MIXING IT UP FOR THE 90s

TWO YEAR COLLEGE CHEMISTRY CONFERENCE
DIVISION OF CHEMICAL EDUCATION
AMERICAN CHEMICAL SOCIETY

October 6-7, 1989

Jefferson Community College

Louisville, KY

COMMITTEE ON CHEMISTRY IN THE TWO-YEAR COLLEGES
Division of Chemical Education, Inc.
American Chemical Society
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Industrial Sponsors Chair	Leo Kling III, Faulkner State Junior College, Bay Minette, AL 36507 (205) 937-9581	(1990)
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American Chemical Society

DEPARTMENT OF ACADEMIC PROGRAMS

1155 SIXTEENTH STREET, N.W.
WASHINGTON, D.C. 20036

September 22, 1989

MEMORANDUM

TO: Chairman and Members
DivCHED COCTYC Executive Committee

FROM: Terri Nally

SUBJECT: Request for Funds

This is a formal request for $3,500 to defray the costs incurred in producing the 2YC3 agenda books and in producing the proposed supplementary materials for the "Guidelines for Chemistry and Chemical Technology Programs in Two-Year Colleges".

Of the $3,500, $1,000 will be dedicated to covering the expenses associated with producing, printing, and distributing the 2YC3 agenda books. Printing costs alone for the 110 copies of the 44-page Cerritos, CA 2YC3 meeting agenda cost $355. Extrapolating the printing costs to the other three agenda books in 1989, and adding mailing and staff expenses to the sum, brings the total to well over $1,000 for this service.

The remaining $2,500 of the $3,500 sought is for a *matching grant* to meet the projected $5,000 costs of printing and distributing supplementary materials for the "Guidelines for Chemistry and Chemical Technology Programs in Two-Year Colleges". The other $2,500 in projected costs, plus the indirect costs of production, will be financed by the ACS Education Division.

The supplementary materials are being developed by the SOCED Task Force on Two-Year Colleges to complement and enrich the standards outlined in "Guidelines". The materials are succinct references to important sources of detailed information, which will be useful for faculty in implementing effectively the guidelines. The materials will be distributed free of charge, as were the "Guidelines", to all science departments at two-year colleges. Moreover, the supplements will be available upon request. Production of the supplementary materials will complete T1 of the "Tomorrow" report's recommendations concerning development of the guidelines.

If you have any questions about these worthwhile projects or the request, I will be happy to respond to your queries at the COCTYC executive meeting on Thursday, October 6, 1989 in Louisville. Thank you for your careful and thoughtful consideration of this funding request.

cc: Ms. J.M. Boese

AGENDA

Committee on Chemistry in the Two-Year Colleges (COCTYC)

106th Conference

Jefferson Community College, Louisville, KY

Friday, October 6, 1989

I. Introductions of Present and New Officers and Past Chairs

II. Minutes from the 105th Conference, Cerritos, CA
Executive meeting minutes, page 3
Approval of COCTYC minutes, page 6

III. Reports

A. 105th Conference (5/19-20) at Cerritos, CA- John Clevenger
B. 106th Conference (10/6-7) at Louisville, KY - Nik Kakolesha
C. Membership - Mike Knoll, page 9
D. Treasurer - Duane Sell, page 11
E. College Sponsors - Duane Sell, page 13
F. Industrial Sponsors - Leo Kling III, page 21
G. Future Meeting Sites - Elliott Greenberg, page 23
H. ACS Two-Year College Programs - Terri Nally, page 29
I. SOCED Task-Force On Two-Year Colleges
J. Chair's Interim Report to DivCHED, page 31

IV. Old Business

A. Results and discussion of survey, Newsletter (Vol. 89-I), page 33
B. Guidelines survey, Newsletter (Vol. 89-II), page 39

V. New Business

VI. Announcements

VII. Self-introductions by those present

VIII. Adjournment

COCTYC EXECUTIVE MEETING
105TH CONFERENCE
HOLIDAY INN, LA MIRADA, CALIFORNIA
THURSDAY, MAY 18, 1989

Members present: Edith Bartley, Ralph Burns, John Clevenger, Elliott Greenberg, Leo Kling III, Duane Sell

Guest present: Christine Romer, Program Chair for the 105th Conference

The meeting was called to order at 7:07 p.m. by the Chair, John Clevenger.

Chris Romer reported that there were 82 pre-registrants and 14 speakers for the conference.

It was moved by Edith Bartley, and seconded by Duane Sell, that the minutes of the previous Executive Committee meeting at Irving, Texas, be approved as presented in the agenda book (pgs. 3-6). Approved by voice vote.

POLICIES AND PROCEDURES

Election
Election ballots were opened and counted. The Chair-Elect for 1990 will be Margot Schumm of Montgomery Community College, Rockville, Maryland. Richard Jones of Sinclair Community College, Dayton, Ohio was elected as Membership Chair to succeed Michael Knoll in 1990.

Nominations
Nominations for Chair-Elect for 1991 are currently open. At present there is one nominee.

OLD BUSINESS

Newsletter Funding
Duane Sell reported that financial support for our Newsletter by industrial or research companies looks promising. Leo Kling has written our Industrial Sponsors to inquire about their possible interest in advertising in the Newsletter, and is awaiting their replies.

Newsletter Survey - Recent Comments
Recent replies to the survey noted the difficulty in travelling to meetings. There was some discussion about alternate ways to disseminate information. Suggestions mentioned were publication of abstracts and publicity about local $2YC_3$ section activities.

ACS Policy on Education
John Clevenger mentioned a letter from Uni Suskind indicating that SOCED is having a meeting to formulate a white paper for ACS policy on Education. It is apparently not well known that items such as the Guidelines are SOCED documents but are **NOT** ACS official policy. The purpose of the SOCED meeting is to prepare issues and documents to be submitted to ACS for adoption as official policy. John Clevenger solicited input on issues to be submitted to SOCED.

House Subcommittee on Science, Space & Technology
John Clevenger reported on his appearance before the committee and noted their very favorable attitude toward two-year colleges.

Long Range Planning/Organizational Improvement
An involved discussion resulted in general agreement on some goals and suggestions for improving operations:

1) An annual 10% increase in membership as well as College and Industrial Sponsors.

2) Update our record keeping and our college rosters.

3) We need more people to do some work. Involve the entire COCTYC to work on the above issues.
   a) When officers and past chairs are introduced at COCTYC meetings, also ask committee members to identify themselves.
   b) Schedule small discussion groups. At each of our conferences schedule a regional COCTYC meeting, probably during the Friday lunch.

4) Streamline Executive Committee and COCTYC meetings by deleting reports which are published in the agenda book, thereby allowing more time for work on substantive issues of long range planning and organizational improvement.

Secretary/Editor Position
There was some discussion about the possibilities of realigning the duties of the Chair and Chair-Elect. Leo Kling moved, seconded by Duane Sell, that we reactivate the Executive Committee position of Secretary/Editor, effective January 1, 1991, contingent upon approval of the COCTYC. All Executive Committee members attending the Louisville Conference will vote on whether or not to submit this proposal to the COCTYC. Those members who will not be able to attend the Louisville Conference are asked to please submit, in writing, their views on this issue so that due consideration can be given to all opinions.

Executive Committee Workshop
Because of the large backlog of critical issues facing the Executive Committee, and the limited time for discussion at regular meetings, there was general agreement on the concept of holding an annual Executive Committee Workshop devoted to issues of long range planning and organizational improvement. This workshop will be scheduled at a conference that is centrally located and is expected to draw a good turnout. Most likely this will be the Midwestern Conference. An Executive Committee Workshop is scheduled for 4 p.m. on Thursday, October 5, 1989 in Louisville. The location of the meeting will be announced later.

DIVCHED 11th Biennial Meeting
Leo Kling reported that he has made overtures to various persons with regard to chairing or co-chairing the $2YC_3$ booth committee. He indicated that progress is being made and is optimistic. Leo also issued a call for papers from two-year college people.

$2YC_3$ Conference Sites for Spring 1994

Elliott Greenberg reported that the Spring ACS National meeting in April, 1994 will be in Mexico City. It will therefore not be possible to select a nearby site for the $2YC_3$ Conference, as is our usual practice. It was agreed to schedule our two conferences independent of the ACS meeting. The possibility of funding the $2YC_3$ Chair to attend the ACS meeting, in addition to the two $2YC_3$ Conferences, was deferred.

ANNOUNCEMENTS

Edith Bartley reported that the 12th DIVCHED Biennial Meeting will be held at the University of California-Davis on either July 26-31 (1st choice) or August 2-7, 1992.

John Clevenger announced that June 14, 1989 is the deadline for submission of material for the next newsletter.

Duane Sell announced receipt of the annual $2,000 check from DIVCHED.

ADJOURNMENT

The meeting was adjourned at 10:17 p.m.

Respectfully submitted,

Elliott Greenberg

Elliott Greenberg
1989 Chair-Elect

COMMITTEE ON CHEMISTRY IN THE TWO-YEAR COLLEGE
105TH CONFERENCE
CERRITOS COMMUNITY COLLEGE, NORWALK, CALIFORNIA
FRIDAY, MAY 19, 1989

ATTENDANCE

There were 23 persons in attendance at this meeting.

OPENING

John Clevenger, Chairperson, called the meeting to order at 9:35 a.m., and asked everyone present to introduce themselves, indicating their participation and/or office (including COCTYC member) in $2YC_3$

John Clevenger indicated that the "theme" of the meeting would be to solicit ideas and thoughts from those present, and to seek ways of increasing the involvement of the membership.

NEW BUSINESS

Recognition of Immediate Past Chair
John Clevenger presented a plaque to Ralph Burns in appreciation of his year as Chairperson of $2YC_3$. John noted that Ralph is this year's CMA National Award winner, and also mentioned the regional award winners.

Election Results
John Clevenger announced that the new Membership Chair, to take office on January 1, 1990, is Richard Jones of Sinclair Community College, Dayton, Ohio.

Margot Schumm of Montgomery Community College, Rockville, Maryland, has been elected to serve as Chair-Elect for 1990.

Newsletter Survey
John Clevenger passed out survey forms, asking those who have not already done so to respond to the survey. He again indicated that we wish to find out what we can do better, etc. John skimmed through the survey form and mentioned the results obtained so far.

Conference Scheduling
Opinions were solicited on the desirable times of the year for $2YC_3$ Conferences. In particular, the question of holding conferences after the end of the academic year was explored. It was apparent, as expected, that there is a wide variation in school schedules and desirable times for conferences. However, there was very strong support voiced for summer conferences.

Cecil Hammonds made a strong point about the need for faculty to attend meetings for professional renewal, etc.

2YC$_3$ Local Section Activities

Elizabeth Armstrong spoke about the San Francisco Bay Area organization and activities. Gilbert Albelo described activities of the Portland Area College Chemistry Teachers (PACCT). John Clevenger asked that news of local activities be forwarded to him for inclusion in the Newsletter. June 14, 1989 is the deadline for submission of material for the next issue.

ANNOUNCEMENTS

John Clevenger announced that SOCED is seeking input for issues to be presented to ACS for approval as the Society's official policy position.

Ralph Burns announced an opening for a faculty position at St. Louis Community College-Meramec.

Elliott Greenberg announced the possibility that the vacancy at Prairie State College, Chicago Heights, IL may still be open.

Gilbert Albelo announced an opening at Mt. Hood Community College, Gresham, OR.

ADJOURNMENT

The meeting was adjourned at 10:32 a.m.

Respectfully submitted,

Elliott Greenberg

Elliott Greenberg
1989 Chair-Elect

1989 2YC3 MEMBERSHIP REPORT

Renewals from 1988	-	438
New Members	-	107
TOTAL		608

545 g.d.c.

Submitted by

Mike Knoll

September 7, 1989

COMMITTEE ON CHEMISTRY IN THE TWO-YEAR COLLEGE

STATEMENT OF INCOME AND EXPENSE

FOR THE PERIOD OF 04/20/89 TO 9/05/89

BEGINNING BALANCE $10227.32

	CURRENT PERIOD RATIO	CURRENT PERIOD AMOUNT	YEAR TO DATE RATIO	YEAR TO DATE AMOUNT	
INCOME					
DUES AND SPONSORS					
Membership Dues	28.7	1094.00	35.7	2432.00	
Industrial Sponsors	1.6	60.00	6.8	460.00	
College Sponsors	4.6	175.00	15.1	1025.00	
Divched	52.5	2000.00	29.4	2000.00	
MEETINGS					
Dallas			1.9	130.00	
Cerritos	1.8	70.00	1.0	70.00	
INTEREST					
Checking Account	4.2	162.45	5.0	343.15	
CD's	6.6	252.18	5.1	346.44	
TOTAL INCOME	100.0	$ 3813.63	100.0	$ 6806.59	14040.95
EXPENSES					
TRAVEL & OFFICE					
Travel	60.0	2289.20	45.7	3109.53	
Office & Postage	5.1	195.92	4.5	309.05	
MEETINGS					
Cerritos	1.3	48.00	0.7	48.00	
OTHER					
Transferred to CD	65.6	2500.00	73.5	5000.00	
TOTAL EXPENSES	132.0	$ 5033.12	124.4	$ 8466.58	
NET INCOME(LOSS)	(32.0)	$(1219.49)	(24.4)	$(1659.99)	

CURRENT BALANCE $ 9007.83

To date there are 113 COLLEGE SPONSORS. Is your college a 2YC$_3$ COLLEGE SPONSOR?

Respectfully Submitted

Duane Sell

Duane Sell, Treasurer

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COMMITTEE ON CHEMISTRY IN THE TWO-YEAR COLLEGE

Division of Chemical Education, American Chemical Society

APPLICATION FOR COLLEGE SPONSORSHIP

College name _____ Date _____

Address: (Street) _____

(City) _____ (State) _____ (Zip) _____

Contact Person _____

[ ] Prof. [ ] Dr. [ ] Mr. [ ] Ms. [ ] Miss [ ] Mrs.

Telephone (Area Code) _____ (Number) _____ (Ext) _____

Address (if different from above) _____

_____

All questions and correspondence concerning the **2YC$_3$** College Sponsorship should be directed to:

Duane Sell
W.R. Harper College
1200 West Algonquin Road
Palatine, IL 60067-7398

Phone: (312) 397-3000 Ext. 2408

Please make check for $25 payable to:

COMMITTEE ON CHEMISTRY IN THE TWO-YEAR COLLEGE

and return with the completed application to:

DUANE SELL

at the above address

We <u>are</u> a tax exempt organization. Our tax I.D. # is 23-7169683.

Thank you for your sponsorship.

REPORT
OF
2YC3 INDUSTRIAL SPONSORS

PAID INDUSTRIAL SPONSORS TO DATE - 23

RESPECTFULLY SUBMITTED,

LEO KLING III

3 MAY 1989

CONFERENCE SUMMARY CALENDAR

SEPTEMBER 1, 1989

ACADEMIC YEAR					
1989-90	**106th** (Midwestern) Oct. 6-7,1989 Louisville, KY	**107th** (Southern) Nov. 17-18,1989 Gulfport, MS	**108th(ACS)** (Eastern) Apr. 20-21,1990 Roxbury Crossing, MA	**109th(C_3)** (Western) June 15-16,1990 N. Vancover, BC	**110th** (Biennial) Aug 6-10,1990 Atlanta, GA
1990-91	**111th** (Midwestern) Oct. 5-6,1990 Elgin, IL	**112th** (Eastern) Nov. 16-17,1990 Mays Landing, NJ	**113th** (Western) Mar. 1-2,1991 Phoenix, AZ	**114th(ACS)** (Southern) Apr. 12-13,1991 Clarkston, GA	
1991-92	**115th**	* **116th** (Southern) Nov. 15-16,1991 Wilmington, NC	**117th(ACS)** (Western) Apr. 3-4,1992	* **118th** (Midwestern) May, 1992 Dearborn, MI	**119th** (Biennial) Aug. 1992
1992-93	**120th** (Southern) Oct. 2-3,1992 Amarillo, TX	* **121st** (Western) Nov. 13-14,1992 Gresham, OR	* **122nd(ACS)** (Midwestern) Apr. 16-17,1993 Bloomington, MN	**123rd**	

* Indicates New or Revised Information

TWO-YEAR COLLEGE CHEMISTRY CONFERENCE SITES

1989-90 ACADEMIC YEAR

107th CONFERENCE(Southern Region)

Nov. 17-18,1989-Mississippi Gulf Coast Community College
Jefferson Davis Campus, Switzer Road,
Gulfport, MS 39507 (601)896-3355

Program Theme: POLYMER CHEMISTRY: ACADEMICS, INDUSTRY, and RESEARCH
Program Chair: James M. Knight, Mississippi Gulf Coast C.C.
OFFICE: (601) 896-3355 Ext. 144 **HOME:** (601) 875-5884
Local Arrangements Chair:
Ed Decker, Mississippi Gulf Coast C.C.
OFFICE: (601) 896-3355 **HOME:** (601) 896-8377
Local Industrial Sponsor Coordinator:
Janie Languirand, Mississippi Gulf Coast C.C.
OFFICE: (601) 896-3355

Comments: Excellant location near beach, motels, restraurants, special rates. Near Gulfport/Biloxi airport; limousine service possible to New Orleans Airport.

108th CONFERENCE(Eastern Region)
(Preceding the ACS National Meeting, Boston,MA, April 22-27, 1990)

Apr.20-21,1990-Roxbury Community College
1234 Columbus Ave, Roxbury Crossing, MA 02120-3400
Program Theme: TBA
Program Chair: Dorothy(Dotty) Stumpf, Middlesex Community College
Springs Rd., Bedford, MA 01730
OFFICE: (617) 275-8910 Ext 287 **HOME:** (508) 756-5594
Local Arrangements Chair:
Dorothy Higgins, Roxbury Community College
OFFICE: (617) 541-5317 **HOME:** (617) 739-1876
Local Industrial Sponsor Coordinator:
Muriel Kanter,
HOME: 130 Rosseter St., Dorchester, MA 02121
(617) 282-7139
OFFICE: Massachusetts General Hospital, Acute Care Laboratory
(617) 726-3856

109th CONFERENCE(Western Region)

(Tentative) **June 15-16,1990-Capillano College**, 2055 Purcell Way
North Vancouver, BC V7T 3H5
(604) 986-1911
Program Theme: CHEMISTRY ON THE PACIFIC RIM
Contact Persons: William Wasserman, Seattle Central Community College
Seattle, WA 98122 (206) 587-4083
Penny LeCouter, Capillano College, North Vancouver
Local Arrangements Chair:
TBA
Local Industrial Sponsor Coordinator:
TBA

NOTE: SPECIAL REGISTRATION FEE IN EFFECT FOR THIS MEETING

110th CONFERENCE

(In conjuction with the 11th Biennial Conference on Chemical Education)
Aug.6-10,1990 - Georgia Institute of Technology, Atlanta, GA

2YC$_3$ Program Chair:
Leo Kling III, Faulkner State Junior College
Bay Minette, AL 36507 **OFFICE:** (205) 937-9581
HOME: (205) 937-4384

* **2YC$_3$ Booth Committee:** Paula Ballard, Jefferson State Jr. College
2601 Carson Rd., Birmingham, AL 35215
Garry McGlaun, Gainesville Jr. College
P.O. Box 1358, Gainesville, GA 30503
Lester Harrison, Hinds Community College
Gulfport, MS

NOTE: SPECIAL REGISTRATION AND EXHIBIT FEES IN EFFECT FOR THIS MEETING

1990-91 ACADEMIC YEAR

111th CONFERENCE(Midwestern Region)
Oct. 5-6,1990 -Elgin Community College, 1700 W. Spartan Ave.,
Elgin, IL 60123
Contact Person: Lavoir Banks, Elgin Community College
(312) 697-1000 Ext. 320

112th CONFERENCE(Eastern Region)
Nov. 16-17,1990-Atlantic Community College
Black Horse Pike (Route 322), Mays Landing, NJ 08330
* **Program Theme:** SCHOLARSHIP, CURRICULUM, GRANTS
(How to Deliver Better Chemical Education Under
Today's Budget Constraints)
Program Chair: Alla O. Romano, New York City Technical College
300 Jay Street, Brooklyn, NY 11201
OFFICE: (718) 643-8242, or 7224 **HOME:** (516) 627-6491
Local Arrangements Co-Chairs: Edmund J. Zoranski & Donald A. Hurff
Chem. Dept., Atlantic C.C.
Zoranski: OFFICE: (609) 343-5023 **HOME:** (609) 546-7396
Hurff: OFFICE: (609) 343-5012 **HOME:** (609) 390-0730
Local Industrial Sponsor Coordinators: Same as above

113th CONFERENCE(Western Region)
March 1-2, 1991 - Phoenix College, 1202 West Thomas Road,
Phoenix, AZ 85013 (602)264-2492
Program Theme: SCIENTIFIC LITERACY FOR THE 21ST CENTURY
Program Chair: Alan Kruse, Chemistry Department, Pima C.C.
2202 W. Anklam Road, Tucson, AZ 85709
(602)884-6036
Local Arrangements Chair: Patricia Moyer, Chem. Dept., Phoenix College
(602)285-7142
Local Industrial Sponsor Coordinator: John Weide
Chemistry Dept., Phoenix College
(602)285-7140

* Indicates New or Revised Information

114th CONFERENCE(Southern Region)
(Preceding the ACS National Meeting, Atlanta, GA, April 14-19, 1991)
Apr. 12-13,1991-DeKalb Community College, 555 N. Indian Creek Drive
Clarkston, GA 30021
Contact Person: Judy Johnston (404) 299-4088

1991-92 ACADEMIC YEAR

115th CONFERENCE
October 1991

*** 116th CONFERENCE**
Nov. 15-16,1991-Cape Fear Community College, 411 N. Front St.
Wilmington, NC 28401 (919) 343-0481
Program Theme: TBA
Program Chair: Dale R. Buck, Chemical Tech. Dept., Cape Fear C.C.
OFFICE: (919) 343-0481, ext. 263 **HOME:** (919) 762-6369
Local Arrangements Chair: Raymond P. Brandi, Marine Tech. Dept., Cape Fear C.C.
OFFICE: (919) 343-0481, ext. 260 **HOME:** (919) 763-7192
Local Industrial Sponsor Coordinator: William E. Cheek, Chemistry Dept Central Piedmont C.C., 1201 Elizabeth Ave at Old Kings Drive, Charlotte, NC 28204
OFFICE: (704) 342-6968 or 6603 **HOME:** (704) 535-3418

117th CONFERENCE(Western Region)
(Preceding the ACS National Meeting, San Francisco, April 5-10,1992)
Apr. 3-4,1992

*** 118th CONFERENCE(Midwestern Region)**
May 1992 - Henry Ford Community College, 5101 Evergreen
Dearborn, MI 48128-1495
Contact Person: Joseph Maguire

119th CONFERENCE
(In conjuction with the 12th Biennial Conference on Chemical Education)
August 1992
NOTE: SPECIAL REGISTRATION AND EXHIBIT FEES IN EFFECT FOR THIS MEETING.

* Indicates New or Revised Information

1992-93 ACADEMIC YEAR

120th CONFERENCE(Southern Region)
October 2-3,1992-Amarillo College, P.O. Box 447, Amarillo, TX 79178
(806) 376-5111, FAX (806) 371-5370
ADDRESS FOR PACKAGES: 24th and Jackson, Amarillo, TX. 79109
Program Theme: TBA
Program Chair: A.G.(Jerry) Foster, Physical Science Dept.
Amarillo College
(806) 371-5327
Local Arrangements Chair:
Peggy W. Alley, Physical Science Dept.
Amarillo College
(806) 371-5334 or 371-5333
Local Industrial Sponsor Coordinator:
Mary Graff, Physical Science Dept.
Amarillo College
(806) 371-5326

*** 121st CONFERENCE(Western Region)**
Nov. 13-14,1992-Mt. Hood Community College, 26000 S.E. Stark St.
Gresham, OR 97030 (503) 667-6422
Contact Person: Gilbert Albelo

*** 122nd CONFERENCE**(Midwestern Region)
(Preceding the ACS National Meeting, Minneapolis, MN, April 18-23,1993)
Apr. 16-17,1993-Normandale Community College, 9700 France Ave. South,
Bloomington, MN 55431 (612) 830-9300
Contact Person: Warren Eidsness

{April 10-15,1994 ACS National Meeting and 5th Chemical Congress of North America, Mexico City}

* Indicates New or Revised Information

All $2YC_3$ members are urged to make suggestions regarding meeting sites, program personnel, themes and programming suggestions, in writing, to Executive Committee members and Program Chairs of the specific conferences. $2YC_3$ can function more effectively to serve all community colleges when all persons participate. We welcome your suggestions.

AMERICAN CHEMICAL SOCIETY

Office of College Chemistry

Highlights of Activities

for the period

March 7, 1989 - August 30, 1989

College Activities

- •3/89 – Produced and distribuited DivCHED's 2YC3 agenda for its 104th conference.
- •4/89 – Assisted chairman of DivCHED's Committee on Chemistry in Two-Year Colléges in developing testimony that was delivered to a U.S. House of Representatives hearing on *"Science and Engineering in Two-Year and Community Colleges."*
- •5/89 – Produced and distributed DivCHED's agenda for its 105th conference.
- •6/89 – Developed, produced, and distributed the *"2YC Distillate"* newsletter.
- •7/89 – Advisory board for the *"College ChemCom"* text met for the first time.
- •7/89 – Participated in workshop for SOCED's science education policy principle paper.
- •8/89 – Editorial board for the *"College ChemCom"* text met for the first time.

Student Affiliates Activities

- •4/89 – The Student Affiliates Poster Session at the Dallas national meeting was a success. Forty-nine studends presented original research.
- •5/89 – Education staff worked with the ACS Marketing staff on a study of the needs of the Student Affiliates. The study methodology consisted of six focus groups held in Washington, DC, Los Angeles, and Chicago.
- •6/89 – Produced and distributed *the pHilter* to approximately 6300 Student Affiliates and interested others.
- •6/89 – Held the Student Affiliates Faculty Advisor Workshop in Washington, DC. The workshop was convened to gather information about declining enrollments in the program and to obtain information about problem solving. This productive weekend was attended by 15 invited faculty advisors who represented different regions of the country, two- and four-year and research universities, and predominantly majority and minority schools.
- •7/89 – Draft of topline report written and distributed for peer review.
- •7/89 – The second effort renewal mailing for Student Affiliates membership was completed.

MEMBERSHIP DATA

	1989	1988
March	308	233
April	164	148
May	309	210
June	382	382
July	110	169
Total Jan.–July:	**2044**	**1497**

SOCED's Two-Year College Task Force

•5/89 – Completed two consultant visits to Minnesota community colleges as part of the program to implement the T2 recommendations of the *"Tomorrow"* report.

•5/89

8/89– Held meetings pulling together supplementary materials for the *"Guidelines"* and meetings to complete the T3 *"Tomorrow"* report recommendation on a chemical technology program approval service.

•6/89 – Conference call contributing ideas for proposed science education policy principle paper.

Minorities in Science and Technology

•3/89 – Attended meeting of the White House's Task Force on Women, Minorities, and the Disabled in Science and Technology.

•4/89 – Attended meeting of the National Urban Coalititon.

•5/89 – Attended national meeting of National Action Council for Minorities in Engineering, Inc.

•6/89 – Completed taping of the high school and college portions of the U.S. Department of Education funded videotape on *"Opportunities for Minorities in Science."*

•8/89 – Began taping the role model interviews for the minorities in science videotape.

College Chemistry Consultants Service

- MSIP (U.S. Department of Education)–funded program
  - Thirteen consultant visits completed between March and August.
  - Eight community outreach programs completed between March and August.
  - Two traditional C3S visits completed.

COMMITTEE ON CHEMISTRY IN THE TWO-YEAR COLLEGES
ACS Division of Chemical Education

INTERIM REPORT, August, 1989
John V. Clevenger, 1989 Chair

This report summarizes activities and conferences conducted by the Committee on Chemistry in the Two-Year Colleges (COCTYC) for the period January-August, 1989.

Conferences. Our major activity continues to be planning and conducting four regional Two-Year College Chemistry Conferences ($2YC_3$) each year. For 1989 these are:

104th $2YC_3$, April 7-8, 1989, North Lake College, Irving, TX

105th $2YC_3$, May 19-20, 1989, Cerritos Community College, Norwalk, CA

106th $2YC_3$, Jefferson Community College, Louisville, KY

107th 2YC3, Mississippi Gulf Coast Community College, Gulfport, MS

The first two of these have each had over 100 participants, with over 70 new members joining $2YC_3$. The 104th, $2YC_3$ had the dual themes of: We Are in the Middle: High School - Community College - University Interfacing and Hazardous Waste Management. There was a great deal of interest in presentations such as "Disposal of Hazardous Laboratory Chemicals - How It's Done" and "Articulation and Transferability Concerns" These are obviously areas of major concern in two-year colleges.

The 105th $2YC_3$ had the program theme, Chemical Education: How Effective Is It? Some of the talks were: "Chemical Education for Public Understanding", "One Third of A Nation, Our Challenge in the 1990's", and "Science and Human Values...One Culture."

We encourage anyone interested, from any level of chemical education, to come to our conferences. Your participation makes them even better.

Grants and Other Resources. We're making a special effort to encourage and help two-year faculty with the process of seeking out and obtaining as many resources as possible. This has always been one of the goals of our conferences. For this year, we've added a special session on writing grants to each of our conferences. Presentors are two-year college instructors who've served as grant review panelists. Staff from the National Science Foundation will participate in at least three of these sessions. For faculty not able to attend the conferences, we've tried to provide sources for various resources through the Newsletter.

Continuing Acitivities. We publish four editions of the $2YC_3$ Newsletter, distributing over 2000 copies of each edition to all two-year colleges and individual $2YC_3$ members. We encourage membership in the Division of Chemical Education and participation in its programs and acitivities. We also participate in the Society Committee on Education (SOCED) Task Force on ACS Involvement in the Two-Year Colleges.

New and Future Directions. We are beginning an ongoing discussion of how we can best continue to serve the two-year college community. With a survey in our $2YC_3$ Newsletter , and continuing with surveys and discussions at our conferences, we're asking questions such as: "What are we doing well?" "What can we do to improve our conferences and other activities?" and "How can we actively involve more people in the organization?" We would welcome all suggestions and participation. Feel free to contact any of the officers or program chairs of the individual conferences with any ideas you may have.

New Officers. At the 105th $2YC_3$ in Cerritos, CA we announced the results of two elections for new officers. Margot Schumm of Montgomery Community College, Rockville, MD is the Chair-Elect for 1990. Richard Jones of Sinclair Community College, Dayton, OH will be the new Membership Chair on January 1, 1990.

Respectfully submitted,

John V. Clevenger

John V. Clevenger
1989 COCTYC Chair

SURVEY

This survey is not designed to produce a series of numbers to be tabulated, but to gather information about strengths, weaknesses, and future directions for the Committee on Chemistry in the Two-Year Colleges.

We'll use your responses as starting points for discussions at future Committee meetings. Please feel free to make any comments that you believe would improve this organization.

CONFERENCES

What factors influence you to attend a conference?
(Please number in order of importance.

- ❑ Program (❑ overall ❑ particular speakers)
- ❑ Location (❑ close to your college ❑ nice location)
- ❑ Funding for your expenses
- ❑ Time of year
- ❑ Workshops with Conference ❑ Other (explain)

_____

What topics would you like to see at a conference in the near future? (Please number in order of importance.)

- ❑ New Instrumentation
- ❑ Developments and Applications of Learning Theory
- ❑ Effective Instructional Techniques
- ❑ New Course Designs/Content
- ❑ Lecture Demonstrations
- ❑ Articulation with other insitutions
- ❑ Hazardous Materials Management
- ❑ Grant Writing ❑ Other (explain)

_____

Would you like more workshops at the conferences?

❑ Yes ❑ No

If you answered yes: What topics would be useful?

_____

When should they be scheduled?
(Please number in order of importance.)

- ❑ Friday afternoon ❑ Saturday morning
- ❑ Saturday afternoon

What could be changed about the conferences to make them more useful to you? _____

_____

_____

If you have never attended one of our conferences, please briefly explain the reason(s). _____

_____

_____

What could our organization do to make the conferences more attractive to you? _____

_____

_____

Additional comments about our conferences

_____

_____

PUBLICATIONS

This Newsletter is our primary means of communication with you. What do you like most about it?

_____

What additional things would you like to see included? _____

_____

_____

We also contribute (financially and $2YC_3$ news) to the publication and distribution of the *2YC Distillate: A Newsletter for Two-Year College Educators*, published by the American Chemical Society.

Do you receive the *Distillate*? ❑ Yes ❑ No
If not, could we add you to the *Distillate* mailing list?

❑ Yes ❑ No

For that part of the *Distillate* relating to $2YC_3$:
What do you like most? _____

_____

What additional things would you like to see included?

_____

_____

Additional comments about our publications:

_____

THE ORGANIZATION

Are you now a member of $2YC_3$? ❑ Yes ❑ No
If you are not a member of $2YC_3$, please briefly explain the reason(s). _____

_____

We are trying to *actively* involve more people in this organization. How could we encourage you to take a more active role? _____

_____

Additional comments about our organization:

(Optional)

Name _____

College _____

College Address _____

City _____ State _____

Zip Code _____

Please return to:
John V. Clevenger, 1989 Chairman COCTYC, Truckee Meadows C.C., 7000 Dandini Blvd, Reno. NV 89512

September 5, 1989

FIRST NEWSLETTER SURVEY

CONFERENCES

Factors To Attend Conference

{higher numbers indicate higher rating}

Program (161)	Overall (30)	Part. Speak. (7)
Location (155)	Close (32)	Location (3)
Funding (104)		
Time of year (82)		
Workshops (54)		
Other		

Topics

{higher numbers indicate higher rating}

New Instrumentation (151)
Learning Theory (174)
Instructional Techniques (234)
New Course Content (175)
Lecture Demonstrations (169)
Articulation (124)
Hazardous Materials (154)
Grant Writing (78)
Other (12)
- inexpensive lab substitutes
- NMR/HPLC
- technician training
- instrumentation[4]

Workshops

Yes (18) No (15)

Topics

- interactive videodiscs, hypercard
- instrumentation[3]
- computer applications in chem[3]
- interfacing computers with instrumentation
- consultatant visits by ACS
- creation & evolution
- microscope work & chemistry
- hazardous materials management [2]
- grant writing[2]
- technical writing
- laboratory experience

Scheduled
{higher numbers indicate higher rating}
Fri afternoon (13) Sat. morn (28) Sat. afternoon (29)

What Could Be Changed About Conferences
smaller/less elaborate in smaller regions/minimize travel & leave
current topics of student life and careers
emphasis on how "experts" present a topic (e.g. acid/base equil)
continue to do what is timely
KC Kansas just fine
open forum
small group discussion [2]
-----(topics: motivating students, test anxiety, etc.)
better accomadate technician instructors
closer to home[2]
one day format
employment/ bulletin board/ interviews
schedule other than school time
-------(no substitutes available/difficult to leave during regular school year)
nothing-timing is good, variety of locations is good
keep speakers on schedule
I think you do a good job
In addition to books, could video publishers show their wares
more opportunity to interact with other participants - mixers

Why Haven't You Attended
time & money-several smaller local ones may be useful [3]
I just began teaching chemistry
time and location

What Could Make Conferences More Attractive?
funding/travel subsidies(NSF, NSTA, NIH) [7]
---one doesn't need to attend every year
coordinate with state organization
conference near Chicago, or Wis., Minn.
more convenient arrangements for lunch
good workshop on Sat. afternoon/makes it worthwhile to come
funding [several times]
{continued next page}

travel stipends
more demonstrations
different time of year [4]
---- during breaks or summer
--- midwest in spring(late April to mid May)
good exhibitions - bigger
tours of local culutal & social events

Additional comments
how can we include more H.S. teachers
tours of cultural and social events in area/ other groups do this as well as the business meeting
technician instructors need more help than others, yet seem to be ignored
they're great-keep up the good work
great diversity of topics to cover needs of all
distribute roster of attendees
good workshops on Sat. aft. make it worthwhile to come

PUBLICATIONS

What Do You Like About The Newsletter?

future meetings/program announcements [14]
news [7]
it reminds me I'm part of a family
informative and up-to-date
this is the first Newsletter that is more than a conference announcement
brevity and concise
knowing what is happening and what to expect at each conference
very thorough
format
timeliness-4 times/yr

What Additional Things Would You Like Included?

articles in addition to conference programs [2]
------textbook reviews
------available grants
------new recruitment ideas
------budget & planning
------salary scales at 2yr schools
------essays
------letters from members
------ dialogue among H.S./2yr/4yr
-------new ideas on instruction and lab ideas

{continued next page}

--------info. on fellow teachers/interests/specializations/ more communication/fellowship
------ more activities reports for technician instructors
------short news of research topics
------news about local $2YC_3$ type groups
------community outreach ideas
is it needed/combine with Distillate?
meeting agendas 6 months in advance
abstracts/summaries of various conference topics
meeting results - decisions, etc.

What Do You Like Most About The Distillate?
crossword puzzle
news
key points of meetings
general information
I read it cover to cover
updates on conferences
abstracts of papers/keypoints [3]
critical comparisons of video-tapes available
aids in my teaching

What Additional Things Would You Like Included?
more material
book reviews/curriculum planning
projects/techniques
new ideas on instruction/computer software
salaries and benefits at other colleges/ curriculum changes/ problems with quarter to semester movement
receive more often--"quarterly" publication seems to be annual
aids to my teaching
more computer applications

Additional Comments
excellent
Distillate should be published more frequently

THE ORGANIZATION

Member of $2YC_3$ Yes (39) No (3)

If Not, Explain

no conf. I can feasibly attend in next 2-3 yrs

How Could We Encourage A More Active Role?

not easy/ many thinking retirement not more involvement
financial aid
continue to invite participation & be open and friendly
keep reminding me of committees and task forces
I have no travel money/ is there something to do while staying at home?
offer more rewards like money
let me know what I can do
ask me to help with committees, etc

Additional Comments

establish a local Bulletin Board Service
thank you for this opportunity and for your efforts
great, I appreciate the interaction with other 2-yr college teachers
concern about increasing spending of funds with little observed benefits to organizaton
more NSF funding for special projects

GUIDELINES SURVEY
PLEASE COPY AND DISTRIBUTE TO INDIVIDUAL FACULTY

Copies of the *Guidelines for Chemistry and Technology Programs in Two-Year Colleges* have been sent to all two-year colleges and several other target groups. These guidelines were developed by the ACS Society Committee on Education's Task Force on Two-Year Colleges established in 1983 with a four-part charge:

1. Revise the 1970 *Guidelines for Chemistry in the Two-Year College* to reflect the diversity of chemistry education responsibilities that have become the norm for individual two-year colleges in the past 15 years.
2. Develop an outreach and consultation program which would make the expertise of the Society staff membership more effectively available to two-year college administrators whose institutions are engaged in substantial efforts at improving the quality of their chemistry programs.
3. When the revised *Guidelines* have been published and the outreach and consultation program is functioning, the ACS should undertake to certify/approve Chemical Technology programs in two-year colleges at the request of such institutions.
4. When recommendations 1,2, and 3 have been implemented, the ACS should undertake to certify/approve college transfer programs in two-year colleges at the request of such institutions.

Charges 1,2, and 3 are completed or are well underway. There has been considerable discussion regarding whether charge 4 should be implemented. The Task-Force is very interested in the distribution and use of the *Guidelines* and anxious to know how the question of implementation is perceived by the people most concerned. Your reaction to these questions would be extremely useful.

Please respond to the following questions and return this survey to:

Jay Bardole, Division of Science & Math
Vincennes University,
Vincennes, IN 47591

Have you seen a copy of the *Guidelines*? ❑ Yes ❑ No

If your answer was no, and you would like a copy, please contact the Office of College Chemistry, American Chemical Society, 1155 16th St., N.W., Washington, D.C. 20036-4899 (202) 872-4587

Have the *Guidelines* been useful to you? ❑ Yes ❑ No Please explain

Should the *Guidelines* be used to certify/approve college transfer programs in two-year colleges?
❑ Yes ❑ No Please explain

REGION I - WESTERN STATES: Alaska, Arizona, California, Colorado, Hawaii, Idaho, Montana, Nevada, New Mexico, Oregon, Washington, Wyoming

---

Christine Romer
Cerritos Community College
11110 E. Alondra Blvd.
Norwalk, CA 90650-9973
(213) 860-2451

PROGRAM CHAIR - 1989

Albelo, Gilbert
Mt. Hood Community College
26000 S. E. Stark Street
Gresham, OR 97030 (1990)
(503) 667-6422

Armstrong, Elizabeth
Skyline College
3300 College Drive
San Bruno, CA 94066-1698 (1991)
(415) 355-7000

Berner, Victor
New Mexico Junior College
Lovington Highway
Hobbs, NM 88240 (1990)
(505) 392-4510 ext. 261

Chase, Jody
Truckee Meadows Community College
7000 Dandini Blvd.
Reno, NV 89512 (1990)
(702) 673-7221

Collins, Carolyn
Clark County Community College
3300 East Cheyenne Avenue
N. Las Vegas, NV 89030 (1991)
(702) 456-4109

Cunningham, Alan
Monterey Penninsula College
Monterey, CA 93940 (1990)
(408) 646-4154

Hubbs, Robert
DeAnza College
Cupertino, CA 95014 (1991)
(408) 996-4774

Hubscher, Arthur
Ricks College
Rexburg, ID 83460-0500 (1991)
(208) 356-1930

Lungstrum, Richard A.
American River College
Sacramento, CA 95841 (1989)
(916) 484-8464

Mooney, William T.
El Camino College
Torrence, CA 90506 (1990)
(213) 532-3670

Peter, James R.
Cerritos Community College
11110 E. Alondra Blvd.
Norwalk, CA 90650 (1990)
(213) 860-2451

Scott, Peter
Linn-Benton Community College
6500 SW Pacific Blvd.
Albany, OR 97321 (1989)
(503) 928-2361

Sherman, Ruth
Los Angeles City College
855 N. Vermont Avenue
Los Angeles, CA 90029 (1989)
(213) 669-4223

Van Dyke, Martin
Front Range Community College-North
3645 W. 112th Avenue
Westminster, CO 80030 (1990)
(303) 466-8811

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106th TWO-YEAR COLLEGE CHEMISTRY CONFERENCE

Jefferson Community College, P.O. Box 1036, Louisville, KY 40202

Friday, October 6 and Saturday, October 7, 1989

Program Theme: Mixing It Up For the 90's
1° Focus: Integrated/Interdisciplinary Approaches to Chemistry
2° Focus: Development & Retention of Science (Chem) Students
3° Focus: New Laboratory Techniques & Concerns

For additional information about the program or arrangements, please contact:

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Local Industrial Sponsors Coordinator: William (Bill) Baker
General Electric, Louisville, KY 40204, (502) 361-3300

Friday, October 6, 1989

9:00-3:00 Registration, exhibits, coffee—Hartford Building Lobby

9:30-10:45 Committee on Chemistry in the Two Year Colleges, John Clevenger, COCTYC Chair, presiding. Room 006. This meeting is open to all interested persons.

10:50-11:10 Opening Ceremonies, Lecture Hall Room 006. Opening remarks: John Clevenger. Welcome: Dr. Ronald J. Horvath, President, Jefferson Community College. Introduced by Patricia McCoy Brown

11:15-noon Keynote Address: Dr. Russel M. Pitzer, Ohio State University.
"Relativity and The Periodic Table"

Noon-1:30 Lunch (area restaurants)

1:30-2:20 Professors Janet Schwitzer and Vera Quinn, Jefferson Community College.
"Interdisciplinary Approaches to Teaching Science"

2:25-3:00 Professor Jay Bardole, Vincennes University and member of the Task Force on Two-Year Colleges.
"What Has The Task Force Done For You?"

3:00-3:15 Coffee break

3:15-3:45 Ms. Terri Nally, Office of College Chemistry, American Chemical Society.
"Two-Year Colleges, ACS, and Capital Hill"

3:50-4:15	Dr. Ted Reid, National Science Foundation, Washington, DC. *"NSF and Two Year Colleges"*
4:20-4:50	Professor Peggy W. Alley, Amarillo College. *"Anatomy of a Winning NSF-ILI Grant"*
4:50-5:15	Professor Katherine E. Weissmann, Charles Stewart Mott Community College. *"Perspectives of the N.S.F. Instrumentation and Laboratory Improvement Proposal Review Process-Suggestions for Two-Year College Proposal Writers"*
5:20-6:15	Break
6:15-6:30	Depart for Riverboat
6:30-9:30	Riverboat Ride

Saturday, October 7, 1989

8:30-noon	Registration, coffee, exhibits
8:30-9:20	Professor S. Smith, University of Illinois. *"Enhancing the General Chemistry Laboratory Experience with Interactive Videodisc"*
9:25-10:05 OR	Professor Leonard C. Grotz, University of Wisconsin-Waukesha Center *"Scientific Methods: Uses and Abuses"*
9:25-11:30	WORKSHOP I: Professor Ron Pike, Merrimack College. Room 907 *"Microscale Organic Laboratory Techniques"*
10:05-10:20	Coffee Break
10:25-11:25	Professors Janice A. Olexia and Estelle K. Gearon, Montgomery College. *"Improving Achievement in Introductory Chemistry Through Structured Supplimentary Instruction"*
11:30-12:30	Lunch
12:35-1:10 OR	Ms. Betty Abraham, East Hardin High School *"Producing Better Science Fairs for Middle and High Schools"*
12:35-1:10	WORKSHOP II: Professor Ron Pike, Merrimack College. Room 907 *"Microscale Organic Laboratory Techniques"*
1:15-2:15	Dr. Daniel J. Antion, Associate Provost, University of South Carolina. *"Project Interaction: An Effective Model for the Continuing Education of High School Chemistry Teachers"*
2:15-2:30	Coffee Break
2:35-4:00	Professors C. Marvin Lang and Donald L. Showalter, University of Wisconsin-Stevens Point—Chemical Demonstration Show *"Yes Virginia, Chemistry Can Be Fun"*

106th TWO-YEAR COLLEGE CHEMISTRY CONFERENCE

Jefferson Community College
Louisville, KY 40202

October 7, 1989

MICROSCALE WORKSHOP

106th TWO-YEAR COLLEGE CHEMISTRY CONFERENCE

Jefferson Community College
Louisville, KY 40202

October 7, 1989

MICROSCALE WORKSHOP

Table of Contents

*Excerpts taken from Mayo, D.W.; Pike, R.M.; Butcher, S.S. *Microscale Organic Laboratory* 2nd ed., Wiley: New York, 1989.

**From a forth coming text by Szafran, Z.; Pike, R.M.; Singh, M.M. *Inorganic Microscale Laboratory* Wiley: New York, 1990.

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Experiment 2 Simple Distillation at the Semimicroscale Level: Separation of Ethyl Acetate from *trans*-1,2-Dibenzoylethylene

Physical Properties of Reactants and Products

Compound	MW	Wt/Vol	mp(°C)	bp(°C)	Density	n_D
Ethyl acetate	88.12	1.0 mL		77	0.90	1.3723
trans-1,2-Dibenzoylethylene	236.27	50 mg	111			

Transfer 1 mL of the yellow stock solution (*trans*-1,2-dibenzoylethylene/ethyl acetate, 50 mg/mL) to a 3-mL conical vial by automatic delivery pipet (remember to place the vial in a small beaker to prevent tipping during the transfer). Place a boiling stone in the vial and assemble the Hickman still head. The still assembly is mounted in a sand bath and placed on a hot plate (see Fig. 5.15). The temperature of the bath is raised to 90–100°C at a rate of 5°C/minute.

CAUTION: *Do not let the temperature of the still rise too rapidly.*

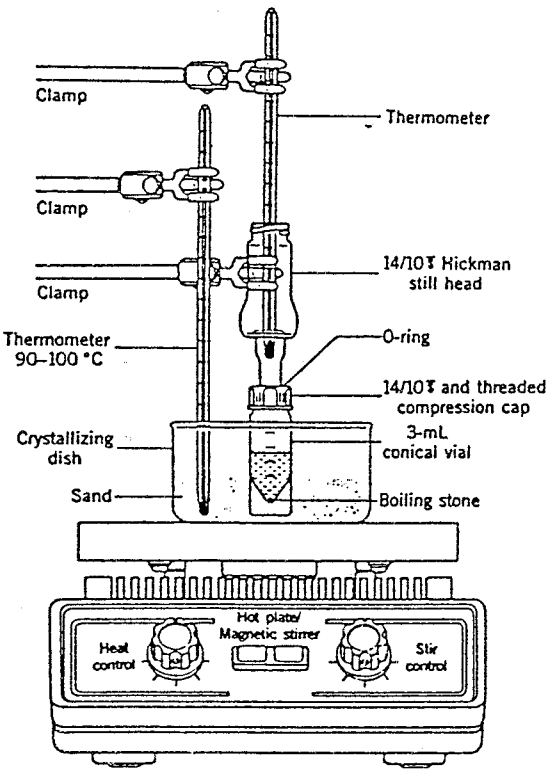


Fig. 5.15 *Hickman still (14/10 with conical vial (3 mL).*

Once boiling commences, the rate of heating should be lowered to 2–3°C/min. A slow distillation rate is very important in establishing equilibrium between the vapor and liquid components in the mixture. Follow the course of the distillation by the rise of condensate on the sides of the Hickman column. When the condensate reaches the trap, adjust the bath temperature so that liquid is removed from the column slowly (approximately 100 μL/minute). A smooth slow distillation will provide a cleaner separation of the components and will also avoid mechanical transfer of nonvolatile components via splattering to the condensate trap (if the condensate appears yellow, mechanical contamination has occurred).

Collect approximately 50–150 μL of the ester in the collar of the still (the first fraction collected is often referred to as the forerun; give the temperature range). As the distillation continues, remove the forerun with a Pasteur pipet having a slightly bent tip (microburner). Place the fraction in a clean, dry 1-dram screw-capped vial (use an aluminum foil liner to avoid cap contamination). Number the fraction with a marking pen. Collect a second fraction of ester (400–500 μL, which may require combining two or even three collections from the collar; give the temperature range), which should be clear and colorless. Remove and store as before. Discontinue the distillation. Allow the distilling flask to cool slowly by leaving it in the warm sand bath while measuring the physical properties of the distillate fractions. Three physical properties of the ester will be measured to establish the identity and purity of the compound by comparison with known literature values.

Determine the refractive index (see Chapter 4) of the two fractions collected. Compare the experimental values to those found in the literature for ethyl acetate. If the values are within 0.0010 unit of each other, the fractions are considered to have the same constitution. Are the values for the two fractions the same? If not, which one deviates the most from the reference data? Attempt to explain the result?

Determine the density (see Chapter 4) of the ester using material contained in the second fraction. This measurement is nondestructive. The material employed may be recovered for use in further tests. Compare your results with those values found in the literature.

Determine the boiling point of the second fraction by the ultramicro boiling point procedure (see Chapter 4). Compare your result with the literature value. Does this fraction appear to be pure ethyl acetate?

In the next step, disconnect and cool the 3-mL conical vial in an ice water bath for 10 min. *trans*-1,2-Dibenzoylethylene will crystallize from the concentrated solution. Remove the remaining solvent from the distillation vial with a Pasteur filter pipet and place the crystals on a porous clay plate to air dry.

Recrystallize this material from 95% ethanol using the Craig tube Determine the melting point after drying the crystals on a clay tile or on filter paper (Lit. value =111-112°C)

Reference values of the physical constants are available in the *CRC Handbook of Chemistry and Physics*. Submit a copy of the table prepared in your laboratory notebook to the instructor after tabulating the experimentally measured values of the physical properties in addition to those reported in the literature for ethyl acetate (see "acetic acid, ethyl ester").

Experiment 3C

Fractional Distillation of 2-Methylpentane and Cyclohexane with a Spinning Band in a Hickman–Hinkle Still

The distillation will separate the same two compounds used in Experiment 3B. The distillate can be analyzed to determine the number of theoretical plates. If careful attention is given to the procedure, the spinning Hickman–Hinkle is capable of more than seven theoretical plates.

In this experiment separation of a 2-mL mixture of 2-methylpentane and cyclohexane is achieved. The purity of the fractions will be determined by gas chromatography and by measurement of the refractive index.

Physical Properties of Components in the Mixture

Compound	MW	bp (°C)	n_D
2-Methylpentane	86.18	60.3	1.3715
Cyclohexane	84.16	80.7	1.4266

Assemble the system as shown in Figure 3.18. In the process, make sure that the Teflon band is aligned as straight as possible in the column. In particular, the pointed section extending into the pot must be straightened to minimize vibration during spinning of the band.

Once the spinning band has been tested and rotates freely, place 1.0 mL of 2-methylpentane and 1.0 mL of cyclohexane in the pot (to be delivered with a Pasteur pipet or an automatic delivery pipet). Reassemble the system and lower the column into the sand bath.

Cover the sand bath with aluminum foil during the distillation to prevent the collar of the still from overheating.

Gently heat the pot until boiling occurs. When heating commences turn on the magnetic stirrer at a low setting. When reflux commences at the base of the column the magnetic stirrer is raised to intermediate settings. Once liquid begins to enter the column the spin rate is increased to the maximum (1000–1500 rpm). *It is extremely important that careful temperature control be exercised at this stage so that the condensing vapors ascend the column very slowly.* Vapor phase enrichment by the most volatile component is limited mainly to this period, as fraction collection commences immediately on arrival of the vapor column at the annular ring. Once condensation occurs fractions are collected by the same technique used in Experiment 3A. Characterization of the fractions, however, follows the procedure given for Experiment 3B.

Characterization of the Fractions

The composition of each of the fractions may be determined by gas chromatography, the refractive index, or both. See Experiment 3B for details.

An alternate approach to the procedures discussed in Experiment 3B is to establish the fraction volume by weight. The curves shown in Figure 5.22 again may be used to estimate the number of theoretical plates. The volume of the first fraction can be estimated or determined more accurately by weighing the fraction in a tared screw-cap vial. The composition of this fraction then may be determined and the fraction of the total represented by this portion calculated. If, for example, the first fraction has a volume of 0.4 mL (20% of the total) and has a composition 0.898 by volume of 2-methylpentane, we would infer that the system had a resolution equivalent to about four theoretical plates.

It is absolutely critical that the temperature of the pot be adjusted so that vapors in the column rise very slowly. It is possible for overheated vapors to be forced through the air condenser.

When the vapors slowly arrive in the unjacketed section of the head of the column the condenser joint acts as a vapor shroud to effectively remove vapors from the receiver cup area. During this total reflux period, maximum separation of the components is achieved. Once total reflux is obtained the system is left for 20–30 minutes to reach thermal equilibrium. During total reflux the head thermometer should read about 57–60°C (at least for the equilibration time).

After the end of equilibration you are ready for collection. Rotate the air condenser 180° so that the beveled edge is over the collection duct. By pulling the Teflon stopper in the side arm out, some of the condensate will be collected. (Putting it back in stops collection.) Collect six drops (approximately 0.30 mL), and put the stopper back into the arm. After removing the collection vial, transfer the contents into a covered shell vial using a Pasteur pipet. *Label all fractions.* Collect two 0.60-mL fractions; then turn off the heat and stirring motor and remove the pot from the sand bath. Transfer the pot solution using a Pasteur pipet to a fourth covered shell vial. You have now collected four fractions.

Characterization of the Fractions

The composition of each of the fractions may be determined by gas chromatography and the refractive index.

The Gow-Mac gas chromatograph should be set up as follows:

Column	DC 710
Attenuation	64
Injection	10 μL
Temperature	80°C
Flow rate (He)	55 mL/minute
Chart speed	1 cm/minute

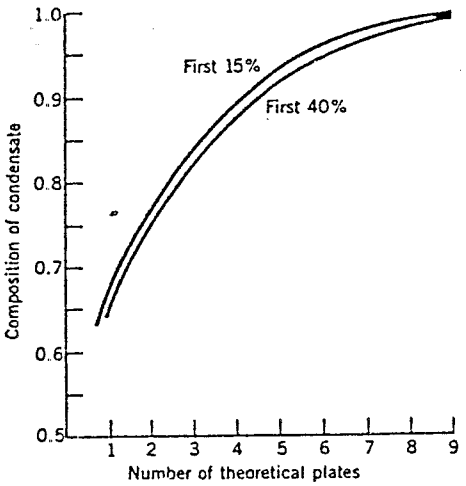


Fig. 5.22 *Composition of the first 15% and the first 40% of the volume collected in the distillation of a 50% (v/v) mixture of 2-methylpentane and cyclohexane.*

If we assume that the refractive index is a linear function of the volume fraction, the following relationship gives us the volume fraction of 2-methylpentane in a mixture. X is the volume fraction and n_D is the measured refractive index.

$$X = (1.4266 - n_D)/(1.4266 - 1.3715)$$

The curve shown below in Figure 5.22 may be used to estimate the number of theoretical plates from the composition of the *first* 0.30-mL fraction. For example, if the composition of the first 0.30 mL is 0.89, we would infer that the system had a resolution equivalent to about four theoretical plates. Note that the number of plates cannot be determined with confidence if the composition is greater than about 0.97. If we really wanted to determine the number of theoretical plates for a system with more than five plates, we could start with a mixture only 10 or 20% in the MVC, rather than the 50% used in this experiment.

ENVIRONMENTAL DATA

Substance	Amount (mL)	TLV (mg/m^3)	Emissions (mg)	Volume (m^3)
2-Methylpentane[a]	1.0	1800	650	0.4
Cyclohexane	1.0	1050	780	0.7

[a] treated as hexane isomers other than *n*-hexane.

Experiment 4A, 4B, 4C Solvent Extraction

PART A: Determination of a Partition Coefficient: Benzoic Acid—Methylene Chloride and Water

This experimental exercise illustrates the procedure that is used to determine a partition coefficient. Weighing small quantities on an electronic balance, the use of automatic delivery pipets, the transfer of solutions with the Pasteur pipet, and the use of a Vortex mixer (if available) are techniques encountered in this experiment.

Physical Properties of Reactants and Products: Parts A and B

Compound	MW	Wt/Vol	mmol	mp(°C)	bp(°C)	Density
Benzoic acid	122.13	50 mg	0.41	122		
Methylene chloride		2.4 mL			40	1.33
Water		600 μL			100	1.00
Sodium bicarbonate (10% solution)		600 μL				

To a 3.0-mL conical vial fitted with a screw cap, add 50 mg (0.41 mmol) of benzoic acid. There is then added 600 μL of methylene chloride followed by 600 μL of water.

These solvents are delivered by aid of automatic delivery pipets. The methylene chloride is dispensed in the hood.

The mixture is shaken (or a Vortex mixer is used) until the benzoic acid dissolves. The two layers are then allowed to separate after being thoroughly mixed (remember to vent the vial).

Draw the lower layer into a Pasteur filter pipet, carefully transfer the methylene chloride solution to a vial containing 100 mg of anhydrous, granular sodium sulfate, and then cap the vial.

If the methylene chloride layer is not transferred totally in the first operation, perform a second transfer.

After drying the methylene chloride solution for a few minutes, transfer it to a previously **tared** vial using a Pasteur filter pipet. The sodium sulfate is rinsed with an additional 600 μL of methylene chloride and the rinse combined with the solution in the tared vial. The solvent is now evaporated under a gentle stream of nitrogen gas or in a sand bath in the **hood**.

HOOD

If a sand bath is used, a boiling stone is placed in the vial before it is tared.

Weigh the vial and determine the weight of benzoic acid in the methylene chloride layer and in the water layer.

For the methylene chloride layer, the weight of the benzoic acid and vial minus the weight of the vial equals the weight of the benzoic acid.

For the water layer, the original weight of benzoic acid minus the amount of benzoic acid in the methylene chloride layer equals the weight of the benzoic acid.

Since equal volumes of both solvents were used, the partition coefficient may be determined from the ratio of the weight of benzoic acid in the methylene chloride solvent to the weight of benzoic acid in the water layer.

Calculate the partition coefficient for benzoic acid in the solvent pair (equal volumes) used in this exercise.

PART B: Benzoic Acid—Methylene Chloride and 10% Sodium Bicarbonate Solution: An Example of Acid–Base Extraction Techniques

Benzoic acid reacts readily with sodium bicarbonate to form sodium benzoate, carbon dioxide, and water. The sodium derivative has saltlike characteristics. It is very soluble in water and nearly insoluble in methylene chloride. **Repeat the identical procedure** carried out in **Part A** but replace the 600 μL of water used with 600 μL of 10% sodium bicarbonate solution. The efficiency of the basic aqueous extraction procedure can be measured by recovering any unreacted benzoic acid from the organic layer. Obtain the melting point of any recovered residue. Sodium benzoate has a melting point above 300°C, whereas benzoic acid melts near 122°C.

When a carboxylic acid is placed in a solution containing bicarbonate ion, bubbles of carbon dioxide are observed. This reaction may be used as a qualitative test for the presence of a carboxylic acid.

$$CH_3C(=O)—OH + HCO_3^- \rightleftharpoons CH_3C(=O)—O^- + H_2CO_3$$

$$H_2CO_3 \rightleftharpoons CO_2\uparrow + H_2O$$

Test for a Carboxylic Acid

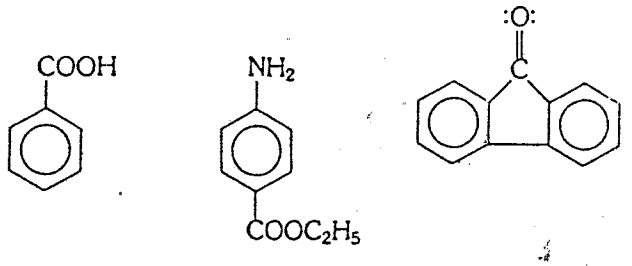
Place 1 mL of 5% sodium or potassium bicarbonate on a small watch glass. Add the pure acid sample, one drop from a Pasteur pipet if the sample is a liquid (~5 mg if a solid), to the bicarbonate solution. Evolution of bubbles of carbon dioxide indicates the presence of an acid.

Perform the preceding test for carboxylic acids on several organic acids such as acetic, benzoic, propanoic, and chloroacetic.

PART C: A Three-Component Mixture: An Example of Separation of an Acid, a Base, and a Neutral Substance by Solvent Extraction

This experiment illustrates a further example of the solvent extraction technique as it is used in the organic laboratory to separate organic acids and bases. As outlined in the discussion section, the solubility characteristics of these important organic compounds in water are dependent on the pH of the solution. The extraction procedure employed for the separation of a mixture of an acid, a base, and a neutral substance takes advantage of this fact.

The components of the mixture to be separated in this experiment are benzoic acid, ethyl 4-aminobenzoate (a base), and 9-fluorenone (a neutral compound prepared in Experiment 40A).



Benzoic acid | Ethyl 4-aminobenzoate | 9-Fluorenone

In carrying out the separation, you should keep a record or flow chart of your procedure (see discussion: separation of acids and bases) in the laboratory notebook and carefully label all flasks.

The estimated time of the reaction is 1.5 hours.

Physical Properties of Reactants and Products

Compound	MW	Wt/Vol	mmol	mp(°C)	bp(°C)	Density
Benzoic acid	122.13	50 mg	0.41	122		
Ethyl 4-aminobenzoate	165.19	50 mg	0.31	89		
9-Fluorenone	180.22	50 mg	0.27	82		
Diethyl ether		4 mL			35	0.71
3 *M* HCl		4 mL				
3 *M* NaOH		4 mL				
6 *M* HCl		—				
6 *M* NaOH		—				

In a stoppered or capped 15-mL centrifuge tube containing 4 mL of diethyl ether are added 50 mg (0.41 mmol) of benzoic acid, 50 mg (0.31 mmol) of ethyl 4-aminobenzoate, and 50 mg (0.27 mmol) of 9-fluorenone. Dissolution of the solids is accomplished by stirring with a glass rod or mixing on a Vortex mixer.

HOOD *The diethyl ether is measured using a 10-mL graduated cylinder. It is dispensed in the* **hood.**

Separation of the Basic Component

Using a calibrated Pasteur pipet, 2 mL of 3 *M* hydrochloric acid are added to the centrifuge tube while cooled in an ice bath, and the resulting two-phase system mixed thoughly for several minutes (a Vortex mixer is excellent for this purpose). After the layers have separated, the bottom aqueous layer is removed,

using a Pasteur filter pipet, and transferred to a **labeled**, 10-mL Erlenmeyer flask.

Note. *A small amount of crystalline material may form at the interface between the layers. The second extraction dissolves this material.*

This step is now repeated with an additional 2 mL of the 3 *M* acid solution and the aqueous layer again transferred to the same Erlenmeyer flask. This flask is now stoppered (capped) and set aside.

Isolation of the Ethyl 4-Aminobenzoate

To the acidic aqueous solution, separated and set aside, add 6 *M* NaOH dropwise until the solution is distinctly alkaline to litmus paper. Cool the flask in an ice bath for about 10–15 minutes. Collect the solid precipitate by reduced-pressure filtration using a Hirsch funnel. Wash the precipitate with two 1-mL portions of distilled water. Dry the material on a clay plate, on filter paper, or in a vacuum drying oven. Weigh your product and calculate the percentage recovery. Obtain a melting point of the dried material and compare your result with the literature value. It is of interest to note that this material is used as a topical anesthetic.

Separation of the Acidic Component

To the remaining ether solution now add 2 mL of 3 *M* NaOH. The system is mixed as before and the aqueous layer is separated and transferred to a **labeled**, 10-mL Erlenmeyer flask.

This step is repeated and the aqueous layer again removed and transferred to the same Erlenmeyer flask. This flask is stoppered and set aside.

Separation of the Neutral Component

After washing with two 1-mL portions of distilled water, to the remaining wet, ether solution in the centrifuge tube add about 250 mg of anhydrous sodium sulfate. Set this mixture aside while working up the other extraction solution. This will allow sufficient time for the solution to dry. If the drying agent clumps, add additional sodium sulfate.

Isolation of the Benzoic Acid

To the aqueous alkaline solution, separated and set aside, add 6 *M* HCl dropwise until the solution is distinctly acidic to litmus paper. Cool the flask in an ice bath for about 10 minutes. Collect the precipitated benzoic acid by reduced-pressure filtration using a Hirsch funnel. Wash the precipitate with two 1-mL portions of distilled water. Dry the product using one of the techniques described earlier for the ethyl 4-aminobenzoate. Weigh the benzoic acid and calculate your percentage recovery. Obtain the melting point of the dried material and compare your result with the literature value. The qualitative test for organic carboxylic acids, given in the discussion section, may also be performed.

Isolation of the 9-Fluorenone

Transfer the dried ether solution by use of a Pasteur filter pipet to a tared 10-mL Erlenmeyer flask containing a boiling stone. Rinse the drying agent with an additional 1 mL of ether and also transfer this rinse to the same Erlenmeyer flask.

HOOD Concentrate the solution on a warm sand bath in the **hood** using a **slow** stream of nitrogen gas. Obtain the weight of the isolated 9-fluorenone and calculate the percentage recovery. Obtain a melting point of the material and compare your result with the literature value.

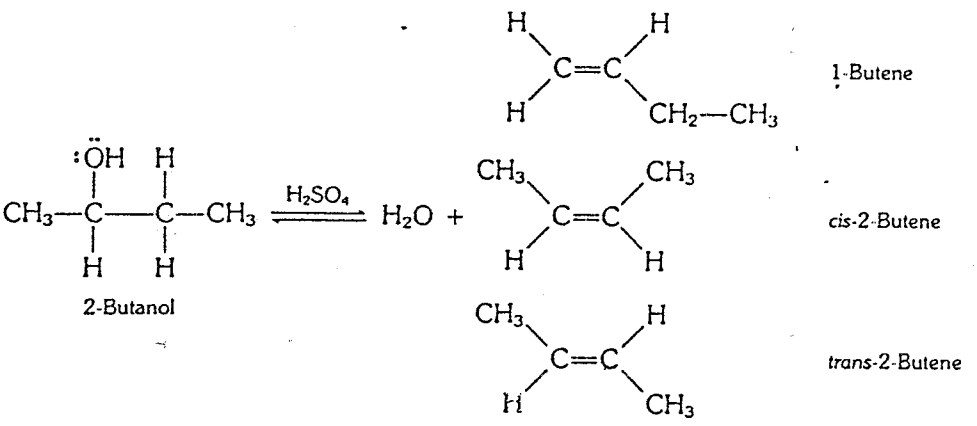
TECHNIQUE 9: PREPARATION AND ANALYSIS OF A GASEOUS PRODUCT

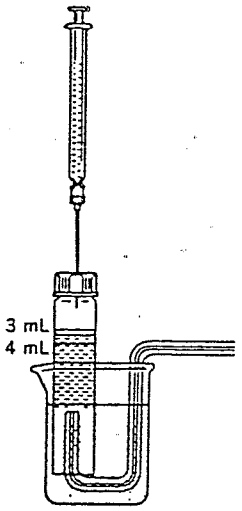
Experiment 9

The Dehydration of a Secondary Alcohol, 2-Butanol: 1-Butene; *trans*-2-Butene; *cis*-2-Butene

(1-butene; 2-butene, *E*-; 2-butene, *Z*-)

This experiment illustrates the acid-catalyzed elimination reaction of the secondary (2°) alcohol 2-butanol. The gaseous products formed in the reaction are separated and analyzed by the use of gas chromatography.





Isolation of Product

Collect about 3–4 mL of gas in the collection reservoir and then, using a hypodermic syringe, withdraw a 0.5 mL sample through the rubber septum for GC analysis.(■)

Remove the delivery tube from the collecting reservoir and then from the water before discontinuing the heat on the reaction vial. This order prevents water from being sucked back into the reaction flask.

Purification and Characterization

The collected gas is analyzed by gas chromatography without further purification.

Gas Chromatography Conditions

Column: ¼ in. × 8 ft packed with 20% silicone DC-710
Room temperature
Flow rate (He gas): 20 mL/minute
Sample size: 0.5 mL of collected gas.

Assuming that the amount of each substance in the gas is proportional to the area of its corresponding peak, determine the percentage ratio of the three components in the gas sample.

Area Under a Curve. *Several techniques may be used. The following method gives reproducible results of ±3–4%: peak height (mm) × width at half-height (mm), measured from the baseline of the curve.*

The order of elution of the butenes is 1-butene, *trans*-2-butene, and *cis*-2-butene. *If the reaction mixture is heated strongly, rearrangement can occur and isobutene is also formed.*

ENVIRONMENTAL DATA

Substance	Amount	TLV (mg/m^3)	Emissions (mg)	Volume (m^3)
2-Butanol	100 μL	305	10	<0.1
Sulfuric acid	50 μL	1		

Losses unknown, but assumed small because of gas trapping

QUESTIONS

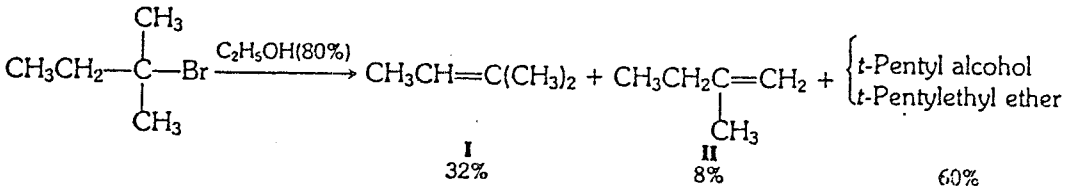
5-51. Gas chromatographic analysis of a mixture of organic compounds gave the following peak areas (cm^2): hexane, 2.7, heptane, 1.6; hexanol, 1.8; toluene, 0.5.

a. Calculate the mole percent composition of the mixture. Assume that the response of the detector (area/mole) is the same for each component.

b. Calculate the weight percent composition of the mixture under the same assumptions used in a.

5-52. It is noted at the end of the experiment that if the mixture is heated strongly, rearrangement can occur and isobutene is also formed. Suggest a mechanism to account for the formation of this compound.

5-53. When *t*-pentyl bromide is treated with 80% ethanol, the following amounts of olefinic products are detected on analysis.

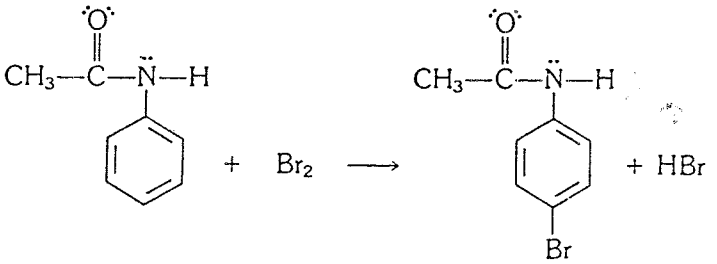


Explain why compound **I** is formed in far greater amount than the terminal alkene.

Experiment 33 Halogenation: 4-Bromoacetanilide

(acetamide, *N*-(4-bromophenyl)-)

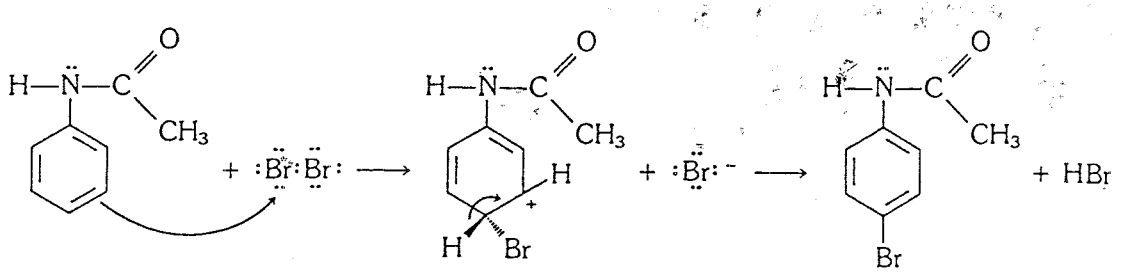
In this experiment an electrophilic aromatic substitution is described. This type of reaction constitutes a very common route by which many aromatic compounds are prepared.



Acetanilide Bromine 4-Bromoacetanilide

DISCUSSION Aromatic compounds may be brominated by treatment with bromine in the presence of a Lewis acid catalyst such as ferric chloride. For very active substrates, such as amines, the reaction may proceed in the absence of a catalyst. In many cases, with amines or phenols, it is impossible to stop the bromination and all possible ortho and para positions are substituted. For this reason, primary aromatic amines are often converted to the corresponding acetanilide derivative, if the monosubstituted compound is desired. This effect is demonstrated in the present experiment. Furthermore, acetylation of the primary amine, aniline, effectively blocks the ortho positions due to steric hindrance. Electrophilic substitution by bromine is thus directed to the para position on the ring. The —$NHCOCH_3$ group is a less powerful ortho, para directing group than —NH_2 due to the presence of the carbonyl. Thus, substitution of the amino group renders the ring less nucleophilic. For these reasons only monosubstitution is observed.

The mechanism of the reaction is an illustration of the classic electrophilic substitution sequence on an aromatic ring. The mechanism shown below is presented as proceeding without the aid of a catalyst.



EXPERIMENTAL Estimated time for completion of the experiment: 1.5 hours.

Physical Properties of Reactants and Products

Compound	MW	Wt/Vol	mmol	mp(°C)	bp(°C)
Acetanilide	135.17	25 mg	0.19	114	
Glacial acetic acid	60.05	4 drops			118
Br_2–acetic acid reagent		3 drops			
4-Bromoacetanilide	214.08			168	

Reagents and Equipment

In a 3.0-mL conical vial fitted with a cap is placed 25 mg (0.19 mmol) of acetanilide to which are added 4 drops of glacial acetic acid using a medicine dropper. Stirring with a glass rod may be necessary to help dissolve the acetanilide. To the clear solution are added 3 drops of bromine–acetic acid reagent

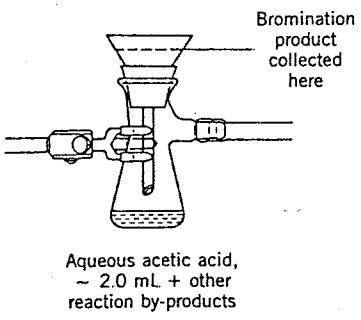
HOOD **[hood]**. The vial is immediately capped.

HOOD

> **WARNING:** ***Bromine is a severe irritant. It is suggested that plastic gloves be worn since Br_2 burns require extended periods of time to heal. Dispense only in the hood. The reagent is prepared by mixing 2.5 mL of liquid bromine with 5.0 mL of glacial acetic acid.***

Reaction Conditions

The reddish-brown solution is allowed to stand at room temperature for 10 minutes with intermittent shaking. During this period yellow-orange crystals deposit from the solution.



Isolation of Product

Add 0.5 mL of water (calibrated Pasteur pipet) to the reaction mixture with swirling, followed by 5 drops of aqueous sodium bisulfite solution (33%). This treatment discharges the residual color, due to the presence of unreacted bromine, and results in the formation of white crystals. The reaction mixture is cooled in an ice bath for 10 minutes to maximize the product yield.

The white crystals of 4-bromoacetanilide are collected by vacuum filtration using a Hirsch funnel.(■) The filter cake is washed with three 0.25-mL portions of cold water (calibrated Pasteur pipet) and dried by drawing air through the crystals under reduced pressure for approximately 5 minutes.

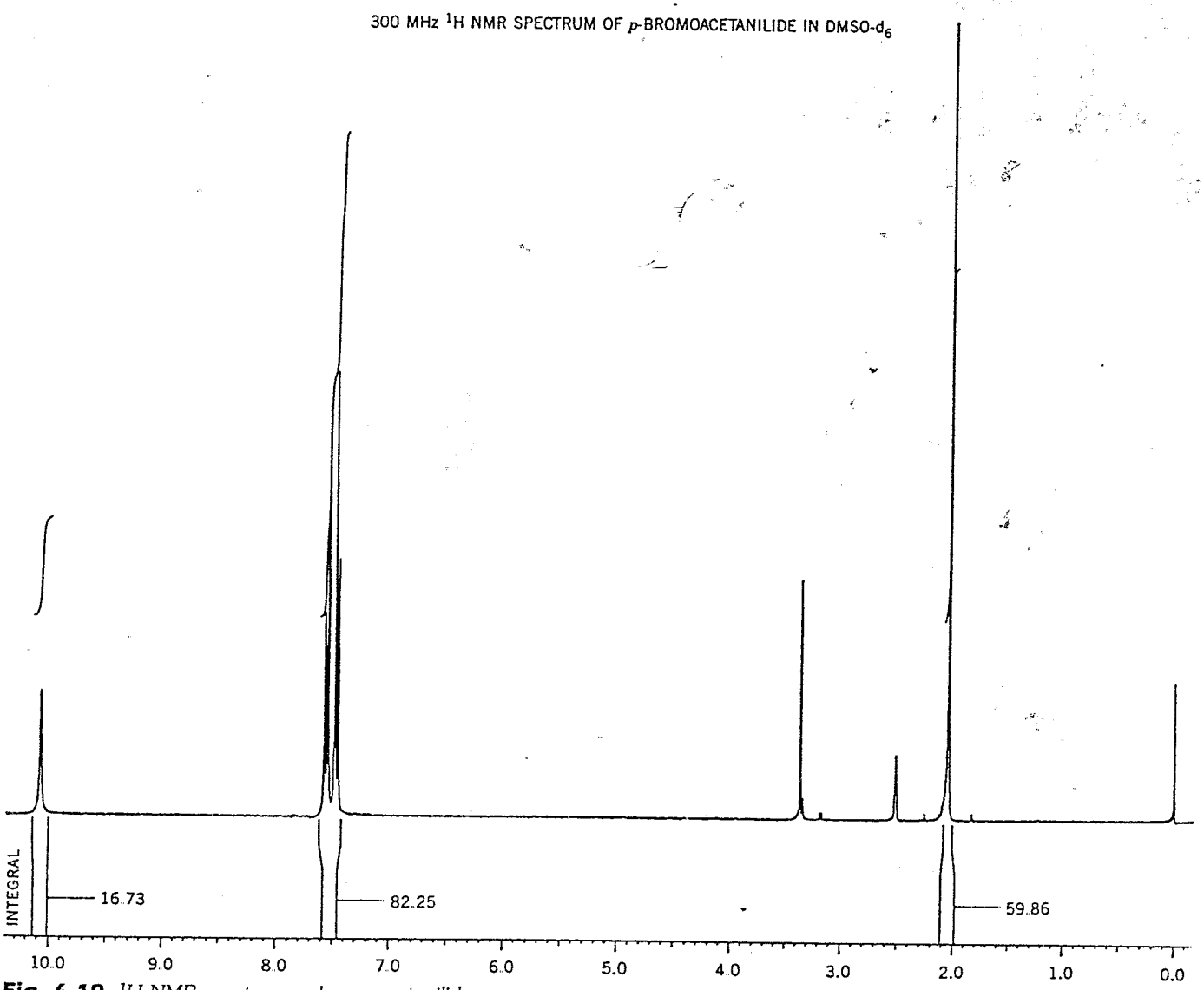


Fig. 6.19 *^{1}H-NMR spectrum: p-bromoacetanilide.*

Isolation and Characterization

The crude 4-bromoacetanilide is purified by recrystallization from 95% ethanol using the Craig tube.

Weigh the dried product and calculate the percentage yield. Determine the melting point and compare your result with the literature value.

Obtain an IR spectrum of the material and compare it with that recorded in the literature.

Nuclear Magnetic Resonance Analysis

Figures 6.19 and 6.20 are the ^{1}H and ^{13}C NMR spectra of *p*-bromoacetanilide in DMSO-d_6. These can be used to compare with NMR spectra you may obtain on your product.

In the ^{13}C spectrum, the DMSO-d_6 appears as a septet at 39.7 ppm. The resonance from the methyl group occurs at 24 ppm and the amide carbonyl carbon resonates at 169 ppm. The carbons of the benzene ring are observed between 110 and 140 ppm.

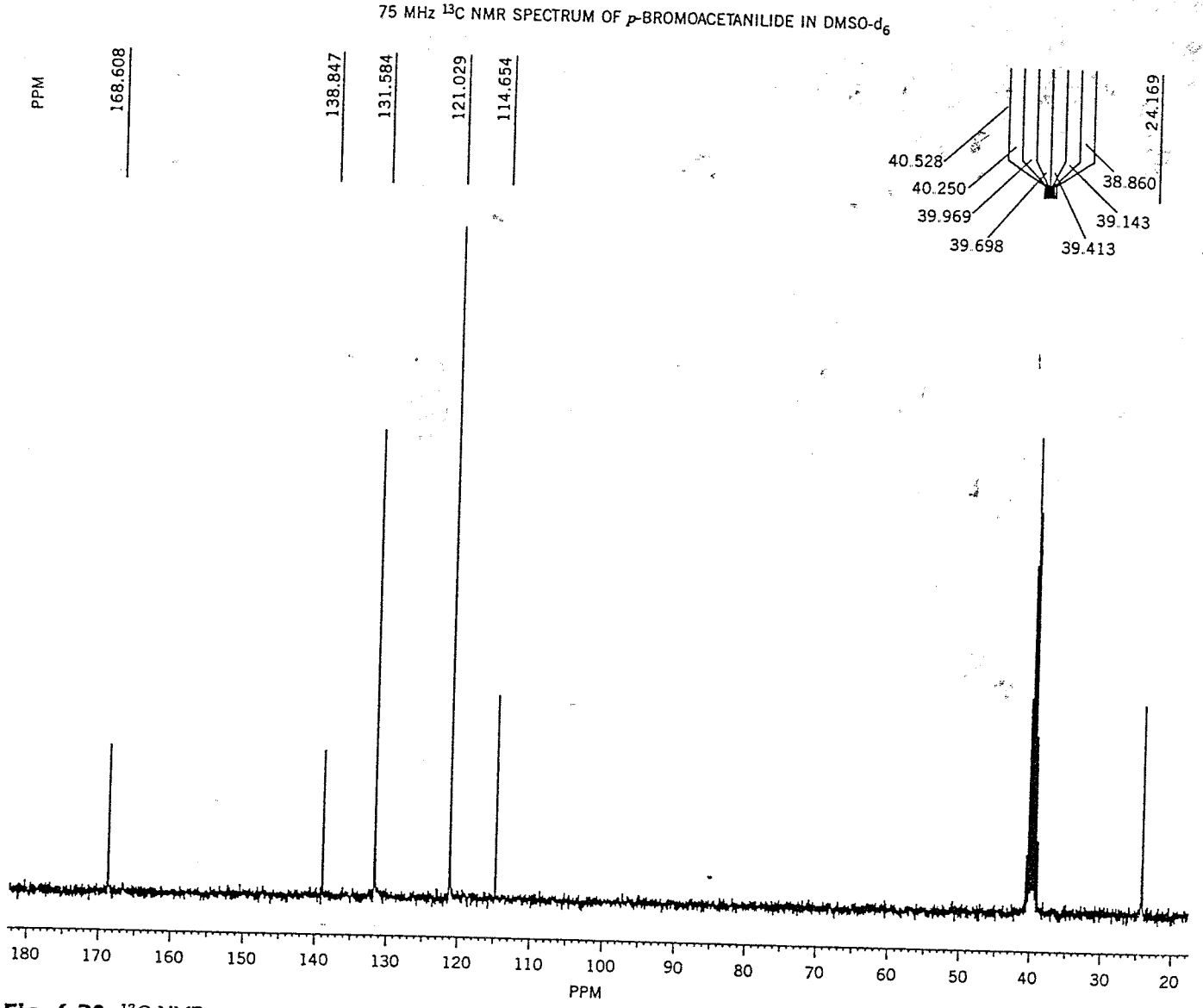


Fig. 6.20 *^{13}C-NMR spectrum:* p-*bromoacetanilide.*

In the ^{1}H spectrum, the peak from trace amounts of DMSO-d_5 is seen at about 2.6 ppm. The peak at 3.4 ppm is probably due to water or another impurity in the sample. Note the two small peaks located equidistant to the tall singlet near 2.0 ppm. The small "satellite" peaks are the result of the 1.1% of the methyl groups that have ^{13}C instead of ^{12}C, and are thus coupled to the carbon to which the protons are attached. The amide NH proton, which is probably hydrogen bound to the basic (Lewis) sulfoxide functionality in DMSO-d_6, occurs rather downfield, near 10.1 ppm. This chemical shift may vary from your sample due to subtle differences in concentration, temperature, and moisture content of your DMSO-d_6.

Chemical Tests

Chemical classification tests may also be performed on the amide product. The ignition and the Beilstein test (Chapter 7) are used to confirm the presence of the aromatic ring and the halogen group, respectively. Does the hydroxamate test for amides (Chapter 7) give a positive result?

Chapter 6F
Chemistry of the Group 15 (VA) Elements

Introduction

Nitrogen is an element which forms few stable compounds. Most nitrogen compounds are thermodynamically unstable with respect to nitrogen gas, due to the extremely strong homonuclear triple bond. The more electronegative elements might be expected to form stable compounds by oxidizing nitrogen to various positive oxidation states. In general, however, such products are also not stable, as bond lengths tend to be short, and such elements have lone pairs of electrons, as does nitrogen in its singly bonded compounds. The resulting lone pair-lone pair repulsions weaken the N-N, N-O, or N-X bonds, making such products quite reactive. The most stable nitrogen compounds, therefore, are with elements having no lone pairs of electrons, most notably hydrogen, carbon, and boron.

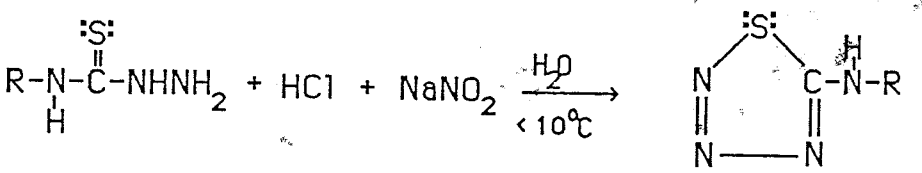
The heavier members of the group form more stable compounds, as the products are thermodynamically stable with respect to the elements. Further, the heavier elements also form longer bonds, lessening lone pair repulsions when present. Finally, the heavier elements posess empty low energy **d** orbitals, into which lone pair electron density can be diffused, lessening repulsions further.

Experiment 12
Preparation of 5-Anilino- and 5-amino-1,2,3,4-thiatriazole

Introduction

The thiatriazole ring is an example of an inorganic heterocyclic ring system, containing nitrogen, sulfur and carbon atoms. It was first prepared in 1896[1]. The system is thermally unstable; the compounds synthesized in this experiment undergoing vigorous decomposition when heated. Extensive studies have been carried out on the catalyzed and uncatalyzed decomposition of these materials[2]. This thermal instability has led to the use of several of these substituted compounds as blowing agents to generate foamed materials[3]. They have also been used as one of the ingredients in corrosion inhibiting formulations for copper and ferrous metals[4] and in antihypertension compositions[5]. An additional use is in the formulation of materials for document reproduction[6].

The heterocyclic ring is formed by the diazotization of the corresponding thiosemicarbazide:



R= H, C_6H_5

Diazotization is a well known oxidation reactions in organic chemistry[7]. It is used to prepare diazonium salts of alkane and arene primary amines. They are key intermediates in the preparation of a wide variety of materials.

Part A: Preparation of 5-Anilino-1,2,3,4-thiatriazole

Safety Recommendations

4-Phenyl-3-thiosemicarbazide [CAS# 5351-69-9]: The compound has not been extensively investigated, however, thiosemicarbazide is extremely toxic. It would be prudent to follow strict safety procedures, and handle this material **only** with gloves. IPR-MUS LD50: 15 mg/kg.

Sodium Nitrite [CAS# 7632-00-0]: Sodium nitrite is harmful if swallowed, inhaled, or absorbed through the skin. It has been shown to have effects on fertility and embryo or fetal development. It should be handled with care, wearing gloves. ORL-HMN LDLO: 71 mg/kg, ORL-RAT LD50: 85 mg/kg.

Chemical Data

Compound	FW	Amount	mmol	mp(°C)
4-phenyl-3-thiosemicarbazide	167.23	167 mg	1.0	138
$NaNO_2$	69.00	69 mg	1.0	271

Time Required for Experiment: 1 hour

Experimental Procedure

Prepare a suspension of 167 mg (1 mmol) of 4-phenyl-3-thiosemicarbazide in 760 µL of 15% hydrochloric acid in a 10 mL round-bottom flask containing a magnetic stir bar. Place the flask in an an ice-salt bath, prepared in a 7.5 cm evaporating dish, set on a magnetic stirring hot plate [see Figure 6F.1]. The bath temperature is monitored using a thermometer and the contents of the flask cooled to 5-10°C with stirring.

Prepare a solution of sodium nitrite in a small test tube or 10 mL Erlenmeyer flask by dissolving 69 mg (1 mmol) $NaNO_2$ in 150 µL water. Add approximately 130 µL of this nitrite solution, dropwise, using a Pasteur pipet, to the stirred reacton mixture. The addition should be made over a 8-12 minute time span, such that the temperature of the mixture is not allowed to exceed 10°C.

NOTE: *A rapid addition of the nitrite solution will result in the decomposition of the diazonium salt intermediate, resulting in a marked decrease in product formation.*

Isolation of Product

When the addition is complete, collect the white precipitate that has formed by suction filtration using a Hirsch funnel. Save the precipitate; it is the product.

Return the filtrate to the reaction flask using a Pasteur pipet, and add the remaining sodium nitrite solution, dropwise. Addition is stopped when the reaction mixture becomes pale yellow (any additional nitrite and the solution becomes intense yellow). If further precipitation of product is observed, collect the product as before. Wash the combined filter cakes of the off-white 5-anilino-1,2,3,4-thiatriazole with three 0.5 mL portions of ice-cold distilled water, and then dry the product on a clay plate or on filter paper. The product may be further dried in a desiccator under vacuum over t.h.e. SiO_2 desiccant. The average yield of product is 142 mg (86 %).

Purification of Product

The compound (50 mg) may be recrystallized from methanol using a Craig tube to yield beautiful colorless needles. Dry the crystals on a clay plate or on filter paper.

Characterization of Product

Obtain the decomposition point for the product in the same manner as a melting point. The pure material decomposes vigorously at 142-143°C. Obtain the IR spectrum of the product as a KBr pellet and compare your spectral data with that reported in the literature[8].

References

1. Lieber, E.; Pillai, C.N.; Oftedahl, E.; Hites, R.D. *Inorganic Syntheses* Wiley: New York, 1960, Vol. 6, p.44 and references therein.
2. For example see Holm, A.; Calsen, L.; Larsen, E. *J. Org. Chem.* **1978,***43,* 4816.
3. Farbenfabriken Bayer Akt. -Ges. *Brit. Pat.* 861,056 (Feb. 1961); Schmidt, K.L.; Lober, F.; Muller, E.; Scheurlen, H. *Ger. Pat.* 1,1
4. Liddell, R.W. *U.S. Pat.* 2,877,188 (Mar. 1959).
5. Schoepke, H.G.; Swett, L.R. *U.S. Pat.* 3,265,576 (1966); also see *Brit. Pat.* 1,033,519 (1966).
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MICROSCALE LAWS

Rules of the Trade for Handling Organic Materials at the Microscale Level

Now that we have briefly looked at the equipment we will be using to carry out the microscale organic reactions, let us examine specific techniques used to deal with the small quantities of material involved. These reactions by our definition start with 15 to 150 mg of the limiting reagent. These quantities sound small, and they are. Although 150 mg of a light powdery material will fill half of a 1-mL conical vial, you will have a hard time observing 15 mg of a clear liquid in the same container even with magnification. On the other hand, this volume of liquid is reasonably easy to observe when placed in a 0.1-mL conical vial. A vital part of the game of working with small amounts of materials is to become familiar with microscale techniques and to practice them as much as possible in the laboratory.

Rules for Working with Liquids at the Microscale Level

1. *Liquids are never poured at the microscale level.* Liquid substances are transferred by pipet or syringe. As we are working with small easy-to-hold glassware at the microscale level, the best technique for transfer is to hold both containers with the fingers of one hand, with the mouths as close together as possible. The free hand is then used to operate the pipet (syringe) to withdraw the liquid and make the transfer. This approach reduces to a minimum the time that the open tip is not in or over the reservoir or the reaction flask. We employ three different pipets and two standard syringes for most experiments in which liquids are involved. This equipment is a prime source of contamination. *Be very careful to thoroughly clean the equipment after each use.*

a. *Pasteur pipet.* Often called a capillary pipet, the Pasteur pipet is a simple glass tube with the end drawn to a fine capillary. These pipets can hold several milliliters of liquid (Fig. 3.26*a*) and are filled using a small rubber bulb or one of the very handy commercially available pipet pumps. You will do many transfers using the Pasteur pipet. It is suggested that you calibrate several of them for approximate delivery of 0.5, 1.0, 1.5, and 2.0 mL of liquid. This is easily done by drawing the measured amount of a liquid from a 10-mL graduated cylinder and marking the level of the liquid in the pipet. This can be done with transparent tape or by scratching with a file. Indicate the level with a marking pen before trying to tape or file the pipet.

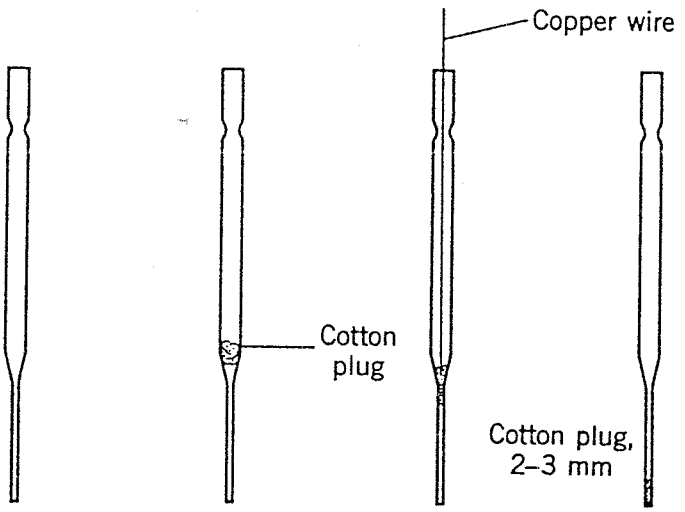


Fig. 3.26 *Preparation of Pasteur filter pipet.*

b. *Pasteur filter pipet.* A very handy adaptation of the Pasteur pipet is a filter pipet. This pipet is constructed by taking a small cotton ball and placing it in the large open end of the standard Pasteur pipet. Hold the pipet vertically and tap gently to position the cotton ball in the drawn section of the tube (Fig. 3.26*b*). Now form a plug in the capillary section by pushing the cotton ball down the pipet with a piece of copper wire (Fig. 3.26*c*). Finish by seating the plug flush with the end of the capillary (Fig. 3.26*d*). The optimum-size plug will allow easy movement along the capillary while it is being positioned by the copper wire. Compression of the cotton will build enough pressure against the walls of the capillary (once the plug is in position) to prevent plug slippage while the pipet is filled with liquid. If the ball is too big, it will wedge in the capillary before the end is reached, and wall pressure will be so great that liquid flow will be shut off. Even some plugs that are loose enough to be positioned at the end of the capillary still will have developed sufficient lateral pressure to make the filling rate unacceptably slow. With a little practice, however, these plugs can be quickly and easily inserted. Once in place the plug is rinsed with 1 mL of methanol and 1 mL of hexane and dried before use.

The purpose of placing the cotton plug in the pipet is twofold: First, a particular problem with the transfer of volatile liquids via the standard Pasteur pipet is the rapid buildup of back-pressure from solvent vapors in the rubber bulb. This pressure quickly tends to force the liquid back out of the pipet, and can cause valuable product to drip on the bench top. The cotton plug tends to resist this back-pressure and allows much easier control of the solution once it is in the pipet. The time delay factor becomes particularly important when the Pasteur filter pipet is employed as a microseparatory funnel (see the discussion on extraction techniques, Experiment 4).

Second, each time a transfer of material is made, the material is automatically filtered. This process effectively removes dust and lint, which are a constant problem when working at the microscale level with unfiltered room air.

c. *Automatic pipet (considered the Cadillac of pipets).* Automatic pipets quickly, safely, and reproducibly measure and dispense specific volumes of liquids. They are particularly valuable at the microscale level, as they generate the precise, accurate liquid measurements that are absolutely necessary when handling microliter volumes of reagent. The automatic pipet adds considerable insurance for the success of an experiment, as any liquid can be efficiently measured, transferred, and delivered to the reaction flask. They become almost an essential instrument in laboratory sections with large numbers of students.

The automatic pipet system consists of a calibrated piston pipet with a specially designed disposable plastic tip. You may encounter any one of three pipet styles: single volume, multirange, continuously adjustable (see Fig. 3.27). The first type is calibrated to deliver only a single volume. The second type is adjustable to two or three predetermined delivery volumes. The third type is the most versatile and can be user set to deliver any volume within the range of the pipet. Obviously, the price of these valuable laboratory tools goes up with increasing attributes. They are expensive and their use must be shared in the laboratory. Treat them with respect!

The automatic pipet is designed so that the liquid comes in contact with the special tip only. Never load the pipet without the tip in place. Never immerse the tip completely in the liquid that is being pipetted. Always keep the pipet vertical when the tip is attached. Follow these three rules and most automatic pipets will give many years of reliable service. The following suggestions are a few general rules for improving reproducibility with an automatic pipet.

Try to effect the same uptake and delivery motion for all samples. Smooth depression and release of the piston will give the most consistent results. Never allow the piston to snap back.

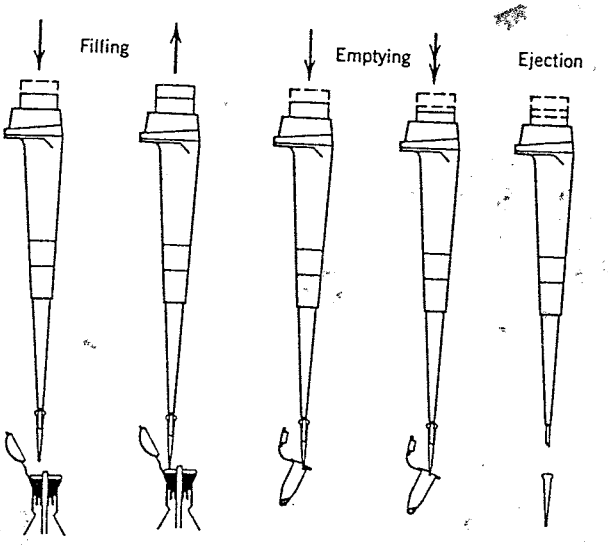


Fig. 3.27 *Operation of automatic delivery pipet.* (Courtesy of Brinkmann Instruments Co., Westbury, N.Y.)

Always depress the piston to the first stop before inserting the tip into the liquid. If the piston is depressed after submersion, formation of an air bubble in the tip becomes likely. Bubble formation will result in a filling error.

Never insert the tip more than 5 mm into the liquid. It is good practice not to allow the body of the pipet to contact any surface or bottleneck that might be wet with a corrosive chemical.

If an air bubble forms in the tip during uptake, return the fluid, discard the tip, and repeat the sampling process.

d. *Syringes.* Syringes are particularly helpful pieces of equipment when transferring liquid reagents or solutions to sealed reaction systems from sealed reagent or solvent reservoirs. They can be inserted through a septum, which avoids opening the apparatus to the atmosphere (see Experiment 13). They are also routinely employed in the determination of ultramicro boiling points (10-μL GC syringe). It is critically important to clean the syringe needle after each use. Effective cleaning of a syringe requires as many as a dozen flushes. The microscale laboratory utilizes a low-cost glass 1-mL insulin syringe in which the rubber plunger seal is replaced with a Teflon seal. For gas chromatographic separation work, the standard 50- or 100-μL syringes are preferred (see Experiment 1).

2. *Liquid volumes may be converted easily to weight measure by the following relationship:*

$$\text{Volume (mL)} = \frac{\text{weight (g)}}{\text{density (g/mL)}}$$

3. *Work with liquids in conical vials* and work in vials that are approximately double the volume of the material. The trick here is to reduce the surface area of the flask in contact with the sample to an absolute minimum. Conical systems are far superior to the spherical surface of the conventional round-bottom flask.

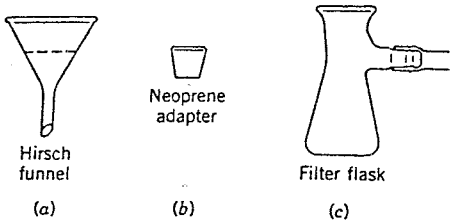


Fig. 5.39 *Component parts for vacuum filtration.*

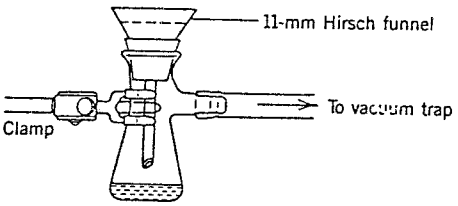


Fig. 5.40 *Vacuum filtration apparatus.*

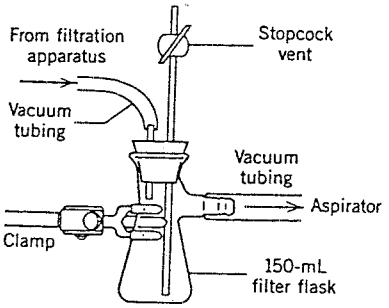


Fig. 5.41 *Vacuum trap.*

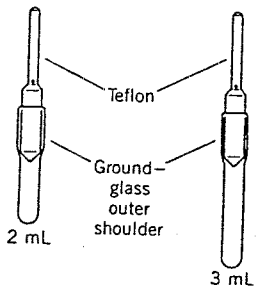


Fig. 5.42 *Craig tubes.*

Filtration Techniques

Use of the Hirsch Funnel

The standard filtration system for collecting products purified by recrystallization in the microscale laboratory is vacuum filtration with an 11-mm Hirsch funnel. In addition, many reaction products that do not require the crystallization step are collected directly by this technique. The funnel is shown in Figure 5.39*a*.

The Hirsch filter funnel is composed of a ceramic cone with a circular flat bed perforated with small holes. The diameter of the bed is 11 mm, and in operation it is covered by a flat piece of filter paper of the same diameter. The funnel is sealed into a filter flask with a Neoprene adapter (see Fig. 5.39*b*).

The filter flask, which is heavy-walled and especially designed to operate under vacuum, is constructed with a side arm (they are often called "side-arm pressure flasks"; see Fig. 5.39*c*).

The side arm is connected with heavy-walled rubber vacuum tubing to a water aspirator or water pump. When water is running through the aspirator, a partial vacuum is formed, which creates a flow of air down the vacuum tubing from the filter flask. With the rubber adapter in place, the entering air is forced through the filter paper, which is held flat by suction. The mother liquors of the crystallization are rapidly forced into the filter flask, while the crystals retained by the filter are quickly dried by the stream of air passing through them (Fig. 5.40).

When you are using a water pump, it is very important to have a safety trap mounted in the vacuum line leading from the filter flask. Any drop in water pressure (easily created by one or two other students on the same water line turning on their aspirators at the same time) can result in the backup of water into the system as the flow through the aspirator decreases (see Fig. 5.41).

Craig Tube Crystallizations

The Craig tube is commonly used for microscale crystallizations in the range of 10–100 mg of material (see Fig. 5.42). The process consists of the following steps.

1. The sample is placed in a small test tube (10 × 75 mm).
2. The solvent (0.5–2 mL) of choice is added, and the sample dissolved by heating in the sand bath. Rapid stirring with a microspatula (roll the spatula rod between your fingers) greatly aids the dissolution and protects against boilover. A modest excess of solvent is added after the sample is completely dissolved. It will be easy to remove this excess at a later stage, as the volumes involved are very small. The additional solvent ensures that the solute will stay in solution during the hot transfer.
3. The heated solution is transferred to the Craig tube by Pasteur filter pipet (the pipet is preheated with hot solvent). This transfer automatically filters the solution (if decolorizing charcoal powder has been added, two filtrations by

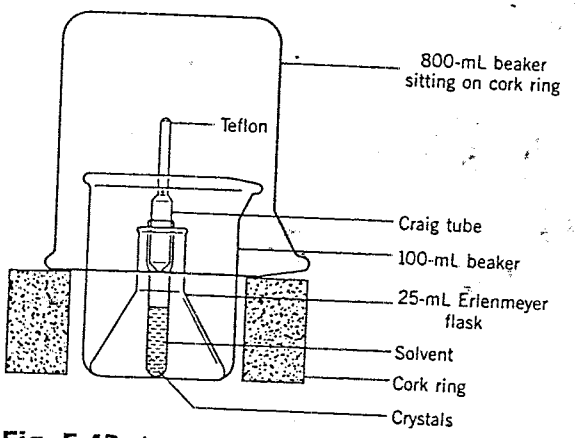


Fig. 5.43 *Apparatus for slow crystallization.*

the pipet may be required. The second filtration is almost always avoided by the use of charcoal pellets.)

4. The hot filtered solution is then concentrated to saturation by gentle boiling in the sand bath. Constant agitation of the solution with a microspatula during this short period will avoid the use of a boiling stone and guarantee that a boilover will not occur. The ready crystallization of product on the microspatula just above the solvent surface serves as a good indication that saturation is close at hand.
5. The upper section of the Craig tube is set in place, and the system allowed to cool in a safe place. As cooling commences, seed crystals, if necessary, may be added by crushing them against the side of the Craig tube with a microspatula just above the solvent line. A good routine, if the time is available, is to place the assembly in a small Erlenmeyer, then place the Erlenmeyer in a beaker, and finally cover the first beaker with a second inverted beaker. This will ensure slow cooling, which will enhance good crystal growth (Fig. 5.43). A Dewar flask may be used when very slow cooling and large crystal growth are required (as in X-ray crystallography).
6. After the system reaches room temperature, cooling in an ice bath will further improve the yield.
7. Solvent is now removed by inverting the Craig tube assembly into a centrifuge tube and spinning the mother liquors away from the crystals (Fig. 5.44). This operation takes the place of the usual filtration step in simple crystallizations. It avoids another transfer of material and also avoids product contact with filter paper.
8. After removal from the centrifuge, the Craig tube is disassembled and any crystalline product clinging to the upper section is scraped into the lower section. If the lower section is tared it can be left to air dry to constant weight or placed in a warm vacuum oven (wrap a piece of filter paper over the open end secured by a rubber band to prevent dust from collecting on the product while drying). The yield can then be directly calculated.

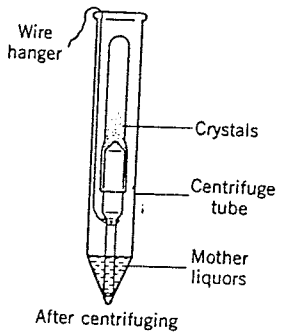


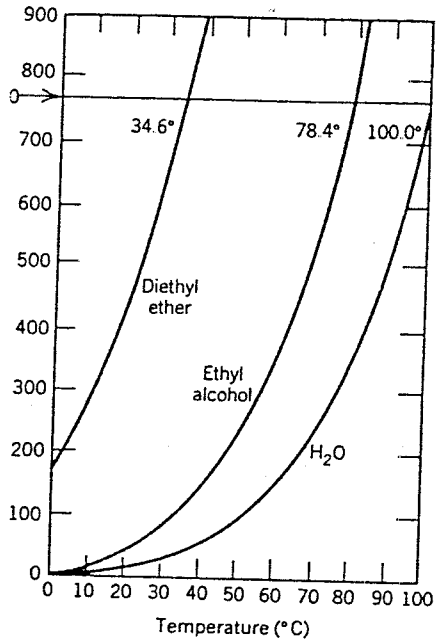
Fig. 5.44 *Crystal collection with a Craig tube.*

The cardinal rule in carrying out the purification of small quantities of solids is *keep the transfers to an absolute minimum!*. The Craig tube is very helpful in this regard.

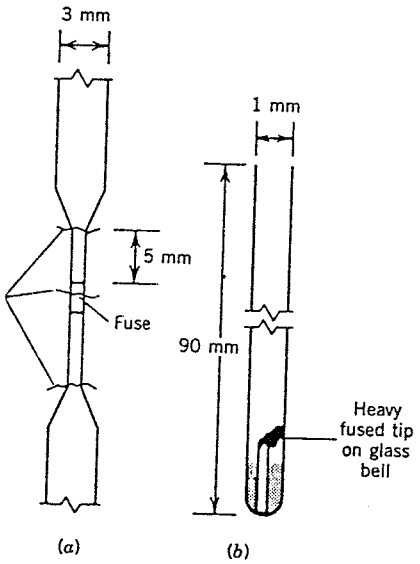
The preceding routine will maximize the crystallization yield. If time is important, the process can be shortened considerably. Shortcuts, however, invariably lead to a corresponding drop in yield.

In the following experiment the techniques of simple crystallization, vacuum filtration, and Craig tube recrystallization are introduced.

ULTRAMICRO BOILING POINT



g. 4.1 *Vapor–pressure curves.* From ady, J. E.; Humiston, G. E. *General emistry*, 3rd ed.; Wiley: New York, 1982. eprinted by permission of John Wiley & ns, New York.)



'. 4.2 *(a) Preparation of small glass bell ultramicro boiling point determination.* n Mayo, D. W.; Pike, R. M.; Butcher, S.; Meredith, M. L. *J. Chem. Educ.* **85,** *62,* 1114. *(b) Ultramicro boiling t assembly.* From Mayo, D. W.; Pike, M.; Butcher, S. S.; Meredith, M. L. *J. m. Educ.* **1985,** *62,* 1114.

The vapor pressure of a liquid increases in a nonlinear fashion on heating. When the pressure reaches the point where it matches the local atmospheric or applied pressure, the liquid boils. That is, internally it spontaneously begins to form large vapor bubbles which rapidly rise to the surface. If heating is continued, both the vapor pressure and the temperature of the liquid will remain constant until the substance has been completely vaporized (Fig. 4.1).

Since microscale preparations generally yield quantities of liquid products in the range 30–70 μL, the allocation of 5 μL or less to boiling point measurements becomes highly desirable. The modification of the Wiegand ultramicro boiling point procedure described here has established that reproducible and reasonably accurate (±1°C) boiling points can be observed on 3–4 μL of many liquids.[1]

Procedure

Ultramicro boiling points can be conveniently determined in standard (90-mm length) Pyrex glass capillary melting point tubes. The melting point tube replaces the conventional 3- to 4-mm (o.d.) tubing used in the Siwoloboff[2] procedure. The sample (3–4 μL) is loaded into the melting point capillary via a 10-μL syringe and centrifuged to the bottom.

A small glass bell (which replaces the conventional melting point tube as the bubble generator in micro boiling point determinations) is formed by heating 3-mm (o.d.) Pyrex tubing with a microburner and drawing it out to a diameter small enough to be readily accepted by the melting point capillary. A section of the drawn capillary is fused and then cut to yield two small glass bells approximately 5 mm long (Fig. 4.2*a*). It is important that the fused section be reasonably large. This section is more than just a seal. The fused glass must add sufficient weight to the bell so that it will firmly seat itself in the bottom of the melting point tube.

One of the glass bells is inserted into the loaded melting point capillary, open end first (down), and allowed to fall to the bottom. The assembled system (Fig. 4.2*b*) is then inserted into the stage of a Thomas–Hoover Uni-Melt Capillary Melting Point Apparatus[3] or similar system (Fig. 4.3).

The temperature is rapidly raised to 15–20°C below the expected boiling point (the temperature should be monitored carefully in the case of unknown substances) and then adjusted to a 2°/minute rise rate until a fine stream of bubbles is emitted from the glass bell. The heat control is then adjusted to drop the temperature. The boiling point is taken at the point where the last escaping bubble collapses (i.e., when the vapor pressure of the substance equals the atmospheric pressure). The heater is then rapidly adjusted to again raise the temperature at 2°/minute and induce a second stream of bubbles. This procedure may then be repeated several times. *It should be emphasized that the precise and sensitive temperature control provided by the Thomas–Hoover system is essential to the successful application of this cycling technique. Although this control is a desirable feature it is not, however, essential for obtaining satisfactory boiling point data.*

[1] Wiegand, C. *Angew. Chem.* **1955,** *67,* 77. Mayo, D. W.; Pike, R. M.; Butcher, S. S.; Meredith, M. L. *J. Chem. Educ.* **1985,** *62,* 1114.
[2] Siwoloboff, A. *Ber.* **1886,** *19,* 795.
[3] Thomas Scientific, 99 High Hill Road, P.O. Box 99, Swedesboro, NJ 08085.

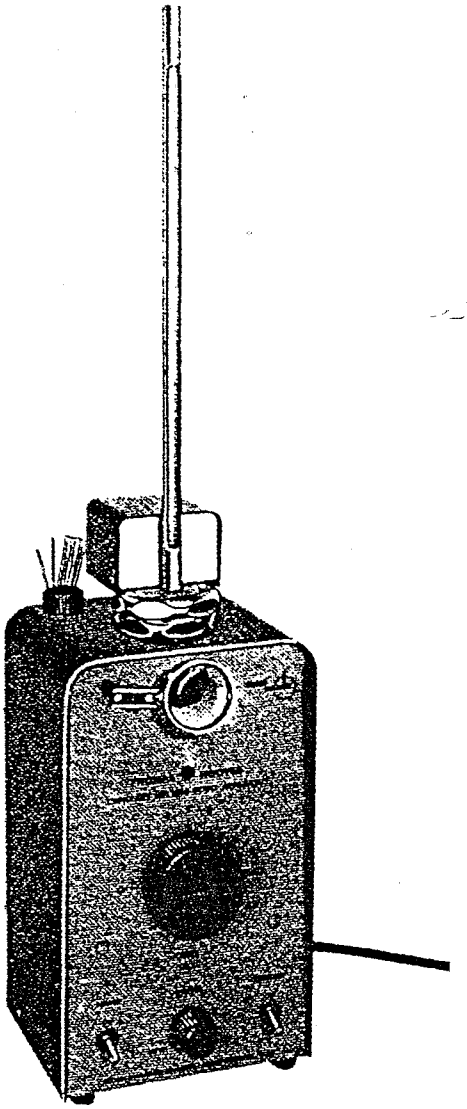


Fig. 4.3 *Thomas–Hoover melting point determination device.* (Courtesy of Arthur Thomas Scientific, Swedesboro, NJ.)

Utilization of the conventional melting point capillary as the "boiler" tube has the particular advantage that the system is ideally suited for observation in a conventional melting point apparatus. The illumination and magnification available make the observation of rate changes in the bubble stream readily apparent. Economical gas chromatographic syringes (10 μL) appear to be the most successful instrument for dealing with the small quantities of liquids involved in these transfers. The 3-in. needles normally supplied with the 10-μL barrels will not reach the bottom of the capillary; however, liquid samples deposited on the walls of the tube are easily and efficiently moved to the bottom by centrifugation. After the sample is packed in the bottom of the capillary tube, the glass bell is introduced. Use of the glass bell is necessary because, if a conventional Siwoloboff fused capillary insert is employed (it would extend beyond the top of the melting point tube), capillary action between the "boiler" tube wall and the capillary insert would draw the majority of the sample from the bottom of the tube up onto the walls. This effect often precludes the formation of the requisite bubble stream.

Little loss of low-boiling liquids occurs (see Table 4.1). Furthermore, if the boiling point is overrun, and the sample is flashed from the bottom section of the "boiler" capillary, it rapidly will condense on the upper cooler sections of the tube which extend above the heat transfer liquid. The sample can easily be recentrifuged to the bottom of the tube and a new determination of the boiling point commenced. Indeed, if the bell cavity fills completely during the cooling point of a cycle, it is often difficult to reinitiate the bubble stream without first emptying the entire cavity by overrunning the boiling point.

Observed boiling points for a series of compounds which boil over a wide range of temperatures are summarized in Table 4.1.

Materials that are thermally stable at their boiling point will give identical values on repeat determinations. Substances that begin to decompose will give values that slowly drift after the first few measurements. The observation of color and/or viscosity changes plus a variable boiling point all signal the need for caution in making extended repeat measurements.

Table 4.1 Observed Boiling Points (°C)[a]

Compound	Observed	Literature Value	Reference
Methyl iodide	42.5	42.4	[b]
Isopropyl alcohol	82.3	82.4	[c]
2,2-Dimethoxypropane	80.0	83.0	[d]
2-Heptanone	149–150	151.4	[e]
Cumene	151–153	152.4	[f]
Mesitylene	163	164.7	[g]
p-Cymene	175–178	177.1	[h]
Benzyl alcohol	203	205.3	[i]
Diphenylmethane	263–265	264.3	[j]

[a] Observed values are uncorrected for changes in atmospheric pressure (corrections all estimated to be less than ±0.5°C).

[b] *CRC Handbook of Chemistry and Physics*, 62nd ed.; CRC Press: Boca Raton, FL, 1981; #9082, p C-373.

[c] Ibid. #11971, p C-470.

[d] *Dictionary of Organic Compounds*, 4th ed.; Oxford University Press: London, 1965; Vol. I, p 11.

[e] *CRC Handbook of Chemistry and Physics*, 62nd ed.; CRC Press: Boca Raton, FL, 1981, #7627, p C-321.

[f] Ibid. #5394, p C-244.

[g] Ibid. #8987, p C-370.

[h] Ibid. #2192, p C-138.

[i] Ibid. #3160, p C-169.

[j] Ibid. #6282, p C-274.

REFRACTIVE INDEX

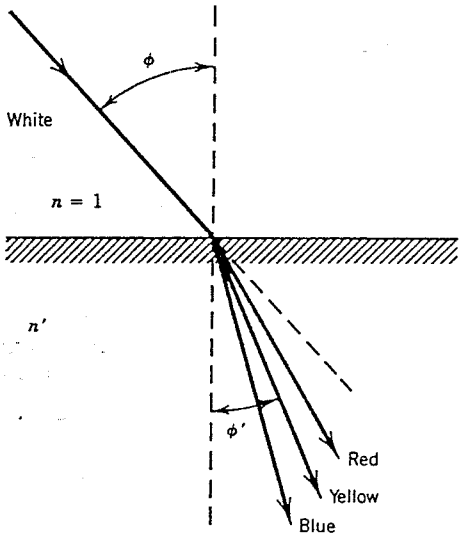


Fig. 4.6 *Upon refraction white light is spread out into a spectrum. This is called dispersion.*

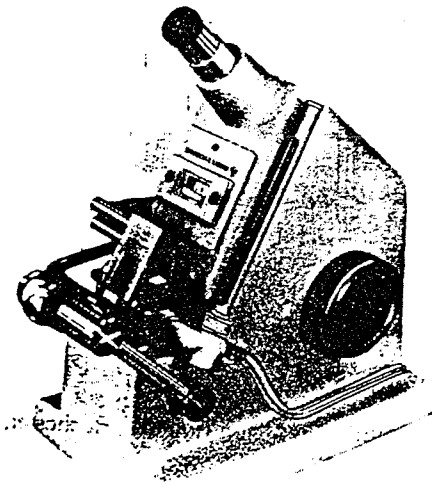


Fig. 4.7 *Abbe-3L refractometer.* (Courtesy of Milton Roy Co., Rochester, NY.)

It is commonly observed that a beam of light "bends" as it passes from one medium to another. For example, an oar looks bent as one views the portion under the water. This effect is a consequence of the refraction of light. It results from the change in velocity of the radiation at the interface of the media, and the angle of refraction is related to the velocity change as follows (see Fig. 4.6).

$$\frac{\sin \phi}{\sin \phi'} = \frac{\text{velocity in vacuum}}{\text{velocity in sample}} = n \text{ (refractive index)}$$

Since the velocity of light in a medium must be less than that in a vacuum, the index of refraction n will always be greater than one. In practice, n is taken as the ratio of the velocity of light in air relative to the medium being measured. The refractive index also is wavelength dependent.

The wavelength dependence gives rise to the effect of dispersion or the spreading of white light into its component colors. When we measure n, therefore, we must specify the wavelength at which the measurement is made. The standard wavelength for refractive index determinations has become the bright yellow sodium 589-nm emission, the sodium D line. Sodium, unfortunately, is a poor choice of wavelength for these measurements with organic substances, but as the sodium lamp represents one of the easiest monochromatic sources of radiation to obtain experimentally, it is widely used. Because the density of the medium is sensitive to temperature, the velocity of radiation also changes with temperature, and therefore, refractive index measurements must be made at constant temperatures. Many values in the literature are reported at 20°C. In the table of data given for each experiment, no record of temperature indicates a value measured at 20°C, as in the case of density values. The index can be measured optically quite accurately to four decimal places. Since this measurement is particularly sensitive to the presence of impurities, the refractive index can be a valuable physical constant for tracking the purification of liquid samples.

The measurement (for example) is reported as

$$n_D^{20} = 1.4628$$

Procedure

In the Abbe-3L refractometer (Fig. 4.7), white light is used as the source, but compensating prisms give indices for the D line. This refractometer is commonly used in many undergraduate organic laboratories.

Samples (approximately 10 μL) are applied between the horizontal surfaces of a pair of hinged prisms (Fig. 4.8). A sampling procedure recently developed by B. P. Ronald significantly reduces the amount of sample required and allows accurate measurements of highly volatile materials. The technique involves placing a small precut 6-mm disk of good-quality lens paper at the center of the bottom prism. The sample is loaded onto the disk with a micro-Pasteur pipet or microliter syringe.[5]

> **CAUTION: *Do not touch the prisms with the Pasteur pipet or syringe tip as they may be easily marred and scratched and will then give erroneous results.***

Refractive Index Measurements Utilizing the Lens Paper Disk Technique

Substance	T (°C)	n^t (normal) 100 μL	n^t (microdisk) 2–4 μL
Water	24.5	1.3224	1.3226
Diethyl ether	24	1.3508	1.3505
Chlorobenzene	24.5	1.5225	1.5219
Iodobenzene	24.5	1.6151	1.6151

The refractometer is adjusted so that the field of view has a well-defined light and dark split image (see your instructor for the correct routine for making instrument adjustments on your particular refractometer).

When using the refractometer, always clean the prisms with alcohol and lens paper before and after use. Record the temperature at which the reading is taken. A reasonably good extrapolation of temperature effects can be obtained by assuming that the index of refraction changes 0 0004 unit per degree Centigrade and varies inversely with the temperature.

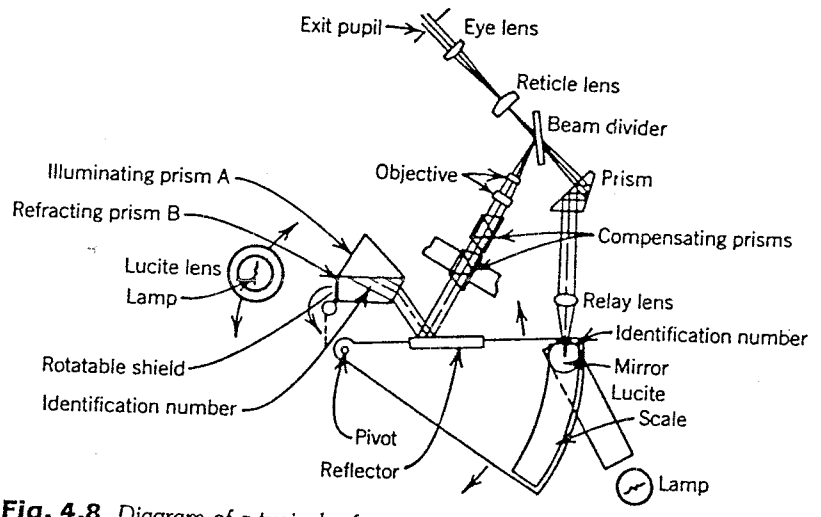


Fig. 4.8 *Diagram of a typical refractometer.* (Courtesy of Milton Roy Co., Rochester, NY.)

[5] B. P. Ronald, Department of Chemistry, Idaho State University, Pocatello, Idaho, Personal Communication.

Size of TLC plate = 1" x 0.5"

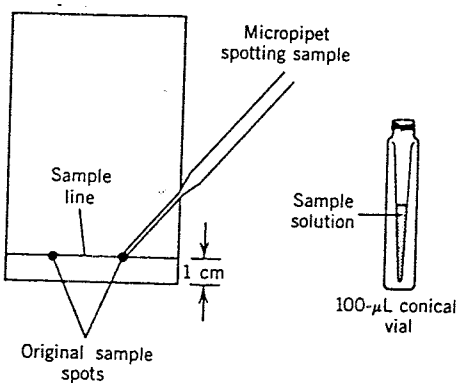


Fig. 5.55 *Sample application to a TLC plate.*

The sequence of operations is as follows.

1. A piece of window glass, a microscope slide, or a sheet of plastic can be used as a support for a thin layer of adsorbent spread over the surface. It is possible to prepare locally the glass surfaces, but plastic-supported thin-layer systems are only commercially available. The plastic-supported layers are particularly attractive because they possess very uniform coatings and are highly reproducible in operation. Another convenient feature of the plastic-backed plates is that they can be cut with scissors into very economical 1 × 3-in. strips. The latter style is used exclusively in the microscale laboratory.
2. A pencil line is drawn parallel to a short side of the plate 1.0 cm from the edge. One or two points evenly spaced are marked on the line. The sample (1 mg) to be analyzed is placed in a 100-μl conical vial and a few drops of solvent are added. A micropipet (prepared by the same technique used for constructing the capillary insert in the ultramicro boiling point determination; see Chapter 4) is used to apply a small fraction of the solution from the vial to the plate (Fig. 5.55).
3. The chromatogram is carried out by placing the spotted thin-layer plate in a screw-capped wide-mouth jar or a breaker with a watch glass cover containing a small amount of developing solvent (Fig. 5.56). The material spot on the TLC plate must initially be positioned above the solvent line. The jar is quickly recapped or the watch glass replaced to maintain an atmosphere saturated with the developing solvent. The elution solvent rapidly ascends the plate by capillary action. The choice of solvent will be similar to that used in column chromatography, but need not be identical. The spotted material is eluted vertically up the plate. Resolution of mixtures into individual spots along the vertical axis occurs by precisely the same mechanism as in column chromatography. Development is interrupted when the solvent line nears the top of the plate.
4. Visualization of colorless separated components is achieved by placing the plate in an iodine vapor chamber for a few seconds. Iodine forms a reversible complex with most organic substances. Thus, dark spots will develop in those areas containing sample material. On removal from the iodine chamber, the spots are marked by pencil, because they will fade rather rapidly. Sample quenching of UV activated fluorescent indicator coated TLC plates is an alternative mode of detection. The elution characteristics are reported as R_f values. The R_f value is a measure of the travel of a substance up the plate during the chromatogram relative to the solvent movement. This value is defined as the length of migration by the substance divided by the distance

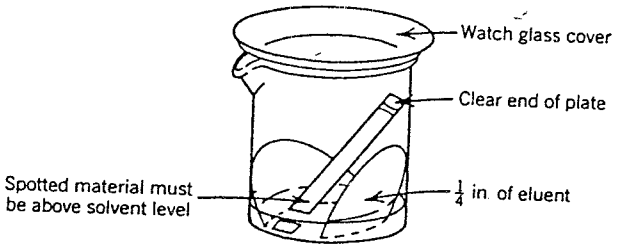


Fig. 5.56 *Development of a TLC plate. From* Zubrick, James W. *The Organic Chem Lab Survival Manual,* 2nd ed.; Wiley: New York, 1988. (Reprinted by permission of John Wiley & Sons, New York.)

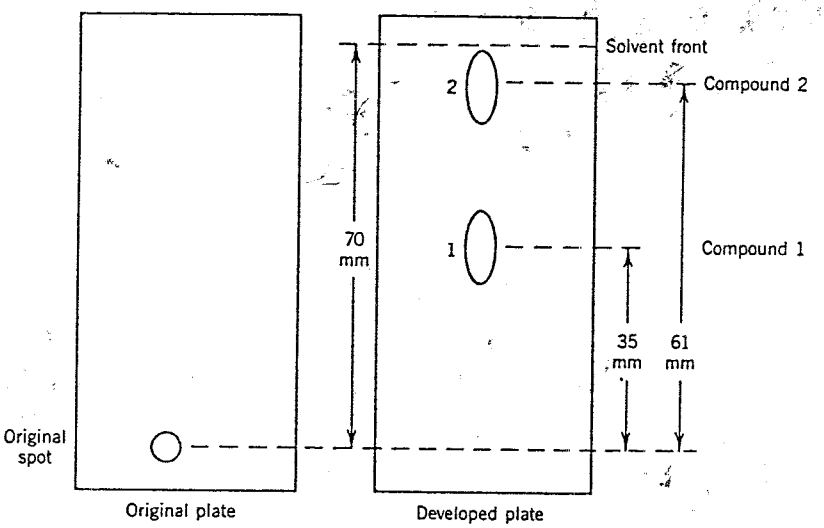


Fig. 5.57 *A sample calculation of R_f values.*

traversed by the solvent front (the position of the solvent front should be quickly marked on the plate when the chromatogram is terminated; see Fig. 5.57).

Thin-layer chromatography is used in a number of applications. The speed of the technique makes it quite useful for monitoring large-scale column chromatograms. Analysis of fractions can guide decisions on the solvent elution sequence. TLC analysis of column-derived fractions can also give an indication of how best to combine collected fractions. Following the progress of a reaction by periodically removing small aliquots for thin-layer analysis is another useful application of thin-layer chromatography. The technique is utilized in several microscale experiments, for example, Experiments 6, 17, 21A, 32, 34D, 40A, 41, and 45.