

MANUSCRIPT FOR THE UNIVERSITY OF HAWAI'I AT HILO  
MARINE OPTION PROGRAM

# A Guide to Operations of an Elemental Analyzer and Isotope- Ratio Mass Spectrometer

Brittany “Maya” Fuemmeler

Marine Science Department  
University of Hawai'i at Hilo

MOP Advisors

Tara Holitzki  
Marine Science Analytical Laboratory Manager  
University of Hawai'i at Hilo

Dr. Tracy Wiegner  
Marine Science Department  
University of Hawai'i at Hilo

SUBMITTED

May 2017

## **Abstract**

The ability to properly use scientific instrumentation is an essential part of research projects. Instrument users need to become familiar with the science behind the functions of the instrument, as well as its operational procedures. This is done by reading the instrument's user manual. However, laboratory managers and technicians will summarize this information in an abbreviated Standard Operating Procedure (SOP) document to guide and train new instrument users. For my Marine Option Program project, I developed an SOP for the CosTech Elemental Analyzer (EA) and Delta V Isotope Ratio Mass Spectrometer (IRMS) housed at the University of Hawai'i at Hilo Analytical Laboratory. The EA combusts solid samples to a gas, which is analyzed for carbon and nitrogen amounts, which then can be diverted into an IRMS, which then quantifies the amounts of specific stable isotopes for those elements. The SOP developed during this project is easier to understand, is organized with reference pictures on how to efficiently use the EA and IRMS. It can serve as a template for other instruments' SOPs in the laboratory.

*Keywords:* elemental analyzer, isotope ratio mass spectrometer, standard operating procedure, SOP

## Introduction

When conducting research projects, analytical instrumentation is commonly used to analyze samples for microscopic variables, such as elements and their isotopes. The most commonly used instruments to do this are: elemental analyzers (EA), and high precision isotope ratio mass spectrometers (IRMS). The EA combusts solid samples to a gas, which is analyzed for carbon and nitrogen amounts within the sample, which then can be diverted into an IRMS, which then quantifies the amounts of specific stable isotopes for those elements.

Carbon (C) and nitrogen (N) are the two most common elements analyzed in trophic level interactions and food web analysis. Stable isotopes of carbon and nitrogen ( $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$ ) are used to identify photosynthetic pathways of primary producers, and for estimating trophic positioning in the food web, respectively (Doucett et al. 2009, Hondula et al. 2014, Layman et al. 2012, Post 2002). Stable isotopes are also used in ecological studies such as diet specialization in marine mammals using  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  (Newsome et al. 2009), biological, oceanographic, and paleo-studies (Craig and Gordon 1965, Peterson and Fry 1987). They are highly useful in solving biogeochemical problems as they can be used as tracers as well as for processing information and can tell us changes in the environment, based on known characteristics of each element analyzed (Pearson and Fry 1987). During the coastal methods course I participated in fall of 2016, my team and I collected 4 seaweed samples at 4 sites along the Keaukaha coast in Hilo, Hawai'i. We dried and prepared the samples to be run in the EA and IRMS at the University of Hawai'i at Hilo's Analytical Laboratory. These dry seaweed samples were analyzed for stable nitrogen and carbon isotopes in order to identify if there were any anthropogenic waste contaminants entering the coastal waters.

For my Marine Option Program (MOP) project, I worked with an EA and IRMS. The EA was a CosTech Elemental Combustion System. This EA combusts solid samples, and is set up to analyze those samples for carbon, nitrogen, and hydrogen. It can also be set up to do sulfur analysis. The IRMS was a Thermofisher Delta V Isotope Ratio Mass Spectrometer which analyzes gas samples for stable carbon and nitrogen isotope ratios. It was directly connected with the CosTech EA so that following the EA combustion, it could quantify the amount of each stable carbon and nitrogen isotope in the samples.

These instruments can range from tens to hundreds of thousands of dollars, and repairs on these instruments can not only be expensive, but they can also cause a backup in jobs. Since it is a fee for service laboratory, clients that submit their samples expect high quality data returned to them in a timely manner so that they may finish their projects. However, if instruments go down, those samples must be placed on hold until the laboratory receives any required parts or they are able to troubleshoot and fix the instruments. It is essential that the laboratory technician using the machines knows how to properly use it, and can troubleshoot when problems arise. When the instrument is new, the instrument users must not only attend extensive training specifically for use of the instrument, but they must also become familiar with the operations manual. The operations manual for the instrument includes not only how to turn the instrument on and off, but how all the components of the instrument are connected, what the critical parts are, how the instrument analyzes and produces the data, and how to properly maintain and repair components of the instrument. However, this can be over 250 pages of reading. This is where the standard operating procedure (SOP) comes into play. An SOP is a set of written, easy to understand and well organized instructions that detail how to properly perform a specific function (EPA 2007;

Amare 2012). When writing an SOP, it is important to consider who the reader will be. There are two kinds of SOPs: a technical SOP and an administrative SOP (EPA 2007).

For the benefit of future University of Hawai'i at Hilo Analytical Laboratory users, I developed a more detailed, user-friendly SOP for the EA and IRMS. This is a technical SOP, which is defined as procedures that can be in a simple hierarchical step format used for any technical procedure or operational process. These are typically used for performing analytical methods in a laboratory (EPA 2007). Therefore, this format was chosen because an administrative SOP is more aimed at listing instruction on reviewing documentation or quality assurance of projects. The current SOP that is supplied in the lab is only text and provides no visual aids to help new users in becoming familiar with the proper usage of the instruments. However, the SOP that I have written is more simplified, with easy to understand step-by-step instructions for how to run a job from start-up of the instruments to processing of the data.

## Methods

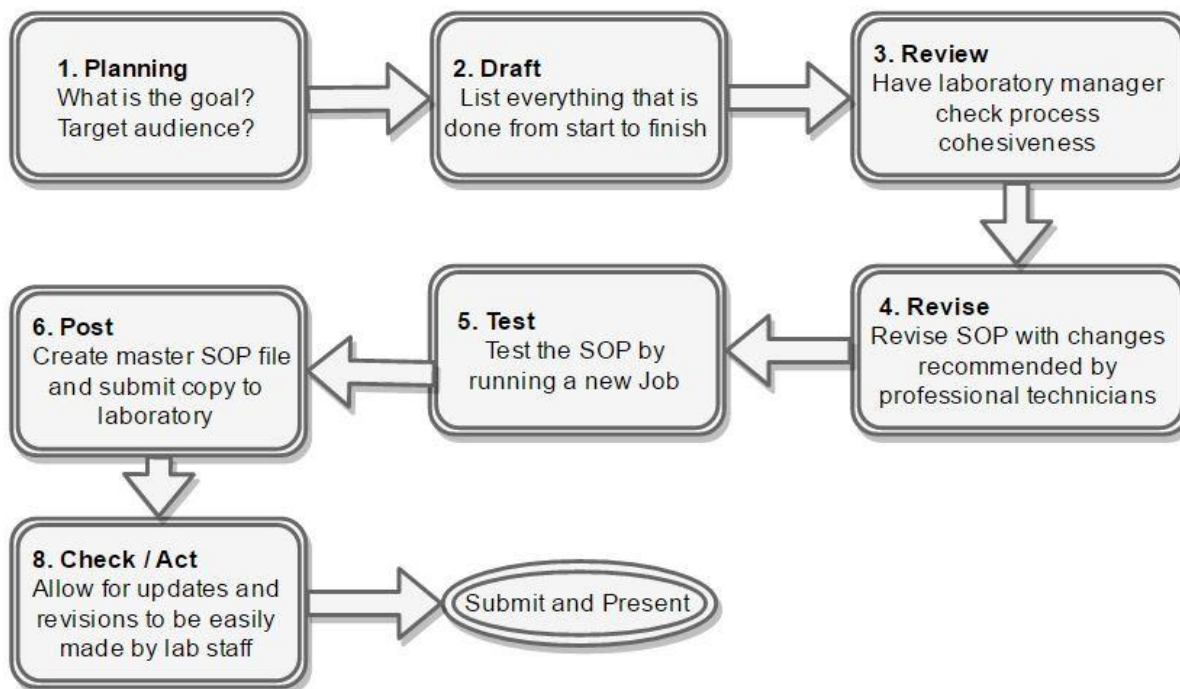


Figure 1. Process of planning and writing a Standard Operating Procedure. Structured to fit my MOP project.

To develop my SOP, I followed a step-by-step process on how to plan and write an SOP outlined by Amare (2012), and Colligon and Rosa (2006). Planning for the SOP began in November 2016 in the University of Hawai‘i at Hilo Analytical Laboratory (UH Hilo AL). The initial plan was to create a SOP for the new Delta V IRMS which was purchased with a National Science Foundation (NSF) Major Research Instrumentation (MRI) grant awarded to Dr. Tracy Wiegner and Tara Holitzki. On 8 November 2016, a ThermoFisher technician arrived to open all the instruments’ boxes and began building and installing the new Thermo-chemical Elemental Analyzer, Delta V Isotope Ratio Mass Spectrometer, and Gas Bench II, as well as start training the laboratory manager and technician how to use the instruments. The installation of the instruments continued through to the middle of the spring 2017 semester. While the analytical laboratory awaited the new gases (C, H, O, N), I observed the process of installing the new machine. However, following winter recess, as the new IRMS was not fully set-up, calibrated,

and running yet, I changed my original MOP project plan. My new plan was to develop an updated, more user-friendly SOP with images for the current EA and IRMS. On 19 January 2017, the first IRMS job of the year was prepped and ready to run on the CosTech EA and Delta V IRMS. During the run, using Microsoft Office: Word notes were recorded.

On 6 March 2017, the technician and I practiced the process of analyzing a sample for nitrogen and carbon from start to finish without a sample. We did this so I could ask questions regarding earlier note taking. Photos of the individual parts of the EA and IRMS, and of the computer screens were taken.

On 29 March 2017, I tested my SOP by running through a job. I instructed the technician on what to do, following each step of my SOP. I checked the SOP to make sure it included all the essential information from the instruments' start-up, to what specific steps needed to be taken to ensure that accurate data would be produced, and finally to processing the instruments' data.

The final SOP is stored in the Analytical Laboratory and with Ms. Holitzki as both a printed copy and as an editable PDF. This SOP is unique to the EA and IRMS in the UH Hilo AL, and the procedures would need to be modified for use in another laboratory that may have different instrumentation.

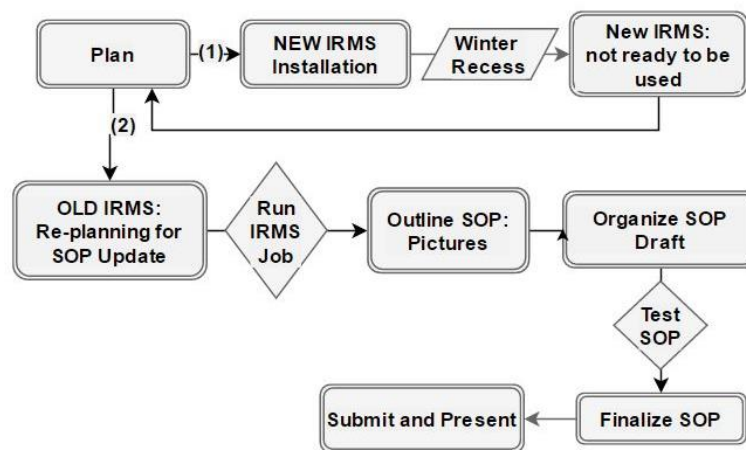


Figure 2. Project workflow from start (November 8, 2016) to finish (May, 2017).

## **Discussion**

Before starting this project, I had very little experience working in an analytical laboratory, and no experience writing SOPs. The amount of planning and coordinating that goes into not only writing the SOP, but also ensuring that it is comprehensive and can be understood by someone with no instrumentation experience was more time consuming and challenging than previously anticipated. However, the feedback that I received from Ms. Holitzki was a crucial part of completing project successfully and helped me to produce the best SOP possible. My SOP has not yet been used to train new IRMS users, however, Ms. Holitzki plans to use it with the next one.

In conclusion, following the completion of this project, I gained a more technical understanding of the “behind-the-scenes” part of analytical laboratories. In addition to observing how incoming samples from clients are put into the job queue, I got to use my own research project to gain experience in drying, preparing, and logging samples and standards to be processed in the EA and IRMS. I got to observe, learn, and log how to use the EA and IRMS, starting with the warm-up procedures, calibrating the gases, loading and running the samples, and finally normalizing the data so that when it is sent back to the client, they can easily use it in their own research. Now, rather than being a researcher that submits samples to the laboratory and trusts that the results will come back perfectly, I have the knowledge of what it takes to process valuable research samples. The pressure and trust that is placed in the staff of analytical laboratories seems greatly underappreciated. However, I hope that with the knowledge gained from this project, I will be able to apply them in my future career.



**Acknowledgements**

I would like to acknowledge Tara Holitzki, Dr. Tracy Wiegner, Dr. Steven Colbert, Jennifer Sims, University of Hawai'i Hilo Analytical Laboratory, and the University of Hawai'i Hilo Marine Science Department.

## References

- Amare G (2012) Reviewing the Values of a Standard Operating Procedure. *Ethiop J Health Sci* 22: 205-208
- Colligon I and Rosa M (2006) GLP SOPs for equipment calibration and maintenance. Part 3: process for mapping SOP development. *Qual Assur J* (10) 279-285
- Craig H and Gordon LI (1965) Stable isotopes in oceanographic studies and paleotemperatures.
- Doucett RR, Marks JC, Blinn DW, Caron M, Hungate BA (2007) Measuring terrestrial subsidies to aquatic food webs using stable isotopes of hydrogen. *Ecol Soc Am* (88) 1587-1592
- Kracht O (2011) Simultaneous N, C and S isotope ratio determination on a Delta V isotope ratio MS using a flash elemental analyzer. ThermoFisher Scientific Application Note: 30194
- Hondula KL, Pace ML, Cole JJ, Batt RD (2014) Hydrogen isotope discrimination in aquatic primary producers: implications for aquatic food web studies. *Aquatic Science* (76) 217-229. Accessed 14 Sep.
- Landais A, Winkler R, Prie F (2014) Triple isotopic composition of oxygen in water from ice cores. ThermoFisher Scientific Application Note: 30287
- LaPorte DF, Holmden C, Patterson WP, Prokopiuk T, Eglington BM (2009) Oxygen isotope analysis of phosphate: improved precision using TC/EACF-IRMS. *J Mass Spectrom* 44: 879-890
- Layman CA, Araujo MS, Boucek R, Harrison E, Jud ZR, Matich P, Hammerschlag-Peyer CM, Rosenblatt CA, Vaudo JJ, Yeager LA, Post D, Bearhop S (2012) Applying stable isotopes to examine food web structure: an overview of analytical tools. *Biol Rev* (87) 542-562
- Michener R, Lajtha K (2007) Stable isotopes in ecology and environmental science. Michener R, Lajtha K (eds) *Stable isotopes in ecological research*, 2<sup>nd</sup> Edition. Blackwell Publishing Ltd, Oxford, UK
- Newsome SD, Tinker MT, Monson DH, Oftedal OT, Ralls K, Staedler MM, Fogel ML, Estes JA (2009) Using stable isotopes to investigate individual diet specialization in California sea otters (*Enhydra lutris nereis*)
- Post DM (2002) Using stable isotopes to estimate trophic position: models, methods, and assumptions. *Ecol* (83) 703-718
- Peterson BJ and Fry B (1987) Stable Isotopes in Ecosystem Studies. *Annual Reviews of Ecology and Systematics* 18: 293-320

ThermoFisher Scientific (2017) Delta V™ isotope ratio mass spectrometer. Accessed 6 Feb.  
<https://www.thermofisher.com/order/catalog/product/IQLAAEGAATFABHMZZZ>

ThermoFisher Scientific (2017) TC/EA high temperature conversion elemental analyzer.  
Accessed 6 Feb.  
<https://www.thermofisher.com/order/catalog/product/IQLAAEGAATFAETMALU>

United States Environmental Protection Agency (2007) Guidance for preparing standard operating procedures (SOPs) EPA/600/B-07/001. Accessed 6 Feb.  
<https://www.epa.gov/sites/production/files/2015-06/documents/g6-final.pdf>