

National Institute of Standards and Technology
Dietary Supplement Laboratory Quality Assurance Program
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January 21, 2022

Dear Colleagues,

Thank you for your interest and/or past participation in NIST Quality Assurance Programs (QAPs). A call for participants in Exercise 1 of the Dietary Supplement Laboratory Quality Assurance Program (DSQAP) is now open. DSQAP is designed to assist laboratories in the development and validation of new analytical methods, in improving the quality of their analytical measurements, and in supporting compliance with a number of federal regulations enforced by the US FDA, USDA, and other international bodies. Exercise 1 will offer opportunities for participants to measure mass fractions of nutritional and toxic elements, water- and fat-soluble vitamins, phenolic content, contaminants, proximates, and withanolides in dietary supplement ingredients and finished products. There will also be a focus on the compositional testing of kelp materials, sponsored by World Wildlife Fund. Laboratories may elect to participate in some or all studies (e.g., only nutritional elements and water-soluble vitamins) or report only selected analytes (e.g., calcium and sodium, but not iron and potassium), as applicable to the work done in their laboratories.

Participation in DSQAP is free of charge with the support of NIH-Office of Dietary Supplements (ODS), although participants will be required to pay for the cost of sample shipment by providing NIST with a shipping account number using UPS, FedEx (including TNT), or DHL. International participants must provide an import shipping account number, if applicable. Participants are responsible for all incurred shipping charges, including those that may result from shipments being returned to NIST because of customs clearance issues. In no cases are participants paid to participate in DSQAP.

Instructions for sign up for new and existing NIST QAP participants are attached. **Registration will open on January 24, 2022 and will close on March 4, 2022. Samples will be distributed in late March/early April 2022.**

We hope that within this exercise you will find studies that are of interest and use to your laboratory. Future DSQAP studies may address additional areas such as authenticity and microbial contaminants, as needs are identified. If you have any suggestions for studies or would like more information regarding DSQAP and other NIST QAPs, please send your request to QAPHUB@nist.gov or visit <https://qa.nist.gov>.

Please let us know if you have questions. We look forward to your participation in this and future DSQAP exercises.

Best regards,
DSQAP Team

Dietary Supplement Laboratory Quality Assurance Program (DSQAP)

Exercise 1 – Spring 2022

Compositional Testing of Kelp

With the rapid growth of the global seaweed and kelp farming industry in particular, reference materials and appropriate analytical techniques are critical for the characterization of product composition. Kelp has been a traditional part of the diet in Asian cultures and is increasing in popularity among the global population for human consumption and animal feeds. The goals of these studies are to determine discrepancies across the industry, narrow focus to the methodologies that need improved standardization, and connect the farming industry with the analytical testing community. Studies including kelp samples are sponsored in part by World Wildlife Fund (WWF). Laboratories can sign up for any of the studies for which they have capabilities and interest.

Nutritional and Toxic Elements

Kelp may serve as an important source of essential nutrients, but accumulation of elements depends on both the bioavailability of these elements in the environment and the uptake capacity of each species of these brown algae. Besides varying between species, elemental concentrations in kelp may vary by season, growing stage, depth of cultivation, location of origin, and genetics. This study will compare measurements of selected nutritional elements and toxic elements. Some elements are essential for the body to function properly, but both deficiency and excess can lead to potential negative health outcomes. For example, high sodium intake and iodine disorders are still major worldwide public health issues. Consumers limiting Na intake to lower health risks combined with the increased use of designer salts has led to decreased iodine intake, as iodized salt has traditionally been a common source of iodine in most diets. Kelp accumulates iodine and thus could be a potential source of iodine for consumers. However, these macroalgae may also uptake toxic elements. This may result in contamination and consumption of contaminated foods, which can cause illness, impairment, or, at very high doses, death. Participants will be asked to use their laboratory's routine measurement technique(s) for the determination of the selected elements of interest in kelp.

Analytes

Ca, Cr, I, K, Mg, Na, S, Se
tAs/iAs, Cd, Pb, Hg, Cu, Zn

Samples

Kelp

Water- and Fat-Soluble Vitamins

Kelp has been studied for its vitamin composition and both vitamin B₃ and vitamin K₁ are commonly found at nutritionally significant levels. Vitamin B₃ (also known as niacin) is involved in cellular metabolism, cell signaling, DNA production and repair, and has antioxidant properties. Symptoms of niacin deficiency include skin rash or discoloration, constipation or diarrhea, fatigue, and memory loss. Even though niacin deficiency is rare in most Western countries, people who are malnourished are at risk. Vitamin K is a group of compounds (vitamin K₁ and K₂) that play a key role in clotting of blood, preventing excessive bleeding. Vitamin K deficiencies are rare in adults but are very common in newborn infants, and a single injection of vitamin K for newborns is standard. Vitamin K has also been used to counteract an overdose of the blood thinner coumadin. Vitamin K₁ is the form obtained from leafy greens and some other vegetables, and reports suggest that *cis*- and *trans*- isomers of vitamin K₁ may have different bioavailabilities. Participants will be asked to use their laboratory's routine measurement technique(s) for the determination of vitamin B₃ (as niacin and niacinamide) and vitamin K₁ (as total phylloquinone, *cis*-phylloquinone, and *trans*-phylloquinone) concentrations in kelp.

Analytes

Vitamin B₃, Vitamin K₁

Samples

Kelp

Total Phenolic Content

Polyphenol-rich diets have been linked to many health benefits. Polyphenols are a class of bioactive compounds found in kelp, which may contribute bioactive profiles. Although many approaches exist for determination of total phenolic content, this study will focus on the measurement of gallic acid and gallic acid equivalents in several different kelp materials. Participants will be asked to determine gallic acid and gallic acid equivalents in kelp and green tea. This study will also include a component for multi-laboratory validation of the AOAC *Official First Action Method* 2017.13 for the measurement of total phenolic content and is also sponsored in part by the National Institutes of Health Office of Dietary Supplements (NIH-ODS).

Analytes	Samples
Gallic acid, gallic acid equivalents	Kelp, Green Tea

Proximates, Dietary Fiber, and Starch

Variations between species, growing season, or growth stage may affect the nutritional make up of kelp. Proximates make up the macronutrient profile of foods and their measurement is critical for crop designation and health, as well as understanding the impact of macronutrient consumption on human health. Fiber and starch are also important product characteristics for both nutritional and industrial use aspects. Participants will be asked to use their laboratory's routine measurement technique(s) for the determination of ash, protein, carbohydrates, fat (crude fat and fatty acids), calories, solids, dietary fiber, and starch in kelp.

Analytes	Samples
ash, protein, carbohydrates, fat (crude fat and fatty acids), calories, solids, dietary fiber, starch	Kelp

Organic Contaminants

Algae are known to be able to take up chemical compounds in their environment, and consumer products must be free from high levels of harmful contaminants. Perfluoroalkyl substances (PFAS) are a class of industrial compounds used in a variety of processes and consumer products and exposure has become a major public health concern due to potential adverse health effects. PFAS are highly resistant to degradation and can persist in the environment and the body for years. Standardization and harmonization of analytical measurement techniques are essential for the detection of PFAS for consumer safety and for the association of PFAS concentrations with exposure sources and health outcomes. Participants will be asked to use their laboratory's routine measurement technique(s) for the determination of select PFAS concentrations in kelp.

Analytes	Samples
Select PFAS Compounds	Kelp

B Vitamins in Meal Replacement Supplements

B vitamins are a group of water-soluble compounds important for maintaining good health and well-being. These vitamins impact energy levels, brain function, and cell metabolism as they play roles in converting food into energy, creating new blood cells, and maintaining healthy cells throughout the body. Vitamin B deficiencies have been linked to anemia, depression, fatigue, muscle weakness, and poor memory. Meal replacement supplements contain many nutritional compounds, including water-soluble vitamins. Products in both powdered and ready-to-consume forms are available on the market and have identical labeling requirements, but the matrix complexities and differences can pose measurement challenges for analytical testing laboratories. A goal for this study is to determine any discrepancies and/or analytical challenges for the measurements of B vitamins across the testing community, using similarly promoted consumer products with different sample preparation requirements. Participants will be asked to use their laboratory's routine measurement technique(s) for the determination of thiamin (B₁), riboflavin (B₂), niacin (B₃), pantothenic acid (B₅), vitamin B₆, biotin (B₇), folic acid (B₉), and Vitamin B₁₂ concentrations in both liquid and powdered meal replacement formulations. This study is sponsored in part by the National Institutes of Health Office of Dietary Supplements (NIH-ODS).

Analytes

thiamin (B₁), riboflavin (B₂), niacin (B₃),
pantothenic acid (B₅), vitamin B₆, biotin (B₇),
folic acid (B₉), vitamin B₁₂

Samples

Liquid and Powdered Meal Replacement
Formulations

Withanolides in Ashwagandha

Ashwagandha (*Withania somnifera*, fam. Solanaceae) is highly regarded in traditional medicines and increasingly popular as a dietary supplement. Along with a high alkaloid content, ashwagandha contains withanolides, which are naturally occurring plant-based steroids that are purported to improve cognitive function and have anti-inflammatory and immune-boosting properties. Reference methods and materials are needed to substantiate product labels and investigate health claims. A goal for this study is to provide ashwagandha materials to determine any community discrepancies and/or analytical challenges in the analysis of withanolides. This study will also include a component for multi-laboratory validation of the AOAC *Official First Action Method* 2015.17 and move towards the development of an ashwagandha reference material which will allow the industry to meet the requirements of regulatory agencies. Participants will be asked to use their laboratory's routine measurement technique or the AOAC method, if possible, for the determination of withanoside IV, withanoside V, withaferin A, 12-deoxywithastromonolide, withanolide A, and withanolide B in ashwagandha root and extract materials. This study is sponsored in part by the National Institutes of Health Office of Dietary Supplements (NIH-ODS).

Analytes

withanoside IV, withanoside V, withaferin A,
12-deoxywithastromonolide,
withanolide A, withanolide B

Samples

Ashwagandha Root and Extract

Registration Instructions

For New NIST QAP Participants

To participate, first request an account by navigating to <https://qa-hub.nist.gov> and clicking *Request new account*. Your account request will be approved by a program administrator, after which you will receive an automated confirmation email with login information. Please modify the temporary password immediately upon logging in to the system and confirm or update your contact information on the *My account* page. *All participants are required to provide shipping account information (e.g., FedEx/TNT, UPS, DHL), to which the cost of sample shipment will be charged. Your account will not be approved until a valid shipping account is provided.* Next, log in to your account at <https://qa-hub.nist.gov>. Click *Enroll to upcoming exercises* on the top menu or scroll to the bottom section under *Upcoming exercises* and click the blue button for *Apply for exercises*. and select the studies of interest to your laboratory, then click the blue button for *Save and confirm*. Your participation request will be approved by a program administrator, and you will receive an automated confirmation email. Study selections can be modified at any time up until the close of the registration period.

For Existing NIST QAP Participants (DSQAP, HAMQAP, and/or CannaQAP)

Navigate to <https://qa-hub.nist.gov> and click *Reset your password*. Enter the email address associated with your registration on a previous QAP site, and click the blue *Submit* button. You will then receive an automated email with instructions to reset your password. Please confirm or update your contact information on the *My account* page (access via the black menu bar) using the red *My laboratory settings* tab at the top. To participate, click *Enroll to upcoming exercises* on the top menu or scroll to the bottom section under *Upcoming exercises* and click the blue button for *Apply for exercises*. and select the studies of interest to your laboratory, then click the blue button for *Save and confirm*. Your participation request will be approved by a program administrator, and you will receive an automated confirmation email. Study selections can be modified at any time up until the close of the registration period. *All participants are required to provide shipping account information (e.g., FedEx/TNT, UPS, DHL), to which the cost of sample shipment will be charged. Your participation request will not be approved until a valid shipping account is provided.*