Preparation of Common Types of Reagent Water and Desk Reagents Specified In This Book

Common Types of Reagent Water

a. Distilled water: Prepare reagent-grade water by distillation. Distilled water quality depends on the type of still and quality of feedwater. Deionized feedwater is preferred. Commercial resin purification trains can produce equivalent or superior water for some applications. Confirm suitability of distilled water or equivalent for specific analyses, including freedom from significant levels of interference and the constituent being determined.

b. Carbon dioxide-free distilled water: Prepare fresh as needed by boiling distilled water for 15 min and cooling rapidly to room temperature. Exclude CO_2 entry during cooling and storage with a tube containing soda lime or a commercially available CO_2-removing agent.*

c. Redistilled water: Prepare by redistilling single-distilled water from an all-borosilicate-glass or quartz distillation apparatus.

d. Deionized distilled water: Slowly pass distilled water through a 25-cm column of glass tubing (1 to 2.5 cm in diameter) that has been charged with 2 parts by volume of a strongly basic anion-exchange resin in the hydroxyl form and 1 part by volume of a strongly acidic cation-exchange resin in the hydrogen form. Use ion-exchange resins of a quality suitable for analytical work. Units using such resins in flow-through sequence can produce water of very high quality and are available commercially.

*Ascarite, Fisher Scientific Co.; Caroxite, A. H. Thomas Co.; or equivalent.

TABLE A: PREPARATION OF UNIFORM ACID SOLUTIONS*

Desired Component	Hydrochloric Acid (HCl)	Sulfuric Acid (H_2SO_4)	Nitric Acid (HNO_3)
Specific gravity (20/4°C) of ACS-grade conc acid	1.174–1.189	1.834–1.836	1.409–1.418
Percent of active ingredient in conc reagent	36–37	96–98	69–70
Normality of conc reagent	11–12 (for .5N)	36	15–16
Volume (mL) of conc reagent to prepare 1 L of:			
18*N* solution	—	500 (1 + 1)†	—
6*N* solution	500 (1 + 1)†	167 (1 + 5)†	380
1*N* solution	83 (1 + 11)†	28	64
0.1*N* solution	8.3	2.8	6.4
Volume (mL) of 6*N* reagent to prepare 1 L of 0.1*N* solution	17	17	17
Volume (mL) of 1*N* reagent to prepare 1 L of 0.02*N* solution	20	20	20

*All values approximate.

†The *a* + *b* system of specifying preparatory volumes appears frequently throughout this manual and means that *a* volumes of the concentrated reagent are diluted with *b* volumes of distilled water to form the required solution.

HF conc = 48%

Acid Solutions

Prepare the following reagents by cautiously adding required amount of concentrated acid, with mixing, to designated volume of proper type of distilled water. Dilute to 1000 mL and mix thoroughly.

See Table A for preparation of HCl, H_2SO_4, and HNO_3 solutions.

Alkaline Solutions

a. Stock sodium hydroxide, NaOH, 15*N* (for preparing 6*N*, 1*N*, and 0.1*N* solutions): Cautiously dissolve 625 g solid NaOH in 800 mL distilled water to form 1 L of solution. Remove sodium carbonate precipitate by keeping solution at the boiling point for a few hours in a hot water bath or by letting particles settle for at least 48 h in an alkali-resistant container (wax-lined or polyethylene) protected from atmospheric CO_2 with a soda lime tube. Use the supernate for preparing dilute solutions listed in Table B.

Alternatively prepare dilute solutions by dissolving the weight of solid NaOH indicated in Table B in CO_2-free distilled water and diluting to 1000 mL.

TABLE B: PREPARATION OF UNIFORM SODIUM HYDROXIDE SOLUTIONS

Normality of NaOH Solution	Required Weight of NaOH to Prepare 1,000 mL of Solution *g*	Required Volume of 15*N* NaOH to Prepare 1,000 mL of Solution *mL*
6	240	400
1	40	67
0.1	4	6.7

Store NaOH solutions in polyethylene (rigid, heavy-type) bottles with polyethylene screw caps, paraffin-coated bottles with rubber or neoprene stoppers, or borosilicate-glass bottles with rubber or neoprene stoppers. Check solutions periodically. Protect them by attaching a tube of CO_2-absorbing granular material such as soda lime or a commercially available CO_2-removing agent.* Use at least 70 cm of rubber tubing to minimize vapor diffusion from bottle. Replace absorption tube before it becomes exhausted. Withdraw solution by a siphon to avoid opening bottle.

b. Ammonium hydroxide solutions: NH_4OH: Prepare 5*N*, 3*N*, and 0.2*N* NH_4OH solutions by diluting 333 mL, 200 mL, and 13 mL, respectively, of the concentrated reagent (sp gr 0.90, 29.0%, 15*N*) to 1000 mL with distilled water.

Indicator Solutions

a. Phenolphthalein indicator solution: Use either the aqueous (1) or alcoholic (2) solution.

1) Dissolve 5 g phenolphthalein disodium salt in distilled water and dilute to 1 L.

2) Dissolve 5 g phenolphthalein in 500 mL 95% ethyl or isopropyl alcohol and add 500 mL distilled water.

If necessary, add 0.02*N* NaOH dropwise until a faint pink color appears in solution 1) or 2).

b. Methyl orange indicator solution: Dissolve 500 mg methyl orange powder in distilled water and dilute to 1 L.

103 EXPRESSION OF RESULTS*

1. Units

This text uses the International System of Units (SI) and chemical and physical results are expressed in milligrams per liter (mg/L). Record only the significant figures. If concentrations generally are less than 1 mg/L, it may be more convenient to express results in micrograms per liter (μg/L). Use μg/L when concentrations are less than 0.1 mg/L.

Express concentrations greater than 10 000 mg/L in percent, 1% being equal to 10 000 mg/L when the specific gravity is 1.00. In solid samples and liquid wastes of high specific gravity, make a correction if the results are expressed as parts per million (ppm) or percent by weight:

$$\text{ppm by weight} = \frac{\text{mg/L}}{\text{sp gr}}$$

$$\text{\% by weight} = \frac{\text{mg/L}}{10\,000 \times \text{sp gr}}$$

In such cases, if the result is given as milligrams per liter, state specific gravity.

The unit equivalents per million (epm), or the identical and less ambiguous term milligram-equivalents per liter, or milliequivalents per liter (me/L), can be valuable for making water treatment calculations and checking analyses by anion-cation balance.

Table 103:I presents factors for converting concentrations of common ions from milligrams per liter to milliequivalents per liter, and vice versa. The term milliequivalent used in this table represents 0.001 of an equivalent weight. The equivalent weight, in turn, is defined as the weight of the ion (sum of the atomic weights of the atoms making up the ion) divided by the number of charges normally associated with the particular ion. The factors for converting results from milligrams per liter to milliequivalents per liter were computed by dividing the ion charge by weight of the ion. Conversely, factors for converting results from milliequivalents per liter to milligrams per liter were calculated by dividing the weight of the ion by the ion charge.

2. Significant Figures

To avoid ambiguity in reporting results or in presenting directions for a procedure, it is the custom to use "significant figures." All digits in a reported result are expected to be known definitely, except for the last digit, which may be in doubt. Such a number is said to contain only significant figures. If more than a single doubtful digit is carried, the extra digit or digits are not significant. If an analytical result is reported as "75.6 mg/L," the analyst should be quite certain of the "75," but may be uncertain as to whether the ".6" should be .5 or .7, or even .4 or .8, because of unavoidable uncertainty in the analytical procedure. If the standard deviation were known from previous work to be ±2 mg/L, the analyst would have, or should have, rounded off the result to "76 mg/L" before reporting it. On the other hand, if the method were so good that a result of "75.61 mg/L" could have been conscientiously reported, then the analyst should not have rounded it off to 75.6.

Report only such figures as are justified by the accuracy of the work. Do not follow the all-too-common practice of requiring that quantities listed in a column have the same number of figures to the right of the decimal point.

a. Rounding off: Round off by dropping digits that are not significant. If the digit 6, 7, 8, or 9 is dropped, increase preceding digit by one unit; if the digit 0, 1, 2, 3, or

*Approved by Standard Methods Committee, 1981.

TABLE 103:I. CONVERSION FACTORS*
(Milligrams per Liter—Milliequivalents per Liter)

Ion (Cation)	me/L = mg/L×	mg/L = me/L×	Ion (Anion)	me/L = mg/L×	mg/L = me/L×
Al^{3+}	0.111 2	8.994	BO_2^-	0.023 36	42.81
B^{3+}	0.277 5	3.603	Br^-	0.012 52	79.90
Ba^{2+}	0.014 56	68.67	Cl^-	0.028 21	35.45
Ca^{2+}	0.049 90	20.04	CO_3^{2-}	0.033 33	30.00
Cr^{3+}	0.057 70	17.33	CrO_4^{2-}	0.017 24	58.00
			F^-	0.052 64	19.00
Cu^{2+}	0.031 47	31.77	HCO_3^-	0.016 39	61.02
Fe^{2+}	0.035 81	27.92	HPO_4^{2-}	0.020 84	47.99
Fe^{3+}	0.053 72	18.62	$H_2PO_4^-$	0.010 31	96.99
H^+	0.992 2	1.008	HS^-	0.030 24	33.07
K^+	0.025 58	39.10	HSO_3^-	0.012 34	81.07
			HSO_4^-	0.010 30	97.07
Li^+	0.144 1	6.941	I^-	0.007 880	126.9
Mg^{2+}	0.082 29	12.15	NO_2^-	0.021 74	46.01
Mn^{2+}	0.036 40	27.47	NO_3^-	0.016 13	62.00
Mn^{4+}	0.072 81	13.73	OH^-	0.058 80	17.01
Na^+	0.043 50	22.99	PO_4^{3-}	0.031 59	31.66
NH_4^+	0.055 44	18.04	S^{2-}	0.062 38	16.03
Pb^{2+}	0.009 653	103.6	SiO_3^{2-}	0.026 29	38.04
Sr^{2+}	0.022 83	43.81	SO_3^{2-}	0.024 98	40.03
Zn^{2+}	0.030 59	32.69	SO_4^{2-}	0.020 82	48.03

* Factors are based on ion charge and not on redox reactions that may be possible for certain of these ions. Cations and anions are listed separately in alphabetical order.

4 is dropped, do not alter preceding digit. If the digit 5 is dropped, round off preceding digit to the nearest even number: thus 2.25 becomes 2.2 and 2.35 becomes 2.4.

b. Ambiguous zeros: The digit 0 may record a measured value of zero or it may serve merely as a spacer to locate the decimal point. If the result of a sulfate determination is reported as 420 mg/L, the report recipient may be in doubt whether the zero is significant or not, because the zero cannot be deleted. If an analyst calculates a total residue of 1146 mg/L, but realizes that the 4 is somewhat doubtful and that therefore the 6 has no significance, the answer should be rounded off to 1150 mg/L and so reported but here, too, the report recipient will not know whether the zero is significant. Although the number could be expressed as a power of 10 (e.g., 11.5×10^2 or 1.15×10^3), this form is not used generally because it would not be consistent with the normal expression of results and might be confusing. In most other cases, there will be no doubt as to the sense in which the digit 0 is used. It is obvious that the zeros are significant in such numbers as 104 and 40.08. In a number written as 5.000, it is understood that all the zeros are significant, or else the number could have been rounded off to 5.00, 5.0, or 5, whichever was appropriate. Whenever the zero is ambiguous, it is advisable to accompany the result with an estimate of its uncertainty.

Sometimes, significant zeros are dropped without good cause. If a buret is read as "23.60 mL," it should be so recorded, and not as "23.6 mL." The first number indicates that the analyst took the trouble to estimate the second decimal place; "23.6 mL" would indicate a rather careless reading of the buret.

c. The plus-or-minus (±) notation: If a calculation yields as a result "1476 mg/L" with a standard deviation estimated as ±40 mg/L, report it as 1480 ± 40 mg/L. However, if the standard deviation is estimated as ±100 mg/L round off the answer still further and report as 1500 ± 100 mg/L. By this device, ambiguity is avoided and the report recipient can tell that the zeros are only spacers. Even if the problem of ambiguous zeros is not present, showing the standard deviation is helpful in that it provides an estimate of reliability.

d. Calculations: As a practical operating rule, round off the result of a calculation in which several numbers are multiplied or divided to as few significant figures as are present in the factor with the fewest significant figures. Suppose that the following calculations must be made to obtain the result of an analysis:

$$\frac{56 \times 0.003\,462 \times 43.22}{1.684}$$

A ten-place calculator yields an answer of "4.975 740 998." Round off this number to "5.0" because one of the measurements that entered into the calculation, 56, has only two significant figures. It was unnecessary to measure the other three factors to four significant figures because the "56" is the "weakest link in the chain" and limits accuracy of the answer. If the other factors were measured to only three, instead of four, significant figures, the answer would not suffer and the labor might be less.

When numbers are added or subtracted, the number that has the fewest decimal places, not necessarily the fewest significant figures, puts the limit on the number of places that justifiably may be carried in the sum or difference. Thus the sum

$$\begin{array}{r@{.}l} 0 & 0072 \\ 12 & 02 \\ 4 & 0078 \\ 25 & 9 \\ 4886 & \\ \hline 4927 & 9350 \end{array}$$

must be rounded off to "4928," no decimals, because one of the addends, 4886, has no decimal places. Notice that another addend, 25.9, has only three significant figures and yet it does not set a limit to the number of significant figures in the answer.

The preceding discussion is necessarily oversimplified. The reader is referred to the bibliography for more detailed sources.

ABBREVIATIONS

The following symbols and abbreviations are used throughout this book:

Abbreviation	Referent
AA	atomic absorption
A or amp	ampere(s)
AC	alternating current
ACS	American Chemical Society
amu	atomic mass units
APHA	American Public Health Association
ASTM	American Society for Testing and Materials
AWWA	American Water Works Association
BOD	biochemical oxygen demand
°C	degree(s) Celsius
c	count(s)
Ci	curie(s)
cm, cm^2, cm^3	centimeter(s), square centimeter(s), cubic centimeter(s)
COD	chemical oxygen demand
conc	concentrated
cpm	counts per minute
cps	counts per second
d	day
DC	direct current
diam	diameter
DO	dissolved oxygen
dpm	disintegrations per minute
g	gram(s)
g	gravity, unit acceleration of
GC	gas chromatograph
GC/MS	gas chromatograph/mass spectrometer
h	hour
IC	ion chromatograph
ICP	inductively coupled plasma
ID	inside diameter
IU	international unit(s)
JTU	Jackson candle turbidity unit(s)
KeV	kiloelectron volt(s)
kg	kilogram(s)
kPa	kilopascal
L	liter(s)
M	mole or molar
m, m^2, m^3	meter(s), square meter(s), cubic meter(s)
MCL	maximum contaminant level
me	milliequivalent(s)
meV	megaelectron volt(s)
mg	milligram(s)
min	minute(s)
mL	milliliter(s)
mm, mm^2, mm^3	millimeter(s), square millimeter(s), cubic millimeter(s)
mol wt	molecular weight
MPN	most probable number
MS	mass spectrometer
mV	millivolt(s)
μA	microampere(s)
μCi	microcurie(s)
μg	microgram(s)
μL	microliter(s)
μm	micrometer(s)
N	normal
NBS	National Bureau of Standards
nCi	nanocurie(s)
ng	nanogram(s)
No.	number
NTU	nephelometric turbidity unit(s)
OD	outside diameter
Pa	pascal
pCi	picocurie(s)

Abbreviation	*Referent*
pg	picogram(s)
PVC	polyvinyl chloride
rpm	revolution(s) per minute
rps	revolution(s) per second
SD	standard deviation
SDI	sludge density index
s	second(s)
sp., spp.	species
sp gr	specific gravity
ST	standard taper
SVI	sludge volume index
TFE	tetrafluoroethylene
THM	trihalomethane(s)
TOC	total organic carbon
T.O.N.	threshold odor number
TOX	total organic halogen
Toxicity terms	See Section 801A
U	unit(s)
USP	United States Pharmacopoeia
UV	ultraviolet
V	volt(s)
v/v	volume ratio
W	watt(s)
WPCF	Water Pollution Control Federation
ZSV	zone settling index

Abbreviations of periodical titles in reference lists and bibliographies are based on those given in *Biosis. List of Serials with Title Abbreviations,* 1970. Biosciences Information Service of Biological Abstracts, Philadelphia, Pa.

TABLE A: UNIT PREFIXES

Symbol	Prefix	Multiples and Submultiples
M	mega-	10^6
k	kilo-	10^3
m	milli-	10^{-3}
μ	micro-	10^{-6}
n	nano-	10^{-9}
p	pico-	10^{-12}

TABLE B: METRIC-ENGLISH EQUIVALENTS

Metric Unit	Multiplied by	= English Unit
m	3.279	ft
lux	0.0929	ft-c
L	0.2642	gal
cm	0.394	in.
kg	2.203	lb
g	0.0353	oz
kPa	0.145	psi

.001 g = 1mg

1g = 1000 mg