glacial acetic acid gave 1.12 g. or a 9.2% yield of 4-methoxy-6-dibenzofurancarboxylic acid melting at 240–242°.

Anal. Calcd. for $C_{14}H_{10}O_4$: neut. equiv., 242. Found: neut. equiv., 237.

The methyl 4-methoxy-6-dibenzofurancarboxylate prepared by means of diazomethane from the methoxy acid melting at 240–242° was proved identical with both the methoxy ester of the single acid obtained from metalation of 4-hydroxydibenzofuran and an authentic sample of methyl 4-methoxy-6-dibenzofurancarboxylate.

Anal. Calcd. for $C_{15}H_{12}O_4$: methoxyl, 24.20. Found: methoxyl, 24.25.

Metalation of Resorcinol.—To an ice-cold solution of approximately 0.3 mole *n*-butyllithium in 400 cc. of ether was added dropwise with stirring 11.0 g. (0.1 mole) of resorcinol in 75 cc. of ether. Gas was evolved but no precipitate formed during the ensuing twenty-one hour period of refluxing. Carbonation, in the usual manner, was followed by acidification with iced dilute hydrochloric acid and extraction with ether. The crude acid obtained from the ether layer[8] was recrystallized from water to give 3.3 g., m. p. 145–147°, and 0.8 g., m. p. 192–195°. An additional 0.7 g., m. p. 150°, was obtained from the filtrate; combined yield, 31.1%.

The 3.3-g. portion, melting at 150–152° after recrystallization from water, was treated with dimethyl sulfate and sodium hydroxide to form 2,6-dimethoxybenzoic acid,[9] m. p. 183–184°, identified by a mixed melting point determination with a reference sample.

The 0.8-g. portion, m. p. 206–207° after recrystallization from water, was converted with diazomethane to methyl 2-hydroxy-4-methoxybenzoate[10] melting at 49° alone or mixed with an authentic specimen. Hydrolysis of the ester with alcoholic potassium hydroxide gave 2-hydroxy-4-methoxybenzoic acid, m. p. 153–154°. The mixed melting point with a known sample showed no depression.

The third portion was not identified but was probably a mixture of the two acids contaminated with resorcinol.

Metalation of Resorcinol Dimethyl Ether.—A mixture of 0.1 mole of resorcinol dimethyl ether and 0.1 mole of *n*-butyllithium in ether was refluxed for twenty hours. Carbonation, by pouring the mixture jet-wise upon solid carbon dioxide, gave 10 g. (55% yield) of 2,6-dimethoxybenzoic acid melting at 186°. The methyl 2,6-dimethoxybenzoate, prepared by refluxing the silver salt of the dimethoxy acid with methyl iodide, melted at 87°.[9]

A small quantity (0.043 g.) of 2,2′,6,6′-tetramethoxybenzophenone,[4] melting at 204°, was also isolated.

Summary

Metalation by *n*-butyllithium of 2-methoxydibenzofuran gives 2-methoxy-1-dibenzofuryllithium and 2-methoxy-3-dibenzofuryllithium; and metalation of 4-methoxydibenzofuran gives 4-methoxy-6-dibenzofuryllithium and 4-methoxy-3-dibenzofuryllithium. However, metalation of the corresponding phenols gives but one product in each case: 2-hydroxydibenzofuran yielding 2-hydroxy-1-dibenzofuryllithium (as a lithium salt of the phenol); and 4-hydroxydibenzofuran yielding 4-hydroxy-6-dibenzofuryllithium.

In contrast, metalation of resorcinol dimethyl ether gives apparently but one product (2,6-dimethoxyphenyllithium), whereas resorcinol gives the two isomers, 2,6-dihydroxyphenyllithium and 2,4-dihydroxyphenyllithium.

AMES, IOWA RECEIVED JANUARY 15, 1940

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[8] Nierenstein and Clibbens, *Org. Syntheses*, **10**, 95 (1930).

[9] Mauthner, *J. prakt. Chem.*, [2] **121**, 259 (1929).

[10] Herzig, Henzel and Batscha, *Monatsh.*, **24**, 888 (1903).

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

BY G. P. BAXTER (*Chairman*), M. GUICHARD, O. HÖNIGSCHMID AND R. WHYTLAW-GRAY

The following report of the Committee covers the twelve-month period September 30, 1938 to September 30, 1939.[1] Three changes in the table of atomic weights have been adopted:

Hydrogen, from 1.0081 to 1.0080, iron, from 55.84 to 55.85, lutecium, from 175.0 to 174.99.

Hydrogen.—Several investigations of the H^1/H^2 ratio in certain natural waters during the last four years have given values higher than that used in computing the atomic weight of hydrogen for the Table, 5000 (see Eighth Report of this Committee).

Johnston, THIS JOURNAL, **57**, 484 (1935)	5900
Tronstad, Nordhagen and Brun, *Nature*, **136**, 515 (1935)	5840
Hall and Jones, THIS JOURNAL, **58**, 1915 (1936)	6550
Gabbard and Dole, *ibid.*, **59**, 181 (1937)	7020
Morita and Titani, *Bull. Chem. Soc. Japan*, **13**, 419 (1938)	6320
Tronstad and Brun, *Trans. Faraday Soc.*, **34**, 766 (1938)	5400
Voskuyl, Thesis, Harvard University (1938)	6700
Swartout and Dole, THIS JOURNAL, **61**, 2025 (1939)	6970 6880

Since with $H^1 = 1.00785$ (chemical scale) and even with $H^1/H^2 = 6000$ the atomic weight of

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[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. M. Guichard, Faculté des Sciences, Sorbonne, Paris, France; Prof. O. Hönigschmid, Sofienstrasse 9/2, Munich, Germany; Prof. R. Whytlaw-Gray, University of Leeds, Leeds, England.

hydrogen in natural waters becomes 1.0080 this value is adopted for the Table, although the nature of the hydrogen in any compound is bound to be subject to a slight uncertainty.

Chlorine.—Hönigschmid and Hirschbold-Wittner[2] have determined the atomic weight of chlorine in hydrogen chloride samples subjected to isotopic separation by the thermal diffusion method by Clausius and Dickel. Weighed amounts of silver were precipitated with excess acid and the silver chloride was collected and weighed. Vacuum weights are given.

Ag, g.	AgCl, g.	AgCl:Ag	At. wt., Cl
	Heavy fractions		
0.59164	0.78666	1.32963	35.560
.48005	.64450	1.34257	36.956
	Light fractions		
.73019	.96925	1.32739	35.310
.61237	.81188	1.32580	35.147
.58001	.76830	1.32463	35.021
.57969	.76765	1.32428	34.979

If the isotopic weights of the two chief isotopes of chlorine are 36.968 and 34.971 on the chemical scale the separation seems to have been nearly complete.

THE ATOMIC WEIGHT OF IRON

Sample	$FeBr_2$, g.	Ag, g.	$FeBr_2$:2Ag	At. wt. Fe	AgBr, g.	$FeBr_2$:2AgBr	At. wt. Fe
I	1.85170	1.85236	0.999644	55.851			
I	3.30576	3.30690	.999655	55.854			
II	2.07499	2.07574	.999639	55.850			
II	3.28783	3.28900	.999644	55.851	5.72554	0.574239	55.848
I	2.92032	2.92137	.999641	55.850	5.08551	.574243	55.849
I	3.30851	3.30966	.999653	55.853	5.76158	.574237	55.847
I	2.32787	2.32870	.999644	55.851	4.05371	.574257	55.854
I	2.83665	2.83765	.999648	55.852	4.93977	.574247	55.851
II	2.80087	2.80188	.999640	55.850	4.87744	.574250	55.852
II	2.61260	2.61354	.999640	55.850	4.54968	.574238	55.847
II	2.85977	2.86078	.999647	55.852	4.98006	.574244	55.849
	Average		.999645	55.851		.574244	55.850

Iron.—Hönigschmid and Liang[3] have compared ferrous bromide with silver and silver bromide. A solution of Mohr's salt was freed from heavy metals with hydrogen sulfide. After oxidation ferric hydroxide was three times precipitated from acid solution, and the third precipitate was dissolved in sulfuric acid and electrolytically reduced to ferrous sulfate which was recovered by crystallization. Electrolytic precipitation of metallic iron from ammonium oxalate solution followed, and the metallic deposit was dissolved in nitric acid. Carbon was removed by filtration and the ferric nitrate solution evaporated to crystallization. Thermal decomposition to oxide and reduction in hydrogen yielded Sample I. Spectroscopic examination by Gerlach revealed no impurities. The remainder of the ferric nitrate was twice more recrystallized from nitric acid and converted to metal (Sample II).

Synthesis of the bromide was effected by heating the pure metal in a current of dry nitrogen and bromine. The sublimed product was resublimed in pure nitrogen and fused in nitrogen in a quartz weighing tube which could be bottled in dry air. After being weighed the salt was dissolved in very dilute sulfuric acid. The solution was clear and free from ferric salt. After careful oxidation with slightly less than the theoretical quantity of bichromate the solution was compared with silver in the conventional way with the help of a nephelometer. In many of the experiments the silver bromide was collected and weighed. Vacuum weights are given.

The average of all the experiments, 55.850, is slightly higher than that found by analysis of ferrous bromide by Baxter, Thorvaldson and Cobb,[4] 55.838, presumably because the material employed by the latter contained traces of carbon, silica and possibly ferric salt. By reduction of ferric oxide Baxter and Hoover[5] found 55.847, while Hönigschmid, Birckenbach and Zeiss[6] by analysis of ferric chloride obtained the value 55.853.

Recent determinations of abundance ratios with the mass spectrograph, when converted to the chemical scale with the packing fraction -7×10^{-4} (Dempster, *Phys. Rev.*, **53**, 869

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(2) Hönigschmid and Hirschbold-Wittner, *Z. anorg. allgem. Chem.*, **242**, 222 (1939).

(3) Hönigschmid and Liang, *Z. anorg. allgem. Chem.*, **241**, 361 (1939).

(4) Baxter, Thorvaldson and Cobb, THIS JOURNAL, **33**, 319 (1911).

(5) Baxter and Hoover, *ibid.*, **34**, 1657 (1912).

(6) Hönigschmid, Birckenbach and Zeiss, *Ber.*, **56**, 1473 (1923).

(1938)), and the conversion factor 1.000275 give the following values.

	Isotope 54	56	57	58	Mean mass number	At. wt.
de Gier and Zeeman[a]	6.5	90.2	2.8	0.5	55.908	55.853
Nier[b]	6.04	91.57	2.11	0.28	55.906	55.851

[a] *Proc. Roy. Soc. Amsterdam*, **38**, 959 (1935). [b] *Phys. Rev.*, **55**, 1143 (1939).

In view of the evidence the value 55.85 for iron seems more probable than the older one 55.84, and has been adopted for the Table, although the value 55.847 obtained by the most direct method, the reduction of the oxide, might well be looked upon as a maximum owing to the possibility of incomplete reduction.

Molybdenum.—Mattauch and Lichtblau[7] have redetermined the relative abundances of the molybdenum isotopes. In the following table their percentages are compared with those found earlier by Aston. The atomic weight is calculated with the packing fraction -6.0×10^{-4} (Dempster) and the conversion factor 1.000275.

	92	94	95	96	97
Aston	14.2	10.0	15.5	17.8	9.6
Mattauch and Lichtblau	15.5	8.7	16.3	16.8	8.7

	98	100	Mean mass number	At. wt. Mo
Aston	23.0	9.8	95.94[a]	95.86
Mattauch and Lichtblau	25.4	8.6	95.98	95.90

[a] Incorrectly calculated by Aston to be 96.03, *Proc. Roy. Soc.* (London), **A130**, 309 (1931).

Both these values are considerably lower than that obtained by Hönigschmid and Wittner by analysis of molybdenum pentachloride, 95.949, which was adopted for the Table two years ago. (See Eighth Report of this Committee.)

Europium.—Lichtblau[8] has determined the abundance ratio of the europium isotopes, Eu^{151}/Eu^{153}, to be 0.963 ± 0.012. With the packing fraction -2×10^{-4} (Dempster) and the conversion factor 1.000275 the atomic weight of europium is 151.95 ± 0.01. This value for europium agrees far better with that recently found by Baxter and Tuemmler by analysis of europous chloride, 151.96, than with Kapfenberger's recent value, 151.90 (see Ninth Report of this Committee).

Lutecium.—Hönigschmid and Wittner[9] have published details of their analyses of lutecium trichloride, already reported[10]; (see Eighth Report of this Committee). The starting material had been purified by von Welsbach and was identical with that used by him in his determination of the atomic weight. From examination of the X-ray spectrum Noddack estimates a content of 1.18% of ytterbium but no more than 0.04% of any other rare earth. Purification consisted in double precipitation of the hydroxide with solution in hydrochloric acid, double precipitation of the oxalate with subsequent ignition in each case, and double crystallization of the chloride from concentrated hydrochloric acid. The chloride was dehydrated in a stream of dry hydrogen chloride at gradually increasing temperatures finally at 450° and after being weighed was compared with silver in the conventional way. The silver chloride was determined in some cases. Vacuum weights are given.

The authors prefer the result of the former method of analysis, 174.966. Corrected for the ytterbium content this becomes 174.986.

Mattauch and Lichtblau,[11] with the mass spectrograph, find a new isotope of lutecium of mass number 176, to the extent of 2.52%. With the packing fraction $+1 \times 10^{-4}$ (Dempster) and the conversion factor 1.000275 the atomic weight of lutecium is 174.994, in close agreement with that found by Hönigschmid and Wittner.

The value 174.99 is adopted for the Table in place of the older value 175.0 which depends on von Welsbach's analysis of the sulfate.

Lead.—Nier[12] has determined the abundance ratios of the lead isotopes in twenty-one speci-

THE ATOMIC WEIGHT OF LUTECIUM

$LuCl_3$, g.	Ag, g.	$LuCl_3$:3Ag	At. wt. Lu	AgCl, g.	$LuCl_3$:3AgCl	At. wt. Lu
2.10076	2.41662	0.86930	174.968	3.21098	0.65424	174.961
2.94416	3.38688	.86928	174.964	4.50009	.65424	174.962
2.20514	2.53662	.86932	174.977			
2.63280	3.02874	.86927	174.960	4.02420	.65424	174.960
2.70083	3.10697	.86928	174.963			
	Average	.86929	174.966		.65424	174.961

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(7) Mattauch and Lichtblau, *Z. physik. Chem.*, B**42**, 288 (1939).

(8) Lichtblau, *Naturwiss.*, **27**, 260 (1939).

(9) Hönigschmid and Wittner, *Z. anorg. allgem. Chem.*, **240**, 284 (1939).

(10) Hönigschmid and Wittner, *Naturwiss.*, **25**, 748 (1937).

(11) Mattauch and Lichtblau, *Z. Physik*, **111**, 514 (1939).

(12) Nier, *Phys. Rev.*, **55**, 153 (1939).

mens of radiogenic lead, and has computed the atomic weights of these specimens using the packing fraction $+1.55 \times 10^{-4}$ and the conversion factor 1.000275. While in most cases the agreement between the atomic weights found in this way and those which had been determined chemically is fair, in some cases considerable discrepancies appear. If Dempster's packing fraction for lead, $+2.3 \times 10^{-4}$, is used the agreement is far less satisfactory. The latter is also true for the results obtained by Nier with common lead (see Ninth Report of this Committee).

INTERNATIONAL ATOMIC WEIGHTS
1940

	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	30.98
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	1.0080	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	55.85	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	174.99	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