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## Fifth Report of the Committee on Atomic Weights of the International Union of Chemistry

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During the year the Committee has suffered an irreparable loss in the death of Mme. P. Curie, Professor in the Faculty of Sciences of the University of Paris.

The following report of the Committee covers the twelve-month period, September 30, 1933, to September 30, 1934.<sup>1</sup>

Only one change has been made in the table of atomic weights,<sup>2</sup> from 93.3 to 92.91 in the case of columbium (niobium).

**Carbon.**—Batuecas<sup>3</sup> has determined the density of propylene at 0° and several pressures. Propylene was prepared by three methods: (1) action of phosphorus pentoxide on normal propyl alcohol, (2) catalytic dehydration of isopropyl alcohol by aluminum phosphate at 250–300°, (3) catalytic dehydration of propyl alcohol by active aluminum oxide at 270–300°. Chemical purification was followed by fractional distillation. Since the first method yielded a product difficult to purify, density determinations were made only with gas prepared by the second and third methods.

(1) Authors of papers bearing on the subject are requested to send copies to each of the four members of the Committee at the earliest possible moment: Prof. G. P. Baxter, Coolidge Laboratory, Harvard University, Cambridge, Mass., U. S. A.; Prof. O. Hönigschmid, Sofienstrasse 9/2, Munich, Germany; Prof. P. LeBeau, Faculté de Pharmacie, 4 Avenue de l'Observatoire, Paris VI<sup>e</sup>, France; Prof. R. J. Meyer, Sigismundstr. 4, Berlin W 10, Germany.

(2) In the report of the Committee for 1933 as published in THIS JOURNAL the atomic weight of erbium is erroneously given in the table of atomic weights as 165.20 instead of the older value 167.64. The decision of the Committee to make no change in the atomic weight of this element was made too late for correction of the American version. This error does not appear elsewhere.

(3) Batuecas, *J. chim. phys.*, **31**, 165 (1934).

Values in the following table are calculated to 760 mm. ( $g = 980.616$ ).

DENSITY OF PROPYLENE				
1 atmosphere				
Method of preparation	Globe G. 1007.55 ml.	Globe N-3 772.58 ml.	Average	
2	1.9140	1.9148	1.9144	
	1.9147	1.9147	1.9147	
	1.9148	1.9158	1.9153	
	1.9145	1.9149	1.9147	
	1.9156		1.9156	
	1.9142	1.9146	1.9144	
	Average	1.9146	1.9150	1.9148
3	1.9154	1.9149	1.9152	
	1.9156	1.9151	1.9153	
	1.9144	1.9153	1.9148	
	1.9145	1.9147	1.9146	
	Average	1.9149	1.9151	1.9150
Average of all		1.9148	1.9150	1.9149
2/3 atmosphere				
2	1.9003	1.9021	1.9012	
	1.9031	1.9031	1.9031	
	1.9016	1.9030	1.9023	
	Average	1.9017	1.9027	1.9022
3	1.9027	1.9011	1.9019	
	1.9023	1.9017	1.9020	
	Average	1.9025	1.9014	1.9020
Average of all		1.9020	1.9022	1.9021
1/2 atmosphere				
2	1.8955	1.8959	1.8957	
	1.8961		1.8961	
	Average	1.8958	1.8959	1.8958
3	1.8954	1.8955	1.8955	
	1.8963	1.8946	1.8955	
	Average	1.8959	1.8951	1.8955
Average of all		1.8958	1.8953	1.8956

On the assumption that the change of  $PV$  with  $P$  is a linear one, the following values are calculated:

$$1 + \lambda = 1.0204$$

$$C_3H_8 = 42.062$$

$$C = 12.005$$

**Carbon and Nitrogen.**—Moles and Salazar<sup>4</sup> have redetermined the densities of oxygen, carbon monoxide and nitrogen using the same (improved) apparatus for all three gases.

Oxygen was prepared by pyrolysis of (a) potassium permanganate and (b) potassium and sodium chlorates with manganese dioxide. Chemical purification was followed by fractional distillation.

Carbon monoxide was prepared (a) from potassium ferrocyanide and sulfuric acid and (b) from formic acid and concentrated sulfuric acid. Chemical and physical purification followed.

Nitrogen, obtained from sodium nitrite and ammonium sulfate, was subjected to chemical purification.

#### THE DENSITY OF OXYGEN

760 mm.			
Method	Globe G, 987.59 ml.	Globe N, 992.04 ml.	Average
a	1.42895	1.42899	1.42897
a	1.42892	1.42897	1.42894
a	1.42903	1.42887	1.42895
a	1.42896	1.42894	1.42895
a	1.42893	1.42897	1.42895
b	1.42897	1.42894	1.42895
b	1.42897	1.42894	1.42895
Average	1.42896	1.42895	1.42895

  

380 mm.			
a	1.42832	1.42827	1.42829
a	1.42826	1.42835	1.42830
a	1.42836	1.42831	1.42833
a	1.42830	1.42841	1.42835
a	1.42830	1.42835	1.42832
b	1.42820	1.42833	1.42826
b	1.42832	1.42829	1.42830
Average	1.42829	1.42833	1.42831

#### THE DENSITY OF CARBON MONOXIDE

760 mm.			
Method	Globe G	Globe N	Average
a	1.24998	1.25000	1.24999
a	1.25012	1.25001	1.25006
a	1.24999	1.25001	1.25000
a	1.25000	1.24998	1.24999
b	1.25001	1.25001	1.25001
b	1.24998	1.25001	1.24999
b	1.25003	1.24998	1.25000
b	1.25002	1.25000	1.25001
Average	1.25002	1.25000	1.25001

(4) Moles and Salazar, *Anales soc. españ. fis. quim.*, **32**, 954 (1934).

380 mm.			
a	1.24978	1.24977	1.24977
a	1.24970	1.24975	1.24972
a	1.24978	1.24979	1.24978
a	1.24976	1.24977	1.24976
a	1.24978	1.24971	1.24974
a	1.24982	1.24975	1.24978
b	1.24974	1.24970	1.24972
b	1.24976	1.24973	1.24974
b	1.24974	1.24975	1.24974
Average	1.24976	1.24975	1.24975

#### THE DENSITY OF NITROGEN

760 mm.		
Globe G	Globe N	Average
1.25049	1.25048	1.25049

  

380 mm.		
1.25020	1.25023	1.25022

From these data are calculated the following values.

	Limiting density	$1 + \lambda$	Molecular weight
Oxygen	1.42767	1.00090	
Carbon monoxide	1.24950	1.00040	28.006(5)
Nitrogen	1.24995	1.00043	28.0165

The atomic weights of carbon and nitrogen, respectively, are thus found to be 12.006(5) and 14.0083.

**Nitrogen.**—Moles and Sancho<sup>5</sup> dispute the claim of Dietrichson, Bircher and O'Brien that very dry glass surfaces adsorb little or no ammonia and attempt to correct the results of these authors and those of Dietrichson, Orleman and Rubin on the density of ammonia by using the adsorption data of Moles and Crespi, and of Crespi and Aleixandre.

They have also redetermined the density of ammonia at 0° and at one atmosphere and one-half atmosphere using a volumeter method, in which the weight of ammonia is found by gain in weight of a receptacle containing sulfuric acid.

Synthetic ammonia was dried with potassium hydroxide and barium oxide and was fractionally distilled from liquid sodium-potassium amalgam.

Improved apparatus was employed. The barometer with its reservoir was maintained at 0° so that no temperature correction was necessary. The volumes of the globes were 5566.52 (G) and 2378.59 (P) ml. Residual pressures in the globes were from 0.3 to 41 mm. and the differences between residual and total pressures from 736 to 769 mm. Corrections for the compressibility of ammonia were therefore made, as well as for ad-

(5) Moles and Sancho, *ibid.*, **33**, 931 (1934).

sorption on the basis of the work of Moles and Crespi.

	Globe G	Globe P	Globes G + P
	1 atmosphere		
	0.77143	0.77145	
	.77131	.77150	
	.77152	.77133	
	.77137	.77150	
Average	.77141	.77144	
Average of all		.77143 <sup>a</sup>	
	$\frac{1}{2}$ atmosphere		
0.76558	0.76558		0.76572
			.76574
			.76540
			.76570
Average	0.76558	0.76558	.76564
		Average of all	.76562

<sup>a</sup> Incorrectly calculated in the paper.

The limiting density of ammonia is calculated on the assumption, which Moles and Sancho prefer, that the compressibility is a linear function of the pressure. The normal molal volume is assumed to be 22.4142 liters. The results on this basis are

Limiting Density	0.75981
Mol. wt. NH <sub>3</sub>	17.0305
At. wt. N	14.007

The authors find 14.008 for nitrogen, but this value involves two arithmetical errors, one in the average density at one atmosphere and one in calculating the molecular weight of ammonia.

**Sodium.**—Johnson<sup>6</sup> has redetermined the ratio of sodium chloride to silver. Sodium chloride was

fusion atmosphere in most cases was air but in analyses 3 and 4 nitrogen was used and in analysis 6 it was hydrogen chloride and nitrogen. A fifth normal solution of the sodium chloride was then precipitated with a fifth normal solution of a very nearly equivalent quantity of the purest silver and the end-point of the comparison was found at 0° with a nephelometer by the "standard solution" method recently developed by Johnson, and also in some cases potentiometrically. In half the analyses the silver solution was added to the chloride, in half the reverse method of precipitation was employed. Finally the silver chloride was collected, dried at 300° and weighed. Dissolved silver chloride was estimated nephelometrically. Loss in weight of the silver chloride on fusion was not determined. Weights are corrected to vacuum.

The fifth analysis was rejected on the basis of disagreement, the sixth analysis on the basis of acidity of the sodium chloride, the ninth and tenth on the basis of impurity in the sodium chloride, and the first analysis for silver chloride on the basis of a known uncertainty.

The final result for sodium is 0.003 lower than the International value.

**Calcium.**—Smith and Tait<sup>7</sup> have compared calcium extracted from geologically old, potassium rich, calcium poor minerals with common calcium. Pegmatites from Portsoy, Banffshire, Scotland, and Rhiconich, Sutherlandshire, Scotland, were extracted with hydrochloric acid. About 30% of the original calcium was thus ob-

THE ATOMIC WEIGHT OF SODIUM

Fraction of NaCl	NaCl, g.	Ag, g.	Ratio NaCl: Ag	At. wt. Na	AgCl, g.	Ratio NaCl: AgCl	At. wt. Na
1	5.48994	10.13246	0.541817	22.994	13.46165	(0.407821)	(22.999)
1	5.43976	10.03981	.541819	22.994	13.33993	.407780	22.993
2	5.48976	10.13223	.541812	22.994	13.46231	.407787	22.994
2	5.47090	10.09724	.541821	22.995	13.41552	.407804	22.996
3	5.42144	10.00502	(.541872)	(23.000)	13.29327	(.407833)	(23.001)
3	5.4360	10.0340	(.54176)	(22.988)	13.3307	(.407780)	(22.993)
4	5.59192	10.32064	.541819	22.994	13.71263	.407793	22.995
4	5.43087	10.02352	.541813	22.994	13.31763	.407796	22.995
5	5.51668	10.18218	(.541798)	(22.992)	13.52883	(.407772)	(22.992)
5	5.42755	10.01778	(.541792)	(22.992)	13.31017	(.407775)	(22.992)
		Average	.541817	22.994		.40779	22.994

very carefully purified by precipitation with hydrogen chloride and crystallization. During the last crystallization it was divided into five fractions. The sodium chloride was fused in a weighed platinum crucible contained in an electrically heated quartz muffle and weighed. The

tained. The purification consisted in five precipitations as oxalate, conversion to nitrate through the oxide, electrolysis of the nitrate solution, five crystallizations of the nitrate, three precipitations as carbonate, conversion to chloride through the oxide and one crystallization as

(6) Johnson, *J. Phys. Chem.*, **37**, 923 (1933).

(7) Smith and Tait, *Proc. Roy. Soc. Edinburgh*, **54**, 88 (1934).

chloride (A). In each case a second sample was similarly prepared from the mother liquors of the third nitrate crystallization (B). For comparison two preparations were made in a similar way from marine shells from Fifeshire, Scotland, and coral limestone from Bermuda.

The calcium chloride was prepared for weighing by preliminary dehydration in a vacuum at low temperature and finally fusion in an atmosphere of nitrogen and hydrogen chloride in a "bottling apparatus." After solution of the weighed salt a correction was determined for deviation from neutrality and then the solution was compared with pure silver in the usual way.

THE ATOMIC WEIGHT OF CALCIUM

Sample	CaCl <sub>2</sub> , g.	Ag, g.	Ratio CaCl <sub>2</sub> :2Ag	At. wt. Ca
Coral A	1.77727	3.45491	0.514419	40.077
	1.82493	3.54743	.514437	40.081
	1.81606	3.53030	.514421	40.077
	1.74543	3.39311	.514404	40.074
	1.75771	3.41695	.514409	40.075
		Average	.514418	40.077
Marine A	2.02793	3.94230	0.514403	40.074
	2.09760	4.07770	.514408	40.075
Shells A	2.23959	4.35369	.514412	40.076
	2.18660	4.25062	.514419	40.077
B	2.13950	4.15897	.514430	40.079
	2.04200	3.96947	.514426	40.079
		Average	.514416	40.076
Portsoy B	1.70015	3.30465	0.514472	40.088
	1.78487	3.46928	.514479	40.090
A	1.74779	3.39721	.514478	40.090
	1.69538	3.29562	.514434	40.080 <sup>a</sup>
B	1.87366	3.64188	.514476	40.089
	2.07253	4.02853	.514463	40.087
		Average	.514468	40.087
Rhiconich B	1.94278	3.77612	0.514491	40.093
	1.98782	3.86364	.514494	40.093
A	1.88677	3.66715	.514505	40.096
	1.96884	3.82687	.514478	40.090
B	2.19803	4.27222	.514494	40.093
	2.17033	4.21850	.514479	40.090
		Average	.514490	40.092

<sup>a</sup> Incorrectly calculated in the original paper.

The atomic weight found for common calcium is not far from that found by Richards and Hönigschmid (40.074) but is lower than that found by Hönigschmid and Kempter (40.084). Some uncertainty exists as to the vacuum corrections for calcium chloride and silver applied by Smith and Tait, who took into consideration the densities of both the brass and the aluminum weights. If this was not done also in the standardization of the weights the vacuum corrections as applied are incorrect. The possible error in

the atomic weight of calcium would vary with the proportion of fractional weights employed and in the experiments of Smith and Tait may amount to from +0.008 to -0.007.

The differences in the atomic weight of calcium from the different sources are used to calculate the half life of K<sup>41</sup> (see Fourth Report of this Committee).

**Krypton.**—Heuse and Otto<sup>8</sup> have redetermined the density of krypton. The gas was carefully purified first with hot calcium, then by fractional distillation. The coefficient of compressibility was found at 0° to be -0.00279 and the temperature coefficient  $0.0036609 + 358 \times 10^{-7} p$  (meters). With a 267.53-ml. globe the following results were obtained, using the density of oxygen 1.42900 and the compressibility coefficient -0.00094.

THE DENSITY OF KRYPTON

Weight, g.	Pressure, mm.	Temp., °C.	Density	At. wt. Kr
0.83479	681.84	20.60	3.7432	83.668
.85258	684.27	15.50	3.7428	83.659
.84484	681.32	16.87	3.7427	83.657
.88017	715.13	19.11	3.7433	83.670
		Average	3.743	83.66

**Columbium (Niobium).**—Hönigschmid and Wintersberger<sup>9</sup> have determined the ratio of columbium pentachloride to silver. Potassium columbium oxyfluoride was fractionally crystallized from hydrochloric acid solution with rejection of the most soluble and the least soluble (containing tantalum) fractions, until the head fraction gave no evidence of tantalum upon x-ray analysis (Prandtl and v. Hevesy). The product was converted to sodium columbate and residual iron was removed with ammonium sulfide. Next columbic acid was precipitated with sulfurous acid, washed and ignited. Further extraction with hydrochloric acid and water followed. This material still contained titanium and a trace of zirconium (Sample I).

Purified columbic acid, prepared by Fetkenheuer but still containing 0.1% of tantalum, was converted to the potassium columbic oxyfluoride and this substance was fractionally crystallized until free from tantalum. Zirconium was removed by fusion with potassium carbonate and extraction, and iron was removed as sulfide. Columbic acid was then precipitated with salicylic

(8) Heuse and Otto, *Physik. Z.*, **35**, 57 (1934).

(9) Hönigschmid and Wintersberger, *Z. anorg. allgem. Chem.*, **219**, 161 (1934).

acid to eliminate titanium, and the precipitate was ignited to oxide (Sample II).

A portion of Sample I was freed from zirconium as in the case of Sample II (Sample III).

Solutions obtained in the course of the analyses were freed from silver by precipitation as chloride and from oxalic acid by ignition. The columbic acid was recovered from alkaline solution by precipitation with sulfur dioxide (Sample IV).

Columbium pentachloride was prepared by treatment of the sulfide with chlorine. To prepare the sulfide, hydrogen sulfide charged with carbon disulfide was conducted over the columbic oxide at 1000°. The pentachloride was then produced by passing a current of chlorine, carefully freed from oxygen, into a nitrogen-filled glass bulb containing the sulfide and warming to 250°. Fractional distillation of the pentachloride in an exhausted glass system followed and the samples for analysis were sealed off in glass bulbs. Considerable difficulty was experienced at first in eliminating the higher boiling oxychloride on the one hand and the lower boiling sulfur chloride on the other, but this difficulty was eventually overcome.

The weight of the pentachloride was found by weighing the bulb and then, after breaking the bulb under saturated oxalic acid solution and acidification with nitric acid, the glass fragments were collected and weighed. Comparison with silver followed. All weights are corrected to vacuum.

THE ATOMIC WEIGHT OF COLUMBIUM

Sample	CbCl <sub>5</sub> , g.	Ag, g.	Ratio CbCl <sub>5</sub> :5Ag	At. wt. Cb
II	3.04386	6.07711	0.50087	92.886
II	4.15231	8.28917	.50093	92.918
II	3.04987	6.08879	.50090	92.900
II	2.50957	5.00994	.50092	92.910
II	1.20611	2.40770	.50094	92.921
III	0.79147	1.57998	.50094	92.920
III	1.44972	2.89407	.50093	92.915
III	0.94171	1.87985	.50095	92.927
II	.79636	1.58976	.50093	92.917
II	1.03393	2.06422	.50088	92.891
II	1.64281	3.27968	.50091	92.903
IV	1.13889	2.27376	.50088	92.892
IV	2.48870	4.96803	.50094	92.924
IV	2.27453	4.54070	.50092	92.912
			.50092	92.909

The average value of the atomic weight of columbium, 92.91, agrees almost exactly with Aston's finding that columbium is probably a simple element with an atomic weight 92.90 on

the chemical scale. The value 92.91 replaces in the table the older value 93.3 which was a compromise value from earlier determinations.

**Molybdenum.**—Lautié<sup>10</sup> has determined the ratio of molybdenum to its trioxide in two ways. Impure trioxide was converted to the volatile chlorohydrine by heating in a current of dry hydrogen chloride, with rejection of the first and last portions. The product was dissolved in ammonia and the solution evaporated to dryness. Nitric acid was then added and after heating to 400° in oxygen the residual molybdenum trioxide was five times resublimed. Portions of about 1.5 g. were heated to constant weight in a quartz boat, and then gradually to 800° in a current of active hydrogen. The resulting metal was weighed in an atmosphere of argon or nitrogen.

In this way the ratio Mo/MoO<sub>3</sub> was found to be 0.66669. By reoxidation of the molybdenum the value 0.66668 was found for the same ratio. The former corresponds to the atomic weight 96.01 the latter to 96.02.

**Iodine, Carbon, Sodium.**—Baxter and Hale<sup>11</sup> have compared sodium carbonate and iodine pentoxide by neutralization. Sodium carbonate was purified by repeated crystallization and was prepared for weighing by fusion in an atmosphere of carbon dioxide. Iodic acid was prepared from purified iodine, through potassium and barium iodates, and was fractionally crystallized until free from non-volatile residue. By gradual heating to 240° the iodic acid was dehydrated to iodine pentoxide and weighed. Nearly equivalent amounts of the two substances were allowed to react in dilute aqueous solution and the resulting solution was brought to neutrality in an atmosphere free from carbon dioxide, with bromophenol-blue as indicator, by adding very dilute acid and base solutions. Weights are corrected

THE RATIO OF IODINE PENTOXIDE TO SODIUM CARBONATE

Na <sub>2</sub> CO <sub>3</sub> , g.	I <sub>2</sub> O <sub>5</sub> , g.	Ratio I <sub>2</sub> O <sub>5</sub> :Na <sub>2</sub> CO <sub>3</sub>
8.98436	28.29617	3.14949
9.56078	30.11314	3.14965
7.55451	23.79256	3.14945
9.33412	29.39833	3.14956
8.46160	26.65025	3.14955
9.29771	29.28312	3.14950
9.70811	30.57533	3.14946
8.42645	26.53892	3.14948
8.81736	27.76915	3.14937
	Average	3.14950

(10) Lautié, *Compt. rend.*, **197**, 1730 (1933).

(11) Baxter and Hale, *THIS JOURNAL*, **56**, 615 (1934).

to vacuum and corrections of  $-0.001\%$  for adsorbed air and of  $-0.0023\%$  for retained moisture are applied to the weights of iodine pentoxide.

In the following tables the atomic weights of iodine, carbon and sodium are computed individually on the basis of various assumed atomic weights for the other two elements.

THE ATOMIC WEIGHT OF IODINE

	C = 12.000	C = 12.005	C = 12.010
Na = 22.997	126.914	126.922	126.930
Na = 22.994	126.905	126.913	126.920

THE ATOMIC WEIGHT OF CARBON

	I = 126.915	I = 126.917	I = 126.920	I = 126.925
Na = 22.997	12.001	12.002	12.004	12.007
Na = 22.994	12.007	12.008	12.010	12.013

THE ATOMIC WEIGHT OF SODIUM

	I = 126.915	I = 126.917	I = 126.920	I = 126.925
C = 12.000	22.997	22.998	22.999	23.000
C = 12.005	22.995	22.996	22.996	22.998
C = 12.010	22.992	22.993	22.994	22.995

**Cesium.**—Baxter and Thomas<sup>12</sup> have continued work on the analysis of cesium chloride, the preliminary results of which were included in the report of this Committee for 1933. Nearly four kilograms of cesium nitrate were fractionally crystallized. The nine less soluble fractions of the last (seventeenth) series, amounting to 80% of the total material, when tested spectroscopically showed no evidence of the presence of potassium or rubidium. A portion of the combined fourth and fifth fractions was converted to perchlorate and recrystallized. The perchlorate was then converted to chloride, which was recrystallized (Sample A). The mother liquors of Sample A were converted to dichloriodide and after recrystallization this salt was converted to chloride and purified as before (Sample B). Sample C was prepared from the next to least soluble of the nitrate fractions by several crystallizations as perchlorate and one as chloride. The least soluble of the nitrate fractions was recrystallized three times as perchlorate, and converted to chloride (Sample D).

To prepare the chloride for weighing it was fused in a platinum boat in various atmospheres as indicated in the table. Analysis followed the conventional method of comparison with silver. Weights are corrected to vacuum.

The value 132.91 was adopted for the International Table of 1933.

(12) Baxter and Thomas, *THIS JOURNAL*, **56**, 1108 (1934).

THE ATOMIC WEIGHT OF CESIUM					
Sam- ple	Fusion atmosphere	Wt. of CsCl	Wt. of Ag	Ratio CsCl:Ag	At. wt. Cs
A	N <sub>2</sub>	8.96291	5.74296	1.56068	132.909
A	N <sub>2</sub>	9.60983	6.15731	1.56072	132.913
A	H <sub>2</sub> + 25% HCl	9.70288	6.21686	1.56074	132.916
A	H <sub>2</sub> + 25% HCl	9.53125	6.10698	1.56071	132.912
A	H <sub>2</sub>	7.52304	4.82025	1.56072	132.913
B	H <sub>2</sub> + 75% HCl	9.38363	6.01254	1.56068	132.909
B	H <sub>2</sub> + 20% HCl	9.33593	5.98207	1.56065	132.906
B	H <sub>2</sub> + trace HCl	10.83528	6.94414	1.56035	132.874
B	N <sub>2</sub>	7.70022	4.93415	1.56060	132.901
B	H <sub>2</sub> + trace HCl	7.78342	4.98770	1.56052	132.892
B	H <sub>2</sub> + 50% HCl	7.83724	5.02201	1.56058	132.898
C	H <sub>2</sub> + 35% HCl	5.78546	3.70748	1.56048	132.888
D	H <sub>2</sub> + 20% HCl	2.16882	1.38968	1.56066	132.907
C	H <sub>2</sub> + 35% HCl	4.02114	2.57655	1.56067	132.908
				Average	1.56063 132.903
				Average excluding Analysis 8	1.56065 132.906
				Average of Analyses 1-7	1.56070 132.911

**The Rare Earths.**—Aston<sup>13</sup> has determined the isotopic composition and atomic weights of the rare earths with the following results.

	Mass spec- trum values	International values	Difference
Lanthanum	138.91	138.92	-0.01
Cerium	140.13	140.13	.00
Praseodymium	140.91	140.92	-.01
Neodymium	143.5	144.27	-.8
Samarium	150.1	150.43	-.3
Europium	151.90	152.0	-.1
Gadolinium	156.9	157.3	-.4
Terbium	158.91	159.2	-.3
Dysprosium	162.5	162.46	.0
Holmium	164.91	163.5	+1.4
Erbium	167.15	167.64	-0.5
Thulium	168.91	169.4	-.5
Ytterbium	173.2	173.04	+.2
Lutecium	174.91	175.0	-.1

While the agreement of the physical with the chemical value is in many cases as close as could be expected, in others the discrepancy is glaring. To explain the larger differences on the basis of impurity in the rare earth material used in the chemical determinations improbably large proportions of impurity would be required. The conventional chloride analysis used for a large proportion of the chemical determinations in

(13) Aston, *Proc. Roy. Soc. (London)*, **A146**, 46 (1934).

## INTERNATIONAL ATOMIC WEIGHTS

1935

	Symbol	Atomic Number	Atomic Weight		Symbol	Atomic Number	Atomic Weight
Aluminum	Al	13	26.97	Molybdenum	Mo	42	96.0
Antimony	Sb	51	121.76	Neodymium	Nd	60	144.27
Argon	A	18	39.944	Neon	Ne	10	20.183
Arsenic	As	33	74.91	Nickel	Ni	28	58.69
Barium	Ba	56	137.36	Nitrogen	N	7	14.008
Beryllium	Be	4	9.02	Osmium	Os	76	191.5
Bismuth	Bi	83	209.00	Oxygen	O	8	16.0000
Boron	B	5	10.82	Palladium	Pd	46	106.7
Bromine	Br	35	79.916	Phosphorus	P	15	31.02
Cadmium	Cd	48	112.41	Platinum	Pt	78	195.23
Calcium	Ca	20	40.08	Potassium	K	19	39.096
Carbon	C	6	12.00	Praseodymium	Pr	59	140.92
Cerium	Ce	58	140.13	Radium	Ra	88	225.97
Cesium	Cs	55	132.91	Radon	Rn	86	222
Chlorine	Cl	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	92.91	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	78.96
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	Ta	73	181.4
Hafnium	Hf	72	178.6	Tellurium	Te	52	127.61
Helium	He	2	4.002	Terbium	Tb	65	159.2
Holmium	Ho	67	163.5	Thallium	Tl	81	204.39
Hydrogen	H	1	1.0078	Thorium	Th	90	232.12
Indium	In	49	114.76	Thulium	Tm	69	169.4
Iodine	I	53	126.92	Tin	Sn	50	118.70
Iridium	Ir	77	193.1	Titanium	Ti	22	47.90
Iron	Fe	26	55.84	Tungsten	W	74	184.0
Krypton	Kr	36	83.7	Uranium	U	92	238.14
Lanthanum	La	57	138.92	Vanadium	V	23	50.95
Lead	Pb	82	207.22	Xenon	Xe	54	131.3
Lithium	Li	3	6.940	Ytterbium	Yb	70	173.04
Lutecium	Lu	71	175.0	Yttrium	Y	39	88.92
Magnesium	Mg	12	24.32	Zinc	Zn	30	65.38
Manganese	Mn	25	54.93	Zirconium	Zr	40	91.22
Mercury	Hg	80	200.61				

many of the more favorable cases has given results agreeing closely with the physical results (lanthanum, cerium, praseodymium), so that there is no evidence against the analytical method. If the chemical result is in error, in certain cases the difficulty may have been due to abnormal composition of the trichlorides analyzed. It is noticeable that the discrepancies are large with many of the more complex elements (neodymium, samarium, gadolinium and erbium) so that it seems far from certain that these determinations of mean mass with the mass spectrograph are

free from error. The Committee feels that without further evidence it is impossible to decide where the difficulty lies and makes no changes in the values for this group of elements in the table.

**Tantalum.**—Hönigschmid<sup>14</sup> has analyzed tantalum pentachloride, purified by sublimation in vacuum. The result for the atomic weight of tantalum, 180.89, agrees exactly with Aston's value, and is lower than the present International value. Details of this investigation were pub-

(14) Hönigschmid, *Naturwiss.*, **22**, 463 (1934).

lished too late for inclusion in the report for this year [*Z. anorg. allgem. Chem.*, **221**, 129 (1935)].

**Lead.**—Marble<sup>15</sup> has determined the atomic weight of radiogenic lead extracted from pitchblende found near Great Bear Lake, North West Territories, Canada. The lead was purified successively as sulfide, sulfate, sulfide, nitrate and chloride and the chloride was finally twice distilled in dry hydrogen chloride. Analysis by the conventional chloride method gave the following results. Weights are corrected to vacuum.

THE ATOMIC WEIGHT OF LEAD			
Wt. of PbCl <sub>2</sub>	Wt. of Ag	Ratio PbCl <sub>2</sub> :2Ag	At. wt. Pb
2.27721	1.77393	1.283709	206.059
2.22354	1.73219	1.283658	206.048
2.22397	1.73249	1.283684	206.054
	Average	1.283684	206.054

The value found by Aston from the isotopic constitution of this same lead, when corrected to the chemical scale, is 206.08. The difference is no greater than present accuracy of the mass spectrograph but is greater than the apparent uncertainty of the chemical method.

(15) Marble, *THIS JOURNAL*, **56**, 854 (1934).

**Radium and Protactinium.**—Two investigations on radioactive elements were published too late for detailed inclusion in this report. Hönigschmid and Sachtleben<sup>16</sup> using 2.2–3.5 g. of radium bromide, from the ratio RaBr<sub>2</sub>:RaCl<sub>2</sub> find the atomic weight of radium to be 226.05, while v. Grosse,<sup>17</sup> from the ratio 2K<sub>2</sub>PaF<sub>7</sub> (0.07–0.09 g.): Pa<sub>2</sub>O<sub>3</sub> finds Pa = 230.6.

Batuecas<sup>18</sup> takes exception to the conclusions of Cawood and Patterson concerning the densities of certain gases and their coefficients of deviation from Boyle's law. His own figures based on average values obtained by several experimenters are:

	Density	1 + λ	M	
C <sub>2</sub> H <sub>4</sub>	1.2605 (4)	1.0076 (6)	28.040	C = 12.004
CO <sub>2</sub>	1.9766 (5)	1.0068 (4)	44.005	C = 12.005
(CH <sub>3</sub> ) <sub>2</sub> O	2.1099	1.0270	46.050	C = 12.001
				(5)
N <sub>2</sub> O	1.9780	1.0073 (5)	44.013	N = 14.0065
SO <sub>2</sub>	2.9263	1.0239	64.061	S = 32.061

(16) Hönigschmid and Sachtleben, *Z. anorg. allgem. Chem.*, **221**, 65 (1935).

(17) A. V. Grosse, *THIS JOURNAL*, **56**, 2501 (1934).

(18) Batuecas, *J. chim. phys.*, **31**, 65 (1934).

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[CONTRIBUTION FROM THE MACKAY CHEMICAL LABORATORY OF THE UNIVERSITY OF NEVADA]

## The Reaction Tungstic Oxide–Carbon–Chlorine<sup>1</sup>

BY GEO. W. SEARS AND FRED LOHSE

The use of chlorine as an extraction agent for certain of the rarer metals from their ores has received the attention of investigators at various times and several processes utilizing the reaction have been patented.<sup>2</sup> Except for the recovery of tungsten from certain tin ores in England, little commercial use of the reaction has apparently been made.<sup>3</sup> Of the different methods described, as applicable for tungsten ores, that involving the reaction of chlorine on the ore mixed with carbon or other reducing agent appeared to be the most promising. Little information, however, could be obtained as to the nature of the reaction. While it seems to have been generally assumed that the oxychlorides, WOCl<sub>4</sub> and WO<sub>2</sub>Cl<sub>2</sub>, are formed and that the carbon acts as a reducing

agent,<sup>4</sup> but little experimental evidence supporting it could be found. It was decided, therefore, to make a more careful study of the nature of the reaction and especially of the role of carbon in it with the hope that information might be obtained that would lead to its more general use.

The yellow c. p. tungstic oxide of commerce was used as a source of tungsten. Except in the preliminary experiments, the chlorine was from a cylinder of the commercial liquid. The carbon was in the form of pulverized wood charcoal or lampblack. The reaction was carried out in an electric tube furnace arranged in such a way that the progress of the reaction could be observed. The mixture of tungstic oxide and carbon was placed in a porcelain boat within the tube and dry chlorine passed over it. The solid reaction products were collected in a series of bulbs which had previously been sealed directly to the furnace tube. The mixed red and yellow colors of the distillation products pointed quite definitely to the formation of both WO<sub>2</sub>Cl<sub>2</sub> and WOCl<sub>4</sub> with possibly some WCl<sub>6</sub>. The mixture, however, was subjected to a process of fractional

(1) Presented before the Pacific Intersectional Division of the American Chemical Society at Salt Lake City, June, 1933.

(2) British Patents 176,428 and 134,891.

(3) Colin J. Smithells, "Tungsten," D. Van Nostrand Co., New York, p. 14.

(4) British Patent 134,891.