4.10689       | .514441            | 40.082                      |
|        |                  | Avera         | ige .514451        | 40.084                      |

<sup>7</sup> Scott and Johnson, This JOURNAL, 52, 3586 (1930).

<sup>&</sup>lt;sup>8</sup> Hönigschmid and Chan, Z. anorg. Chem., 163, 315 (1927).

<sup>&</sup>lt;sup>9</sup> Hönigschmid and Kempter, *ibid.*, 195, 1 (1931).

|        | -                |                 |                      |                             |
|--------|------------------|-----------------|----------------------|-----------------------------|
| Sample | Wt. of CaCl2, g. | Wt. of AgCl, g. | Ratio<br>CaCl2:2AgCl | Atomic weight<br>of calcium |
| Ι      | 1.97942          | 5.11225         | 0.387191             | 40.083                      |
| I      | 2.35393          | 6.07937         | .387199              | 40.086                      |
| I      | 1.67385          | 4.32284         | .387210              | 40.089                      |
| Ι      | 1.62314          | 4.19217         | .387183              | 40.082                      |
| Ι      | 1.42216          | 3.67297         | .387196              | 40.085                      |
| I      | 2.21933          | 5.73153         | .387214              | 40.090                      |
| II     | 1.03950          | 2.68467         | .387198              | 40.086                      |
| II     | 1.45783          | 3.76499         | .387206              | 40.088                      |
|        |                  | Average         | .387200              | 40.086                      |

#### ATOMIC WEIGHT OF CALCIUM (Concluded)

to solidify in nitrogen. The solutions of the weighed chloride were corrected for deviations from the neutral point by titration with N/100 solutions of acid and base and then were compared with silver in the usual way, and the silver chloride was collected and weighed. Weights are corrected to vacuum.

The average of both series, 40.085, is slightly higher than that found earlier by Richards and Hönigschmid, 40.071. For the present 40.08 is recommended.

A. V. and O. Frost<sup>10</sup> claim to have discovered a concentration of Ca<sup>41</sup> by beta-ray emission from K<sup>41</sup> in a potassium feldspar containing 0.042% of calcium oxide. Only 0.15 g. of calcium oxide was available. From the ratio CaCl<sub>2</sub>: CaBr<sub>2</sub> the atomic weight of calcium was found in two experiments to be 40.23. Similar experiments with ordinary calcium which had been purified in the same way gave 40.10.

Hönigschmid and Kempter<sup>11</sup> attacked the same problem with calcium extracted from sylvin by von Hevesy. After preliminary purification the average atomic weight through the chloride was found to be 40.22. Spectroscopic investigation, however, revealed the presence of strontium. After removal of this impurity by fractional precipitation of the oxalate, the observed atomic weight was lowered to 40.093. The material still contained 0.015 atom per cent. of strontium so that the value to be expected is 40.091. Since the sylvin is a geologically younger mineral than the feldspar, a smaller concentration of Ca<sup>41</sup> is to be expected, so that the question as to appreciable variation of Ca<sup>41</sup> in nature is still an open one.

Vanadium.—Scott and Johnson,<sup>12</sup> have analyzed vanadyl trichloride. This was made by heating purified vanadium trioxide in a current of chlorine, and the product was purified by vacuum distillation, after removal of excess chlorine with mercury and sodium. Portions for analysis were removed in sealed glass bulbs in the later stages of the distillation. After being weighed the bulbs were broken under either nitric acid or ammonia.

<sup>&</sup>lt;sup>10</sup> Frost and Frost, Nature, 125, 48 (1930).

<sup>&</sup>lt;sup>11</sup> Hönigschmid and Kempter, Z. anorg. Chem., 195, 9 (1931).

<sup>&</sup>lt;sup>12</sup> Scott and Johnson. THIS JOURNAL, 52, 2638 (1930).

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In the former case the glass was washed with nitric acid and collected on a filter. In the latter, after the supernatant liquid had been filtered, the precipitate was dissolved in nitric acid and the glass was washed and collected. The solutions were then compared with silver in the usual way, and in some cases the silver chloride was collected. The analyses are arranged in the order of decreasing volatility of the chloride samples. Weights are corrected to vacuum.

#### THE ATOMIC WEIGHT OF VANADIUM

| Acid Hydrolysis     |               |                    |                           |                    |                      |                        |
|---------------------|---------------|--------------------|---------------------------|--------------------|----------------------|------------------------|
| Wt. of<br>VOCl3, g. | Wt. of Ag, g. | Ratio<br>VOCl3:3Ag | Atomic wt.<br>of vanadium | Wt. of<br>AgCl, g. | Ratio<br>VOCla:3AgCl | At. wt. of<br>vanadium |
| 8.15697             | 15.23143      | 0.535535           | 50.950                    | 20.23483           | 0.403115             | 50.973                 |
| 8.29538             | 15.48986      | . 535536           | 50.950                    | 20.57872           | .403105              | 50.969                 |
| 7.60527             | 14.20111      | .535541            | 50.951                    | 18.86755           | .403087              | 50.961                 |
| 7.01143             | 13.09218      | . 535543           | 50.952                    |                    |                      |                        |
|                     | Average       | 535539             | 50.951                    |                    | .403102              | 50.968                 |
|                     |               | Alka               | aline Hydrol              | ysis               |                      |                        |
| 7.75120             | 14.47384      | 0.535532           | 50.949                    |                    |                      |                        |
| 7.88453             | 14.72386      | .535493            | 50.936                    | 19.56218           | .403050              | 50.945                 |
| 9.19783             | 17.17614      | . 535500           | 50.938                    | 22.81924           | .403073              | 50.955                 |
| 6.69572             | 12.50344      | . 535510           | 50.941                    |                    |                      |                        |
| 8.04970             | 15.03136      | . 535527           | 50.947                    | <b>19.969</b> 76   | .403094              | 50.964                 |
| 7.62984             | 14.24666      | . 535553           | 50.955                    |                    |                      |                        |
|                     | Average       | .535519            | 50.945                    |                    | .403072              | 50.955                 |

Experimental evidence was found that the nephelometric end-point was slightly affected by the presence of vanadic acid, but the effect on the atomic weight of vanadium is less than 0.005. The average of the comparisons with silver, 50.948, agrees almost exactly with the recent results obtained by McAdam, and by Briscoe and Little.

**Chromium**.—Gonzales<sup>13</sup> has applied to chromyl chloride the recently developed method of preparing volatile inorganic compounds by fractional distillation in vacuum. The compound was prepared by the action of concentrated sulfuric acid on a mixture of sodium chloride and potassium bichromate and after fractional distillation under low pressure was collected in sealed glass bulbs. The bulbs were broken under water and the halogen was determined by comparison with silver in the usual way. Ultimately the silver chloride was determined. Weights are corrected to vacuum.

THE ATOMIC WEIGHT OF CHROMIUM

| Wt. of<br>CrO2Cl2, g. | Wt. of<br>Ag, g. | Ratio<br>CrO <sub>2</sub> Cl <sub>2</sub> :2Ag | Atomic wt.<br>of chromium | Wt. of<br>AgCl, g. | Ratio<br>CrO2Cl2:2AgCl | At. wt.<br>of<br>chromium |
|-----------------------|------------------|--|---------------------------|--------------------|------------------------|---------------------------|
| 9.56543               | 13.32143         | 0.718049                                       | 52.012                    | 17.69786           | 0.540485               | 52.029                    |
| 9.54415               | 13.29120         | .718080  | 52.019                    | 17.65929           | .540460                | 52.022                    |
|                       | Average          | .718065  | 52.016                    |                    | . 540473               | 52.026                    |

13 Gonzales, Anales soc. españ. fís. quím., 28, 579 (1930).

The average result 52.02 is only 0.01 unit higher than the current one and no change is recommended for the present.

Arsenic.—Krepelka<sup>14</sup> has published details of the analysis of arsenic trichloride noted earlier.<sup>18</sup> Recrystallized arsenic trioxide was reduced with sugar charcoal and the metal resublimed. Conversion of the metal to chloride was followed by repeated vacuum distillation of the latter. Samples were collected for weighing in sealed evacuated bulbs. Hydrolysis with ice water was followed by comparison with silver in the usual way. In two cases the silver chloride was collected and weighed. Vacuum weights are given.

#### THE ATOMIC WEIGHT OF ARSENIC

| Wt. of<br>AsCl₃, g. | Wt. of<br>Ag, g. | Ratio<br>AsCl₃:3Ag | At. wt. of<br>arsenic <sup>a</sup> | Wt. of<br>AgCl, g. | Ratio<br>AsCla:3AgCl | At. wt. of<br>arsenic <sup>e</sup> |
|---------------------|------------------|--------------------|------------------------------------|--------------------|----------------------|------------------------------------|
| 3.98710             | 7.11681          | 0.560237           | 74.944                             |                    |                      |                                    |
| 4.81766             | 8.59961          | .560218            | 74.938                             |                    |                      |                                    |
| 6.27437             | 11.20020         | . 560201           | 74.933                             |                    |                      |                                    |
| 2.42721             | 4.33242          | .560244            | 74.946                             | 5.75672            | 0.421631             | 74.934                             |
| 3.86442             | 6.89796          | .560227            | 74.941                             |                    |                      |                                    |
| 5.09819             | 9.10041          | .560215            | 74.937                             |                    |                      |                                    |
| 5.46890             | 9.76222          | .560211            | 74.936                             |                    |                      |                                    |
| 5.10039             | 9.10415          | .560227            | 74.941                             |                    |                      |                                    |
| 5.71146             | 10.19540         | . 560200           | 74.932                             |                    |                      |                                    |
| 3.05992             | 5.46180          | .560240            | 74.945                             |                    |                      |                                    |
| 1.49994             | 2.67755          | .560191            | 74.929                             | 3.55734            | .421646              | 74.941                             |
|                     | Average          | .560219            | 74.938                             |                    | . 421638             | 74.938                             |
|                     |                  |                    |                                    |                    |                      |                                    |

<sup>a</sup> Calculated with Cl = 35.457. The figures given by the authors are calculated with Cl = 35.458.

This value is slightly lower than the value which has been in use for some time, and slightly higher than that found by Aston with the mass spectrograph after correction for the presence of  $O^{18}$ , 74.927. The value 74.93 is adopted in the table of atomic weights.

**Tantalum.**—Krishnaswami<sup>16</sup> has analyzed the chloride and bromide of tantalum. Metallic tantalum was first obtained by reducing purified potassium tantalum fluoride with sodium in an atmosphere of argon. When examined spectroscopically the metal appeared to be free from impurities, although it contained a small percentage of oxide. The metal was converted to halides by the action of pure dry halogens and the halides were twice distilled in vacuum and collected in sealed glass bulbs. After being weighed the bulbs were broken under ammonia and the solutions filtered to remove glass and tantalic acid. To find the weight of the glass the tantalic acid was dissolved in oxalic acid and the glass was collected on a weighed crucible. The solutions were then compared with silver, and

<sup>14</sup> Krepelka, Collect. trav. chim. Tchecoslovaquie, 2, 255 (1930).

<sup>16</sup> Krishnaswami, J. Chem. Soc., 1277 (1930).

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<sup>&</sup>lt;sup>15</sup> Krepelka, Nature, 123, 944 (1929).

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the silver halides were collected and weighed. Weights are corrected to vacuum.

| THE ATOMIC WEIGHT OF TANTALUM |                  |                           |                           |                    |                      |                        |
|-------------------------------|------------------|---------------------------|---------------------------|--------------------|----------------------|------------------------|
| Wt. of<br>TaBrs, g.           | Wt. of<br>Ag, g. | Ratio<br>TaBrs:5Ag        | Atomic wt.<br>of tantalum | Wt. of<br>AgBr, g. | Ratio<br>TaBr₅:5AgBr | At. wt. of<br>tantalum |
| 3.07127                       |                  |                           |                           | 4.96415            | 0.61869              | 181.36                 |
| 3.72095                       |                  |                           |                           | 6.01413            | .61870               | 181.37                 |
| 3.81890                       | 3.54594          | 1.07698                   | 181.34                    | 6.17267            | .61868               | 181.35                 |
| 3.59654                       | 3.33939          | 1.07700                   | 181.36                    | 5.81303            | .61870               | 181.37                 |
| 2.69071                       | 2.49831          | 1.07701                   | 181.37                    | 4.34926            | .61866               | 181.33                 |
| 2.61163                       | 2.42488          | 1.07702                   | 181.37                    | 4.22133            | .61868               | 181.35                 |
| 3.92094                       | 3.64064          | 1.07699                   | 181.35                    | 6.33750            | .61869               | 181.36                 |
| 2.04583                       | 1.89956          | 1.07700                   | 181.36                    | 3.30681            | .61867               | 181.34                 |
|                               | Average          | 1.07700                   | 181.36                    |                    | .61868               | 181.35                 |
| Wt. of<br>TaCls, g.           | Wt. of<br>Ag, g. | <b>Ratio</b><br>TaCls:5Ag | Atomic wt.<br>of tantalum | Wt. of<br>AgCl, g. | Ratio<br>TaCl5:5AgCl | At. wt. of<br>tantalum |
| 3.15350                       | 4.74301          | 0.66488                   | 181.35                    | 6.30152            | 0.50044              | 181.37                 |
| 2.96215                       | 4.45549          | .66483                    | 181.33                    | 5.91874            | .50047               | 181.40                 |
| 4.08061                       | 6.13756          | .66486                    | 181.34                    | 8.15438            | 50042                | 181.36                 |
| 3.21073                       | 4.82972          | .66479                    | 181.30                    | 6.41613            | .50042               | 181.36                 |
| 3.49922                       | 5.26278          | .66490                    | 181.36                    | 6.99201            | .50046               | 181.39                 |
|                               | Average          | .66485                    | 181.34                    |                    | . 50044              | 181.37                 |

The average value, 181.36, is lower than that found by Balke in 1910, 181.50. Balke's method, in which the ratio  $TaCl_5:Ta_2O_5$  was determined, has been found to be unreliable because of the uncertain composition of most oxides. The new value therefore has been adopted.

**Rhenium**.—Hönigschmid and Sachtleben<sup>17</sup> have taken advantage of the increased quantities of rhenium now available by analyzing silver perrhenate. Three specimens of material were prepared. (I) Potassium perrhenate was recrystallized and the silver salt precipitated. Retained potassium was removed by reprecipitation and crystallization. (II) Metallic rhenium was burned to heptoxide in oxygen, and after solution of the oxide in water, silver perrhenate was precipitated with silver nitrate. (III) The third sample was prepared by dissolving silver oxide in perrhenic acid.

| The Atomic Weight of Rhenium |                      |                    |                      |                             |  |  |
|------------------------------|----------------------|--------------------|----------------------|-----------------------------|--|--|
| Sample                       | Wt. of<br>AgReO4, g. | Wt. of<br>AgBr, g. | Ratio<br>AgReO4:AgBr | Atomic weight<br>of rhenium |  |  |
| I                            | 5.36365              | <b>2.</b> 81186    | 1.90751              | 186.34                      |  |  |
| II                           | 7.83577              | 4.10795            | 1.90747              | 186.33                      |  |  |
| II                           | 8.55829              | 4.48684            | 1.90742              | 186.33                      |  |  |
| II                           | 6.34973              | 3.32894            | 1.90743              | 186.33                      |  |  |
| III                          | 8.90918              | 4.67111            | 1.90729              | 186.30                      |  |  |
| III                          | 6.95494              | 3.64684            | 1.90712              | 186.27                      |  |  |
| III                          | 7.85704              | 4.11955            | 1.90726              | 186.30                      |  |  |
|                              |                      | Average            | 1.90735              | 186.31                      |  |  |

<sup>17</sup> Hönigschmid and Sachtleben, Z. anorg. Chem., 191, 309 (1930).

The silver salt was prepared for weighing by fusion in air of a mixture of the perrhenate with an excess of acid. Weighed amounts of salt were dissolved in water and the silver was precipitated as silver bromide. Weights are corrected to vacuum.

This result is 2.4 units lower than the preliminary value found by W. and I. Noddack by analysis of the disulfide, but in view of the inferiority of the latter method and the small quantities weighed, the new value 186.31 is adopted for the table.

**Thallium.**—Hönigschmid and Striebel<sup>18</sup> prepared thallous bromide by precipitation from a solution of the purified sulfate. After distillation in nitrogen the salt was weighed in a quartz tube. Solution in hot water was followed by hot precipitation with a nearly equivalent amount of silver. The end-point was found with a nephelometer in the usual way. Weights are corrected to vacuum.

|                    | The Atomic Weight | OF THALLIU       | М                            |  |  |  |  |  |
|--------------------|-------------------|------------------|------------------------------|--|--|--|--|--|
| Preliminary Series |                   |                  |                              |  |  |  |  |  |
| Wt. of<br>TlBr, g. | Wt. of<br>Ag, g.  | Ratio<br>TlBr:Ag | Atomic weight<br>of thallium |  |  |  |  |  |
| 3.86281            | 1.46582           | 2.63526          | 204.38                       |  |  |  |  |  |
| 3.78429            | 1.43583           | 2.63561          | 204.41                       |  |  |  |  |  |
| 3.96949            | 1.50639           | 2.63510          | 204.36                       |  |  |  |  |  |
| 3.94471            | 1.49669           | 2.63562          | 204.42                       |  |  |  |  |  |
|                    | Average           | 2.63540          | 204.39                       |  |  |  |  |  |
| Final Series       |                   |                  |                              |  |  |  |  |  |
| 4.01222            | 1.52251           | 2.63527          | 204.377                      |  |  |  |  |  |
| 3.97142            | 1.50692           | 2.63546          | 204.397                      |  |  |  |  |  |
| 3.90498            | 1.48170           | 2.63547          | 204.399                      |  |  |  |  |  |
| 4.07193            | 1.54509           | 2.63540          | 204.391                      |  |  |  |  |  |
| 3.68886            | 1.39974           | 2.63539          | 204.390                      |  |  |  |  |  |
| 4.04739            | 1.53580           | 2.63536          | 204.387                      |  |  |  |  |  |
|                    | Average           | 2.63539          | 204.390                      |  |  |  |  |  |

This value agrees exactly with that found earlier by Hönigschmid, Berckenbach and Kothe through the analysis of thallous chloride.

Lead.—Baxter and Bliss<sup>19</sup> have determined the atomic weight of two specimens of RaG. The first was extracted from Swedish kolm, the second from uraninite from Wilberforce, Ontario, Canada. Purification was effected by precipitation as chromate and sulfate and crystallization as nitrate and chloride. After resublimation the chloride was fused preparatory to weighing. Comparison with silver was carried out as usual. Weights are corrected to vacuum.

The kolm lead has a lower atomic weight than any other specimen yet examined and seems to consist almost entirely of the isotope  $Pb^{206}$ .

<sup>&</sup>lt;sup>18</sup> Hönigschmid and Striebel, Z. anorg. allgem. Chem., 194, 293 (1930).

<sup>&</sup>lt;sup>19</sup> Baxter and Bliss, THIS JOURNAL, **52**, 4848, 4851 (1930).

# INTERNATIONAL ATOMIC WEIGHTS

|                  | Sym-<br>bol            | Atomic<br>Number | Atomic<br>Weight |              | Sym-<br>bol            | Atomic<br>Number | Atomic<br>Weight |
|------------------|------------------------|------------------|------------------|--------------|------------------------|------------------|------------------|
| Aluminum         | Al                     | 13               | 26.97            | Molybdenum   | Mo                     | 42               | 96.0             |
| Antimony         | $\mathbf{Sb}$          | 51               | 121.76           | Neodymium    | $\mathbf{Nd}$          | 60               | 144.27           |
| Argon            | Α                      | 18               | 39.944           | Neon         | Ne                     | 10               | 20.183           |
| Arsenic          | As                     | 33               | 74.93            | Nickel       | Ni                     | 28               | 58.69            |
| Barium           | Ba                     | 56               | 137.36           | Nitrogen     | Ν                      | 7                | 14.008           |
| Beryllium        | Be                     | 4                | 9.02             | Osmium       | Os                     | <b>7</b> 6       | 190.8            |
| Bismuth          | Bi                     | 83               | 209.00           | Oxygen       | 0                      | 8                | 16.0000          |
| Boron            | В                      | 5                | 10.82            | Palladium    | Pd                     | 46               | 106.7            |
| Bromine          | Br                     | 35               | 79.916           | Phosphorus   | Р                      | 15               | 31.02            |
| Cadmium          | Cd                     | 48               | 112.41           | Platinum     | Pt                     | 78               | 195.23           |
| Calcium          | Ca                     | <b>20</b>        | 40.08            | Potassium    | K                      | 19               | 39.10            |
| Carbon           | С                      | 6                | 12.00            | Praseodymium | Pr                     | 59               | 140.92           |
| Cerium           | Ce                     | 58               | 140.13           | Radium       | Ra                     | 88               | 225.97           |
| Cesium           | Cs                     | 55               | 132.81           | Radon        | Rn                     | 86               | 222              |
| Chlorine         | C1                     | 17               | 35.457           | Rhenium      | Re                     | 75               | 186.31           |
| Chromium         | Cr                     | 24               | 52.01            | Rhodium      | Rh                     | 45               | 102.91           |
| Cobalt           | Co                     | 27               | 58.94            | Rubidium     | Rb                     | 37               | 85.44            |
| Columbium        | Cb                     | 41               | 93.3             | Ruthenium    | Ru                     | 44               | 101.7            |
| Copper           | Cu                     | 29               | 63.57            | Samarium     | Sm                     | 62               | 150.43           |
| Dysprosium       | Dy                     | 66               | 162.46           | Scandium     | Sc                     | 21               | 45.10            |
| Erbium           | Er                     | 68               | 167.64           | Selenium     | Se                     | 34               | 79.2             |
| Europium         | Eu                     | 63               | 152.0            | Silicon      | Si                     | 14               | 28.06            |
| Fluorine         | F                      | 9                | 19.00            | Silver       | Ag                     | 47               | 107.880          |
| Gadolinium       | Gd                     | 64               | 157.3            | Sodium       | Na                     | 11               | 22.997           |
| Gallium          | Ga                     | 31               | 69.72            | Strontium    | Sr                     | 38               | 87.63            |
| Germanium        | Ge                     | 32               | 72.60            | Sulfur       | S                      | 16               | 32.06            |
| Gold             | Au                     | 79               | 197.2            | Tantalum     | Та                     | 73               | 181.4            |
| Hafnium          | $\mathbf{H}\mathbf{f}$ | 72               | 178.6            | Tellurium    | Te                     | 52               | 127.5            |
| Helium           | He                     | <b>2</b>         | 4.002            | Terbium      | $\mathbf{Tb}$          | 65               | 159.2            |
| Holmium          | Ho                     | 67               | 163.5            | Thallium     | T1                     | 81               | 204.39           |
| Hydrogen         | н                      | 1                | 1.0078           | Thorium      | $\mathbf{Th}$          | 90               | 232.12           |
| Indium           | In                     | 49               | 114.8            | Thulium      | Tm                     | 69               | 169.4            |
| Iodine           | Ι                      | 53               | 126.932          | Tin          | Sn                     | 50               | 118.70           |
| Iridium          | Ir                     | 77               | 193.1            | Titanium     | Ti                     | 22               | 47.90            |
| Iron             | Fe                     | 26               | 55.84            | Tungsten     | W                      | <b>74</b>        | 184.0            |
| K <b>rypt</b> on | Kr                     | 36               | 82.9             | Uranium      | U                      | 92               | 238.14           |
| Lanthanum        | La                     | 57               | 138.90           | Vanadium     | V                      | 23               | 50.95            |
| Lead             | Pb                     | 82               | 207.22           | Xenon        | Xe                     | 54               | 130.2            |
| Lithium          | Li                     | 3                | 6.940            | Ytterbium    | $\mathbf{Y}\mathbf{b}$ | 70               | 173.5            |
| Lutecium         | Lu                     | 271              | 175.0            | Yttrium      | Y                      | 39               | 88.92            |
| Magnesium        | Mg                     |                  | 24.32            | Zinc         | Zn                     | 30               | 65.38            |
| Manganese        | Mn                     |                  | 54.93            | Zirconium    | Zr                     | 40               | 91.22            |
| Mercury          | Hg                     | 80               | 200.61           |              |                        |                  |                  |

| Sample    | Wt. of PbCls, g. | Wt. of Ag. g. | Ratio<br>PbCl2:2Ag | Atomic weight<br>of lead |
|-----------|------------------|---------------|--------------------|--------------------------|
| Common    | 2.74332          | 2.12809       | 1.28910            | 207.222                  |
|           | 3.60741          | 2.79852       | 1.28904            | 207.209                  |
|           | 3.07537          | 2.38565       | 1.28911            | 207.224                  |
|           | 2.81471          | 2.18351       | 1.28908            | 207.218                  |
|           |                  |               | 1.28909            | 207.218                  |
| Kolm      | 1.61294          | 1.25678       | 1.28339            | 205.990                  |
|           | 1.60407          | 1.24983       | 1.28343            | 205.999                  |
|           | 2.56499          | 1.99842       | 1.28351            | 206.016                  |
|           | 1.83748          | 1.43167       | 1.28345            | 206.003                  |
|           | 3.32075          | 2.58729       | 1.28349            | 206.011                  |
|           | 3.07451          | 2.39530       | 1.28356            | 206.027                  |
|           |                  |               | 1.28347            | 206.008                  |
| Uraninite | 3.74779          | 2.91808       | 1.28433            | 206.194                  |
|           | 5.63102          | 4.38436       | 1.28434            | 206.196                  |
|           |                  |               | 1.28434            | 206.195                  |

#### THE ATOMIC WEIGHT OF LEAD

Aston<sup>20</sup> has extended the usefulness of the mass spectrograph to the determination of the chemical atomic weight of complex elements by microphotometric measurements of the intensities of the isotopic lines in a mass spectrogram. The following table gives the percentages of the components, as well as the packing fractions and the atomic weights calculated on the basis of chemical oxygen.

| Isotopic weights and percentages |      |      |       |       |       | Packing Atomic wt.<br>fraction 0 = 16.0000 |       |       |                              |
|----------------------------------|------|------|-------|-------|-------|--|-------|-------|------------------------------|
| Chromium                         | 50   | 52   | 53    | 54    |       |  |       |       |                              |
|                                  | 4.9  | 81.6 | 10.4  | 3.1   |       |  |       |       | $-10 \times 10^{-4}$ 52.011  |
| Zine                             | 64   | 65   | 66    | 67    | 68    | 69   | 70    |       |                              |
|                                  | 48.0 | 2.5  | 25.9  | 5.3   | 17.1  | 0.85                                       | 0.38  |       | $-9.9 	imes 10^{-4} 65.380$  |
| Molyb-                           | 92   | 94   | 95    | 96    | 97    | 98   | 100   |       |                              |
| denum                            | 14.2 | 10.0 | 15.5  | 17.8  | 9.6   | 23.0                                       | 9.8   |       | $-5.5 \times 10^{-4}$ 95.97  |
| Krypton                          | 78   | 80   | 82    | 83    | 84    | 86   |       |       |                              |
|                                  | 0.42 | 2.45 | 11.79 | 11.79 | 56.86 | 16.70                                      |       |       | $-8.8 \times 10^{-4}$ 83.77  |
| Tin                              | 112  | 114  | 115   | 116   | 117   | 118  | 119   | 120   | 121  122  124                |
|                                  | 1.07 | 0.74 | 0.44  | 14.19 | 9.81  | 21.48                                      | 11.02 | 27.04 | 2.96 5.03 6.19               |
|                                  |      |      |       |       |       |  |       |       | $-7.3 \times 10^{-4} 118.72$ |
| $\mathbf{X}$ enon                | 124  | 126  | 128   | 129   | 130   | 131  | 132   | 134   | 136                          |
|                                  | 0.08 | 0.08 | 2.30  | 27.13 | 4.18  | 20.67                                      | 26.45 | 10.31 | 8.79                         |
|                                  |      |      |       |       |       |  |       |       | $-5.3 \times 10^{-4} 131.27$ |
| Mercury                          | 196  | 198  | 199   | 200   | 201   | 202  | 204   |       |                              |
|                                  | 0.10 | 9.89 | 16.45 | 23.77 | 13.67 | 29.27                                      | 6.85  |       | $+0.8 \times 10^{-4} 200.62$ |

The close agreement of the calculated atomic weights with those found by chemical means in the case of chromium, zinc, molybdenum, tin and mercury, indicates that the method is capable of giving results of a high

<sup>20</sup> Aston, Proc. Roy. Soc. (London), A126, 511 (1930); A130, 302 (1931); Nature, 126, 200, 348 (1930).

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degree of accuracy. In the cases of krypton and xenon the calculated and experimental (density) values are discrepant and, as pointed out by Aston, new determinations of the densities and compressibilities of these gases should be made.

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## THE CRYSTAL STRUCTURE OF SOME FORMS OF GLUCOSE. A PRELIMINARY PAPER

BY O. L. SPONSLER AND W. H. DORE Received June 18, 1930 Published May 6, 1931

In anticipation of throwing more light upon the atomic arrangement of the cellulose unit, several crystalline forms of glucose have been under investigation. The structural relationship between cellulose and the beta modification of the amylene oxidic ring form of glucose has been pointed out by the authors.<sup>1</sup> In order to gain a clearer conception of cellulose it has seemed desirable to determine the structure of the crystals of the beta form and also of other forms of glucose. The present report deals with some preliminary results on the alpha and the beta forms of *d*-glucose (C<sub>6</sub>H<sub>12</sub>O<sub>6</sub>) and on the alpha glucose monohydrate (C<sub>6</sub>H<sub>12</sub>O<sub>8</sub>·H<sub>2</sub>O).

The first two forms were prepared<sup>2</sup> by the method of Hudson and Dale<sup>3</sup> and the monohydrate by that of Hudson and Vanovsky.<sup>4</sup> The fine crystals so obtained were examined by the powder method of x-ray crystal analysis. Radiation from a molybdenum target Coolidge tube was employed for obtaining the diffraction patterns. The existence of crystallographic data for the two alpha forms made it possible to determine the lattice dimensions and to assign space groups to these forms. The absence of similar data for beta glucose precluded those deductions regarding its structure, but the diffraction patterns made it evident that the alpha and beta forms have different crystal structures.

After this paper was written Hengstenberg and Mark<sup>5</sup> reported a lattice for "d-glucose" without stating whether the alpha or beta form was used. They employed a single crystal rotation method. The agreement of their data with those appearing in this paper, together with the method they used for preparing their material, makes it clear that they were working with alpha d-glucose.

<sup>1</sup> Sponsler and Dore. "Fourth Colloid Symposium Monograph," The Chemical Catalog Co., New York, 1926, pp. 174–202; Meyer and Mark, *Ber.*, 61, 593–614 (1928).

 $^2$  The thanks of the authors are due to Mr. T. C. Broyer, of the Plant Nutrition Laboratory, for preparing these two forms.

<sup>3</sup> Hudson and Dale, THIS JOURNAL, 39, 320 (1917).

<sup>4</sup> Hudson and Yanovsky, *ibid.*, 39, 1013 (1917).

<sup>6</sup> Hengstenberg and Mark, Z. Krist., 72, 301 (1929).