Laboratory Physical Chemistry

Oelke/M.A.C.T.L.A.C

LABORATORY PHYSICAL CHEMISTRY

Part One: Reference

by William C. Oelke, *Grinnell College*
with a Chapter on High Vacuum Techniques
by Richard W. Zuehlke, *Lawrence University*

Part Two: Experiments

Contributed by members of the
Midwestern Association of Chemistry Teachers
in Liberal Arts Colleges, and others.
Edited by William C. Oelke.

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Table I Elements and Atomic Weights, Based on Carbon-12 = 12.000

	SYMBOL	ATOMIC NO.	ATOMIC WEIGHT
Actinium	Ac	89	
Aluminum	Al	13	26.9815
Americium	Am	95	
Antimony	Sb	51	121.75
Argon	Ar	18	39.948
Arsenic	As	33	74.9216
Astatine	At	85	
Barium	Ba	56	137.34
Berkelium	Bk	97	
Beryllium	Be	4	9.0122
Bismuth	Bi	83	208.980
Boron	B	5	10.811[a]
Bromine	Br	35	79.909[b]
Cadmium	Cd	48	112.40
Calcium	Ca	20	40.08
Californium	Cf	98	
Carbon	C	6	12.01115[a]
Cerium	Ce	58	140.12
Cesium	Cs	55	132.905
Chlorine	Cl	17	35.453[b]
Chromium	Cr	24	51.996[b]
Cobalt	Co	27	58.9332
Copper	Cu	29	63.54
Curium	Cm	96	
Dysprosium	Dy	66	162.50
Einsteinium	Es	99	
Erbium	Er	68	167.26
Europium	Eu	63	151.96
Fermium	Fm	100	
Fluorine	F	9	18.9984
Francium	Fr	87	
Gadolinium	Gd	64	157.25
Gallium	Ga	31	69.72
Germanium	Ge	32	72.59
Gold	Au	79	196.967
Hafnium	Hf	72	178.49
Helium	He	2	4.0026
Holmium	Ho	67	164.930
Hydrogen	H	1	1.00797[a]
Indium	In	49	114.82
Iodine	I	53	126.9044
Iridium	Ir	77	192.2
Iron	Fe	26	55.847[b]
Krypton	Kr	36	83.80
Lanthanum	La	57	138.91
Lawrencium	Lw	103	
Lead	Pb	82	207.19
Lithium	Li	3	6.939
Lutetium	Lu	71	174.97
Magnesium	Mg	12	24.312
Manganese	Mn	25	54.9380
Mendelevium	Md	101	
Mercury	Hg	80	200.59
Molybdenum	Mo	42	95.94
Neodymium	Nd	60	144.24
Neon	Ne	10	20.183
Neptunium	Np	93	
Nickel	Ni	28	58.71
Niobium	Nb	41	92.906
Nitrogen	N	7	14.0067
Nobelium	No	102	
Osmium	Os	76	190.2
Oxygen	O	8	15.9994[a]
Palladium	Pd	46	106.4
Phosphorus	P	15	30.9738
Platinum	Pt	78	195.09
Plutonium	Pu	94	
Polonium	Po	84	
Potassium	K	19	39.102
Praseodymium	Pr	59	140.907
Promethium	Pm	61	
Protactinium	Pa	91	
Radium	Ra	88	
Radon	Rn	86	
Rhenium	Re	75	186.2
Rhodium	Rh	45	102.905
Rubidium	Rb	37	85.47
Ruthenium	Ru	44	101.07
Samarium	Sm	62	150.35
Scandium	Sc	21	44.956
Selenium	Se	34	78.96
Silicon	Si	14	28.086[a]
Silver	Ag	47	107.870[b]
Sodium	Na	11	22.9898
Strontium	Sr	38	87.62
Sulfur	S	16	32.064[a]
Tantalum	Ta	73	180.948
Technetium	Tc	43	
Tellurium	Te	52	127.60
Terbium	Tb	65	158.924
Thallium	Tl	81	204.37
Thorium	Th	90	232.038
Thulium	Tm	69	168.934
Tin	Sn	50	118.69
Titanium	Ti	22	47.90
Tungsten	W	74	183.85
Uranium	U	92	238.03
Vanadium	V	23	50.942
Xenon	Xe	54	131.30
Ytterbium	Yb	70	173.04
Yttrium	Y	39	88.905
Zinc	Zn	30	65.37
Zirconium	Zr	40	91.22

[a] *The atomic weight varies because of natural variations in the isotopic composition of the element. The observed ranges are boron, ±0.003; carbon, ±0.00005; hydrogen, ±0.00001; oxygen, ±0.0001; silicon, ±0.001; sulfur, ±0.003.*

[b] *The atomic weight is believed to have an experimental uncertainty of the following magnitude: bromine, ±0.002; chlorine, ±0.001; chromium, ±0.001; iron, ±0.003; silver, ±0.003. For other elements the last digit given is believed to be reliable to ±0.5.*

Table II   Mendeleev's Periodic Table Brought Up to Date

I	II	III	IV	V	VI	VII	VIII		
1 H 1.0080							2 He 4.0026		
3 Li 6.939	4 Be 9.0122	5 B 10.811	6 C 12.011	7 N 14.007	8 O 15.999	9 F 18.998	10 Ne 20.183		
11 Na 22.990	12 Mg 24.312	13 Al 26.981	14 Si 28.086	15 P 30.974	16 S 32.064	17 Cl 35.453	18 Ar 39.948		
19 K 39.102	20 Ca 40.08	21 Sc 44.956	22 Ti 47.90	23 V 50.942	24 Cr 51.996	25 Mn 54.938	26 Fe 55.847	27 Co 58.933	28 Ni 58.71
29 Cu 63.54	30 Zn 65.37	31 Ga 69.72	32 Ge 72.59	33 As 74.922	34 Se 78.96	35 Br 79.909	36 Kr 83.80		
37 Rb 85.47	38 Sr 87.62	39 Yt 88.905	40 Zr 91.22	41 Nb 92.906	42 Mo 95.94	43 Tc ...	44 Ru 101.07	45 Rh 102.905	46 Pd 106.4
47 Ag 107.870	48 Cd 112.40	49 In 114.82	50 Sn 118.69	51 Sb 121.75	52 Te 127.60	53 I 126.904	54 Xe 131.30		
55 Cs 132.905	56 Ba 137.34	57 La 138.91	72 Hf 178.49	73 Ta 180.95	74 W 183.85	75 Re 186.2	76 Os 190.2	77 Ir 192.2	78 Pt 195.09
79 Au 196.97	80 Hg 200.59	81 Tl 204.37	82 Pb 207.19	83 Bi 208.98	84 Po ...	85 At ...	86 Rn ...		
87 Fr ...	88 Ra ...	89 Ac ...	90 Th 232.04	91 Pa ...	92 U 238.03	93 Np ...	94 Pu ...	95 Am ...	96 Cm ...

58 Ce 140.12	59 Pr 140.91	60 Nd 144.24	61 Pm ...	62 Sm 150.35	63 Eu 151.96	64 Gd 157.25	65 Tn 159.2	66 Ds 162.50	67 Ho 164.93	68 Er 167.26	69 Tm 168.93	70 Yb 173.04	71 Lu 174.97
90 Th 232.04	91 Pa ...	92 U 238.03	93 Np ...	94 Pu ...	95 Am ...	96 Cm ...	97 Bk ...	98 Cf ...	99 E ...	100 Fm ...	101 Md ...	102 No ...	103 Lw ...

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Table III   Extended Form of the Periodic Table

IA	IIA	IIIB	IVB	VB	VIB	VIIB	VIII		
1 **H** 1.00797 ±0.00001									
3 **Li** 6.939 ±0.0005	4 **Be** 9.0122 ±0.00005								
11 **Na** 22.9898 ±0.00005	12 **Mg** 24.312 ±0.0005								
19 **K** 39.102 ±0.0005	20 **Ca** 40.08 ±0.005	21 **Sc** 44.956 ±0.0005	22 **Ti** 47.90 ±0.005	23 **V** 50.942 ±0.0005	24 **Cr** 51.996 ±0.001	25 **Mn** 54.9380 ±0.00005	26 **Fe** 55.847 ±0.003	27 **Co** 58.9332 ±0.00005	28 **Ni** 58.71 ±0.005
37 **Rb** 85.47 ±0.005	38 **Sr** 87.62 ±0.005	39 **Y** 88.905 ±0.0005	40 **Zr** 91.22 ±0.005	41 **Nb** 92.906 ±0.0005	42 **Mo** 95.94 ±0.005	43 **Tc** (99)	44 **Ru** 101.07 ±0.005	45 **Rh** 102.905 ±0.0005	46 **Pd** 106.4 ±0.05
55 **Cs** 132.905 ±0.0005	56 **Ba** 137.34 ±0.005	57 ***La** 138.91 ±0.005	72 **Hf** 178.49 ±0.005	73 **Ta** 180.948 ±0.0005	74 **W** 183.85 ±0.005	75 **Re** 186.2 ±0.05	76 **Os** 190.2 ±0.05	77 **Ir** 192.2 ±0.05	78 **Pt** 195.09 ±0.005
87 **Fr** (223)	88 **Ra** (226)	89 †**Ac** (227)							

***Lanthanum Series**

58 **Ce** 140.12 ±0.005	59 **Pr** 140.907 ±0.0005	60 **Nd** 144.24 ±0.005	61 **Pm** (147)	62 **Sm** 150.35 ±0.005	63 **Eu** 151.96 ±0.005

†Actinium Series

90 **Th** 232.038 ±0.0005	91 **Pa** (231)	92 **U** 238.03 ±0.005	93 **Np** (237)	94 **Pu** (242)	95 **Am** (243)

( ) Numbers in parentheses are mass numbers of most stable or most common isotope.

Atomic weights corrected to conform to the 1963 values of the Commission on Atomic Weights.

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IB	IIB	IIIA	IVA	VA	VIA	VIIA	INERT GASES	
						1 **H** 1.00797 ±0.00001	2 **He** 4.0026 ±0.00005	2
		5 **B** 10.811 ±0.003	6 **C** 12.01115 ±0.00005	7 **N** 14.0067 ±0.00005	8 **O** 15.9994 ±0.0001	9 **F** 18.9984 ±0.00005	10 **Ne** 20.183 ±0.0005	2 8
		13 **Al** 26.9815 ±0.00005	14 **Si** 28.086 ±0.001	15 **P** 30.9738 ±0.00005	16 **S** 32.064 ±0.003	17 **Cl** 35.453 ±0.001	18 **Ar** 39.948 ±0.0005	2 8 8
29 **Cu** 63.54 ±0.005	30 **Zn** 65.37 ±0.005	31 **Ga** 69.72 ±0.005	32 **Ge** 72.59 ±0.005	33 **As** 74.9216 ±0.00005	34 **Se** 78.96 ±0.005	35 **Br** 79.909 ±0.002	36 **Kr** 83.80 ±0.005	2 8 18 8
47 **Ag** 107.870 ±0.003	48 **Cd** 112.40 ±0.005	49 **In** 114.82 ±0.005	50 **Sn** 118.69 ±0.005	51 **Sb** 121.75 ±0.005	52 **Te** 127.60 ±0.005	53 **I** 126.9044 ±0.00005	54 **Xe** 131.30 ±0.005	2 8 18 18 8
79 **Au** 196.967 ±0.0005	80 **Hg** 200.59 ±0.005	81 **Tl** 204.37 ±0.005	82 **Pb** 207.19 ±0.005	83 **Bi** 208.980 ±0.0005	84 **Po** (210)	85 **At** (210)	86 **Rn** (222)	2 8 18 32 18 8

64 **Gd** 157.25 ±0.005	65 **Tb** 158.924 ±0.0005	66 **Dy** 162.50 ±0.005	67 **Ho** 164.930 ±0.0005	68 **Er** 167.26 ±0.005	69 **Tm** 168.934 ±0.0005	70 **Yb** 173.04 ±0.005	71 **Lu** 174.97 ±0.005	2 8 18 32 9 2

96 **Cm** (247)	97 **Bk** (247)	98 **Cf** (249)	99 **Es** (254)	100 **Fm** (253)	101 **Md** (256)	102 **No** (256)	103 **Lw** (257)	2 8 18 32 ? 9 2

LABORATORY PHYSICAL CHEMISTRY

To Dr. Harry Lewis, Dean Emeritus of the Institute of Paper Chemistry, Appleton, Wisconsin — known and loved by chemists throughout the United States and the world for his devoted and untiring efforts on behalf of chemical education. Dr. Lewis has the honor of being the "father" of MACTLAC, for it was he who made the first move to initiate this organization.

Preface

Physical chemistry, perhaps more than any other undergraduate chemistry course, has been in the process of evolution during the last several years. What the end result will be is not yet apparent. In the majority of colleges, however, the present course has a far different emphasis from that of ten or even five years ago, with increasing interest in independent work. Changes have been brought about not only by advances in the field itself but also by the efforts of the American Chemical Society to introduce physical chemistry earlier in the curriculum.

The almost exponential increase in the use of instruments in all fields of chemistry emphasizes the need for practical as well as theoretical training. Yet when physical chemistry is placed on the sophomore or junior level, the student more often lacks functional, rather than theoretical, background. Students of even exceptional intelligence vary widely in their ability and perception in dealing with physical apparatus, and some are markedly deficient in this regard. This is more than a matter of physical dexterity or of eye–hand coordination and stems from lack of early experience in tool utilization. There is increasing need for young scientists to learn tool utilization: mathematical, mechanical, and instrumental. Especially necessary is the skill of transferring concepts from print or diagrams to tangible equipment. This can only be acquired through actual practice — hence the value of having physical chemistry laboratory early in the undergraduate years, embracing a wide variety of techniques.

Reference

The reference manual contains pertinent information on all phases of tool use. Here the word "tool" is used in its broadest sense, including writing and mathematics as well as physical and electrical equipment. With regard to the latter, there is information of the purpose of equipment and its applications, limitations, availability, proper use, and safety precautions. The coverage here has been limited to the immediate needs of the student in subject areas for which one or two excellent and readily available reference works are current. Thus the chapter on electronic equipment is quite short. Subjects in which the needed information is fragmentary, widely scattered, or perhaps unpublished, are covered in much greater detail.

This manual is intended to serve as an introduction to the experiments, as a ready reference, and as a supplement to guide the course of independent work. The rapid growth of undergraduate research, with exploration of the widest variety of chemical and ins‚ techniques, makes necessary a general compendium of this kind. It no 'y supplies much of the information needed for preliminary explorations, but also points the way for safe and effective use of equipment for further discoveries. Unique in its field, its value as a reference can well extend into the years of graduate research and beyond.

Experiments

The collection of experiments is the result of a cooperative effort by physical chemistry instructors of the Midwestern Association of Chemistry Teachers in Liberal Arts Colleges. The plan has been to collect experiments or ideas for experimental work from available sources, to rewrite or edit these into conveniently usable form, to submit them to the membership for testing by their students, and to revise before publication on the basis of comments received.

This procedure has not been uniformly applied to all the experiments in the manual. In spite of their being written or revised by experts in the subjects, there undoubtedly still remain rough spots and factors which may cause students difficulty. We do not believe this to be entirely bad. Only when a student is challenged to the limits of his ability does he advance most rapidly. We often learn more from our mistakes than from our successes.

The experiments so far included in this manual vary greatly in difficulty and in treatment. A few involve simple techniques for which detailed directions are given, such that any student should be able to follow them successfully. Most are of intermediate difficulty, and are open-ended in the sense that the method can be extended beyond the examples given into exploratory work verging on research. For several, only general directions are given, thus putting more responsibility upon the student. Two or three require fairly sophisticated apparatus and difficult techniques. This variety should meet the needs of class in most undergraduate colleges and universities.

In general, we believe it is better to assign fewer experiments in physica chemistry, perhaps five to eight each semester, and give the student time to explore several in depth. This way he gets a chance to apply his newly won knowledge of techniques to other chemical systems of his own choosing.

Note. The experiments have been grouped as indicated in the table of contents, but the selection is such as to allow other interesting and stimulating combinations. For example, Experiments 8, 16, and 25 and 26 might equally well be considered under the heading of equilibrium phenomena. Similarly Experiments 10 and 11 and 13 could be grouped under photometric methods. The field of thermodynamics might include Experiments 2 and 3 and 6 as well as those listed as 16–19. Other regroupings are possible.

The construction projects are included for those who consider physical chemistry laboratory to be an introduction to physical methods of research and a preparation for such work on the graduate level. There, nearly every research project involves either personal construction or direction of shop personnel in the fabrication of specialized apparatus. We believe this is a most

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valuable experience for those students orward to graduate training and recommend the projects in this manual as well within the capacity of most undergraduate physical chemistry students.

It is our intention to add to and revise this collection of experiments from time to time. We invite the submission of ideas and manuscripts.

The members of the present Editorial Board wish to thank all who have so generously contributed their time, thought, and effort to this cooperative undertaking. This includes not only the authors, whose names will be found on the individual experiments, but also the many others who have supplied ideas, tested experiments, and supplied helpful criticism of the trial forms. Credit also goes to the several colleges that contributed secretarial time and the use of duplicating equipment. Lastly, we express our appreciation to the unsung students who really helped to pinpoint the weak spots in the experiments.

The Editorial Board

Dr. A. L. Hanson	Saint Olaf College
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Dr. P. M. Wright	Wheaton College

by W. C. Oelke, chairman.

Authorship

This laboratory reference manual, with the accompanying collection of experiments, has resulted from the joint efforts of a group of experienced teachers from MACTLAC. The name MACTLAC, the Midwestern Association of Chemistry Teachers in Liberal Arts Colleges, speaks for itself.

At the 1957 meeting of MACTLAC at Park College a lively discussion took place on the role of the laboratory in physical chemistry. Stimulated by this discussion, W. C. Oelke arose at the end of the meeting and asked whether any would be interested in cooperatively developing better laboratory experiments for physical chemistry classes. A small group responded with enthusiasm. The next year a steering committee was elected, which grew into the Editorial Board of the Cooperative Physical Chemistry Manual Project. This board presently consists of the following:

Dr. A. L. Hanson, *Saint Olaf College*
Dr. J. P. Huselton, *formerly of William Jewell College*, secretary
Dr. W. C. Oelke, *Grinnell College*, editor
Dr. A. C. Wilcox, *Wisconsin State University*
Dr. P. M. Wright, *Wheaton College*
Dr. R. M. Rosenberg, *Lawrence College*, added 1965

It was early agreed that Dr. Oelke would be personally responsible for writing the Reference Manual, and the Board, jointly, for the Manual of Experiments. The Reference Manual is thus the work of Dr. Oelke, except for Chapter 11, which is the work of Dr. Richard W. Zuehlke of Lawrence University.

The senior author acknowledges with the greatest respect and admiration the direct contributions of the gentlemen just mentioned, as well as the continued help, advice, and encouragement of the members of the Editorial Board and his many friends among the larger group of cooperators.

W. C. Oelke

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