

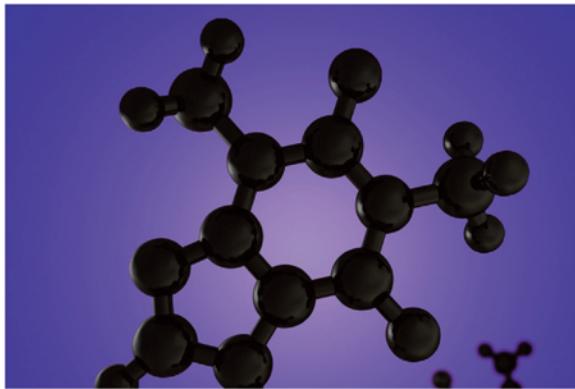


Food Safety Action Plan

REPORT

2010-2011 Targeted Surveys

Chemistry



Microcystins in Bottled Water

TS-CHEM-10/11

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Executive Summary

The Food Safety Action Plan (FSAP) aims to modernize and enhance Canada's food safety system. As part of the FSAP enhanced surveillance initiative, targeted surveys are used to test various foods for specific hazards.

The main objective of the Microcystins in Bottled Water targeted survey was to:

- generate baseline data regarding microcystin levels in bottled water available on the Canadian retail market

Cyanobacteria, also known as blue-green algae, are commonly found in surfacewater algae blooms or blue-green scums¹. Cyanobacteria can produce a group of hepatotoxins called microcystins, which are the most common cyanobacterial toxins found in water². The presence of microcystins in water may result in an unpleasant taste and odour, and may cause illness in people consuming these toxins. It is also possible that extended exposure to low levels of these cyanobacterial hepatotoxins may have long-term or chronic effects in humans³. Microcystins have been identified as a significant threat to freshwater supplies by the World Health Organization (WHO)⁴. Microcystins may be present in bottled waters available at retail if they are present in the water source used to manufacture the final bottled product and if the water is inadequately treated.

Health Canada has proposed a maximum limit of 1.5 micrograms per litre ($\mu\text{g/L}$) for total microcystins in drinking water⁵.

The 2010-2011 Microcystins in Bottled Water survey targeted both imported and domestic bottled water (unflavoured, carbonated and non-carbonated) packaged in plastic and glass bottles. Three hundred samples were collected in various locations across Canada. Each sample was analyzed for the most commonly-occurring hepatotoxins, microcystins and nodularin.

All 300 samples tested did not contain detectable levels of microcystins or nodularin. Thus, 100% of the samples tested were compliant with Health Canada's proposed maximum limit for total microcystins in drinking water.

1 Introduction

1.1 Food Safety Action Plan

In 2007, the Canadian government launched a five-year initiative in response to a growing number of product recalls and concerns about food safety. This initiative, called the Food and Consumer Safety Action Plan (FCSAP), aims to modernize and strengthen the food safety regulatory system. The FCSAP initiative unites multiple partners in ensuring safe food for Canadians.

The CFIA's Food Safety Action Plan (FSAP) is one element of the government's broader FCSAP initiative. The goal of FSAP is to identify risks in the food supply, limit the possibility that these risks occur, improve import and domestic food controls, and identify food importers and manufacturers. FSAP also looks to verify that the food industry is actively applying preventive measures and that there is a rapid response when/if these measures fail.

Within FSAP, there are twelve main areas of activity, one of which is risk mapping and baseline surveillance. The main objective of this area is to better identify, assess, and prioritize potential food safety hazards through risk mapping, information gathering, and testing foods from the Canadian marketplace. Targeted surveys are one tool used to test for the presence and level of a particular hazard in specific foods. Targeted surveys are largely directed towards the 70% of domestic and imported foods that are regulated solely under the *Food and Drugs Act and Regulations*, and are generally referred to as non-federally registered commodities.

1.2 Targeted Surveys

Targeted surveys are pilot surveys used to gather information regarding the potential occurrence of chemical residues in defined commodities. The surveys are designed to answer specific questions; therefore, unlike monitoring activities, testing of a particular chemical hazard is targeted to commodity types and/or geographical areas.

Due to the vast number of chemical hazards and food commodity combinations, it is not possible, nor should it be necessary, to use targeted surveys to identify and quantify all chemical hazards in foods. To identify food-hazard combinations of greatest potential health risk, the CFIA uses a combination of scientific literature, media reports, and/or a risk-based model developed by the Food Safety Science Committee (FSSC).

Microcystins (and nodularin) are potent liver toxins, and can cause unpleasant tastes and odours in drinking water. The major route for human exposure is through the ingestion of contaminated drinking water⁶. Microcystins may be present in bottled waters available at retail if they are present in the water source used to manufacture the final bottled product, and if the water has not undergone adequate treatment to remove these microcystins prior to use.

Bottled water is a widely-consumed beverage for Canadians, comprising 9% of Canada's commercial beverage market share in 2006⁷. Various drinking water sources are used in the manufacture of bottled water, including natural artesian springs, glacial run-off, or municipal water supplies. Conventional water treatment processes (e.g. filtration/flocculation) are ineffective in eliminating microcystins^{4,8,9}. Advanced water treatment options (ozonation, UV photolysis) show various degrees of success in removing microcystins¹⁰. Currently, these treatments are more complex (often requiring more time and/or more steps in the process), expensive, and are less common in water treatment facilities.

Currently, little to no literature or studies have been published that examine bottled water for microcystins or nodularins. Therefore, it was deemed appropriate to generate baseline data and determine what, if any, level of microcystins exist in bottled water available to Canadian consumers.

1.3 Acts and Regulations

The *Canadian Food Inspection Agency Act* stipulates that the CFIA is responsible for enforcing restrictions on the production, sale, composition, and content of foods and other food products as outlined in the *Food and Drugs Acts and Regulations* (FDAR).

Health Canada (HC) establishes the limits (which are health-based) of contaminants and pesticide residues in food. Tolerances are established as a risk management tool, and generally only for foods that significantly contribute to the total dietary exposure. Pre-packaged (bottled) waters are regulated as a food and therefore are subject to all the provisions of the FDAR.

Health Canada has proposed a maximum acceptable concentration (guideline) of 1.5 µg/L for total microcystins in drinking water⁵. This proposed Canadian guideline is similar to existing guidelines for microcystins in drinking water in Australia, New Zealand, France, and the World Health Organization. Some of these guidelines are provisional or proposed due to the limited amount of toxicological data available. Microcystin-LR (the form of microcystins first isolated and most studied) is often used as an indicator for other forms of microcystins in water¹⁶. Relative to microcystins, nodularin has similar toxicity but occurs infrequently, and is usually seen in brackish waters or seawater¹¹. There are currently no guidelines for nodularin in drinking water in Canada. Please refer to **Table 1** below for guideline details.

Table 1: Guidelines for Drinking Water – Maximum Acceptable Microcystin Concentration

Country	Proposed or Established Microcystin Guideline ¹²	Notes
Canada*	1.5 µg/L	Proposed Guideline -Total microcystins in drinking water (based on the toxicity of microcystin-LR)
United States	No current federal guidelines	
World Health Organization (WHO)	1 µg/L	Provisional Guideline - Microcystin-LR
Australia	1.3 µg/L	Total microcystins expressed as microcystin-LR toxicity equivalents
France, Czech Republic, Poland, New Zealand	WHO Provisional Guideline	Microcystin-LR
Brazil	1 µg/L	Total microcystins

*note: The Canadian proposed acceptable microcystin concentration in recreational water (generally, untreated water used by Canadians for recreational purposes that may or may not be used as a raw drinking water source also) is ≤ 20 µg/L¹¹

2 Microcystins in Bottled Water

2.1 Microcystins Overview

Cyanobacteria are commonly found in surfacewater algae blooms or scums¹. Cyanobacteria can produce hepatotoxins, namely microcystins (and nodularin), which are the most common cyanobacterial toxins found in water². Microcystins have been identified as a significant threat to freshwater supplies⁴. Microcystins may also impart an unpleasant taste and odour to water. Not all blue-green algae produce toxins¹³ and some cyanobacteria produce different toxins altogether (i.e. neurotoxins)¹⁴.

Microcystins have more than 60 congeners (variant forms)¹⁵, which differ based on the combination of amino acids found in the toxin. Microcystin-LR is the most common and the most studied form of the toxin¹⁶. Other microcystin forms include microcystin-RR, -YR, -LY, and -LA. Nodularin hepatotoxins are similar but contain fewer amino acids⁴.

Microcystin formation is difficult to predict, as the occurrence of algal blooms is highly variable from year-to-year, and the mechanisms that promote algal growth are complex¹⁷. Additionally, it is difficult to predict whether these blooms will be harmful¹³. The greatest release of toxins from cyanobacteria cells comes from cell death rather than excretion⁶. In Canada, cyanobacterial blooms tend to appear in summer months and are prevalent in the prairies. The degree to which these blooms occur across Canada is

unknown, although it is presumed that the duration of exposure to the blooms and their associated microcystins would be shorter in Canada than in those with milder climates³. Several studies in multiple provinces in the 1990s showed frequent detection of microcystins in raw water supplies, municipal water supplies, dugouts used for domestic and/or livestock water consumption, and recreational use sites, although these levels were well below the water quality guidelines¹⁸. The relationship between these occurrences and the residual presence of microcystins in bottled water is unknown.

Human exposure to microcystins primarily occurs through consumption of contaminated drinking water⁴. People can be exposed to microcystins by accidental ingestion of contaminated water used for recreational activities. Exposure may also be through consumption of algal health products or by eating the liver of fish taken from water with blue-green algae blooms, although these routes are less likely³. Acute exposure to high levels of microcystins may result in stomach cramps, vomiting, diarrhoea, fever, headache, joint and muscle pain, weakness, and skin, eye, and throat irritation¹⁹. Long-term exposure to microcystins, which are hepatotoxins, may cause liver damage³.

Eliminating microcystins from contaminated water is difficult. Microcystins are typically stable at high temperatures and at extreme pH^{8,9}. Microcystins are not degraded by boiling, sedimentation, sand filtration, chlorination, or other conventional water treatments. Extreme treatment conditions are necessary to degrade microcystins over a period of weeks⁴. Applying a high level of chlorine or ozonation is the best way to treat drinking water contaminated with microcystins²⁰. While treating surface waters exhibiting blue-green algae blooms with ozone or chlorination will kill the cyanobacteria, it will also release microcystins into the water⁶.

2.2 Microcystins in Bottled Water

Both domestic and imported bottled water can come from a variety of sources. These include natural aquifers, springs, glacier run-off, and municipal water supplies²¹, and these sources could potentially be contaminated with microcystins. As normal water treatment techniques do not readily degrade microcystins, these toxins, if present in the source water, could remain in the finished bottled water product.

In 2006, 29% of Canadian households drank primarily bottled water as opposed to tap water²². The per capita consumption of bottled water in Canada was 66 L, and that volume had been steadily increasing over the previous decade⁷. Bottled water, of both domestic and imported origin, is consumed in large quantities by Canadians and is a significant trade commodity.

2.3 Rationale

To our knowledge, only one study on levels of microcystins in bottled water has been published. This single Italian study analysed two domestic bottled spring water samples for the presence of various microcystins and nodularin, and did not detect cyanotoxins²³. The FSAP 2010-2011 Microcystin Targeted Survey was used to determine whether microcystin contamination exists in bottled water sold in Canada.

2.4 Targeted Survey Sample Distribution

In the 2010-2011 Microcystins in Bottled Water targeted survey, a total of 300 samples were collected at grocery and specialty stores in 10 Canadian cities (Halifax, Saint John, Ottawa, Toronto, Montréal, Québec City, Calgary, Saskatoon, Vancouver, and Winnipeg). The samples included 110 carbonated waters, 17 mineral waters, 113 spring waters, and 60 waters for which type was not verifiable (“Other”). Bottled water with added flavour or those supplemented with vitamins were not sampled.

2.5 Method Details

In 2008-2009, the CFIA lab developed and validated an LC-MS/MS method for the analysis of microcystins in commercially bottled water. The method analyzed for microcystin-LR, -YR, -RR, -LA, and nodularin. A multi-point calibration was used, ranging from 0.05 to 10 µg/L. Sample spikes, quality control samples, and negative controls were also used. The limits of detection (LOD) ranged from 0.02 to 0.1 µg/L, and the limits of quantitation (LOQ) ranged from 0.05 to 0.5 µg/L. This method was used for analysis in the 2010-2011 survey.

2.6 Limitations

The microcystin survey was designed to provide a snapshot of the levels of microcystins in bottled waters available to Canadian consumers. In comparison to the total number of bottled waters available in the Canadian market, 300 samples represent a small fraction of products available to consumers. Therefore, care must be taken when interpreting and extrapolating these results. Regional differences, year-to-year trends, impact of product shelf life, or cost of the commodity on the open market are not examined in this survey.

3 Results

This survey sampled imported and domestically produced bottled water, including carbonated and non-carbonated waters, packaged in glass and plastic. A variety of artesian, spring, glacial, and municipal-source waters were sampled for this survey. See Figure 1 below for distribution of bottled water sample types. Both microcystins (microcystin-LR, -YR, -RR, -LA and a sum total) and nodularin were analyzed and reported. None of the samples tested contained detectable levels of microcystins or nodularin.

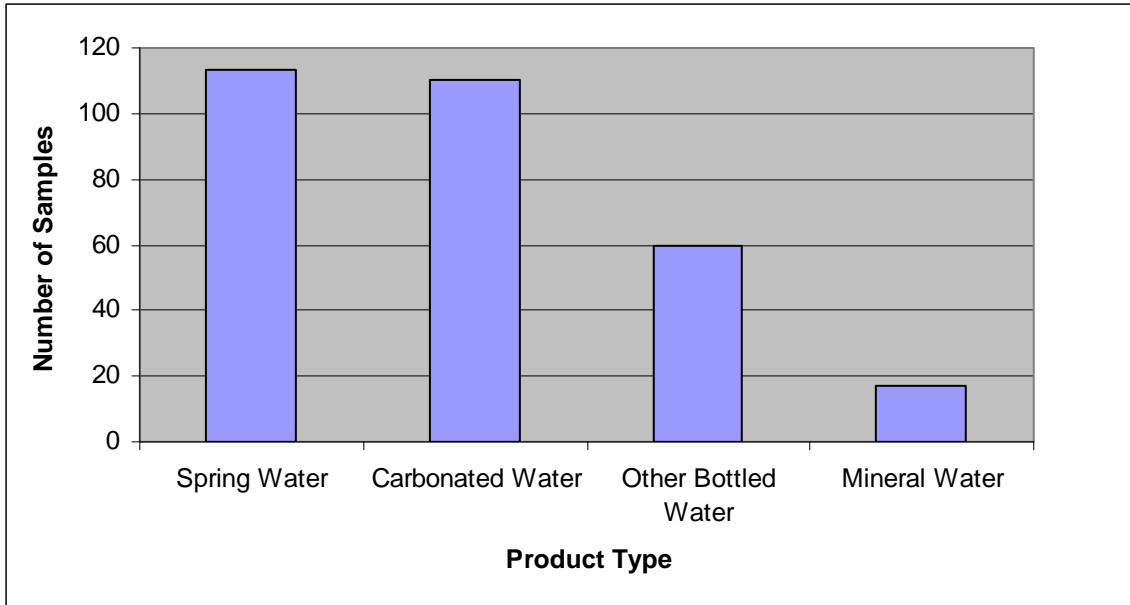


Figure 1: Type of Bottled Water Sampled

Of the 300 samples analyzed, 130 samples were of domestic origin and 170 were imported. The distribution of samples with respect to country of origin is depicted in Figure 2. This figure also shows the water sample types for each country of origin, specifically carbonated, mineral, spring, and those waters for which type was not verifiable (other).

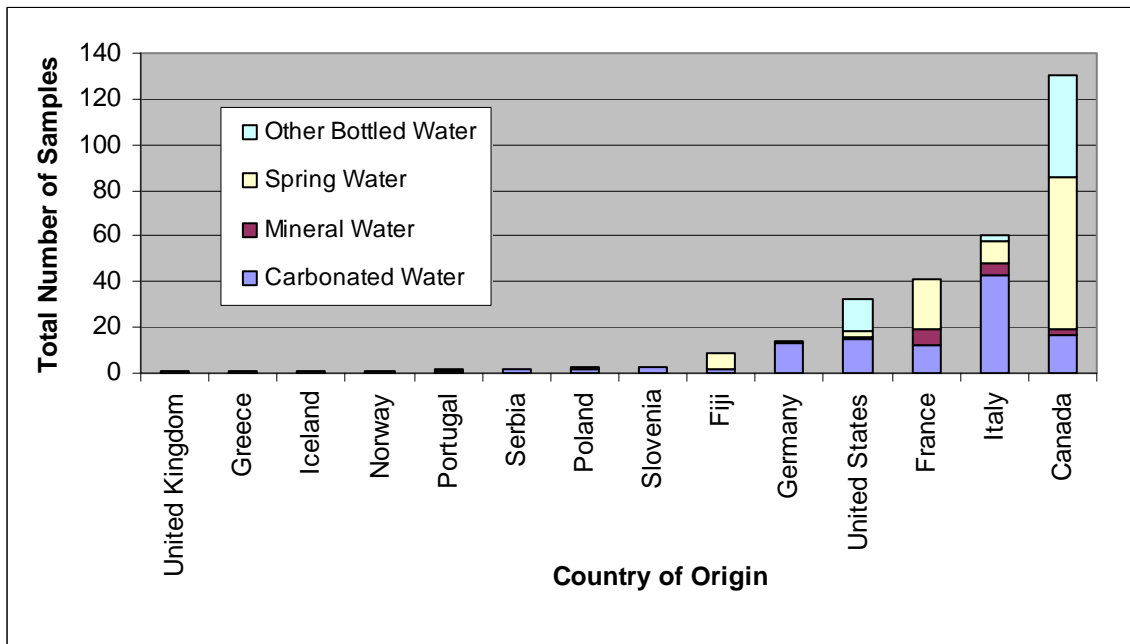


Figure 2: Country of Origin and Type of Bottled Water Sampled

4 Discussion

No residues of microcystins or nodularin were detected in any bottled water sample tested. The compliance rate with Canada's proposed guideline of 1.5 µg/L of total microcystins in drinking water was 100%. The lack of positive results may be due to the water sources used for these products. These sources could be cold, faster-moving, pre-treated, or lean in nutrients that would support the growth of algae (e.g. glacial run-off). Algae tend to thrive in waters that are nutrient-rich, shallow, slow-moving or still, and relatively warm, and generally do not persist through the winter months in temperate climates like Canada (although the hepatotoxins may remain). If there were few algal blooms in the water source, it is less likely that microcystins will be present in the final bottled product.

5 Conclusion

Three hundred bottled water samples were collected and analysed for microcystins and nodularin. These toxins were not detected in any sample. The limits of detection for the microcystins and nodularin tested herein ranged from 0.02 to 0.1 µg/L. All samples were compliant with Canada's proposed guideline for microcystins in drinking water.

References

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- ¹ United States Centers for Disease Control and Prevention (CDC). "Harmful Algal Blooms (HABs)-Facts about Cyanobacteria and Cyanobacterial Harmful Algal Blooms". Undated. *Web.* <http://www.cdc.gov/hab/cyanobacteria/facts.htm>
 - ² Kotak, B.G., Zurawell, R.W., Prepas, E.E., and Holmes, C.F.B. "Microcystin-LR concentration in aquatic food web compartments from lakes of varying tropic status". *Canadian Journal of Fisheries and Aquatic Science.* 53 (1996): 1974-85.
 - ³ Health Canada. "Blue-green Algae (Cyanobacteria) and their Toxins". 2008. *Web.* <http://www.hc-sc.gc.ca/ewh-semt/pubs/water-eau/cyanobacter-eng.php>
 - ⁴ World Health Organization. "Toxic Cyanobacteria in Water: A guide to their public health consequences, monitoring and management". 1999. *Web.* http://www.who.int/water_sanitation_health/resourcesquality/toxcyanbegin.pdf
 - ⁵ Health Canada. "Guidelines for Canadian Drinking Water Quality – Summary Table". December 2010. *Web.* http://www.hc-sc.gc.ca/ewh-semt/pubs/water-eau/2010-sum_guide-res_recom/index-eng.php
 - ⁶ World Health Organization. "Cyanobacterial toxins: Microcystin-LR in Drinking-water". 2003. *Web.* http://www.who.int/water_sanitation_health/dwq/chemicals/cyanobactoxins.pdf
 - ⁷ Agriculture and Agri-Food Canada. "The Canadian Bottled Water Industry". March 2009. *Web.* <http://www4.agr.gc.ca/AAFC-AAC/display-afficher.do?id=1171644581795>
 - ⁸ Harada, K.I., Tsuji, K., Watanabe, M.F., Kondo, F. "Stability of microcystins from cyanobacteria. III. Effect of pH and temperature". *Phycologia*, 1996. 35(6): p. 83-88

- ⁹ Tsuji, K., Watanuki, K., Kondo, F., Watanabe, M.F., Suzuki, S., Nakazawa, H., Suzuki, M., Uchida, H., and Harada, K.I. "Stability of microcystins from cyanobacteria--II. Effect of UV light on decomposition and isomerization". *Toxicon*, 1995. 33(12): p. 1619-31
- ¹⁰ Svrcek, Clark and Smith, Daniel W. "Cyanobacteria toxins and the current state of knowledge on water treatment options: a review". *Journal of Environmental Engineering and Science*. 2004. 3:(3) 155-185
- ¹¹ Health Canada. "Guidelines for Canadian Recreational Water Quality – Document for Public Comment". September 2009. *Web*. http://www.hc-sc.gc.ca/ewh-semt/consult/2009/water_rec-eau/draft-ebauche-eng.php
- ¹² Burch, Micheal D. "Effective doses, guidelines & regulations." *Adv Exp Med Biol*. 2008; 619:831-53.
- ¹³ Environment Canada. "Cyanobacteria and other Harmful Algal Blooms". 2011. *Web*. <http://www.ec.gc.ca/inre-nwri/default.asp?lang=En&n=99B93178-1>
- ¹⁴ Environment Canada. "Threats to Sources of Drinking Water and Aquatic Ecosystem Health in Canada". 2008. *Web*. <http://www.ec.gc.ca/inre-nwri/default.asp?lang=En&n=235D11EB-1&offset=3&toc=show>
- ¹⁵ van Apeldoorn, M.E., van Egmond, H.P., Speijers, G.J.A., and Bakker, G.J.I. "Toxins of Cyanobacteria". *Molecular Nutrition and Food Research*. 51 (2007):7-60.
- ¹⁶ Jacoby, J.M., Collier, D.C., Welch, E.B., Hardy, F.J., and Crayton, M. "Environmental factors associated with a toxic bloom of *Microcystis aeruginosa*". *Canadian Journal of Fisheries and Aquatic Science*. 57 (2000): 231-40.
- ¹⁷ Agriculture and Agri-Food Canada. "Algae, Cyanobacteria, and Water Quality". September 2007. *Web*. <http://www4.agr.gc.ca/AAFC-AAC/display-afficher.do?id=1189714026543&lang=eng>
- ¹⁸ Health Canada. "Cyanobacterial Toxins – Microcystin-LR". April 2002. *Web*. http://www.hc-sc.gc.ca/ewh-semt/pubs/water-eau/cyanobacterial_toxins/toxin-toxines-eng.php
- ¹⁹ Toxicology Data Network (TOXNET). U.S. National Library of Medicine. "Microcystin-LR". December 2009. *Web*. <http://toxnet.nlm.nih.gov/cgi-bin/sis/search/a?dbs+hsdb:@term+@DOCNO+7751>
- ²⁰ World Health Organization. "New section for Microcystin-LR background document". 2007. *Web*. http://www.who.int/water_sanitation_health/dwq/chemicals/microcystin_sections.pdf
- ²¹ Health Canada. "Frequently Asked Questions about Bottled Water". May 2009. *Web*. http://www.hc-sc.gc.ca/fn-an/securit/facts-faits/faqs_bottle_water-eau_embouteillee-eng.php
- ²² Statistics Canada. "The bottle or tap: Sources of drinking water (2006)". November 2008. *Web*. <http://www.statcan.gc.ca/pub/11-526-x/2007001/5100146-eng.htm>
- ²³ Ferretti, E., Lucentini, L., Veschetti, E., Bonadonna, L., Stamatii, A., Turco, L., Ottaviani, M. "Screening and identification of unknown contaminants in water destined to human consumption: A case study". *Microchemical Journal*. 85.1 (2007): 57-64.