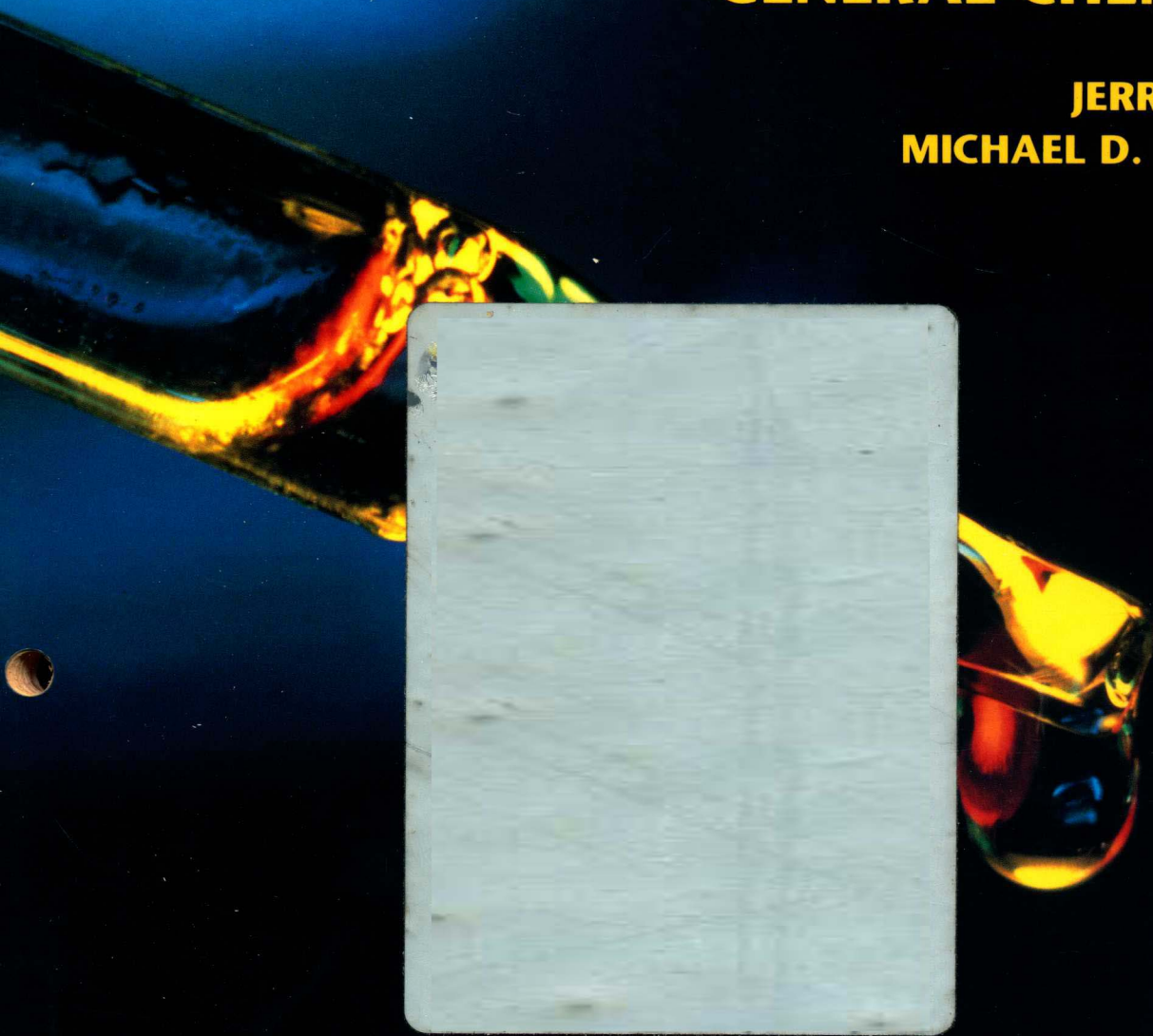


# **MICROSCALE AND MACROSCALE EXPERIMENTS FOR GENERAL CHEMISTRY**

**JERRY L. MILLS  
MICHAEL D. HAMPTON**



# **MICROSCALE AND MACROSCALE EXPERIMENTS FOR GENERAL CHEMISTRY**

**JERRY L. MILLS**

Texas Tech University

**MICHAEL D. HAMPTON**

University of Central Florida

**McGraw-Hill, Inc.**

New York St. Louis San Francisco Auckland Bogotá  
Caracas Lisbon London Madrid Mexico Milan  
Montreal New Delhi Paris San Juan Singapore  
Sydney Tokyo Toronto

**MICROSCALE AND MACROSCALE EXPERIMENTS  
FOR GENERAL CHEMISTRY**

Copyright © 1991 by McGraw-Hill, Inc. All rights reserved.  
Printed in the United States of America. Except as permitted  
under the United States Copyright Act of 1976, no part of  
this publication may be reproduced or distributed in any  
form or by any means, or stored in a data base or retrieval  
system, without the prior written permission of the  
publisher.

10 11 12 QPD QPD 9 0 9

**ISBN 0-07-042442-X**

The editor was Kirk Emry;  
the production supervisor was Annette Mayeski.

Quebecor Printing/Dubuque was printer and binder.

Library of Congress Catalog Card Number: 90-5718



1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																
1A	2A											3A	4A	5A	6A	7A	8A																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																
H	He	Li	Be	B	C	N	O	F	Ne			Al	Si	P	S	Cl	Ar																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																
1.008	4.003	6.941	9.012	10.81	12.01	14.01	16.00	19.00	20.18			26.98	28.09	30.97	32.07	35.45	39.95																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																
11	12	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																											
Na	Mg	K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr	Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe	Cs	Ba	La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																											
22.99	24.31	39.10	40.08	44.96	47.88	50.94	52.00	54.94	55.85	58.93	58.69	63.55	65.39	69.72	72.59	74.92	78.96	79.90	83.80	85.47	87.62	88.91	91.22	92.91	95.94	(98)	101.1	102.9	106.4	107.9	112.4	114.8	118.7	121.8	127.6	126.9	131.3	132.9	137.3	138.9	140.1	140.9	144.2	150.4	152.0	157.3	158.9	162.5	164.9	167.3	168.9	173.0	175.0																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																												
87	88	89	90	91	92	93	94	95	96	97	98	99	100	101	102	103	104	105	106	107	108	109	110	111	112	113	114	115	116	117	118	119	120	121	122	123	124	125	126	127	128	129	130	131	132	133	134	135	136	137	138	139	140	141	142	143	144	145	146	147	148	149	150	151	152	153	154	155	156	157	158	159	160	161	162	163	164	165	166	167	168	169	170	171	172	173	174	175	176	177	178	179	180	181	182	183	184	185	186	187	188	189	190	191	192	193	194	195	196	197	198	199	200	201	202	203	204	205	206	207	208	209	210	211	212	213	214	215	216	217	218	219	220	221	222	223	224	225	226	227	228	229	230	231	232	233	234	235	236	237	238	239	240	241	242	243	244	245	246	247	248	249	250	251	252	253	254	255	256	257	258	259	260	261	262	263	264	265	266	267	268	269	270	271	272	273	274	275	276	277	278	279	280	281	282	283	284	285	286	287	288	289	290	291	292	293	294	295	296	297	298	299	300	301	302	303	304	305	306	307	308	309	310	311	312	313	314	315	316	317	318	319	320	321	322	323	324	325	326	327	328	329	330	331	332	333	334	335	336	337	338	339	340	341	342	343	344	345	346	347	348	349	350	351	352	353	354	355	356	357	358	359	360	361	362	363	364	365	366	367	368	369	370	371	372	373	374	375	376	377	378	379	380	381	382	383	384	385	386	387	388	389	390	391	392	393	394	395	396	397	398	399	400	401	402	403	404	405	406	407	408	409	410	411	412	413	414	415	416	417	418	419	420	421	422	423	424	425	426	427	428	429	430	431	432	433	434	435	436	437	438	439	440	441	442	443	444	445	446	447	448	449	450	451	452	453	454	455	456	457	458	459	460	461	462	463	464	465	466	467	468	469	470	471	472	473	474	475	476	477	478	479	480	481	482	483	484	485	486	487	488	489	490	491	492	493	494	495	496	497	498	499	500	501	502	503	504	505	506	507	508	509	510	511	512	513	514	515	516	517	518	519	520	521	522	523	524	525	526	527	528	529	530	531	532	533	534	535	536	537	538	539	540	541	542	543	544	545	546	547	548	549	550	551	552	553	554	555	556	557	558	559	560	561	562	563	564	565	566	567	568	569	570	571	572	573	574	575	576	577	578	579	580	581	582	583	584	585	586	587	588	589	590	591	592	593	594	595	596	597	598	599	600	601	602	603	604	605	606	607	608	609	610	611	612	613	614	615	616	617	618	619	620	621	622	623	624	625	626	627	628	629	630	631	632	633	634	635	636	637	638	639	640	641	642	643	644	645	646	647	648	649	650	651	652	653	654	655	656	657	658	659	660	661	662	663	664	665	666	667	668	669	670	671	672	673	674	675	676	677	678	679	680	681	682	683	684	685	686	687	688	689	690	691	692	693	694	695	696	697	698	699	700	701	702	703	704	705	706	707	708	709	710	711	712	713	714	715	716	717	718	719	720	721	722	723	724	725	726	727	728	729	730	731	732	733	734	735	736	737	738	739	740	741	742	743	744	745	746	747	748	749	750	751	752	753	754	755	756	757	758	759	760	761	762	763	764	765	766	767	768	769	770	771	772	773	774	775	776	777	778	779	780	781	782	783	784	785	786	787	788	789	790	791	792	793	794	795	796	797	798	799	800	801	802	803	804	805	806	807	808	809	810	811	812	813	814	815	816	817	818	819	820	821	822	823	824	825	826	827	828	829	830	831	832	833	834	835	836	837	838	839	840	841	842	843	844	845	846	847	848	849	850	851	852	853	854	855	856	857	858	859	860	861	862	863	864	865	866	867	868	869	870	871	872	873	874	875	876	877	878	879	880	881	882	883	884	885	886	887	888	889	890	891	892	893	894	895	896	897	898	899	900	901	902	903	904	905	906	907	908	909	910	911	912	913	914	915	916	917	918	919	920	921	922	923	924	925	926	927	928	929	930	931	932	933	934	935	936	937	938	939	940	941	942	943	944	945	946	947	948	949	950	951	952	953	954	955	956	957	958	959	960	961	962	963	964	965	966	967	968	969	970	971	972	973	974	975	976	977	978	979	980	981	982	983	984	985	986	987	988	989	990	991	992	993	994	995	996	997	998	999	1000
(223)	(226)	(227)	(257)	(260)	(263)	(262)	(265)	(266)	(267)	(268)	(269)	(270)	(271)	(272)	(273)	(274)	(275)	(276)	(277)	(278)	(279)	(280)	(281)	(282)	(283)	(284)	(285)	(286)	(287)	(288)	(289)	(290)	(291)	(292)	(293)	(294)	(295)	(296)	(297)	(298)	(299)	(300)	(301)	(302)	(303)	(304)	(305)	(306)	(307)	(308)	(309)	(310)	(311)	(312)	(313)	(314)	(315)	(316)	(317)	(318)	(319)	(320)	(321)	(322)	(323)	(324)	(325)	(326)	(327)	(328)	(329)	(330)	(331)	(332)	(333)	(334)	(335)	(336)	(337)	(338)	(339)	(340)	(341)	(342)	(343)	(344)	(345)	(346)	(347)	(348)	(349)	(350)	(351)	(352)	(353)	(354)	(355)	(356)	(357)	(358)	(359)	(360)	(361)	(362)	(363)	(364)	(365)	(366)	(367)	(368)	(369)	(370)	(371)	(372)	(373)	(374)	(375)	(376)	(377)	(378)	(379)	(380)	(381)	(382)	(383)	(384)	(385)	(386)	(387)	(388)	(389)	(390)	(391)	(392)	(393)	(394)	(395)	(396)	(397)	(398)	(399)	(400)	(401)	(402)	(403)	(404)	(405)	(406)	(407)	(408)	(409)	(410)	(411)	(412)	(413)	(414)	(415)	(416)	(417)	(418)	(419)	(420)	(421)	(422)	(423)	(424)	(425)	(426)	(427)	(428)	(429)	(430)	(431)	(432)	(433)	(434)	(435)	(436)	(437)	(438)	(439)	(440)	(441)	(442)	(443)	(444)	(445)	(446)	(447)	(448)	(449)	(450)	(451)	(452)	(453)	(454)	(455)	(456)	(457)	(458)	(459)	(460)	(461)	(462)	(463)	(464)	(465)	(466)	(467)	(468)	(469)	(470)	(471)	(472)	(473)	(474)	(475)	(476)	(477)	(478)	(479)	(480)	(481)	(482)	(483)	(484)	(485)	(486)	(487)	(488)	(489)	(490)	(491)	(492)	(493)	(494)	(495)	(496)	(497)	(498)	(499)	(500)	(501)	(502)	(503)	(504)	(505)	(506)	(507)	(508)	(509)	(510)	(511)	(512)	(513)	(514)	(515)	(516)	(517)	(518)	(519)	(520)	(521)	(522)	(523)	(524)	(525)	(526)	(527)	(528)	(529)	(530)	(531)	(532)	(533)	(534)	(535)	(536)	(537)	(538)	(539)	(540)	(541)	(542)	(543)	(544)	(545)	(546)	(547)	(548)	(549)	(550)	(551)	(552)	(553)	(554)	(555)	(556)	(557)	(558)	(559)	(560)	(561)	(562)	(563)	(564)	(565)	(566)	(567)	(568)	(569)	(570)	(571)	(572)	(573)	(574)	(575)	(576)	(577)	(578)	(579)	(580)	(581)	(582)	(583)	(584)	(585)	(586)	(587)	(588)	(589)	(590)	(591)	(592)	(593)	(594)	(595)	(596)	(597)	(598)	(599)	(600)	(601)	(602)	(603)	(604)	(605)	(606)	(607)	(608)	(609)	(610)	(611)	(612)	(613)	(614)	(615)	(616)	(617)	(618)	(619)	(620)	(621)	(622)	(623)	(624)	(625)	(626)	(627)	(628)	(629)	(630)	(631)	(632)	(633)	(634)	(635)	(636)	(637)	(638)	(639)	(640)	(641)	(642)	(643)	(644)	(645)	(646)	(647)	(648)	(649)	(650)	(651)	(6																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																							

# MICROSCALE AND MACROSCALE EXPERIMENTS FOR GENERAL CHEMISTRY

JERRY L. MILLIS

MICHAEL D. HAMPTON

Also available from McGraw-Hill

## **Schaum's Outline Series in Science**

Each outline includes basic theory, definitions, and hundreds of solved problems and supplementary problems with answers.

TITLES ON THE CURRENT LIST INCLUDE:

*Analytical Chemistry*  
*Applied Physics, 2d edition*  
*Biochemistry*  
*College Chemistry, 7th edition*  
*College Physics, 8th edition*  
*Earth Sciences*  
*General Biology*  
*Genetics, 2d edition*  
*Human Anatomy and Physiology*  
*Lagrangian Dynamics*  
*Modern Physics*  
*Optics*  
*Organic Chemistry*  
*Physical Chemistry*  
*Physical Science, 2d edition*  
*Physics for Engineering and Science*  
*Zoology*

## **Schaum's Solved Problems Books**

Each title in this series is a complete and expert source of solved problems containing thousands of problems with worked out solutions.

TITLES ON THE CURRENT LIST INCLUDE:

*3000 Solved Problems in Biology*  
*2500 Solved Problems in Chemistry*  
*2000 Solved Problems in Physical Chemistry*  
*3000 Solved Problems in Physics*

Available at your College Bookstore. A complete list of Schaum titles may be obtained by writing to:

Schaum Division  
McGraw-Hill, Inc.  
Princeton Road, S-1  
Hightstown, NJ 08520

## PREFACE

### Second Edition

The first edition of this laboratory manual contained twenty microscale experiments and no conventional scale experiments. That version was intended to supplement other manuals and was not intended to be used as a stand-alone manual. In this second edition, we have added five microscale experiments and fifteen conventional scale experiments. The manual now contains sufficient experiments for a complete two-semester laboratory course sequence with a mixture of conventional scale and microscale experiments.

The experiments are organized by scale rather than by the more conventional method of order of coverage: all of the microscale experiments are listed first, followed by all of the conventional scale experiments.

The main purpose of the first edition of this manual was to popularize the use of microscale experiments in the general chemistry laboratory. While we are providing sufficient experiments for an entire general chemistry laboratory course sequence with this second edition, the primary thrust is still microscale. For this reason, the preface for the first edition is still appropriate.

### First Edition

This laboratory manual for general chemistry is totally based on microscale experiments. It is designed to be a companion or supplement to any other conventional general chemistry laboratory manual. In general the experiments are aimed at the science-engineering students rather than at liberal arts students, but many of the experiments are appropriate for both classes. There is no intent for this manual to be used alone as a text. Rather the intent is to replace approximately one-half of the experiments in a conventional two-semester (or three-quarter) sequence with microscale experiments. We strongly feel that a student who passes through a general chemistry laboratory sequence should recognize and use conventional chemical apparatus and equipment such as glass burets and pipets, volumetric flasks, and spectro-photometers.

The advantages of microscale general chemistry laboratory experiments are several. First, and most important, is safety. The microscale experiments use extremely small quantities of chemicals. Small quantities of chemicals mean a simple and relatively inexpensive chemical disposal problem, an inexpensive laboratory, and the use of laboratory experiments that might otherwise be impractical because of safety problems associated with a conventional-scale experiment. Microscale experiments demonstrate the chemical principles as well as, and frequently better than, their macroscale counterparts. Microscale experiments are generally performed more quickly. Little time is required for washing and laboratory housekeeping, since much of the equipment is disposable. Finally and not insignificantly, microscale experiments are fun to perform.

The approach in this manual is to use very inexpensive (essentially disposable) equipment. All of the experiments use 3.5 mL plastic Pasteur pipets. The pipets are multipurpose; they can be used as a means of transferring liquids, as a microburet (by counting drops), and as a reaction vessel. Also every experiment uses plastic plates with indented wells. The plates used in a particular experiment will be one or more of the following: 6-well plate, 12-well plate, 24-well plate, and 96-well plate. These plates in general replace beakers and test tubes, but they are much more versatile than that.

An alternate approach to microscale experiments is to miniaturize conventional equipment. Currently there is a strong move to this approach in organic chemistry laboratories, both in academic and industrial settings. Although microscale glassware is very expensive, it is necessary for organic chemistry laboratory experiments. For the reasons discussed above, microscale is the direction in which academic laboratories are moving. Until such time as the normal college or university laboratories are equipped with microscale glassware, we feel that the experiments described in this manual are a very inexpensive way to introduce microscale into the general chemistry laboratory.

To reiterate, the philosophy of this laboratory manual is aimed at partial replacement or supplementation of existing laboratory experiments. It is not a radical change, but rather an easy and natural supplement (replacement). Chemistry is a laboratory science. We hope that these experiments will help make your laboratory experience more enjoyable, more educational, more economical, and safer.



## **Acknowledgments**

The inspiration for this manual came from a seminar presented by Professor Stephen Thompson of Colorado State University. Professor Thompson, a pioneer in the area of microscale chemistry, has designed some extremely clever and innovative equipment and experiments.

Discussions with Professor Roy Mitchell of Texas Tech University were extremely valuable. His suggestions, comments, and criticisms are gratefully acknowledged. Robin Damico, an undergraduate student at University of Central Florida, was of invaluable help in the development and testing of these experiments. Stephen Hall and Roger Wolcott provided extended and stimulating discussions during the formative stages of this manual.

## LABORATORY SAFETY GUIDELINES

As stated in the Preface, it is assumed that this microscale laboratory manual is being used in conjunction with a conventional scale laboratory manual. You should have read and understood the safety section in that manual. Safety in the laboratory cannot be overemphasized. Therefore we present here an abbreviated list of safety rules.

1. Safety glasses or safety goggles must be worn **at all times** when you are in the laboratory. Even if you are performing an experiment that is using no potentially dangerous chemicals, you are still in a laboratory where numerous potential hazards exist.
2. Unless your instructor indicates otherwise, contact lenses are not to be worn in the laboratory.
3. You should know where to find and how to use first aid equipment.
4. Your instructor will show you where the eye wash is located. If a corrosive chemical gets in your eyes, **immediately wash the affected area with large quantiles of water from the eye wash**. Notify the instructor of the accident.
5. Your instructor will show you where the safety shower is located. If a corrosive chemical gets on your skin or clothes, **immediately wash the affected area with large quantiles of water**. If your clothing catches fire or if a large quantity of chemicals is spilled on your clothing, then use the safety shower. Notify the instructor of the accident.
6. Never taste anything in the laboratory. Smoking, chewing, eating, or drinking in the laboratory under any circumstances is strictly prohibited.
7. Never directly smell the source of a vapor, but rather bring a small quantity of the vapor to your nose with your cupped hand.
8. Reactions involving malodorous, noxious, or dangerous chemicals should be performed in the hood.
9. You should know the location of and know how to use a fire extinguisher. A small fire, such as a liquid burning in a beaker, can be extinguished by covering the beaker to remove the source of oxygen.
10. No unauthorized experiments are to be performed.
11. Clean up broken glassware immediately.
12. When pouring one liquid into another, do so slowly and cautiously. To dilute an acid, pour the acid into the water: **never pour water into acid**.
13. When heating a test tube, make certain that the open end is pointed away from you and your fellow students. Then, if overheating causes the contents to bump out, they will not splash anyone.

14. If you are transferring corrosive chemicals or are mixing chemicals in your plastic Pasteur pipet, make certain that the open end is pointed away from you and your fellow students.
15. Do not rub your eyes while in the laboratory, as your hands might have chemicals on them.
16. Pay particular attention to the **CAUTION** statements in the Procedure section of the experiments. If you do not understand the caution, ask your instructor for clarification.
17. **In case of any accident, immediately notify the instructor.** In the event that your instructor is not available, notify the stockroom personnel.

# CONTENTS

Preface v

Laboratory Safety Guidelines viii

## Microscale Experiments

1	Microscale Laboratory Techniques.....	1
2	Horizontal Paper Chromatography of Dyes .....	7
3	Two-Dimensional Paper Chromatography of Inks .....	15
4	Activity Series.....	21
5	Identification of Silver, Lead, and Mercurous Ions.....	29
6	Ion Drop-Matrix Test.....	37
7	Colorimetry of Dyes.....	43
8	Colorimetric Determination of Copper.....	51
9	Preparation of Tetraamminecopper(II) Sulfate Monohydrate.....	57
10	Determination of the Purity of a Solid Acid by Titration .....	63
11	Le Chatelier's Principle.....	69
12	Potentiometric Titration of Phosphoric Acid.....	83
13	Determination of Zinc Ions by Complexometric Titration with EDTA.....	97
14	Metal Ion Determination by Back Titration with EDTA .....	103
15	Oxidation States of Manganese .....	109
16	Ion-Exchange Chromatography.....	115
17	Kinetics.....	123
18	The Effect of Dilution on the pH of Buffers.....	131
19	The Effect of the Addition of Acid on the pH of Buffers.....	145
20	The Effect of the Addition of Base on the pH of Buffers.....	155
21	Single Replacement Reactions of the Halogens .....	165
22	Identification of Anions.....	171

23	Determination of Lead Ions by Volumetric Precipitation and EDTA Back Titration.....	175
24	Intermolecular Attraction, Solubility, and Surface Effects .....	183
25	Boyle's Law .....	191
<b>Macroscale Experiments</b>		
26	Recovery of Aluminum.....	197
27	Coefficients of a Chemical Reaction .....	203
28	Molecular Weight of a Volatile Liquid .....	209
29	Formula of Magnesium Oxide .....	213
30	Measurement of pH .....	217
31	Acid - Base Equilibria.....	223
32	Equilibrium Constant of a Complex.....	233
33	Spectrophotometric Determination of Chromium .....	241
34	Determination of Phosphate in Water.....	245
35	Titration With Permanganate.....	249
36	Heat of Reaction .....	255
37	Group II Cations .....	263
38	Group III Cations.....	273
39	Group IV and V Cations .....	281
40	Cation Unknown .....	289



# Experiment 1

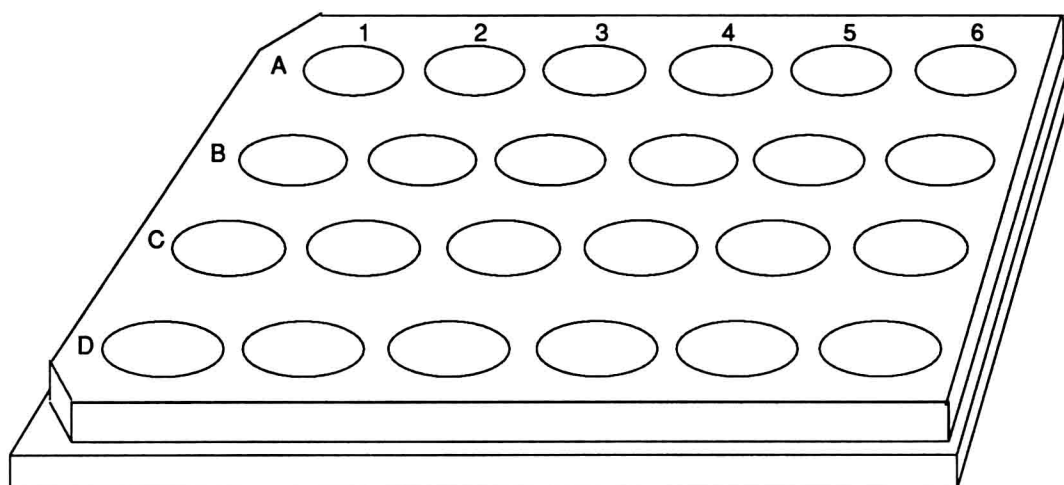
## MICROSCALE LABORATORY TECHNIQUES

### Objective

In this experiment you will learn to use equipment and develop the techniques for the microscale experiments in this laboratory manual.

### Background

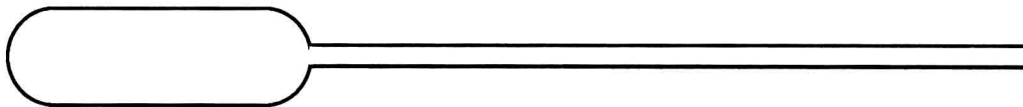
All of the experiments in this laboratory manual require the use of two pieces of equipment that are not commonly encountered in more traditional general chemistry laboratories: plastic well plates and plastic Pasteur pipets. The plastic well plates, such as the 24-well plate,



all have approximately the same outside dimensions. The size of the wells, and thus the number of wells, varies. Depending on the particular experiment, we will use one or more of the following: 96-well plates (8 x 12 wells), 24-well plates (4 x 6 wells), 12-well plates (3 x 4 wells), and 6-well plates (2 x 3 wells). You should notice that the wells are labeled by letters for the horizontal rows and by numbers for the vertical columns. Thus well **B3** would be the well in the second row and in the third column from the top left of the plate.

## 2 Experiment 1

The second piece of equipment unique to the microscale experiments is the disposable plastic Pasteur pipet that is shown below:



The Pasteur pipet will be used in many ways, even as a reaction vessel, during the course of the laboratory. The pipet can be used instead of a buret or volumetric flask by counting drops. In fact, it will be very important for you to be able to deliver drops that are not only very small but also uniform in size from the pipet to wells in the plate.

The purpose of today's experiment is to investigate how the drop size from a plastic Pasteur pipet varies depending on the method of delivery and on the configuration of the pipet tip. You will also learn the proper way to prepare a *stretched-tip* pipet, which you will use in many of the experiments in this laboratory manual.

### Procedure

#### Part 1. Drop Count

In the first part of the experiment, you will ascertain how the size of drops relates to the angle of the pipet (the angle of delivery) and to the size and configuration of the pipet tip. The results that you obtain and the technique that you develop will be very important in the acquisition of accurate data in other experiments in this laboratory.

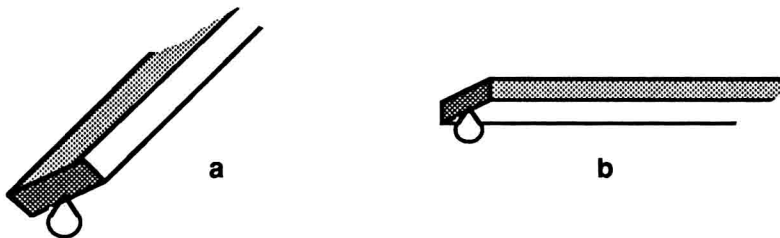
1. Obtain a clean 96-well plate, a 12-well plate, and a new plastic Pasteur pipet from your instructor. Fill one of the wells in your 12-well plate with distilled water to which a small quantity of food color has been added. (The colored water will normally be provided in a plastic squeeze bottle.) The purpose of the food coloring is simply to make the water level in the well more visible.
2. Fill your pipet with the colored water. Select a well in the 96-well plate and, holding the pipet vertically (at a 90° angle), count the number of drops required to exactly fill one of the wells completely. The level of the water in the well should be touching the side of the well top, with about one-third of a sphere extending beyond the top, but not overflowing. Be very careful that the drops on the pipet tip fall freely into the well. If you allow the drops to touch the side of the well, you will obtain poor results. If you make an error, simply repeat the procedure in another well. Record the number of drops required on your answer sheet.
3. Repeat step 2, but hold the pipet at a 45° angle during delivery. Fill the well to the same height. Record the results. Repeat step 2, but hold the pipet horizontally during delivery. Record the results. If any of the wells overflow, cotton swabs are very effective at cleaning the plate. They are also useful in cleaning and drying the wells.
4. It will be very important in many of the microscale experiments to use a pipet with a tip that is smaller than that present on a new Pasteur pipet. Such a tip can be made by stretching the stem and then cutting the plastic in the stretched area. Take your empty pipet and hold the stem with your dominant hand about an inch from the tip,

using a piece of cloth (for better friction). With the bulb of the pipet in the palm of your other hand, grasp the stem near where you are holding it with your dominant hand. Gently stretch the stem using your dominant hand until the stretched area is 1.0 - 1.5 inches long as is shown below:

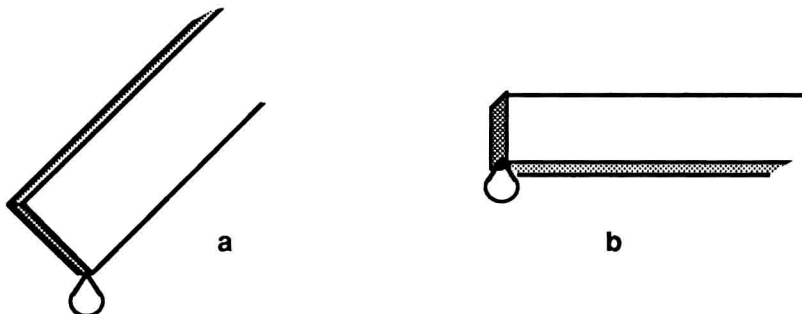


Using scissors or a knife, cut the stem in two in the stretched area. Discard the short piece. Either using your fingernails or the *back* of the knife blade and the desktop, *gently* flatten the last quarter inch of the stem to the tip. If you are too forceful with the flattening procedure, you will split the plastic tip. If this happens, make another attempt with the same pipet or start over with a new pipet. Practice holding the flattened pipet first so that the flattened tip is parallel to the bench top, then twist the flattened tip vertical to the bench top.

5. Repeat step 2, using the flattened end pipet. Record the results on your answer sheet. Keeping the flattened end of the tip parallel to the bench top, repeat step 3, counting the number of drops required to fill the wells when holding the pipet (a) at a 45° angle, and (b) horizontally:



Record your results. Now, keeping the flattened end of the tip vertical to the bench top (that is, keeping the flattened end pointing as much up and down as possible), repeat step 3, counting the number of drops required to fill the wells when holding the pipet (a) at a 45° angle, and (b) horizontally:



Record your results. If the well will not easily hold 25 drops when holding the pipet at 45° with the flattened area pointing up and down, then you have not stretched and flattened the tip properly. Try the procedure again with another pipet.

### Part 2. Drop Volume

In this part of the experiment you are to determine the reproducibility (variation) of drop size and the volume of one drop. If you do not have access to an analytical balance (or a balance accurate to at least 1 mg), then you will skip this part of the experiment. Your instructor may wish to furnish you with some typical data rather than your measuring it.

*Note: In all experiments in this laboratory manual that call for the use of a stretched-tip pipet, the pipet should be prepared as in step 4. The pipet should be held at 45° with the flattened end pointing up and down. Unstretched tips also always should be held at 45°.*

6. Using either a clean, dry 24-well plate or a small beaker (whichever you are provided with that fits on the balance), determine the weight of 100 drops of water using your stretched-tip pipet. Record the data. Repeat the 100-drop weight determination again. You can most simply perform the weighing by adding the second 100 drops to the first. (If you are using a 24-well plate, then add the water to a new well.) The weight of the second 100 drops is determined by difference. Repeat the 100-drop weighing a third time.