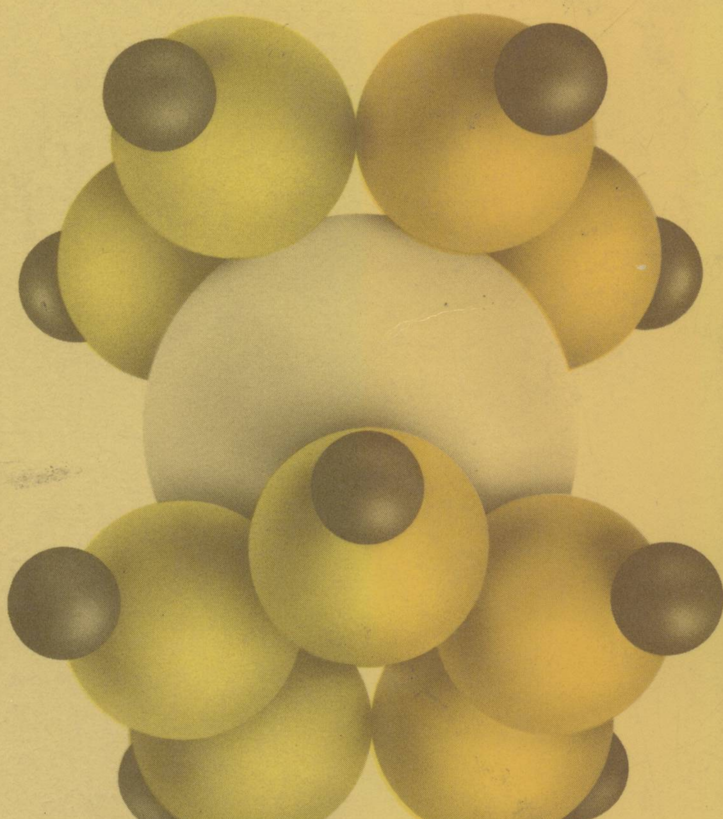
Experimental General Chemistry



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PREFACE

This is a laboratory text for a college general chemistry course. It is designed to show some ways in which chemists synthesize desired compounds, and how they obtain information by experiment and then use the collected data to draw conclusions about the properties and behavior of matter. It is planned to help students learn not only to perform essential laboratory operations, but also to observe, to correlate and interpret data, and to think and act in laboratory situations.

The 35 experiments include portions of a variety of topics, including separations, identification, stoichiometry, qualitative and quantitative analysis, coordination chemistry, chemical equilibrium, electrochemistry, thermodynamics, organic chemistry and biochemistry. They were selected to illustrate both modern and traditional techniques, and to provide teachers and students with a large number of options in choosing experiments to be used and in determining the amount of time and study to be given to any one experiment.

Approximately one-third of the experiments consist of several parts, any or all of which may be assigned or elected. This provides the opportunity for individualized assignments and for some students to spend more or less time on a given experiment than others do. Thus, one student may complete three or four parts of a single experiment, while others in the same laboratory are working on parts of several different experiments. For example, many students, having learned a new and moderately complicated technique, such as measurement of vapor pressure, especially like to continue to use this technique in more challenging ways; other students prefer to move on to other experiments rather than repeat a learned activity. Options such as these in several variations are included in numerous combinations of experiments.

Some experiments relate to the chemistry of everyday life. For example, in Experiment 33 students can synthesize at least four medicinally useful compounds; in Experiment 34 they can identify amino acids obtained from the palms of their hands; in Experiment 6 they can discover for themselves some relationships between color, wavelength and energy of electronic transitions. Other experiments illustrate exciting modern chemistry, such as the synthesis and properties of ferrocene (Experiment 7), the structure of ionic crystals from x-ray diffraction data (Experiment 26) and the electronic spectra of coordination compounds (Experiment 25).

Because lecture and textbook presentations often emphasize the results rather than the details of experiments, each experiment begins with an introduction, giving background information needed to bridge the gap between lecture presentation of the topic and laboratory study of a small segment of it.

Many experiments require more than one laboratory period for completion. Because each experiment msut be done carefully and new techniques constantly learned, it is not expected that all the experiments can be completed in a one-year course. With the large number provided, it is possible to select a different group of experiments in successive years or to give students a choice of experiments during one year.

Among the techniques included are: use of the analytical balance; paper, column and thin-layer chromatography; spectroscopic and spectrophotometric methods; qualitative analysis of cations, anions and simple organic substances; precipitation, ion exchange and solvent extraction quantitative separations; colorimetric, gravimetric and volumetric analysis, includir acid-base, precipitation, oxidation-reduction and complexometric titration measurement of vapor pressure, vapor density, enthalpy change, free energ change, and equilibrium, reaction rate, and electrochemical data; certa aspects of inorganic and organic synthesis.

All experiments are designed for students with little previous laborato experience, but students are expected to work carefully and to learn go laboratory habits. The apparatus and instruments used are, with exceptions, inexpensive and simple to work with. All experiments requi written report, and there is a section dealing with record-keeping in laboratory notebook. Because the evaluation of numerical data is essenti many experiments, special sections on errors of measurement and treatment, use of significant figures and graphical representation of dat provided (Experiment 35).

Most of these experiments have been used in the general chem laboratories at the Ohio State University for several years. They have scrutinized by thousands of students and hundreds of instr Pre-laboratory films prepared to accompany certain of these expe also have been used for several years at Ohio State. They have been f increase significantly the speed and efficiency with which students e use the many experimental techniques called for.

No laboratory experiments that have been developed at a large u can be considered to be the work of three people alone. To the stu teaching assistants, to those who have shared their experiences th literature, and especially to our colleagues at Ohio State, Ar elsewhere who have used or examined this laboratory pro acknowledge with grateful appreciation our debt for assistance and

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TO THE STUDENT

Chemistry, like all other sciences, is built on experimentation. This means that the knowledge accumulated by chemists is obtained or inspired by experiment. In order to appreciate the ecological, medical, and biochemical problems confronting us all in this technological age, and to appreciate science more fully, one needs to become actively involved in the process of collecting and interpreting data. No description of an observed phenomenon, no development of a theory, no classification of facts has complete meaning until a person has experienced for himself the difficulties in obtaining a reliable experimental result, understood the implications of an assumption made in developing a theory, or verified the results of another worker.

It is impossible for anyone to repeat even a small percentage of the chemical experiments that have been performed during the past 200 years. Thus, in planning an introductory course, most chemical educators elect to present the important principles of the science in the lecture portion of the course and to illustrate some of these principles with appropriate laboratory exercises. In addition to illustrating principles in a quantitative manner, we have attempted in this laboratory program to incorporate some new techniques, manipulative skills, and ingenuity into the experiments and methods of experimentation.

For example, Experiment 12 illustrates the methods by which vapor
essure data of a liquid can be obtained and the way these data can be used
determine the heat of vaporization of the liquid. In this case, a value for a
perty–specifically, the heat of vaporization of the liquid–is obtained
out actually measuring that property. Since it is often difficult or
ssible to measure certain properties of matter, such techniques are
oyed frequently. Experiment 7 involves the synthesis of ferrocene,
$H_5)_2$, and a study of its oxidation to the ferrocinium ion,
$H_5)_2^+$, and the reduction back to ferrocene. Ferrocene was prepared in
for the first time; its unusual "sandwich" structure and chemical
rties stimulated intense research into a new class of organometallic
ounds that now encompasses almost all of the transition metal
nts and very recently has been extended to "sandwich" compounds of
nthanide and actinide elements. By judging the relative colors of
es containing ferrocene and ferrocinium ion, you will be asked to
the reducing and oxidizing agents in a relative order. Experiment 33
es the transformation of a common organic chemical into several
t compounds either by one reaction step or by a sequence of steps. A
reactions originating from one compound is often used by modern
ists and by organic and pharmaceutical chemists for the synthesis of
cated new chemical or a drug.

As mentioned above, several of these laboratory experiments are sequential. For example, in Experiment 2 you separate a mixture and determine its quantitative composition. Then in Experiment 3 you identify qualitatively the organic and inorganic salt components of the mixture. In Experiment 9 you prepare and standardize a sodium hydroxide solution and then use the solution in Experiment 10 to determine the equivalent weight of an unknown acid. Thus, both Experiments 9 and 10 must be performed carefully to obtain the correct equivalent weight. Similarly, Experiments 29, 30 and 31 involve three different quantitative analyses of the same unknown.

Many of the experiments have a quantitative basis; consequently, you will generally be asked to determine a value or to make calculations based on your data. Thus, careful efficient work is required throughout the laboratory program. Chemicals generally will be in the laboratory one week before an experiment is scheduled and for one to two weeks afterward, so that a student may proceed at his own pace. However, experience has shown that certain deadlines for experiments must be established if the work is to be completed during the term. The instructor will remind you of these deadlines.

No chemist can afford to begin an experiment without having planned it. Similarly, students of chemistry must be prepared *before* entering the laboratory. This means that you must study and *understand* the principles and experimental procedures before going to the laboratory. Your instructor will help you through the difficult aspects of an experiment if you are prepared, but he cannot be of much help if you do not study the experiment before the laboratory period.

Experimental data are recorded as a permanent record in a laboratory notebook in an organized and legible manner. You should study the experiment before class and prepare the necessary data tables in your notebook in which to record the observations *as you collect them.* Never record numbers or observations on a loose piece of paper with the intent of transferring the data to your record book later. Time spent before class in organizing your data collection and recording steps is invested wisely.

A report of the experiment should be prepared as described in the following section or as modified by your instructor. Careful analysis and interpretation of the data are just as important as making the observations. As you collect the data and as you correlate the results, be aware of instances in which quantitative techniques are required, sources of error, and whether the inherent precision of the measuring device or of the investigator is the determining factor to accuracy in a given experiment. During the first six experiments you will gain experience with the types of problems encountered in collecting and evaluating data. Because the experiments have a quantitative emphasis, at this point it is important to study "Evaluation of Experimental Data" carefully; include a treatment of errors and an evaluation of the precision in your reports of subsequent experiments.

Some experiments give you a choice of projects, any or all of which may be undertaken; others are open-ended so that you can pursue an interesting aspect of the problem as a small individual research problem. Discuss your proposed extensions with the instructor and obtain his permission before proceeding. **NEVER ATTEMPT TO CONDUCT AN UNAUTHORIZED EXPERIMENT.**

The experiments in this program are intended to challenge you. Whereas the lecture material gives you an overview of chemistry, these experiments should give you a good idea of the methods by which a chemist obtains information by experimentation and how he uses the data to formulate conclusions about the properties and behavior of matter. All the chemical information that you will be reading, discussing and studying during the year has been accumulated from data collected in experiments and from the interpretation of those data. This laboratory program will help you gain experience in carefully performing experiments, learning chemical techniques and recording your observations. Also, it should serve as a guide for relating the data to those obtained in other experiments, questioning the significance of the data and creatively interpreting the results.

SAFETY AND LABORATORY RULES

Accidents in a chemical laboratory usually result from improper judgment on the part of the victim or one of his neighbors. *Learn and observe the safety and laboratory rules listed below.* If an accident to you or your neighbor does occur, summon help immediately from the laboratory instructor.

1. *Wear safety glasses.* Because the eyes may be permanently damaged by spilled chemicals and flying broken equipment, be sure to wear safety goggles or safety glasses *at all times* in the laboratory. If you get anything in your eye, report it immediately to your instructor. In laboratories equipped with an *eye wash fountain,* learn its location and how to use it the first day in the laboratory. Note that routine eye-washing with a contact lens in place will not clear a splashed chemical from the eye; wash, remove your contacts and wash again.

2. *Locate safety equipment.* During the first laboratory period familiarize yourself with the location of the safety features of the laboratory, including the *safety shower, fire extinguisher, fire blanket, laboratory first aid kits* and *first aid room.* The safety shower should be used if your clothing catches on fire or if a corrosive chemical is spilled on you in quantities that cannot be easily flushed away at laboratory faucets.

3. *Don't cut yourself.* Cuts and burns are the most common injuries occurring in chemistry laboratories. Cuts can be prevented by following a few simple rules:

a. When inserting glass tubing or thermometers into rubber stoppers, *always* use glycerin (avilable on the shelf) or soapy water as a lubricant on both the glass tubing and the hole. *Always* protect your hand by wrapping the glass tubing with a towel.
b. Fire-polish all sharp edges of broken glass.
c. Discard cracked or broken glassware immediately.
d. Never heat a graduated cylinder with a burner flame.

4. *Wash chemicals from skin.* If you receive a chemical burn from acid, alkali or bromine, immediately wash the burned area with *large quantities* of water. Ask another student to summon the laboratory instructor.

5. *Be careful with flames.* A lighted gas burner can be a major fire hazard. The burner should be burning only for the period of time in which it is actually utilized. Carefully position it on the desk away from flammable materials and overhanging reagent shelves. Before lighting your burner, make sure that flammable reagents (such as acetone, benzene, ether and alcohol)

on neighboring desks are well separated from your burner. Be careful not to extend your arm over a burner while reaching for something.

When heating a test tube over a burner or carrying out a reaction in one, never point the test tube toward your laboratory neighbor or yourself.

Long or tousled hair, especially if covered with hair spray, is a major hazard to the wearer in the laboratory. Keep long hair tied back so that it cannot fall forward into a flame. Sprayed hair, long or short, presents a large surface area of flammable plastic to the oxygen of the air, causing almost explosive combustion if ignited. Beards, too, present a hazard to their owners. *Keep hair away from flames.*

6. *Wear suitable clothing.* Clothing is a protection against spilled chemicals or flaming liquids. Dress which exposes large areas of bare skin is a laboratory hazard, and open-toed shoes or sandals are an invitation to maimed feet. *Footwear must be worn in the laboratory at all times.*

7. *Smell cautiously.* Many chemicals used in the laboratory are toxic. If you are instructed to smell a chemical, do so by pointing the vessel away from your face and carefully fanning the vapors toward your face with your hand and sniffing gently. Never taste a chemical unless specifically directed to do so.

8. *Don't pipet by mouth.* Always use a rubber bulb or rubber tubing connected to an aspirator to fill your pipet. Never fill the pipet by using your mouth.

9. *Assemble safe apparatus.* Makeshift equipment and poor apparatus assemblies are the first steps to an accident. Always assemble an apparatus as outlined in your instructions.

10. *Pour acid into water.* Never pour water into concentrated acid. Concentrated acids and bases may be diluted by pouring the reagent into water while stirring it carefully and constantly.

Never add concentrated acid to concentrated base or vice versa.

11. *Use the hood.* Any experiment involving the use of or production of objectionable (i.e., poisonous or irritating) gases must be performed in the hoods.

12. *Read the label.* Read the label carefully before taking anything from a bottle. Many chemicals have similar names, such as sodium sulfate and sodium sulfite; it is obvious that use of the wrong reagent can spoil an experiment or, in some cases, cause a serious accident.

13. *Leave reagents on the shelves.* Do not carry reagent bottles to your desk. This is a matter of courtesy to the other students in the class, and it minimizes the possibility of contamination of the reagent. Obtain the required quantities of chemicals from the reagent shelf by taking clean test tubes or beakers to the reagent area. Take the amount you need and return the bottle to the shelf. Make it a practice not to take much more material than is required for the experiment, because many chemicals are quite expensive.

14. *Don't contaminate reagents.* Do not insert spatulas or pipets into reagent bottles. Remove a solid reagent from the stock bottle by pouring it onto a clean watch glass or filter paper by gentle rotation of the bottle. To transfer a solid material to a test tube, first place the solid on a creased narrow strip of paper. Then insert the paper into the test tube and shake the solid down the crease by gentle tapping with a finger.

Never put a stopper on the table or working surface where it can pick up dirt or another chemical; hold it in the hand while removing reagent. A stopper with a flat head is conveniently removed by grasping the stopper between the two middle fingers, with the *palm of the hand facing upward.* Hold the stopper in this position until you return it to the bottle.

Never return excess materials to reagent bottles. Pour them down the sink drain if they are water-soluble and nontoxic, and flush with water. Water-insoluble and toxic materials should be placed in the waste jars provided for this purpose. If you are not certain how to dispose of excess chemicals, consult your instructor.

15. *Insoluble liquids and solids go into waste cans.* Place paper, glass and similar items in the proper waste cans, *not in the sinks.* Insoluble solvents, such as benzene and dichloromethane, go into capped waste-solvent containers.

16. *Perform only authorized experiments. Anyone attempting to conduct unauthorized experiments will be subject to immediate and permanent expulsion from the laboratory.*

17. *Plan ahead.* Carefully study each experiment prior to the laboratory period in which the experiment is to be performed. To use the laboratory time efficiently, you must have a clear plan of action, and all data tables must be outlined in the laboratory notebook before performing the experiment.

18. *Keep your work space orderly.* Never place coats, books, and other belongings on the laboratory bench where they will interfere with the experiment and are likely to be damaged. Place tall items, such as graduated cylinders, toward the back of the workbench so they will not be overturned by reaching over them.

19. *Clean up for the next student.* At the end of each laboratory period, wash and wipe off your desk top. Be sure the gas and water are turned off. *Return all special equipment to the stockroom.* Be sure to put everything back into your locker drawer, and also *be sure that your locker drawer is locked before you leave the laboratory.*

THE LABORATORY NOTEBOOK

The importance of keeping an up-to-the-minute record of what you are doing in the laboratory cannot be overemphasized.

A section entitled Notebook and/or Report is included with each experiment to assist you in organizing the recording of observations and data. When completed, this section should become a portion of the notebook and report.

The authors recommend that each student provide himself with a spiral-bound notebook of the size specified by the instructor (8 inch × 10 inch is convenient). A loose-leaf notebook is not acceptable, and some instructors may require a bound notebook. The spiral-bound type is convenient because it folds back on itself when open and requires less space on the laboratory bench. All data and comments should be entered *directly in the notebook, never on a loose sheet of paper* with the intention of copying it into the notebook later. Scraps of paper or loose sheets can get lost between laboratory periods; notebooks are much less likely to be misplaced. A Report Sheet is included with each experiment, and that should provide a neat summary of your notebook data for the experiment.

Enter the work carried out in each laboratory period under that day's date, and add nothing to these entries at a later date. If you discover on Wednesday that something additional should have been recorded on Monday, write it under Wednesday's date, with a note that it should have been a part of Monday's record. Never erase anything in a notebook. If a mistake has been made, cross out the erroneous material with a single line *so that it can still be read,* and rewrite the correct statement in place of it. Even this should not be done except on the day the error is made; if the error is discovered at a later date, simply make an entry on that date (October 14, for example) to the effect that "the material in the second paragraph under the date of October 12 is incorrect and should be ignored; the corrected version follows," and record the correct information under the date of October 14. The material in quotation marks indicates the informal style in which a notebook may be kept; it is usually written in the first person; e.g., "Although the procedure suggested that about 7 ml of 2 M HCl would be required to neutralize the base present, I had to add 9.5 ml before the indicator changed color. The greater volume apparently did no harm, because the precipitate formed readily, as it was supposed to."

WHAT IS TO BE RECORDED IN THE NOTEBOOK?

The criterion of an acceptable laboratory notebook is that the record should be so complete that a second person, with the notebook at hand, could repeat your experiments exactly as you did them, and would know the reason you selected the particular procedure you followed. This means that you should carry out the following steps: (1) Record all numerical data in such a way that the reader knows to what the numbers refer. A three line entry that appears as

1.2463
0.8297
0.4166

for example, is meaningless to a second person (and will become meaningless to you, too, after a few weeks have passed). But an entry such as the following

Wt. of crucible and salt	1.2463 g
Wt. of empty crucible	0.8297 g
Wt. of salt	0.4166 g

is perfectly clear (provided that the salt is described elsewhere). The weights given should have been recorded at the balance; take the notebook there, and copy the balance reading directly. (2) Other observations should be indicated. If the solution turns red when acidified, record this information. If the precipitate is gelatinous rather than crystalline, write it down. Nothing is too trivial to record; if there is any doubt whether something should or should not be recorded, the answer is always "Record it!" (3) The notebook must include the calculations based on the data. Many laboratory workers find it convenient to use the right-hand pages for recording procedures, observations, and data, and to use the left-hand pages, opposite the data, for calculations based on those data. Again, no calculation is too trivial to record; if you are to weigh out 0.10 mole of KCl, show on the left-hand page your addition of the atomic weights of potassium and chlorine to find the weight of 1 mole and your multiplication by 1/10 to determine the weight required. (4) Write chemical equations for all reactions that occur. (5) Sometimes, in the experiments, you are instructed to display graphically the results of your experiments or calculations. You would normally paste these graphs on one of the pages of your notebook. However, your instructor may request that you hand them in as part of your report; if so, make a free-hand sketch in the notebook of the way the graph looked, and paste the graph in the notebook when the report is handed back to you. (6) The procedures you followed in carrying out the experiments would normally be part of the notebook record. In most of the experiments under discussion here, however, detailed procedures are given in the laboratory manual. There is no need to copy these into the notebook; simply refer, in the notebook, to the instructions in the manual, but again in sufficient detail so that the reader will know exactly what you did; e.g., "Following the procedure outlined in the paragraphs below Figure 9-1 on p. 109, I set up two plastic funnels with ashless filter paper, and . . . ." (7) Record discussions of the work with your neighbors or the instructor: "I showed the precipitate to Mr. Blank, telling him what I had done, and he suggested that I add 4 ml of . . . ."

It is not easy to keep a satisfactory notebook. One naturally desires to keep it clean. This stands in the way of recording at any instant the observed data or thoughts in connection with the work. The notebook must be available for use at the time the observations are made; if this availability causes the notebook to become spotted, regard the spotting as a small price to pay. But this does not mean that a notebook need be *illegible* as well as spotted. Neatness in keeping a notebook will pay off in understanding what you did when you refer to the notebook at a later date. Make it a habit to tabulate data whenever possible (be sure to label column headings), separate the text into paragraphs, numbered if necessary, and use as much space as is needed.

THE REPORT

In all cases, the essential features of the experiment and its results should be summarized at the end of the notebook record. Often this summary, plus the notebook record, will be sufficient to serve as a report, if this is required by the instructor. The essential features of a report include answers to the six questions: What did you do? Why did you do it? What were the results? What conclusions did you draw from these results? How valid or precise are these conclusions? How could you improve the precision or definiteness of the conclusions? A two-page report is usually ample, and a shorter one often is adequate.

	IA	IIA	IIIB	IVB	VB	VIB	VIIB	VIII	VIII	VIII	IB	IIB	IIIA	IVA	VA	VIA	VIIA	O
1	1 1.00797 H Hydrogen																	2 4.0026 He Helium
2	3 6.939 Li Lithium	4 9.0122 Be Beryllium											5 10.811 B Boron	6 12.01115 C Carbon	7 14.0067 N Nitrogen	8 15.9994 O Oxygen	9 18.9984 F Fluorine	10 20.183 Ne Neon
3	11 22.9898 Na Sodium	12 24.312 Mg Magnesium											13 26.9815 Al Aluminum	14 28.086 Si Silicon	15 30.9738 P Phosphorus	16 32.064 S Sulfur	17 35.453 Cl Chlorine	18 39.948 Ar Argon
4	19 39.102 K Potassium	20 40.08 Ca Calcium	21 44.956 Sc Scandium	22 47.90 Ti Titanium	23 50.942 V Vanadium	24 51.996 Cr Chromium	25 54.938 Mn Manganese	26 55.847 Fe Iron	27 58.933 Co Cobalt	28 58.71 Ni Nickel	29 63.54 Cu Copper	30 65.37 Zn Zinc	31 69.72 Ga Gallium	32 72.59 Ge Germanium	33 74.922 As Arsenic	34 78.96 Se Selenium	35 79.909 Br Bromine	36 83.80 Kr Krypton
5	37 85.47 Rb Rubidium	38 87.62 Sr Strontium	39 88.905 Y Yttrium	40 91.22 Zr Zirconium	41 92.906 Nb Niobium	42 95.94 Mo Molybdenum	43 (98) Tc Technetium	44 101.07 Ru Ruthenium	45 102.905 Rh Rhodium	46 106.4 Pd Palladium	47 107.870 Ag Silver	48 112.40 Cd Cadmium	49 114.82 In Indium	50 118.69 Sn Tin	51 121.75 Sb Antimony	52 127.60 Te Tellurium	53 126.904 I Iodine	54 131.30 Xe Xenon
6	55 132.905 Cs Cesium	56 137.34 Ba Barium	57 138.91 La* Lanthanum	72 178.49 Hf Hafnium	73 180.948 Ta Tantalum	74 183.85 W Wolfram	75 186.2 Re Rhenium	76 190.2 Os Osmium	77 192.2 Ir Iridium	78 195.09 Pt Platinum	79 196.967 Au Gold	80 200.59 Hg Mercury	81 204.37 Tl Thallium	82 207.19 Pd Lead	83 208.980 Bi Bismuth	84 (210) Po Polonium	85 (210) At Astatine	86 (222) Rn Radon
7	87 (223) Fr Francium	88 (226) Ra Radium	89 (227) Ac** Actinium															

* 6	58 140.12 Ce Cerium	59 140.907 Pr Praseodymium	60 144.24 Nd Neodymium	61 (147) Pm Promethium	62 150.35 Sm Samarium	63 151.96 Eu Europium	64 157.25 Gd Gadolinium	65 158.924 Tb Terbium	66 162.50 Dy Dysprosium	67 164.930 Ho Holmium	68 167.26 Er Erbium	69 168.934 Tm Thulium	70 173.04 Yb Ytterbium	71 174.97 Lu Lutetium
** 7	90 232.038 Th Thorium	91 (231) Pa Protactinium	92 238.03 U Uranium	93 (237) Np Neptunium	94 (242) Pu Plutonium	95 (243) Am Americium	96 (245) Cm Curium	97 (249) Bk Berkelium	98 (251) Cf Californium	99 (254) Es Einsteinium	100 (255) Fm Fermium	101 (256) Md Mendelevium	102 (254) No Nobelium	103 (257) Lw Lawrencium

ATOMIC NUMBER ATOMIC WEIGHT SYMBOL NAME

Explanation:

Symbol
BLACK—solid
DARK GRAY—synthetically prepared
LIGHT GRAY BLOCK—gas
OUTLINED—liquid

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