Molar Mass Determination by Freezing Point Depression

A case study prepared by Beyond Benign as part of the Green Chemistry in Higher Education program: A workshop for EPA Region 2 Colleges and Universities

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Summary:
This experiment involves the determination of the freezing point of a pure solvent and a solution of an unknown organic substance dissolved in the solvent. The molar mass of the unknown is calculated based on the freezing point depression of the solution.

Organic solvents are typically used in this experiment, such as 2-methyl-2-propanol or cyclohexane. Unknowns are used, such as naphthalene, p-nitrotoluene, or a similar halogenated aromatic compound. The organic solvents have high flammability and many of the unknowns have high human health hazards associated with them.

Reduction in waste and purchasing costs:

For every semester this reaction is implemented with 100 students, there are comparable purchasing and waste disposal costs and an overall ***reduction in waste from 0.5 gallons to 0 gallons***.* The greener version of the Molar Mass Determination also ***eliminates the use of 0.33 gallons of 2-methyl-2-propanol or cyclohexane and 0.2 lbs of one or more unknowns such as p-nitrotoluene, naphthalene, and 1-4-dibromobenzene***, all of which have human and/or aquatic hazards.

Background:
This case study is a result of an EPA Region 2 Source Reduction grant[1] titled *Green Chemistry in Higher Education: A Workshop for Region 2 Colleges and Universities*. The Green Chemistry in Higher Education workshop was carried out at Siena College on July 18-21, 2013. 29 faculty members participated from 20 different institutions in New York and New Jersey. The workshop consisted of three main focus areas: green chemistry case studies for lecture and course work, green chemistry laboratory exercises, and toxicology and environmental impact.

During the workshop participants were able to test a variety of greener laboratory exercises for introductory and organic chemistry courses. One of the labs was a “Greener Approach for Measuring Colligative Properties” for the general chemistry course.[2] Three faculty members indicated that they would be implementing the laboratory in their general chemistry courses in the 2013 – 2014 or 2014 – 2015 academic year: Abby O’Connor, The College of New Jersey (NJ), Elizabeth Sprague, RPI (NY), and Matthew Fountain, SUNY Fredonia (NY). Others also expressed initial interest and therefore more are expected to adopt the lab. The reduction in hazardous chemicals used and cost savings for this greener experiment are outlined in the following pages.

* The waste can be reduced to close to zero if the waste is used as starting materials for other laboratory experiments, such as biodiesel or soap making.

[1] **Disclaimer: Although the information in this document has been funded wholly or in part by the United States Environmental Protection Agency under assistance agreement X9-96296312 to Beyond Benign, it has not gone through the Agency’s publications review process and, therefore, may not necessarily reflect the views of the Agency and no official endorsement should be inferred.**

[2] Adapted from: McCarthy, S. M., and Gordon-Wylie, S. W., “A Greener Approach for Measuring Colligative Properties”, J. Chem. Ed., 82 (1), 2005, 116-119.

Additional Resources for Green Chemistry in General Chemistry and Beyond:

Greener Educational Materials (GEMs) Database (University of Oregon)

- Website: http://greenchem.uoregon.edu/gems.html
- Description: Searchable database with Green Chemistry educational materials uploaded by faculty members and educators world-wide
- Most curriculum is available for download (free-of-charge) or with primary literature information
- Google map of Green Chemistry educators

American Chemical Society's Green Chemistry Institute

- Website: www.acs.org/greenchemistry
- Description: Green Chemistry Resources for educators and students
- Experiments and Curriculum available for download
- List of ACS books on Green Chemistry

Green Chemistry Commitment

- Website: www.greenchemistrycommitment.org
- Description: A program of Beyond Benign to adopt Green Chemistry Learning Objectives in higher education.
- Case studies are available, university highlights, and curriculum resources

Beyond Benign

- Website: www.beyondbenign.org
- Description: Green Chemistry Curriculum available on-line (free-of-charge)
- Regional Outreach and Community Educational Events

GCEdNet - Green Chemistry Education Network

- Website: http://cmetim.ning.com/
- Description: A place where Green Chemistry educators share resources
- Blogs, discussions and chat rooms

University of Scranton Greening Across The Chemistry Curriculum

- Website: http://www.scranton.edu/faculty/cannm/green-chemistry/english/drefusmodules.shtml
- Description: Green Chemistry modules available for download
- Power point presentations, hand-outs available

Carnegie Mellon University Institute for Green Science

- Website: http://igs.chem.cmu.edu/
- Description: Green Chemistry modules available for download
- Power point presentations, hand-outs available

Traditional Experiment:

Colligative Properties laboratory exercises are commonly performed in general chemistry in order to introduce students to colligative properties and to use the properties to determine the molar mass of a substance. The experiment is typically performed with an organic solvent that has a melting point around room temperature, such as 2-methyl-2-propanol (25°C), or cyclohexane (6.5°C). The warming curve for the pure solvent is typically observed, followed by the introduction of an unknown compound. The freezing point depression is observed and can be measured to determine the molar mass of the unknown compound.

Molar Mass Determination by Freezing Point Depression *Traditional Experiment*

Chemicals avoided per class of 100 students:
1.25L (0.3 gal) 2-methyl-2-propanol or 500 mL (0.13 gal) cyclohexane
0.22 lbs of unknowns such as naphthalene, p-nitrotoluene and 1,4-dibromobenzene

Chemicals used and hazards:

The chemicals that are typically used in this experiment are listed below, along with a list of the hazards. The amounts are estimated based on a common procedure from one of the most widely used General Chemistry textbooks[3], along with a procedure from Monmouth University's General Chemistry II Laboratory Manual.

Table 1. Chemicals used, human health and aquatic toxicity data:

Chemical:	Amount per group of 2 students:	Flammability:*	Human health toxicity:[4]	Aquatic toxicity:[4]
2-methyl-2-propanol	25 mL (0.066 gal)	Flammable; NFPA Code: 3; Flash Point: 11°C	*Low toxicity* LD50 (oral, rat) 2,743 mg/kg; LD50 (dermal, rabbit) 2,000 mg/kg	*Low toxicity* LC50 (fish, 96 hr) 6,140 mg/l; EC50 (daphnia, 48 hr) 933 mg/l
Cyclohexane	10 mL (0.0026 gal)	Flammable; NFPA Code: 3; Flash Point: -20°C	*Causes CNS depression, drowsiness, dizziness,* Low acute toxicity, LD50 (oral, rat) 12,705 mg/kg; LC50 (inh, rat) 34,000 mg/l; LD50 (dermal, rabbit) 2,000 mg/kg	High toxicity: LC50 (fish, 96 hr) 4.53 mg/l; EC50 (daphnia, 48 hr) 0.9 mg/l; EC50 (algae, 72 hr) 3.4 mg/l
Naphthalene	2 g (0.0044 lb)	n/a	*High toxicity* IARC Group 2B: Possibly carcinogenic to humans LD50 (oral, rat) 490 mg/kg; LC50 (inh, rat) 340 mg/m3; LD50 (dermal, rabbit) 20,000 mg/kg	*High toxicity* LC50 (fish, 96 hr) 0.9-9.8 mg/l; LC50 (daphnia, 48 hr) 1-3.4 mg/l
p-nitrotoluene	2 g (0.0044 lb)	n/a	*Moderate toxicity* IARC Group 3: Not classifiable as to its carcinogenicity to humans. LD50 (oral, rat) 2,250 mg/kg; LC50 (inh, rat) 975 mg/m3	*High toxicity* LC50 (fish, 96 hr) 49.7 mg/l; EC50 (algae, 96 hr) 22 mg/l
1,4-dibromo-benzene	0.4 g (0.00088 lb)	n/a	*Moderate toxicity, causes respiratory irritation* LD50 (oral, mouse)3,120 mg/kg	*High toxicity* LC50 (fish, 96 hr) 0.68 mg/l

[3] "Colligative Properties: Freezing Point Depression and Molar Mass", Experiment 19 in Chemistry The Central Science Laboratory Experiments, 12th Edition, by Nelson, J.H., Kemp, K.C., and Stoltzfus, M., Pearson Education, 2012, p. 237-250.
[4] Human health and aquatic toxicity data was gathered from Globally Harmonized Safety Data Sheets, which can be obtained from Sigma-Aldrich [http://www.sigmaaldrich.com/united-states.html].
* NFPA (National Fire Protection Association) codes can be found here: http://en.wikipedia.org/wiki/NFPA_704#Red

Traditional Experiment, Continued:

The purchasing and waste disposal costs associated with this procedure are estimated in the following table. Purchasing costs were estimated based on prices available from Sigma-Aldrich:[5]

Total amounts of chemicals used and disposed of per class of 100 students:

- ***0.33 gal of 2-methyl-2-propanol or 0.13 gal of cyclohexane***
- ***0.22 lbs. naphthalene, p-nitrotoluene, or 1,4-dibromobenzene***
- ***Estimated 0.5 gallons of liquid waste****

Molar Mass Determination by Freezing Point Depression

Traditional Experiment

Volume of waste and purchasing and waste disposal costs per class of 100 students:

0.5 gallons of liquid waste

$52.98-$149.12 in purchasing and disposal costs

Table 2. Purchasing and waste disposal costs:

Chemical:	**Amount per 100 students:**	**Waste disposal cost**[6]	**Purchasing cost:**[5]	**Purchasing cost per 100 students:**	**Waste disposal cost per 100 students:**	**Total cost (per 100 students)**
2-methyl-2-propanol	1,250 mL (0.33 gal)	$11.27/gal	$100, 1L	$125.00	$3.72	$128.72
Cyclohexane	500 mL (0.13 gal)	$11.27/gal	$91.40, 1 L	$45.70	$1.47	$47.17
Naphthalene	100 g (0.22 lb)	$1.35/lb	$42.20, 1 kg	$4.20	$0.30	$4.50
p-nitrotoluene	100 g (0.22 lb)	$11.27/gal	$20.10, 100 g	$20.10	$0.30	$20.40
1,4-dibromobenzene	20 g (0.044 lb)	$1.35/lb	$28.80, 100 g	$5.76	$0.06	$5.82
TOTAL (per 100 students):	**0.13 - 0.33 gal and 0.04 - 0.22 lb**			**$49.90 - $145.10**	**$1.53 - $4.02**	**$52.98 - $149.12**

Total purchasing and waste disposal costs per class of 100 students:

- ***$49.90 - $145.10 in purchasing costs***
- ***$1.53 - $4.02 in waste disposal costs***
- ***$52.98 - $149.12 total cost***

* 0.5 gallons of liquid waste is estimated due to the solid waste (naphthalene, p-nitrotoluene, or 1-4-dibromobenzene) being dissolved in the solvent (i.e., cyclohexane) and therefore increasing the volume of the liquid waste.

[5] Sigma-Aldrich [http://www.sigmaaldrich.com/united-states.html, Accessed July 18, 2014].
[6] Waste disposal costs are based on the EPA Cost Calculator Tool [http://www.epa.gov/p2/pubs/resources/measurement.html#calc, accessed December 2014].

Molar Mass Determination by Freezing Point Depression
A Greener Approach

Volume of waste and purchasing and waste disposal costs per class of 100 students:
0-1.2 lbs. of waste*
$38.14 - $272.34 in purchasing and disposal costs

A Greener Approach:

The greener version of the colligative properties laboratory exercise uses fatty acids to measure the freezing point depression of a fatty acid as an unknown is added. The greener procedure has slightly higher purchasing costs associated with the materials (range of $36.50 - $270.70 versus $49.90 - $145.10 for the traditional procedure), depending on which of the unknown fatty acids is used (see tables in the following pages). However, the waste can be reduced to essentially zero since the fatty acids can be used as starting materials for other laboratory exercises, such as a biodiesel experiment, making soap, or making wax.

Dr. Matthew Fountain at SUNY Fredonia has implemented a greener version of the typical colligative properties laboratory experiment that uses fatty acids instead of organic solvents and halogenated aromatic compounds.[1] The fatty acids used in the greener version of the laboratory exercise can then be used as starting materials for other laboratory exercises, such as making biodiesel, soap, or wax. The freezing point is first observed for pure stearic acid. An unknown fatty acid is then added into the pure fatty acid and the freezing point depression is observed.

Table 3. Chemicals used, human health and aquatic toxicity data:

Chemical:	**Amount per group of 2 students:**	**Human health toxicity:**[4]	**Aquatic toxicity:**[4]
stearic acid	9 g (0.02 lb)	*Low toxicity* LD50 (oral, rat) > 2,000 mg/kg; LD50 (dermal, rabbit) > 5,000 mg/kg	*Low toxicity*
lauric acid	2 g (0.0044 lb)	*Moderate toxicity* LD50 (oral, rat) > 5,000 mg/kg; Can cause eye damage	*High toxicity* LC50 (fish, 96 hr) 5 mg/l; LC50 (daphnia, 48 hr) 3.6 mg/l
palmitic acid	2 g (0.0044 lb)	*Low toxicity* LD50 (oral, rat) > 5,000 mg/kg	*High toxicity*, LC50 (fish, 96 hr) > 1,000 mg/l; EC50 (daphnia, 48 hr) > 4.8 mg/l
myristic acid	2 g (0.0044 lb)	*Low toxicity* LD50 (oral, rat) > 10,000 mg/kg; Can cause skin irritation	*Low toxicity*

* The volume of waste can be reduced to close to zero if the fatty acids are used as starting materials for other laboratory experiments.

Molar Mass Determination by Freezing Point Depression

A Greener Approach

Volume of waste and purchasing and waste disposal costs per class of 100 students:

0-1.2 lbs. of waste*

$38.14 - $272.34 in purchasing and disposal costs

A Greener Approach, Continued:

The purchasing and waste disposal costs associated with this procedure are estimated in the following table. Purchasing costs were estimated based on prices available from Sigma-Aldrich:[5]

Total amounts of chemicals used and disposed of per class of 100 students:

- ***1 lb of stearic acid***
- ***0.22 lbs of lauric acid, palmitic acid or myristic acid***
- ***1.22 lbs of fatty acids total***
- ***0-1.22 lbs of waste generated****

Table 4. Purchasing and waste disposal costs:

Chemical:	**Amount per 100 students:**	**Waste disposal cost**[6]	**Purchasing cost:**[5]	**Purchasing cost per 100 students:**	**Waste disposal cost per 100 students:**	**Total cost (per 100 students)**
stearic acid	450 g (0.99 lb)	$1.35/lb	$66.00, 1 kg	$29.70	$1.34	$31.04
lauric acid	100 g (0.22 lb)	$1.35/lb	$68.00, 1 kg	$6.80	$0.30	$7.10
palmitic acid	100 g (0.22 lb)	$1.35/lb	$241.00, 100 g	$241.00	$0.30	$241.30
myristic acid	100 g (0.22 lb)	$1.35/lb	$64.60, 100 g	$64.60	$0.30	$64.90
TOTALS:*	**1.2 lbs.**			$36.50 - $270.70	$1.64	$38.14 - $272.34

Total purchasing and waste disposal costs per class of 100 students:

- ***$36.50-$270.70 in purchasing costs***
- ***$1.64 in waste disposal costs***
- ***$38.14 - $272.34 total cost***

* The volume of waste can be reduced to close to zero if the fatty acids are used as starting materials for other laboratory experiments.

Molar Mass Determination by Freezing Point Depression
Summary

***Waste comparison**:*
Eliminates use of organic solvents and halogenated compounds
Cost comparison:
Variable purchasing costs depending on materials used

Traditional Experiment Summary:

Total amounts of chemicals used and disposed of per class of 100 students:
- ***0.33 gal of 2-methyl-2-propanol or 0.13 gal of cyclohexane***
- ***0.22 lbs. naphthalene, p-nitrotoluene, or 1,4-dibromobenzene***
- ***Estimated 0.5 gallons of liquid waste***

Total purchasing and waste disposal costs per class of 100 students:
- ***$49.90 - $145.10 in purchasing costs***
- ***$1.53 - $4.02 in waste disposal costs***
- ***$52.98 - $149.12 total cost***

A Greener Approach Summary:

Total amounts of chemicals used and disposed of per class of 100 students:
- ***1 lb of stearic acid***
- ***0.22 lbs of lauric acid, palmitic acid or myristic acid***
- ***1.22 lbs of fatty acids total***
- *0-1.22 lbs of waste generated*

Total purchasing and waste disposal costs per class of 100 students:
- ***$36.50-$270.70 in purchasing costs***
- ***$1.64 in waste disposal costs***
- *$38.14 - $272.34 total cost*

Conclusions:

The greener version for measuring colligative properties of a material shows a reduction in the hazards associated with the materials, however there are some hazards that remain for the fatty acids (i.e., lauric acid has moderate human toxicity and high aquatic toxicity). If the instructor limits the unknown to the safer fatty acid, myristic acid, then the hazards will be low for the materials. The most expensive material for the greener laboratory exercise is palmitic acid, therefore, if that unknown is avoided, then the purchasing costs can be kept low. If the instructor uses stearic acid and myristic acid, then the following benefits can be realized:

- Purchasing costs of $94.30 for 100 students
- Waste disposal costs of $1.64 for 1.2 pounds of waste per 100 students (However, this can be dropped to almost $0.00 and no waste if the waste fatty acids are used as starting materials for other experiments).
- The elimination of the organic solvents and halogenated organic compounds used in the traditional laboratory exercise, along with the associated waste.

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