JOURNAL OF THE
AMERICAN CHEMICAL SOCIETY

VOLUME 60 APRIL 8, 1938 NUMBER 4

Eighth Report of the Committee on Atomic Weights of the International Union of Chemistry

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The following report of the Committee covers the twelve-month period, September 30, 1936, to September 30, 1937.[1] The following changes in the table of atomic weights have been made.

Hydrogen, from 1.0078 to 1.0081
Helium, from 4.002 to 4.003
Carbon, from 12.01 to 12.010
Molybdenum, from 96.0 to 95.95
Erbium, from 167.64 to 167.2
Tungsten, from 184.0 to 183.92
Osmium, from 191.5 to 190.2

Hydrogen and Helium.—Mass spectroscopic values for these elements obtained by Aston[2] and by Bainbridge and Jordan[3] based on the physical scale are as follows

	Aston	Bainbridge and Jordan
H^1	1.00812	1.00813
He	4.00391	4.00389

On the chemical scale these become H^1 = 1.00785 and He = 4.00285 (conversion factor 1.00027). Allowance for H^2, with the abundance ratio 1/5000, gives 1.00805 for chemical hydrogen. The values 1.0081 and 4.003 for hydrogen and helium have been adopted for the Table, since they seem more reliable than those obtained by other methods.

Carbon.—Baxter and Hale[4] have continued the quantitative combustion of heavy hydrocarbons, with certain improvements in technique. Chrysene used previously (I) was further purified (II) and a third sample was synthesized from naphthalene (III). Triphenylbenzene used previously (II) was further purified (III) and a new sample was synthesized from benzaldehyde (from amygdalin) (IV). A new sample of anthracene was purified as before (II) and a third sample was synthesized from phthalic acid and benzene (III). As previously reported the purification of pyrene was not successful. The old as well as the new results are corrected for small increases in weight of the absorption tubes when gases were passed through the combustion train in the absence of any hydrocarbon. The earlier results are further corrected for a small error in weight calibration. Results are calculated with H = 1.0078. The use of the more probable value 1.0081 for hydrogen gives a value for carbon only 0.0003 lower.

The average atomic weight of carbon is 0.0013 higher than the preliminary value (see Seventh Report of this Committee).

Carbon.—Scott and Hurley[5] have determined the atomic weight of carbon by comparison of benzoyl chloride with silver. Thiophene-free toluene was oxidized to benzoic acid by means of potassium permanganate. The product was twice precipitated with hydrochloric acid, re-

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[1] Authors of papers bearing on the subject are requested to send copies to each of the three 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^e, France.

[2] Aston, *Nature*, **137**, 357, 613 (1936).

[3] Bainbridge and Jordan, *Rev. Modern Phys.*, **9**, 370 (1937).

[4] Baxter and Hale, THIS JOURNAL, **59**, 506 (1937).

[5] Scott and Hurley, *ibid.*, **59**, 1905 (1937).

ATOMIC WEIGHT OF CARBON

Sample	Hydrocarbon, g.	H_2O, g.	H, g.	C, g.	CO_2, g.	O, g.	Ratio C: O_2	At. wt. of C
				Chrysene				
I	2.78052	1.31192	0.14678	2.63374	9.65237	7.01863	0.375250	12.008(0)
I	2.69266	1.27591	.14275	2.54991	9.34366	6.79375	.375332	12.010(6)
I	2.97790	1.41044	.15780	2.82010	10.33440	7.51430	.375298	12.009(5)
I	2.99659	1.41906	.15877	2.83782	10.39868	7.56086	.375330	12.010(6)
II	3.01102	1.42558	.15950	2.85152	10.44739	7.59587	.375404	12.012(9)
II	2.97646	1.40901	.15764	2.81882	10.32819	7.50937	.375374	12.012(0)
II	2.97260	1.40723	.15744	2.81516	10.31566	7.50050	.375330	12.010(6)
II	1.56689	0.74145	.08295	1.48394	5.43767	3.95373	.375327	12.010(5)
III	3.08222	1.45976	.16332	2.91890	10.69608	7.77718	.375316	12.010(1)
III	2.07420	0.98195	.10986	1.96434	7.19819	5.23385	.375315	12.010(1)
						Average	.375328	12.010(5)
				Triphenylbenzene				
I	3.00022	1.58994	0.17788	2.82234	10.34128	7.51894	0.375364	12.011(6)
II	2.99781	1.58711	.17757	2.82024	10.33453	7.51429	.375317	12.010(1)
II	2.99647	1.58580	.17742	2.81905	10.33018	7.51113	.375316	12.010(1)
III	3.00284	1.58874	.17775	2.82509	10.35196	7.52687	.375334	12.010(7)
III	6.00641	3.17865	.35563	5.65078	20.70670	15.05592	.375320	12.010(2)
IV	2.99682	1.58563	.17740	2.81942	10.33123	7.51181	.375332	12.010(6)
IV	3.00217	1.58866	.17774	2.82443	10.35028	7.52585	.375297	12.009(5)
IV	2.99844	1.58649	.17750	2.82094	10.33739	7.51645	.375302	12.009(7)
						Average	.375323	12.010(3)
				Anthracene				
I	2.99495	1.51439	0.16943	2.82552	10.35385	7.52833	0.375318	12.010(2)
I	2.04939	1.03659	.11597	1.93342	7.08535	5.15193	.375281	12.009(0)
III	2.87189	1.45078	.16231	2.70958	9.92877	7.21919	.375330	12.010(6)
III	2.95847	1.49521	.16729	2.79118	10.22821	7.43703	.375308	12.009(9)
III	2.88436	1.45742	.16306	2.72130	9.97193	7.25063	.375319	12.010(2)
II	6.06324	3.06430	.34284	5.72040	20.96070	15.24030	.375347	12.011(1)
II	5.44882	2.75414	.30814	5.14068	18.83572	13.69504	.375368	12.011(8)
II	2.81287	1.42365	.15928	2.65359	9.72451	7.07092	.375282	12.009(0)
II	5.54044	2.80123	.31340	5.22704	19.15276	13.92572	.375352	12.011(3)
						Average	.375323	12.010(3)
						Average of all results	.375325	12.010(4)

crystallized from water and sublimed. To prepare benzoyl chloride the benzoic acid was warmed with redistilled phosphorus trichloride. Then the benzoyl chloride was purified by fractional distillation in exhausted all-glass systems. During the later stages of the fractionation samples for analysis were sealed off in glass bulbs. Analysis was effected by first breaking the weighed bulbs under aqueous pyridine and collecting the glass on filters for weighing. The solutions were then compared with weighed nearly equivalent quantities of pure silver by the equal opalescence method. In the following table the fractions are listed in the order of decreasing volatility. The weights of the bulbs and glass as well as of the silver were corrected for the buoyancy of the air during weighing.

The authors reject the result obtained with Fraction 4, which was the last volatile fraction analyzed, since it was suspected to contain hydrogen chloride, and that obtained from Fraction 6 on the score of disagreement. This, however, was the least volatile fraction examined.

If the atomic weight of hydrogen is taken as 1.0081 instead of the current value, that of carbon is lowered only 0.0002.

The final result is in excellent agreement with that of Baxter and Hale (see above) and with

THE ATOMIC WEIGHT OF CARBON

Fraction of benzoyl chloride	Benzoyl chloride, g.	Ag, g.	Ratio C_7H_5OCl: Ag	At. wt. C
4	13.08649	10.04596	1.302662	12.0050
5	13.12011	10.06925	1.302988	12.0100
8	13.09014	10.04617	1.302998	12.0102
11	12.43004	9.53960	1.302994	12.0101
12	13.48239	10.34736	1.302979	12.0099
15	14.70047	11.28211	1.302989	12.0101
16	13.80987	10.59833	1.303023	12.0106
14	10.71976	8.22688	1.303016	12.0105
9	14.61669	11.21768	1.303004	12.0103
6	12.53791	9.62157	1.303104	12.0118
Average, excluding the first and last			1.302999	12.0102

that obtained from mass spectroscopic evidence.

The values for C^{12} found by Aston[6] and by Bainbridge and Jordan[3] are 12.00355 and 12.00398 on the physical scale. Corrected to the chemical scale (conversion factor 1.00027) these become 12.0003 and 12.0007. Even with the abundance ratio for C^{13} as low as 1/100 the atomic weight of carbon becomes 12.0103 and 12.0107.

On the basis of the chemical evidence the value 12.010 has been adopted for the atomic weight of carbon in the International Table but from the physical evidence it seems possible that the correct value is nearer 12.011.

Carbon and Nitrogen.—Moles and Toral[7] have redetermined the densities of oxygen, carbon dioxide and nitrous oxide with improved apparatus. Oxygen was prepared from potassium permanganate and from a mixture of potassium and sodium chlorates with manganese dioxide. Carbon dioxide was obtained by heating sodium acid carbonate, and after purification was several times resublimed. Nitrous oxide was prepared from ammonium nitrate and was fractionated by sublimation.

THE DENSITY OF OXYGEN (760 MM.)

	Globe A 580.995	Globe B 454.735	Average
		760 mm.	
		1.42892	1.42892
	1.42892	1.42890	1.42891
	1.42897	1.42899	1.42898
	1.42893	1.42896	1.42895
Average	1.42894	1.42894	1.42894
		506.67 mm.	
	1.42849	1.42844	1.42847
	1.42854	1.42853	1.42854
Average	1.42852	1.42848	1.42850
		380 mm.	
	1.42819	1.42812	1.42816
	1.42836	1.42829	1.42833
	1.42856	1.42831	1.42844
	1.42823	1.42832	1.42828
	1.42824	1.42833	1.42829
Average	1.42832	1.42827	1.42830

THE DENSITY OF CARBON DIOXIDE (760 MM.)

	Globe A	Globe B	Average
		760 mm.	
	1.97695	1.97696	1.97695
	1.97693	1.97691	1.97692
	1.97696	1.97695	1.97695
Average	1.97695	1.97694	1.97694
		570 mm.	
	1.97355	1.97349	1.97352
	1.97352	1.97358	1.97355
	1.97353	1.97351	1.97352
Average	1.97353	1.97353	1.97353
		475 mm.	
	1.97187	1.97174	1.97180
	1.97190	1.97189	1.97189
		1.97186	1.97186
		1.97188	1.97188
Average	1.97189	1.97184	1.97185
		380 mm.	
	1.97016	1.97012	1.97014
	1.97008	1.97017	1.97012
	1.97011	1.97016	1.97013
Average	1.97012	1.97015	1.97013

THE DENSITY OF NITROUS OXIDE (760 MM.)

	Globe A	Globe B	Average
		760 mm.	
	1.97826	1.97819	1.97822
	1.97822	1.97823	1.97822
	1.97820	1.97822	1.97821
Average	1.97823	1.97821	1.97822
		380 mm.	
	1.97103	1.97105	1.97104
	1.97096	1.97092	1.97094
Average	1.97100	1.97099	1.97099

From these values the following limiting densities and molecular weights are calculated.

	D_L	Mol. wt.
O_2	1.42764	32.0000
CO_2	1.96333	44.0072
N_2O	1.96377	44.0167

The atomic weights of carbon and nitrogen are then 12.007 and 14.008.

Moles[8] discusses the calculation of molecular weights by the method of limiting densities.

Nitrogen.—Moles and Sancho[9] recalculate the earlier results of Moles and Sancho (see Fifth Report of this Committee) on the density of ammonia after applying a correction for the coefficient of expansion of the barometer scale. The new values are 0.77140 and 0.76560 at one and one-half atmosphere, respectively. A similar correction for the scale used by Moles and Batuecas (see First Report of this Committee) gives 0.77140 for the density at one atmosphere.

New determinations were made by the volumeter method with ammonia prepared by heating nickel ammonia bromide.

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(6) Aston, *Nature*, **139**, 922 (1936).

(7) Moles and Toral, *Sitzber. Akad. Wiss. Wien*, **IIb**, **145**, 948 (1936); *Monatsh.*, **69**, 342 (1936); *Anales soc. españ. fis. quim.*, **35**, 42 (1937).

(8) Moles, *J. chim. phys.*, **34**, 49 (1937).

(9) Moles and Sancho, *Anales soc. españ. fis. quim.*, **34**, 865 (1936).

THE DENSITY OF AMMONIA (760 MM.)

Pressure of filling, mm.		Density
762		0.77135
760		.77147
	Average	.77141
583		.76856
509		.76758
397		.76562
396		.76560
	Average	.76561
264		.76376
263		.76372
	Average	.76374

The equation

$$D_p = 0.759877 + 0.001153p$$

expresses these results within the limit of accuracy of the experiments. With the limiting densities 0.75988 and 1.42761 for ammonia and oxygen the molecular weight of ammonia is 17.0327. With the atomic weight of hydrogen 1.0078, nitrogen is 14.009. The value 1.0081 for hydrogen gives 14.008 for nitrogen.

Carbon, Nitrogen and Fluorine.—Cawood and Patterson,[10] using an improved micro-displacement balance, have compared the density of oxygen with those of carbon dioxide, ethylene, carbon tetrafluoride, nitrous oxide and methyl fluoride under two different pressure conditions. By extrapolation the ratios at zero pressure were found.

The gases were subjected to chemical purification and fractional distillation or sublimation. Each ratio in the following tables is the mean of a long series of measurements at 21° ±.

CARBON DIOXIDE

p_{O_2}	p_{O_2}/p_{CO_2}
418.3	1.37764
234.5	1.37662
0	1.37532
$M_{CO_2} = 44.0101$	$C = 12.010$

ETHYLENE

p_{O_2}	$p_{O_2}/p_{C_2H_4}$
428.1	0.879963
234.9	.878507
0	.876735
$M_{C_2H_4} = 28.0556$	$C = 12.012\ (H = 1.0078)$

CARBON TETRAFLUORIDE

p_{O_2}	p_{O_2}/p_{CF_4}
484.2	2.75106
252.8	2.75040
0	2.74967
$M_{CF_4} = 87.989$	$F = 18.995\ (C = 12.011)$

METHYL FLUORIDE

p_{O_2}	p_{O_2}/p_{CH_3F}
454.8	1.06839
229.0	1.06596
0	1.06350
$M_{CH_3F} = 34.0318$	$F = 18.997 \left\{ \begin{array}{l} H = 1.0078 \\ C = 12.011 \end{array} \right.$

NITROUS OXIDE

p_{O_2}	p_{O_2}/p_{N_2O}
418.6	1.37794
229.1	1.37680
0	1.37542
$M_{N_2O} = 44.0135$	$N = 14.007$

Sodium.—Scott and Hurley[11] point out that with the atomic weight of carbon 12.010 and the current values of International atomic weights, various recent experimental values for ratios involving sodium carbonate indicate a value for the atomic weight of sodium lower than the International value and in agreement with that recently found by Johnson, 22.994 (see Fifth Report of this Committee).

Ratio	
$Na_2CO_3 : 2Ag$	22.993
$Na_2CO_3 : 2AgBr$	22.993
$Na_2CO_3 : I_2O_5$	22.994

Aluminum.—Hoffman and Lundell[12] have determined the ratio of aluminum to aluminum oxide. Weighed quantities of aluminum were dissolved in hydrochloric acid and in one set of experiments aluminum hydroxide was precipitated, collected and ignited (Series I). In another the aluminum chloride was converted to sulfate and this compound in turn to oxide by ignition (Series II). Analysis of the two samples of aluminum employed revealed only traces of a few impurities, including oxygen. The oxide obtained in the main experiments was ignited at 1200–1300° in platinum crucibles. Tests for residual gases, sulfate and water were negative provided the oxide was cooled and weighed in a closed receptacle. In every case blank experiments carried on simultaneously with very small weighed quantities of aluminum were used for comparison, the weights of metal and oxide being *subtracted from* those *of* the *experiments proper*.

The weights given in the following tables are corrected for the small amounts of impurities found in the original metal, and for the buoyancy of the air.

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[10] Cawood and Patterson, *Trans. Roy. Soc.*, **A236**, 77 (1936).

[11] Scott and Hurley, THIS JOURNAL, **59**, 2078 (1937).

[12] Hoffman and Lundell, *Bur. Standards J. Research*, **18**, 1 (1937).

THE ATOMIC WEIGHT OF ALUMINUM

Al, g.	Al_2O_3, g.	Ratio 2Al:3O	At. wt. Al
	Series I		
2.00100	3.78105	1.124126	26.979
1.89511	3.58079	1.124241	26.982
1.83837	3.47351	1.124289	26.983
1.88787	3.56752	1.123966	26.975
1.90155	3.59348	1.123894	26.973[a]
2.33772	4.41805	1.123726	26.969[a]
1.99419	3.76859	1.123867	26.973
	Average	1.124015	26.976
	Series II		
1.88650	3.56504	1.123893	26.973
2.00812	3.79482	1.123927	26.974
1.63804	3.09555	1.123862	26.973
2.65087	5.00956	1.123874	26.973
2.64428	4.99696	1.123944	26.975
2.04031	3.85588	1.123785	26.971
1.72393	3.25736	(1.124231)	(26.982)
Average, omitting the last analysis		1.123881	26.973
Average of Series I and II			26.975

[a] Recalculated.

The average agrees extraordinarily well with the earlier results of Křepelka and of Křepelka and Nikolic by analysis of the chloride, 26.975 and 26.974. Richards and Křepelka found 26.963 by analysis of the bromide while Aston's latest figure[13] corrected to the chemical scale with the conversion factor 1.00027 is 26.984.

Arsenic.—Křepelka and Kočnar[14] have determined the ratio of arsenic tribromide to silver and silver bromide. Pure arsenic and pure bromine were caused to react at 180–200° in an all-glass apparatus. The product was twice fractionally distilled in a current of nitrogen and collected over powdered arsenic. After being digested for three hours it was again distilled in nitrogen and finally was once fractionated in exhausted apparatus into small bulbs for analysis.

The bulbs, after being weighed in air and under water, were broken under ammonia and the glass was collected and weighed. Comparisons of the solutions with solutions of weighed, very nearly equivalent quantities of pure silver were carried out by the equal opalescence method. In one analysis the silver bromide was collected, dried and weighed. Weights are corrected to vacuum.

THE ATOMIC WEIGHT OF ARSENIC

$AsBr_3$, g.	Ag, g.	$AsBr_3$:3Ag	At. wt. As
2.46237	2.53249	0.972312	74.931
3.10332	3.19176	.972291	74.924
5.24485	5.39448	.972262	74.915
1.83326	1.88549	.972299	74.927
1.18537	1.21921	.972244	74.909
2.67066	2.74673	.972305	74.929
	Average	.972286	74.923
	AgBr		
4.09965	7.33996	0.558539	74.926

Molybdenum.—Hönigschmid and Wittmann[15] have analyzed molybdenum pentachloride by comparison with silver. Molybdenum trioxide first was fractionally sublimed. The middle portions were combined and fractionally volatilized as chlorohydrine in a current of hydrogen chloride. After solution in water, ammonia was added and the ammonium molybdate, after evaporation, was ignited to oxide. Reduction in hydrogen to metal followed (Sample I). Sample II was prepared from the head and tail fractions of molybdenum pentachloride rejected in the preparation of this substance.

The pentachloride was prepared by heating the pure metal in a current of oxygen-free chlorine and fractionally subliming the pentachloride four times in chlorine in an all-glass apparatus. The samples of pentachloride for analysis were sealed in glass tubes after evacuation without exposure to the air.

The tubes were weighed in air and in water and then were broken under ammonia containing hydrogen peroxide in a stoppered flask. The glass fragments were collected and weighed. After several days' standing to allow peroxy-

THE ATOMIC WEIGHT OF MOLYBDENUM

Sample	$MoCl_5$, g.	Ag, g.	$MoCl_5$: 5Ag	At. wt. Mo
I	3.92664	7.75178	0.506546	95.946
I	1.15477	2.27969	.506546	95.946
I	1.97299	3.89488	.506559	95.953
I	1.70337	3.36269	.506550	95.948
I	0.54405	1.07400	.506564	95.955
I	1.61924	3.19664	.506544	95.945
I	0.69492	1.37182	.506568	95.958
I	3.35249	6.61842	.506539	95.942
I	1.84113	3.63462	.506554	95.950
I	2.84577	5.61795	.506550	95.948
I	1.12271	2.21639	.506549	95.948
I	1.29219	2.55093	.506556	95.952
I	1.81107	3.57528	.506553	95.950
I	1.89693	3.74477	.506554	95.950
I	1.33890	2.64321	.506543	95.944
I	3.75537	7.41382	.506536	95.941
II	0.58655	1.15788	.506572	95.960
II	1.91751	3.78537	.506558	95.952
II	1.62848	3.21487	.506546	95.946
		Average	.506552	95.949

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(13) Aston, *Nature*, **137**, 163 (1936).

(14) Křepelka and Kočnar, *Collection Czechoslov. Chem. Commun.*, **VIII**, 485 (1936).

(15) Hönigschmid and Wittmann, *Z. anorg. allgem. Chem.*, **229**, 65 (1936).

molybdic acid to decompose, a large excess of nitric acid was added and the solution was compared with weighed, nearly equivalent quantities of pure silver by the equal opalescence method. Weights are corrected to vacuum.

The average result 95.95, which is supported by Aston's isotopic analysis of molybdenum, has been adopted for the Table.

Iodine.—Moles,[16] on the basis of a redetermination of the density of iodine pentoxide (5.28) by means of a mercury volumeter,[17] has recalculated the atomic weight of iodine (1) from the ratio I_2:I_2O_5,[18] and (2) from the ratio I_2O_5:Na_2CO_3[19] with results supporting the value for iodine recently found by Hönigschmid and Striebel, 126.917.

In the investigations by Baxter, Butler and Hale the weights of iodine pentoxide already have been subjected to a correction of −0.001% for adsorbed air. This correction was determined by displacement of air, using the density 4.80. If the real density is 5.28, the apparent adsorption of air disappears. The correction applied by Moles on the basis of the higher density, −0.002%, is, therefore, twice as large as it should be. The following table gives the values (1) as calculated by Moles and (2) corrected as above.

THE ATOMIC WEIGHT OF IODINE

C = 12.006 Na = 22.997

Ratio	Moles	Moles corr.
I_2/I_2O_5	126.915	126.911
I_2O_5/Na_2CO_3	126.919	126.922

If the atomic weights of carbon and sodium are assumed to be 12.010 and 22.994 the second ratio yields 126.918 for iodine.

While the differences between the values for the atomic weight of iodine, found from the above ratios, and the more probable value recently found by Hönigschmid and Striebel, 126.917, are lessened by assuming the density for iodine pentoxide found by Moles and Villan, these differences still remain pronounced and must be dependent on other causes.

Europium.—Baxter and Tuemmler,[20] working with material purified by McCoy, have analyzed europous chloride. Europium originally containing about 70% of rare earth impurities was five times precipitated as europous chloride. Spectroscopic examination by King then revealed less than 0.001% of other rare earths. Further purification consisted in several precipitations as europic oxalate from acid solution, fractional crystallization of europic nitrate from nitric acid and fractional crystallization of europic chloride from hydrochloric acid.

Attempts to prepare anhydrous europic chloride failed because of instability of this salt at high temperatures even in a chlorine atmosphere. Anhydrous europous chloride was, however, prepared easily by slow dehydration and eventual fusion in hydrogen chloride and hydrogen. Weighed amounts of the anhydrous dichloride were dissolved in very dilute nitric acid and allowed to oxidize in the air. Comparison of the solution with silver followed conventional lines. Weights are corrected to vacuum (density $EuCl_2$ assumed to be 5.0).

THE ATOMIC WEIGHT OF EUROPIUM

$EuCl_2$, g.	Ag, g.	$EuCl_2$: 2Ag	At. wt. Eu
2.37131	2.29571	1.03293	151.95
3.08192	2.98364	1.03294	151.95
2.81855	2.72847	1.03301	151.97
4.88930	4.73350	1.03291	151.95
	Average	1.03295	151.95

The average value, which is preliminary, is not far from the International value, 152.0, and the mass spectrum value, 151.90 (Aston).

Erbium.—Hönigschmid and Wittner[21] have published detailed results of analyses of erbium chloride.[22] The erbium material, purified by Feit, was several times precipitated as oxalate and after crystallization the chloride was carefully dehydrated by efflorescence at gradually increasing temperatures up to 450°. Analysis in the usual way by comparison with silver followed. Weights are corrected to vacuum.

ATOMIC WEIGHT OF ERBIUM

$ErCl_3$, g.	Ag, g.	$ErCl_3$: 3Ag	At. wt. Er	AgCl, g.	$ErCl_3$: 3AgCl	At. wt. Er
2.51386	2.97656	0.84455	166.960	3.95499	0.63562	166.952
3.53255	4.18243	.84462	166.981	5.55730	.63566	166.970
2.15972	2.55725	.84455	166.959	3.39780	.63562	166.954
3.03007	3.58787	.84453	166.953	4.76709	.63562	166.954
2.62962	3.11371	.84453	166.953	4.13701	.63563	166.958
4.53536	5.37025	.84453	166.954	7.13535	.63562	166.952
		.84455	166.960		.63563	166.957

X-ray analysis of this material by Noddack indicated 0.37 atom per cent. of yttrium and 0.42 of thulium. Corrected for these impurities the atomic weight of erbium becomes 167.24.

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(16) Moles, *Anales soc. españ. fís. quím.*, **34**, 959 (1936).

(17) Moles and Villan, *ibid.*, **34**, 787 (1936).

(18) Baxter and Butler, Second Report of this Committee.

(19) Baxter and Hale, Fifth Report of this Committee.

(20) Baxter and Tuemmler, THIS JOURNAL, **59**, 1133 (1937).

(21) Hönigschmid and Wittner, *Z. anorg. allgem. Chem.*, **232**, 113 (1937).

(22) See Seventh Report of this Committee.

Aston's mass spectrographic analysis yielded 167.15. On the basis of these results the atomic weight of erbium in the Table has been changed from 167.64 to 167.2.

Tungsten.—Hönigschmid and Wenn[23] have compared tungsten hexachloride with silver. Tungsten material was purified first by synthesizing the hexachloride and distilling it fractionally. Solution in ammonia and precipitation of tungstic acid with nitric acid followed and this process was repeated three times. Ignition and reduction of the oxide in hydrogen was the next step. At this point spectroscopic examination gave doubtful indication of a trace of molybdenum while X-ray spectra showed none of this element.

The pure metal was then converted to hexachloride in a current of oxygen-free chlorine in quartz and the hexachloride was fractionally sublimed in a current of chlorine into a glass tube which could be exhausted and sealed.

After being weighed in air and under water the sealed tube was broken under ammonia and the glass fragments were collected on a platinum sponge crucible. The solution was then compared with weighed, nearly equivalent quantities of pure silver by the equal opalescence method. In precipitating the silver chloride it was found desirable first to add the silver nitrate to the ammoniacal solution and then to acidify with nitric acid in the presence of tartaric acid. Weights are corrected to vacuum.

The Atomic Weight of Tungsten

WCl_6, g.	Ag, g.	WCl_6: 6Ag	At. wt. W
1.75701	2.86712	0.612814	183.920
1.73590	2.83255	.612840	183.937
1.93036	3.15007	.612799	183.911
2.60625	4.25263	.612856	183.948
1.86801	3.04814	.612836	183.935
2.70714	4.41774	.612788	183.904
3.39835	5.54586	.612772	183.893
2.80394	4.57536	.612835	183.934
4.95955	8.09324	.612802	183.912
2.77074	4.52115	.612840	183.937
1.69490	2.76594	.612775	183.895
1.72253	2.81100	.612782	183.900
1.62596	2.65332	.612802	183.913
3.26518	5.32827	.612803	183.913
0.58492	0.95442	.612854	183.946
3.12581	5.10073	.612816	183.922
4.55270	7.42866	.612856	183.948
2.75996	4.50378	.612810	183.918
2.28497	3.72869	.612808	183.916
3.39738	5.54385	.612820	183.924
3.56066	5.81059	.612788	183.904
3.15808	5.15369	.612780	183.899
	Average	.612812	183.920

The average result agrees well with Aston's mass spectrographic value and has been adopted for the Table.

Osmium.—Nier[24] recently has redetermined the isotopic abundance ratios of osmium. These lead to a chemical atomic weight 190.21 (packing fraction -1×10^{-4}; conversion factor 1.00027), while the measurements by Aston[25] give 190.28. It therefore seems probable that the present International value for this element, 191.5, is too high, and accordingly the value for osmium in the Table has been changed to 190.2.

Lead.—Baxter, Faull and Tuemmler have[26] determined the atomic weights of several radio-

The Atomic Weight of Lead

$PbCl_2$, g.	Ag, g.	Ratio $PbCl_2$: 2Ag	At. wt. of Pb
	Common lead		
4.39335	3.40822	1.28905	207.211
3.49797	2.71356	1.28907	207.216
4.21579	3.27033	1.28910	207.222
4.27224	3.31427	1.28904	207.210
5.99791	4.65298	1.28905	207.211
4.74688	3.68250	1.28904	207.209
3.99080	3.09581	1.28910	207.222
	Average	1.28906	207.214
	Samarskite		
1.28803	1.00238	1.28497	206.331
0.75523	0.58769	1.28508	206.355
	Average	1.28503	206.343
	Beaverlodge Lake pitchblende		
2.61248	2.03489	1.28384	206.088
2.75235	2.14398	1.28373	206.070
3.17452	2.47283	1.28376	206.070
6.38415	4.97247	1.28392	206.099
4.01745	3.12921	1.28384	206.089
2.72167	2.11990	1.28387	206.093
2.17947	1.69765	1.28382	206.082
	Average	1.28383	206.084
	Katanga pitchblende, hydrochloric acid extract		
3.43131	2.67306	1.28366	206.049
3.52881	2.74901	1.28367	206.050
2.54121	1.97960	1.28370	206.057
4.28996	3.34206	1.28363	206.041
4.84228	3.77217	1.28369	206.054
4.50429	3.50889	1.28368	206.053
	Average	1.28367	206.051
	Katanga pitchblende, unaltered		
1.70238	1.32624	1.28361	206.038
1.60461	1.25003	1.28366	206.048
4.61797	3.59755	1.28364	206.045
3.73794	2.91206	1.28361	206.037
	Average	1.28363	206.042

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(23) Hönigschmid and Wenn, *Z. anorg. allgem. Chem.*, **229**, 49 (1936).

(24) Nier, *Phys. Rev.*, **52**, 885 (1937).

(25) Aston, *Proc. Roy. Soc.* (London), **A132**, 492 (1931).

(26) Baxter, Faull and Tuemmler, THIS JOURNAL, **59**, 702 (1937).

INTERNATIONAL ATOMIC WEIGHTS
1938

	Symbol	Atomic Number	Atomic Weight		Symbol	Atomic Number	Atomic Weight
Aluminum	Al	13	26.97	Molybdenum	Mo	42	95.95
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	190.2
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	12.010	Praseodymium	Pr	59	140.92
Cerium	Ce	58	140.13	Protactinium	Pa	91	231
Cesium	Cs	55	132.91	Radium	Ra	88	226.05
Chlorine	Cl	17	35.457	Radon	Rn	86	222
Chromium	Cr	24	52.01	Rhenium	Re	75	186.31
Cobalt	Co	27	58.94	Rhodium	Rh	45	102.91
Columbium	Cb	41	92.91	Rubidium	Rb	37	85.48
Copper	Cu	29	63.57	Ruthenium	Ru	44	101.7
Dysprosium	Dy	66	162.46	Samarium	Sm	62	150.43
Erbium	Er	68	167.2	Scandium	Sc	21	45.10
Europium	Eu	63	152.0	Selenium	Se	34	78.96
Fluorine	F	9	19.00	Silicon	Si	14	28.06
Gadolinium	Gd	64	156.9	Silver	Ag	47	107.880
Gallium	Ga	31	69.72	Sodium	Na	11	22.997
Germanium	Ge	32	72.60	Strontium	Sr	38	87.63
Gold	Au	79	197.2	Sulfur	S	16	32.06
Hafnium	Hf	72	178.6	Tantalum	Ta	73	180.88
Helium	He	2	4.003	Tellurium	Te	52	127.61
Holmium	Ho	67	163.5	Terbium	Tb	65	159.2
Hydrogen	H	1	1.0081	Thallium	Tl	81	204.39
Indium	In	49	114.76	Thorium	Th	90	232.12
Iodine	I	53	126.92	Thulium	Tm	69	169.4
Iridium	Ir	77	193.1	Tin	Sn	50	118.70
Iron	Fe	26	55.84	Titanium	Ti	22	47.90
Krypton	Kr	36	83.7	Tungsten	W	74	183.92
Lanthanum	La	57	138.92	Uranium	U	92	238.07
Lead	Pb	82	207.21	Vanadium	V	23	50.95
Lithium	Li	3	6.940	Xenon	Xe	54	131.3
Lutecium	Lu	71	175.0	Ytterbium	Yb	70	173.04
Magnesium	Mg	12	24.32	Yttrium	Y	39	88.92
Manganese	Mn	25	54.93	Zinc	Zn	30	65.38
Mercury	Hg	80	200.61	Zirconium	Zr	40	91.22

genic leads. After extraction from the mineral the lead salts were purified by crystallization as nitrate and chloride, followed by distillation of the chloride in a current of hydrogen chloride. In preparation for weighing the lead chloride was fused in hydrogen chloride. Comparison of the lead chloride with silver followed in the conventional way. Weights are corrected to vacuum.

Since the Beaverlodge Lake and Katanga pitchblendes are free from thorium, it appears that both contain appreciable amounts of common lead. Allowing for the thorium–uranium ratio of this specimen of samarskite, 0.442, this seems also to be the case with the samarskite lead.

Marble[27] has determined the atomic weight of lead from a specimen of galena occurring in a vein which cuts one of the pitchblende veins of the Great Bear Lake deposit and from a point not far from the pitchblende. Purification included crystallization of the nitrate, and of the chloride as well as distillation of the chloride in hydrogen chloride. Analysis was by the conventional method of comparison with silver. Weights are corrected to vacuum.

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(27) Marble, THIS JOURNAL, **59**, 654 (1937).

THE ATOMIC WEIGHT OF LEAD

$PbCl_2$, g.	Ag, g.	Ratio $PbCl_2:2Ag$	At. wt. Pb
0.54549	0.42318	1.28903	207.206
2.77993	2.15663	1.28902	207.204
1.17288	0.90990	1.28902	207.205
	Average	1.28902	207.205

The sample appears to be common lead and if so is one of the oldest to be examined.

Radium.—Attention is called to the fact that in the recent determination of the atomic weight of radium by Hönigschmid and Sachtleben (Sixth Report of this Committee) no correction is made for the effect of the temperature of radium salts on their weights. Allowance for this will presumably raise the atomic weight of radium by 0.01–0.02 unit.

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, NEW YORK UNIVERSITY]

The Synthesis of Δ³-Tetrahydropyridine

BY R. R. RENSHAW AND R. C. CONN[1,2]

In order to determine the physiological effect of introducing the double bond into the piperidine nucleus, we have prepared the Δ³-tetrahydropyridine by the following series of reactions

$$\text{4-Hydroxypyridine} \xrightarrow[C_2H_5OH]{Na} \text{4-hydroxypiperidine} \xrightarrow{HBr}$$

$$\text{4-bromopiperidine} \xrightarrow{NaOCH_3} \Delta^3\text{-tetrahydropyridine}$$

Koenigs and Neumann,[3] in describing the preparation of 4-iodo- and 4-bromopiperidines, state that these 4-halogenopiperidines split halogen acid, in alkaline solution, to give tetrahydropyridine. Apparently, this compound was not isolated nor characterized as no experimental details are given for the reaction. Paal and Hubaleck[4] believed that they obtained the gold salt of Δ³-tetrahydropyridine when they submitted a C-sulfonic acid derivative of piperidine of unknown structure to a dry distillation with potassium hydroxide.

The only step in the series of reactions giving any difficulty was the reduction of the 4-hydroxypyridine. We were unable to obtain any reduction with Adams platinum oxide catalyst under varied experimental conditions, although Emmert[5] has patented a process for the reduction of 4-hydroxypyridine using a large amount of platinum black. The reduction was, therefore, carried out by means of sodium and alcohol according to Koenigs and Neumann,[6] and Emmert and Dorn.[7] The yield was only 30% although we were able to recover as much as 50% of the 4-hydroxypyridine.

The 4-halogenopiperidines do not readily split out hydrohalide. In an experiment in which 4-iodopiperidine was treated with alcoholic potassium hydroxide 4-hydroxypiperidine and unchanged 4-iodopiperidine were the only products isolated. This elimination of hydrohalide is best brought about by the use of sodium methylate or ethylate. The Δ³-tetrahydropyridine was isolated as the hydrochloride and not as the free base because of the demonstrated ease of the polymerization of the latter.

Experimental Part

4-Hydroxypiperidine.—This substance was prepared by the sodium–alcohol reduction of 4-hydroxypyridine,[8] according to the directions of Koenigs and Neumann.[6] The yield was 30% with about 50% of the 4-hydroxypyridine recoverable.

4-Halogenopiperidine Hydrohalides.—4-Iodo- and 4-bromopiperidines were prepared by the method of Koenigs and Neumann.[3] The use of a somewhat larger (tenfold) excess of hydrobromic acid increased the yield of the bromo compound from 66 to 80%.

Alcoholic Potassium Hydroxide and 4-Iodopiperidine Hydriodide.—A solution of 1.6 g. (0.028 *M*) of potassium hydroxide in 18 cc. of absolute ethyl alcohol was added to 4.85 g. (0.014 *M*) of 4-iodopiperidine hydriodide and the suspension, after prolonged shaking, was allowed to stand overnight. Part of the alcohol was removed under reduced pressure at room temperature. Ether was added to precipitate the last traces of potassium iodide. The solution was filtered and the filtrate was saturated with dry hydrogen chloride. One and five-tenths grams of crystalline hydrochloride was obtained. Approximately 50% of this product dissolved readily in alcohol, the remainder being much more insoluble.

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[1] This is the second paper constructed from the thesis presented by R. C. Conn, June, 1935, for the degree of Doctor of Philosophy at New York University.

[2] University Fellow in Chemistry.

[3] Koenigs and Neumann, *Ber.*, **48**, 961 (1915).

[4] Paal and Hubaleck, *ibid.*, **34**, 2761 (1901).

[5] H. Emmert, German Patent 292,456.

[6] Koenigs and Neumann, *Ber.*, **48**, 957 (1915).

[7] Emmert and Dorn, *ibid.*, **48**, 688 (1915).

[8] Koenigs and Greiner, *ibid.*, **64**, 1049 (1931); German Patent 536,891.