Volume 24, Issue 4

# $C_3$ News

December 1999

Newsletter of College Chemistry Canada/La Chemie Collégialle au Canada

27TH COLLEGE CHEMISTRY CANADA CONFERENCE

JUNE 1-4, 2000

KAMLOOPS, BC

FOCUS: CHEMISTRY AND WATER QUALITY

### NEXT ISSUE

IN THE FIRST ISSUE OF THE NEW MILLENIUM $C_3$ NEWS WILL FOCUS ON LOOKING BACK AND LOOKING AHEAD. READERS ARE INVITED TO SUBMIT ARTICLES ON THE EVOLUTION OF $C_3$ TO DATE, AND THE DIRECTION THEY ENVISAGE IT TAKING IN THE 21ST CENTURY.

EDS.

### INSIDE THIS ISSUE



## 27th College Chemistry Canada Conference, June 1-4, 2000 — details and 1st call for papers

***Doug Bickley and Norm Reed***
***University College of the Cariboo, Kamloops, BC.***

The 27th College Chemistry Canada Conference will be hosted by The University College of the Cariboo in Kamloops, BC, from June 1 - 4, 2000. The theme for our conference is Chemistry and Water Quality. The UCC Organizing Committee invites you to visit us in Kamloops, situated in the Thompson River Valley in South Central BC, enjoy our spectacular setting, and take part in what should be a very interesting and relevant conference. Registration fees listed below do not include (the necessary) $C_3$ membership. Prices are in $CDN:

| | |
|---|---|
| Before May 1: | $50 |
| After May 1: | $70 |
| One Day: | $25 |

Current plans are for a number of fifteen- and thirty-minute blocks for presentations either along our theme or, optionally, about any aspects of teaching chemistry. Teaching tips or favorite demonstrations fit nicely in the fifteen-minute time blocks. In addition, a Poster Session is planned to allow flexibility in presentation styles.

For more information visit the conference web site at:

http://www.cariboo.bc.ca/schs/chem/c3conf/c3conf.htm

or contact the conference organizers:

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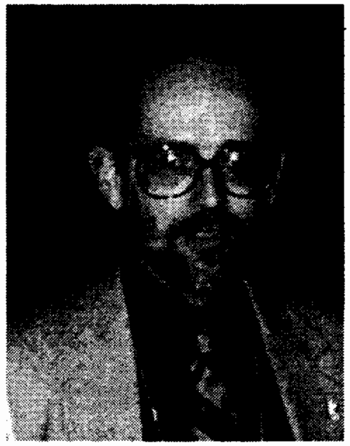

# President's Report

## *by Bob Perkins*

Well, here we are with two weeks left in the semester and it is time for another issue of $C_3$ News. I have had a busy time with my 75% position with the Faculty Association and my 25% teaching assignment (2nd year organic chemistry). $C_3$ is in good shape: the Executive has kept in touch via a monthly conference call as well as frequent e-mail messages back and forth across the country, and the planning for the Kamloops Conference is proceeding well. Check this issue for additional conference details as well as a call for papers. Doug Bickley and Norm Reed will be happy to accept your submissions by e-mail/voice mail/snail mail. Our beloved co-editors feel the same way about submissions to $C_3$ News too; they will welcome any tidbit of news you may care to send along. Remember that this is your newsletter, and instructors in the rest of the country may well be able to make use of your ideas. One of mine appears in this issue; look for "What's the Question ?"

We have just heard from the President of CSC that the proposal from $C_3$ to allow our members to become Affiliate Members in the CED division of the CSC has been approved. There will be more details to follow, but one of the benefits will be to allow us to attend the CSC conferences at a greatly reduced rate (yet to be determined). I have been in discussions with the organizer (Dr. Sharon Bennett — UQAM) of the Chem. Ed. symposia about a $C_3$ presence at the 2001 CSC Conference in Montreal. More details will follow as they become available, but we are looking for support from the $C_3$ members in the Montreal area (*hint hint*) to help coordinate with the CSC. This fits in well with $C_3$ conference plans; our $C_3$ conference in 2001 was due to be back in Eastern Canada, hence a joint effort would make sense. If you have any thoughts on the matter, please let me know.

That's about it for now. I hope that you all have a wonderful break at Xmas and come back to classes in January fully refreshed and ready to go again.

Bob Perkins.

***NEXT $C_3$ CONFERENCE***

1 JUNE - 4 JUNE 2000, KAMLOOPS, BC
FOCUS ON: CHEMISTRY AND WATER QUALITY
CHECK: HTTP://WWW.CARIBOO.BC.CA/SCHS/CHEM/C3CONF/C3CONF.HTM

# Isolation of Carvacrol from Summer Savory: A Natural Products Experiment.

*Mary D. Secord, Don-Roger Parkinson and Julian M. Dust*

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*Sir Wilfred Grenfell College*

Background

Experiments involving the isolation of a natural product from various types of plant and animal material are commonly found in the introductory Organic Chemistry laboratory course. The inclusion of these experiments in the curriculum is generally justified on a number of grounds.

First, these experiments permit exposure of the student to a number of laboratory techniques such as steam and fractional distillation, extraction, use of a mortar and pestle and so forth. A second pedagogical purpose is that of enhancing student interest by connecting everyday things, like the spices in the kitchen cupboard, to organic chemistry, i.e. the structure and properties of the active ingredients of the spices in this example.

Regardless of the rationale, however, natural products laboratory experiments are among those most often described in the literature. For example, one laboratory text gives recipes for the isolation of nicotine from tobacco (1), caffeine from tea or coffee (2), eugenol from cloves or allspice, cuminaldehyde from cumin and cinnaldehyde from cinnamon (3). The isolation or identification of caffeine in various products has been particularly popular and there have been recent reports of its isolation by classical methods (4), and its identification and quantification by more esoteric techniques such as micellar electrokinetic chromatography (5) and solid-phase microextraction/GC-MS (6).

The question of relevance, however, does arise. Exactly how relevant is it that students learn to use steam distillation, for example, if that technique is never used again in the course? How relevant is it to students to isolate eugenol from cloves, if cloves are not among the spices commonly used?

One distinction that is often drawn between "spices" and "herbs" is a geographical one: spices tend to derive from tropical plants, while herbs grow in more temperate climates. It might be argued, then, that natural products involving herbs would be more appropriate to many North American students.

In the context of Newfoundland, savory and its essential oils certainly are relevant. After salt and, perhaps, pepper, summer savory is the most commonly used herb in Newfoundland. It is found in such Newfoundland delicacies as "fish and brewis", codfish cakes, cod-au-gratin, moose stews and soups, various stuffings for turkey and chicken and a multitude of other dishes. As such, it is found in virtually every Newfoundland kitchen, grown in many kitchen gardens and the dried herb is packaged and marketed by a local company.

There are a number of species of savory. A recent herbal states:

> The active ingredient in the various species of savory is carvacrol [2-methy-l-5-(1-methylethyl)phenol or 2-methyl-5-*iso*propylphenol] or its structural isomer, thymol [5-methyl-2-(1-methylethyl)phenol]. Thus, the essential oil of *Satureia thymbra* contains up to 19% thymol, that of *Satureia montana* or Winter Savory contains 30-40% carvacrol and Summer Savory (*Satureia hortensis*) has an essential oil that is reported to consist of up to 80% carvacol (8).In this article we present details of the isolation of carvacol from dried summer savory. The procedure is based on the published method of extraction of carvacol from oil of Spanish oregano (9), using aqeous sodium hydroxide, and relies on the acidity of phenols, as compared to other hydrocarbon or alkanolic components in the plant matter. The summer savory used in this procedure was dried and readily available in that form from a local bulk foods store. This is the form of this herb that most of our students would immediately recognize. No attempt was made to compare the result of this extraction with those using fresh summer savory. Finally note that the dried herb is available in quantity throughout the year, while fresh savory would only be available in the Fall semester of the academic year.

Experimental

A weighed amount of dried summer savory (ca. 15 g) was ground to a powder in a mortar. The ground savory was transferred to a 600 mL beaker. Diethyl ether (200 mL) was added and the slurry stirred for 15 min, then allowed to settle. The supernatent ether was decanted into a beaker. The residual damp savory was gravity filtered through fluted filter paper into the same beaker. The savory was rinsed twice with diethyl ether (20 mL portions).

The combined green-coloured ethereal solution was transferred to a separatory funnel (500 mL), washed thrice with 3 M NaOH (50 mL portions) and the gold-coloured alkaline aqueous layers combined in a beaker. The beaker was cooled in ice. Once cold the pH of the aqueous solution was adjusted to 5 to 6 using 6 M $HC\ell$ (ca. 80 mL).

The cool solution was returned to the separatory funnel. Back extraction with ether (3 x 50 mL), followed by drying ($MgSO_4$) and gravity filtration left a solution which was evaporated under reduced pressure (rotovap) to give 0.020 g (0.13% by mass) of a golden oil. The low melting point of carvacol (lit. 3.5°C(9)) precluded identification by this simple method. However, the UV-vis spectrum of commercial carvacol (Aldrich) and that obtained by this procedure were identical ($\lambda_{max}$ = 220, 280 nm (EtOH)). TLC (silica, 1:1 ligroin: ether) gave a single spot ($R_f$ = 0.61), coincident with the spot for commercial carvacrol.

Gas chromatography (thermal conductivity detector; column: Supelcowax-10 polar, fused silica, capillary 30 m, 0.25 i.d., 0.25 mm film thickness; carrier gas: He; injector temperature: 240°C; detector temperature: 270°C; initial oven temperature: 40°C; ramp: 40°C/ min; final temperature: 260°C) gave a single sharp peak with a retention time of 9.65 min for a 2-L injection of the oil in ether. Spiking with carvacol confirmed the assignment. (While no attempt was made to optimize the analytical protocol, we note that a 1:1 mixture of thymol and carvacol could be separated under these conditions.)

Conclusions

The current experiment is relatively straight-forward that could be completed by students within the typical three-hour laboratory session. Although the yield of carvacol is not high, it is sufficient to permit students to record a UV-Visible spectrum, obtain a thin-layer chromatogram and a GC trace.

We have undertaken preliminary comparative experiments to determine whether other techniques would be more appropriate than our simple extraction method for the isolation of carvacol. Table 1 shows some comparative initial data for the isolation of the essential oil of summer savory by internal steam distillation and Soxhlet extraction with ether. In each case the initial extract or distillate was treated with aqueous NaOH, washed with ether, the aqueous extract acidified and back extracted with ether. Removal of the ether yielded the raw essential oil.

**Table 1. Comparison of isolation methods.**

| Method | Yield | % Yield |
|---|---|---|
| Simple extraction (described above) | 0.020 g | 0.13% (*ca.* 15 g savory used) |
| Internal steam distillation | 0.043 g | 0.17% (*ca.* 25 g savory used) |
| Soxhlet extraction with ether | 0.030 g | 0.21% (*ca.* 14 g savory used) |

As can be gleaned from Table 1, no method was clearly superior to the others. The Soxhlet extraction proceeded overnight and normally would be too time consuming for use in an introductory Organic Chemistry course. Furthermore, the capital cost of kitting out a typical lab section would likely be prohibitive, unless the equipment was also incorporated into other experiments in other courses. It should also be noted that TLC of the crude product of steam distillation, even after the subsequent workup procedure, showed six spots indicative of other unidentified products.

Therefore, while steam distillation may isolate more of the essential oil, it is not clear that it permits isolation of more of the active ingredient, carvacrol.

Finally, we wish to comment on the relevance of this experiment. The experiment does not introduce the student to as many novel bench techniques as some other procedures do. However, this leaves time for the student to use techniques such as TLC, GC and UV-vis in the identification of the product. Clearly, other spectroscopic techniques, such as NMR or IR, could also be introduced as this stage.

The cost of the savory for the experiment is relatively low (*ca.* $0.80/student for the amount used as outlined above).

The experiment uses an herb that is locally grown in Newfoundland, commonly used in Newfoundland cuisine and therefore, arguably, the experiment is more relevant to our students in that it connects the lab experience and everyday life.

We hope that this report will stimulate others to consider natural products experiments that may be more relevant to our students.

Acknowledgements

We acknowledge support of this work by the Principal's Research Fund of Sir Wilfred Grenfell College. This work was presented in preliminary form at the joint $C_3$-2YC3 (Eastern US) conference held at Sir Wilfred Grenfell College, June 12-14, 1997. The article is dedicated to the memory of Dr. Reg Friesen, University of Waterloo, a pioneering Canadian Chemical Educator.

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# What's The Question ?
## *by Bob Perkins*

In the last issue of $C_3$ News I discussed my "7 Deadly Sins" for beginning organic chemistry students. The response has been quite positive and I have had several requests for any more tricks that I may have up my sleeve to keep students actively engaged in the learning process. In response to this, I present below an activity that may be helpful in accomplishing this. I use it myself with some success.

My teaching style in the classroom is very interactive with questions directed to individual students. I indicate when asking the question that I always expect an answer (whether correct or not), and that they will not be penalized for providing an incorrect response. I don't keep track of how many correct or incorrect answers that they provide; I merely want them to take an active role in the presentation of the material. One very useful technique when a student provides an incorrect answer is to say, "That's an excellent answer, but unfortunately it's the correct answer to a different question." I will then turn to another student and ask if they can formulate a question to which the previous answer is the correct one. I'll give a few examples in the paragraphs to follow to demonstrate the idea in action.

Let's suppose that you're trying to determine whether the class has a good understanding of electron configuration and you have asked a student to tell you the number of valence electrons in a ground state Mn atom. The student responds with the number 5, believing that the five 3*d* electrons are actually the valence electrons. You could then ask another student what question would have 5 as the correct answer. One possible question might be, "How many half-filled orbitals does a ground state Mn atom have ?" Another one could be, "How many electrons in a ground state Mn atom have the quantum number $\ell = 2$ ?" This is a very open-ended area for getting the students familiar with shells, subshells, orbitals, etc.

As another example, you might ask about the relationship between $Fe^{2+}$ and $Fe^{3+}$. If the student responds with the word "isotope", you could then have another student provide examples that would illustrate what an isotope is. If they respond with $Na^{1+}$ and $A\ell^{3+}$, you could then turn to another student to hopefully have them come up with the word "isoelectronic."

You could also ask how 2-pentene and 3-pentene are related. If the student to whom the question was put responds that they are positional isomers (a sub-set of structural/constitutional isomers), you could ask another student to draw a pair of compounds with the same formula ($C_5H_{10}$) that would be positional isomers. You could then expand the question by including other types of isomers (skeletal, functional, unsaturated etc.).

My point here is that our subject is vocabulary rich, and the more we can engage our students in using the words, rather than simply memorizing what they mean, the more we will enable them to succeed.

Feel free to send along any comments or provide additional examples.

### RENEWALS

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