

EA-IRMS: Detection of squalane from animal and vegetable sources

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Keywords

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Goal

To determine if cosmetics contain a skin hydrating compound derived from shark liver oil.

Introduction

Cosmetic products, such as skin creams, sunscreens, lipstick and anti-ageing creams have excellent skin softening and hydrating properties. The skin softening and hydrating qualities come from squalene ($C_{30}H_{50}$), a saturated hydrocarbon derived from the naturally occurring, unsaturated hydrocarbon squalene ($C_{30}H_{50}$).

Shark liver oil and olive oil are the source of squalene for skin care cosmetic products. The production of squalene from shark liver oil is less complex, much faster, with higher yields and purity and it is cheaper than from olive oil,¹ although the sharks used to source squalene are part of an endangered species, and their use does not represent a sustainable or renewable practice. These conditions create an opportunity for economically motivated mislabeling of cosmetics, deceiving consumers, ethically and financially, whilst potentially increasing producer profit.

European Union EC Regulation (No. 1223/2009) and USA Federal Food, Drug, and Cosmetic Act (U.S.C. 321-392) require cosmetic products to be accurately labeled for ingredient origin and amount. In addition, EU legislation (EU Directive 2003/15/EC) forbids cosmetic product testing on animals, leading many countries to impose bans on the use of raw materials from animals in cosmetic products. Consequently, the use of raw materials derived from animals has become ethically unacceptable for cosmetic producers and consumers.

The potential for product mislabeling calls for an analytical technique that provides conclusive answers on whether cosmetic products contain shark liver oil. This application brief shows that the carbon isotope fingerprint of cosmetics can be used for product labeling fraud.

Analytical configuration

Carbon isotope fingerprints ($\delta^{13}\text{C}$) of squalene and squalane from shark liver oil, olive oil, and mixtures of both oils can be measured by a Thermo Scientific™ EA IsoLink™ IRMS System.

For sample analysis, around 200 μg were weighed into tin containers and introduced to the reactor from the Thermo Scientific™ MAS Plus Autosampler, where they were combusted in the presence of oxygen. The isotope analysis of CO_2 from the sample combustion was performed by Thermo Scientific™ DELTA V™ Isotope Ratio Mass Spectrometer. The analysis time per sample is less than 5 minutes, using than 0.4 liters of helium.

Carbon isotope fingerprint of squalane and squalene

The carbon isotope fingerprint ($\delta^{13}\text{C}$) of squalane and squalene can be used to differentiate whether it is derived from shark liver oil or olive oil. Shark liver oil squalene is characterized by $\delta^{13}\text{C}$ values between -19.9‰ and -20.9‰ whereas olive oil squalene shows has lower $\delta^{13}\text{C}$ values, between -27.8‰ and -28.4‰ . This difference in $\delta^{13}\text{C}$ between the two sources can be employed to detect (a) the origin of squalane and (b) an adulteration of cosmetics using a mixture of olive oil and shark liver oil.²

Do cosmetics contain shark liver oil?

Table 1 shows $\delta^{13}\text{C}$ of squalene and squalane derived from olive oil and shark liver oil and a 50:50 squalane mixture of squalane from these two sources. The carbon isotope fingerprint of squalane and squalene from shark liver oil and olive oil can be differentiated and used to identify cosmetic products containing purely shark liver oil and therefore verify product label accuracy.

Do cosmetics contain mixes of shark liver oil and olive oil?

Using carbon isotope fingerprints, it is possible to detect mixtures of squalane and squalene from shark liver oil and olive oil and identify fraud and mislabeling resulting from the substitution of olive oil with shark liver oil, which is the commercially cheaper source (Figure 1).

Table 1. $\delta^{13}\text{C}$ analyses of squalane and squalene.

Sample	Mean $\delta^{13}\text{C}$ VPDB [‰; n = 5]	1SD [‰; n = 5]
Olive oil squalene	-28.06	0.06
Olive oil squalane	-27.99	0.12
Shark oil squalene	-20.56	0.07
Shark oil squalane	-20.28	0.15
50:50 mix of shark-olive squalane	-24.25	0.11

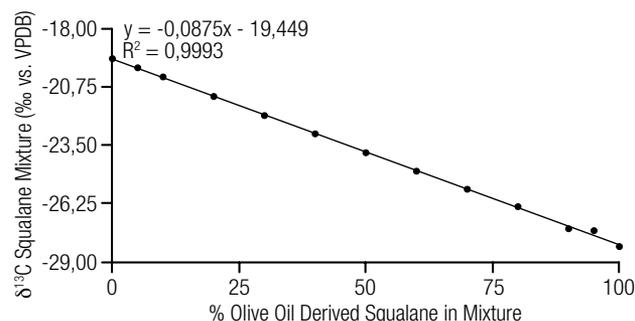


Figure 1. Mixtures of squalane from shark liver oil and olive oil. The linear regression provides a correlation that can be used to calculate the percentage of squalane from olive oil in cosmetics.

The experiment measured pure shark liver oil squalane and then measured samples with the subsequent addition of olive oil squalane. This experiment reflects a practice of mixing squalane sources later included in cosmetic products.

From Figure 1, the correlation predicts a $\delta^{13}\text{C}$ of -23.86‰ for a 50:50 mixture of shark and olive oil derived squalane. Our analysis of a 50:50 mixture of shark liver oil and olive oil squalane gave a $\delta^{13}\text{C}$ of -24.25‰ (Table 1), suggesting that 55% of squalane came from olive oil and 45% from shark liver oil.

The correlation provides a simple tool to identify cosmetic products with mixed sources of squalane and squalene that can be used to verify the accuracy of product labels.

Summary

Carbon isotope fingerprints measured using the EA IsoLink IRMS System enables to detect the origin of oil to be used in cosmetic products allowing product label claims to be easily verified.

References

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The natural difference in $\delta^{13}\text{C}$ between the shark liver oil and olive oil allows products using one of these sources alone, or mixtures of these sources, to be identified. The unique answers provided, combined with fast sample analysis times, low cost of sample analysis and analytical automation, highlight the EA IsoLink IRMS System as an ideal solution for analysis aiming at determining the origin of samples.

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