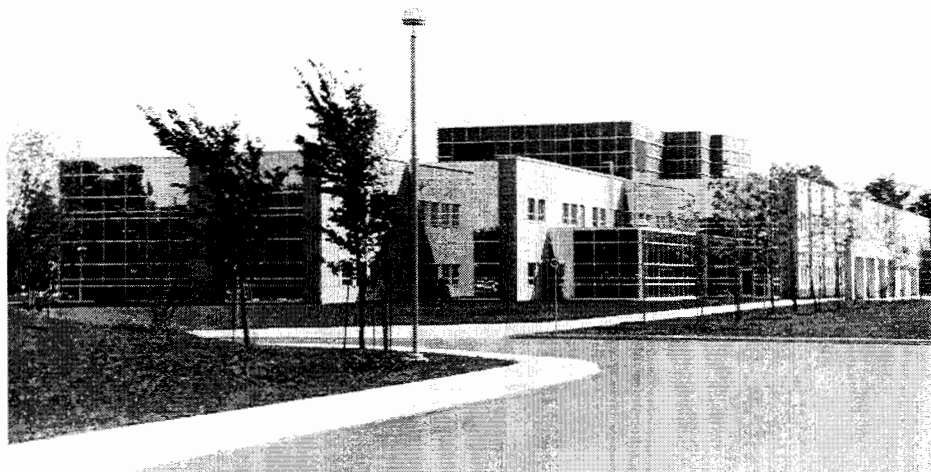


C₃ NEWS

Newsletter of College Chemistry Canada / La Chimie Collégiale au Canada

22nd Conference

This issue of C₃ News contains the first set of information on the 1995 College Chemistry Conference to be held at Heritage College in the National Capital Region. Heritage College is located in Hull, Quebec, on the edge of the Gatineau Hills. This beautiful area is a mecca for cross country skiers in the winter and cyclists, hikers, joggers, and picnickers in the summer. So bring along your hiking boots and picnic baskets and plan on some time to explore the area. The conference theme will be "Teaching Chemistry for the 21st Century"; so start thinking of what you will be presenting. The preliminary call for papers is included inside.



EXTRACTION OF CAFFEINE FROM TEA

David R. Taylor and

Mark K. Thomas

Kwantlen College

Surrey, B.C., V3T 5H8

Neeland has recently reported¹ major improvements in the procedure for extracting caffeine from tea. We now report some minor, but significant, modifications to Neeland's procedure.

We extracted the tea bags with hot water as described by Neeland. However, we found that the same amount of caffeine was extracted but less emulsion was produced by extracting the aqueous solution by stirring (with a magnetic stir bar) with CH₂Cl₂ in an Erlenmeyer flask (the CH₂Cl₂ phase is transferred with a Pasteur pipette) instead of using a separatory funnel. In addition, we dry

the CH₂Cl₂ extract (after washing with aqueous NaOH) with paper instead of Na₂SO₄.² Finally, for the sublimation of caffeine at atmospheric pressure, we use the small and very cheap apparatus shown in the Figure.

We find it educational to have the

students compare the caffeine before and after washing with aqueous NaOH by thin layer chromatography. This shows that the aqueous NaOH removes the more acidic theophylline (pK_a = 8.77). Chlorophyll is also removed, presumably via hydrolysis to the acid salt.

Continued on page 2

In this issue:

Extraction of Caffeine From TeaPage 1

Drying Dichloromethane.....Page 3

Christmas QuizSpecial Insert

EXTRACTION OF CAFFEINE FROM TEA, continued from page 1

Experimental

Three tea bags (ca 9.3 g tea) were extracted with hot water as described by Neeland. The cooled tea solution was extracted by stirring (1" x 5/16" magnetic stir bar; medium speed - ca 10 cps) with two 10-mL portions of CH_2Cl_2 ³ in a 125-mL Erlenmeyer flask. The combined CH_2Cl_2 extract⁴ was washed with two 10-mL portions of 10% NaOH and 20 mL of distilled water, dried by passing through a small wad of Kimwipes in a glass funnel, and evaporated to dryness in 25-mL Erlenmeyer flask. The residue (73 mg) was transferred with the minimum of CH_2Cl_2 to the sublimation vial. Evaporation of the solvent and sublimation on a hot plate gave colourless crystals (55 mg; mp xxx-zzz°C, lit. 238°C).

TLC (Merck silica gel 60 F254, 0.2 mm thick, 3.3 x 6.7 cm plastic sheets; $\text{CHCl}_3:\text{CH}_3\text{OH} = 40:1$) of the unwashed extract showed caffeine, chlorophyll and theophylline (in order of decreasing Rf) whereas the NaOH washed extract showed only caffeine.

References and Notes

- 1 Neeland, Edward G.. C3 News, Spring 1993, p. 7.
- 2 Taylor, David R. see paper below.
- 3 About 50% of the caffeine was extracted but the amount obtained was more than adequate.
- 4 A small portion of the unwashed CH_2Cl_2 was saved for TLC.

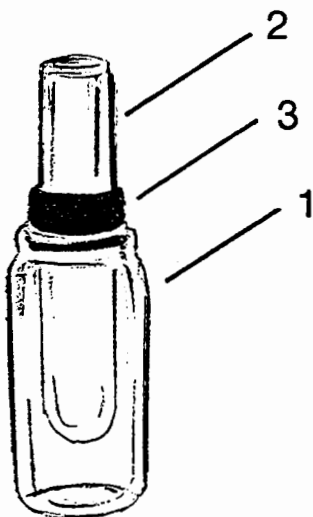


FIGURE. SUBLIMATION APPARATUS

- 1 = KIMBLE Opticlear/ 5 dram vial (27.25 x 55 mm)
- 2 = BAXTER 15 x 85 mm diSPo/ culture tube
- 3 = AHS black rubber tubing (12.5 mm ID, 3.0 mm wall; ca 5 mm wide)



C3 News

Volume 19, No.3, December 1994

Published quarterly by College
Chemistry Canada Inc

President: Suhrir Abhyankar

Editor: Suzanne Gardner

Mailing Address:
Kwantlen College
P.O. Box 9030
Surrey, B.C.
V3T 5H8

Tel: (604)-599-2656
Fax: (604)-599-2716

E-Mail: suzanneg@kwantlen.bc.ca

Articles of any length will be gladly accepted. Please send typewritten copy to the Editor at the above address or send by fax. Copy can also be sent on a 3 1/2" disk, MAC format using Microsoft Word, or any wordprocessor producing ASCII output, or by e-mail.

© 1994 College Chemistry Canada Inc
ISSN 0843-4956

Designed and printed at the
Open Learning Agency.

**The 22nd C3 Conference:
Teaching Chemistry for the 21st Century**

June 1st, 2nd, and 3rd, 1995

to be held at
Heritage College
in the
National Capital Region

Theme: Teaching Chemistry for the 21st Century

Location: Heritage College, Hull, Quebec

Highlights: Wine and Cheese

Banquet and Tour of the Museum of Civilization

Keynote Speaker: A. Carty, President of NRC

Call for Papers

This is a preliminary call for papers for the conference on the theme of "Teaching Chemistry for the 21st Century". Papers on new approaches to or ideas on teaching chemistry would be particularly welcome. If you wish to be considered for inclusion in the C₃ conference program, please mail or fax one of the following persons as soon as possible:

D.J. Kroeger
Algonquin College
200 Lees Ave.
Ottawa, Ontario
K1S 0C5

Pierre Dupont
College de l'Outaouais
333 boul. cite des Jeunes
C.P. 5220, Hull, P.Q.
J8Y 6M5

(613) 727-4723 ext. 5940
FAX: (613) 598-3357

(819) 770-4012 ext. 259
FAX: (819) 770-3855

Name: _____

Address: _____

Affiliation: _____

Telephone: _____ Fax: _____

E-Mail: _____

Title: _____

Preferred Format: 10 minutes 20 minutes 30 minutes

Abstract (50 words approximately):

Audio Visual Requests: _____

Drying Dichloromethane: Analysis of Water in Dichloromethane by NMR

David R. Taylor

Kwantlen College

Surrey, B.C., V3T 5H8

Drying dichloromethane

Dichloromethane is a very useful solvent for liquid-liquid extraction of organic compounds. Most laboratory texts recommend the use of MgSO₄ or Na₂SO₄ for drying the CH₂Cl₂ layer obtained from extraction of an aqueous solution or mixture. However, the solubility of water in CH₂Cl₂ is only 0.20% at 25°C while CH₂Cl₂ forms an aqueous azeotrope (bp 38.1 C) which is 1.5% water.¹ Hence, we have, for many years, merely removed the droplets of water from the CH₂Cl₂ layer by filtering through paper (usually a wad of Kimwipes for small-scale extractions) and then evaporated the CH₂Cl₂ at atmospheric pressure. The CH₂Cl₂ solution must be transferred without transferring most of the water droplets by using a separatory funnel on a large scale or a Pasteur pipet on a small scale. For cloudy CH₂Cl₂ layers, more paper is needed. Using too much exposed paper results in the formation of crystals of CH₂Cl₂ hydrate² on the paper because evaporation and cooling of the CH₂Cl₂ (due to the draft in the fumehood) leads to condensation of water.

Analysis of water in dichloromethane by ¹H NMR

The water content of CH₂Cl₂ can be determined easily with fair accuracy by comparing the peak for the dissolved water (ca δ 1.4) with one of the peaks (¹³C satellites) for the ¹³CH₂Cl₂ doublet (89.5 Hz on either side of the CH₂Cl₂ peak at ca δ 5.2). To avoid overlap of the ¹³C satellites with the spinning sidebands for CH₂Cl₂, the spin rate should not be close to 90 or 45 Hz. Since both water and CH₂Cl₂ contain two H atoms, the ratio of the area for the water peak to the area for both ¹³C satellites (or twice the area of one ¹³C satellite) is equal to the mole ratio of water to ¹³CH₂Cl₂. For ACS grade CH₂Cl₂, the area for the water to that of the upperfield ¹³C satellite was found to be 2.7:9.2 (Fig.) and hence the water:¹³CH₂Cl₂ ratio is 2.7:18.4. Since the natural abundance of ¹³C is 1.11%, the mass ratio of water:CH₂Cl₂ can be calculated as follows:

$$\frac{\text{mass H}_2\text{O}}{\text{mass CH}_2\text{Cl}_2} = \frac{2.7 \text{ mol H}_2\text{O}}{18.4 \text{ mol } ^{13}\text{CH}_2\text{Cl}_2} \times \frac{18.0 \text{ g H}_2\text{O}}{1 \text{ mol H}_2\text{O}} \times \frac{1.11 \text{ mol } ^{13}\text{CH}_2\text{Cl}_2}{100 \text{ mol CH}_2\text{Cl}_2} \times \frac{1 \text{ mol CH}_2\text{Cl}_2}{84.9 \text{ g CH}_2\text{Cl}_2}$$

$$= 0.00034. \text{ Hence \% water} = 0.034\%$$

For CH₂Cl₂ saturated with water, the percent water was determined to be 0.23%, in good agreement with the literature value of 0.20% (see above). After filtering the water-saturated CH₂Cl₂ (ca 5 mL) through a wad of Kimwipes (ca 3 cm x 8 cm) in a Pasteur pipet, the percent water was found to be 0.086%. Thus, the paper removed more than half of the dissolved water as well as the water droplets. Further drying with MgSO₄ for about 2 minutes reduced the water content to 0.082%. Phase separation paper gave results comparable to Kimwipes.

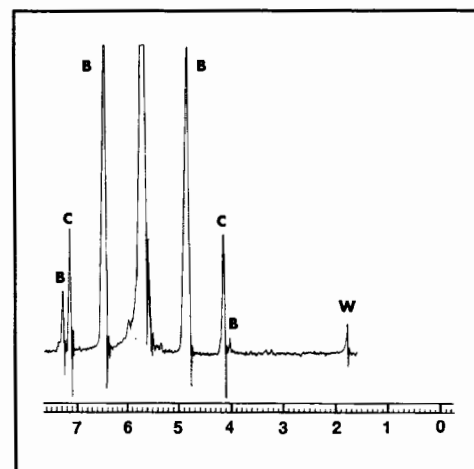


FIGURE.
60 MHz ¹H NMR of ACS grade CH₂Cl₂
W = water;
C = ¹³C satellite;
B = spinning sideband

References

- 1 John A. Riddick, William B. Bunger and Theodore K. Sakano, Organic Solvents, 4th ed., In Techniques of Chemistry, A. Weissberger (Ed.), Vol. II, John Wiley & Sons, New York, 1986; p.491.
- 2 Ref.1, p. 1010.

**College Chemistry
Canada Inc.
Board of Directors
1994-95**

Executive:

President

Sudhir Abhyankar
Sir Wilfred Grenfell College
University Drive
Corner Brook, NF
A2H 6P9
e-mail: sudhir@morgan.ucs.mun.ca

Past-President

Bob Browne
Douglas College
P.O. Box 2503
New Westminster, BC
V3L 5B2
e-mail: bob_browne@douglas.bc.ca

Secretary

Bob Perkins
Kwantlen College
P.O. Box 9030
Surrey, BC
V3T 5H8
e-mail: bobp@kwantlen.bc.ca

Treasurer

Phyllis Lake
1140 Kildonan Place S.W.
Calgary, AB
T2V 4B1

Editor

Suzanne Gardner
Kwantlen College
P.O. Box 9030
Surrey, BC
V3T 5H8
e-mail: suzanneg@kwantlen.bc.ca

Program Coordinators

Dick Kroeger
Algonquin College
200 Lees Avenue
Ottawa, ON
K1S 0C5

Pierre Dupont
College de L'Outaouais
333 boul. de la Cite des Jeunes
C.P. 5220
Hull, PQ
J8Y 6M5

Conference Coordinators

Rod Restivo & Mita Saha
Heritage College
205 rue Laurier
Hull, PQ
J8X 3Y8

CIC Liaison

Leroy Pazdernik
Universite du Quebec
C.P. 500
Trois-Rivieres, PQ
G9A 5H7

2YC3 Liaison

Shahid Jalil
John Abbott College
21275 Lakeshore Road
St. Anne de Bellevue, PQ
H9X 3L9

CSCT Liaison

Murray Morello
Seneca College
1750 Finch Avenue East
North York, ON
M2J 2X5

Directors:

Atlantic Provinces

Geoff Rayner-Canham
Sir Wilfred Grenfell College
University Drive
Corner Brook, NF
A2H 6P9

Quebec

Rod Restivo
Heritage College
205 rue Laurier
Hull, PQ
J8X 3Y8

Harry Wilson
John Abbott College
21275 Lakeshore Road
St. Anne de Bellevue, PQ
H9X 3L9

Ontario

Dinesh Bhatnagar
Algonquin College
200 Lees Avenue
Ottawa, ON
K1S 0C5

MB, SK, AB, and NT

Bill Blann
Keyano College
8115 Franklin Avenue
Fort McMurray, AB
T9H 2H7

Brad Pavelich
Medicine Hat College
299 College Avenue
Medicine Hat, AB
T1A 3Y6

BC and YT

Kelly Sveinson
Langara College
100 West 49th Avenue
Vancouver, BC
V5Y 2Z6

Peter Slade
University College of the Fraser
Valley
33844 King Road
Abbotsford, BC
V2S 4N2



*C3 News
Suzanne Gardner
Kwantlen College
P.O. Box 9030
Surrey, B.C.
V3T 5H8*

OOOPS: In the last issue Cynthia Mutch was incorrectly listed as a regional director for the Prairie region. The regional directors for Manitoba, Saskatchewan, Alberta, and the Northwest Territories are Bill Blann and Brad Pavelich. My apologies to both Cynthia and Brad.