

## Analytical, Nutritional and Clinical Methods

## Seasonal variation in the C, N and S stable isotope composition of retail organic and conventional Irish beef

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**Abstract**

The objective of this study was to investigate seasonal variation in the C, N and S stable isotope composition of retail organic and conventional Irish beef. A total of 242 beef samples (127 organic, 115 conventional) was collected in a one-year survey and analysed by isotope ratio mass spectrometry. The  $\delta^{13}\text{C}$  time series in conventional beef was significantly non-random, with a pronounced seasonal positive shift of  $>2\text{‰}$  between December and June, whilst  $\delta^{13}\text{C}$  in organic beef was less variable and significantly more negative. In conventional beef,  $\delta^{15}\text{N}$  was remarkably invariant (remaining close to  $7\text{‰}$ ) throughout the year, while organic beef was more variable and also significantly lower in  $\delta^{15}\text{N}$ . The S isotope composition ( $\delta^{34}\text{S}$ ) exhibited a complex seasonal pattern in both types of beef. These results show that seasonal patterns can occur in the isotopic composition of beef, probably reflecting seasonality in animal feeding practices modulated by tissue turnover rates. Such seasonal variation needs to be considered in the isotopic authentication of beef and other animal-derived products.

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**1. Introduction**

Organic farm products command premium prices and their market share continues to rise. A number of analytical studies have compared the nutritional value, sensory quality and food safety aspects of organically and conventionally produced foods (Bourn & Prescott, 2002; Woese, Lange, Boess, & Bogl, 1997). However, existing analytical approaches cannot provide a reliable designation of the production system of foods (Ulberth, 2004). A robust scientific technique, or combination of techniques, for the authentication of foods labelled organic would be of

immense value for consumers, government agencies and the agri-food industry.

Stable isotope ratio analysis (SIRA) provides a promising tool for authenticity tests of organically produced foods (Kelly, 2003; Schmidt, Rossmann, Stöckigt, & Christoph, 2005a). For production system investigations, SIRA exploits variations in the natural stable isotope composition in farm products, which can reflect different crop cultivation and animal husbandry practices. For example, SIRA of C can potentially identify the production origin of beef and milk based on the extent of  $\text{C}_4$  photosynthetic plant materials (maize) consumed by cattle (Bahar et al., 2005; Boner & Förstel, 2004; De Smet, Balcaen, Claeys, Boeckx, & van Cleemput, 2004; Knobbe et al., 2006).

SIRA is an authenticity strategy that relies on empirical databases of the chemical composition of genuine

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products, encompassing the full range of variation caused by various factors (Ulberth, 2004). Temperate agriculture is highly seasonal in nature and animal husbandry practices are likely to result in transient seasonal variations in the isotope composition of animal-derived products such as meat and milk. Therefore, information is required on the likely extent of seasonal isotopic variations in animal products from different production systems.

Ireland is a temperate humid country with an annual weather cycle and distinct seasons (Keane & Sheridan, 2004). Husbandry practices, especially the feeding of live-stock animals indoors and outdoors at different periods of the year, are governed by seasons (Lynch, 2004). As the difference in isotopic composition between indoor (mostly conserved forages and grain-based diets) and outdoor (mostly grass-based diets) dietary constituents is likely to result in altered isotopic compositions of animal tissues, seasonal variations in the isotopic composition of meat can be expected. The objectives of the present study were to investigate the annual seasonal variation in the stable isotope composition of light elements (C, N and S) of organic and conventional retail Irish beef and to assess its implications for the isotopic authentication of organic beef.

## 2. Materials and methods

### 2.1. Sources of samples

Organic beef samples were collected from two large supermarkets (referred to as A and B) and two certified butcher shops dealing exclusively with organic beef (C and D) (Table 1). All sources were approved either by the Irish Organic Farmers' and Growers' Association (IOFGA) or the Organic Trust, Ireland, as certified organic beef retailers. Conventional beef samples were collected from the same supermarkets and three further butcher shops (grouped as E) dealing only with conventional beef produced in Ireland. Of the total organic samples collected ( $n = 127$ ), 96.5% were striploin; in instances where this was unavailable, round steak (2%) and sirloin (1.5%) were sampled. All conventional beef samples ( $n = 115$ ) were striploin.

Samples were collected once a week between July 2003 and June 2004. An uneven availability of organic beef throughout the year and the selling of beef from the same sources on consecutive weeks, especially by the small butchers, caused slightly uneven numbers of beef samples collected each week. At the time of collection, the animal

ear-tag number and the supplier/farmer information were recorded wherever available. This survey was originally designed to relate patterns in isotopic compositions of beef to geographical and husbandry background information; however, due to the lack of disclosure of farm-level information by the suppliers, the interpretation is mainly restricted to the description of the overall seasonality of Irish organic and conventional beef.

Upon collection, samples were labelled according to an internal coding system to facilitate tracing of samples during subsequent storage and handling. Collected samples were vacuum-packaged and stored at  $-20^{\circ}\text{C}$  until preparation for analysis.

### 2.2. Preparation of samples

Muscle sub-samples (5 g) were sliced into thin pieces, freeze-dried (Edwards Pirani 501 Freeze Dryer, Edwards Ltd., Crawley, UK) for 48 h and pulverised using a vibrating ball mill (Type MM2, Glen Creston Ltd, Stanmore, UK). The milled samples were then stored in plastic bags under desiccation. Lipids were extracted from 100 mg of milled sub-samples using hexane:isopropanol (3:2 v/v) as described by Radin (1981). Defatted muscle residue (0.9–1.1 mg) was loaded into ultra clean tin capsules for dual C and N isotope analysis. For S isotope analysis, defatted muscle residue (5.0–5.5 mg) and vanadium pentoxide (6.5–7.5 mg) were loaded into tin capsules. All samples were analysed for C and N, but only the samples supplied by one large supermarket (A) were analysed for S (organic  $n = 46$ , conventional  $n = 54$ ).

### 2.3. Isotopic analysis

Natural abundance stable isotope ratios of C ( $^{13}\text{C}/^{12}\text{C}$ ), N ( $^{15}\text{N}/^{14}\text{N}$ ) and S ( $^{34}\text{S}/^{32}\text{S}$ ) in defatted muscle were measured by elemental analyser continuous-flow isotope ratio mass spectrometry (EA-CF-IRMS), using a Europa Scientific ANCA-NT 20-20 Stable Isotope Analyser with ANCA-NT Solid/Liquid Preparation Module (Scrimgeour & Robinson, 2004). The analytical precision, as estimated by replicate analysis ( $n = 24$ ) of bovine liver standard (NIST Standard Reference Material 1577b, Bovine Liver) analysed along with the samples, was 0.1‰ (SD) for both C and N. Working standard was 1 mg of leucine prepared by freeze-drying 50  $\mu\text{l}$  of a 20  $\text{mg ml}^{-1}$  stock solution in tin cups and then calibrated against 'Europa flour' and IAEA standards N1 and N2.

For S isotope ratio analysis, the bovine liver standard (1577b), quality control reference material and beef samples (all previously mixed with vanadium pentoxide catalyst) were analysed (Scrimgeour & Robinson, 2004). During analysis of samples, NBS 127 (barium sulfate, IAEA,  $\delta^{34}\text{S}_{\text{V-CDT}} = +20.3\text{‰}$ ), IAEA-S-1 (silver sulfide,  $\delta^{34}\text{S}_{\text{V-CDT}} = -0.3\text{‰}$ ) and IA-R025 (barium sulfate,  $\delta^{34}\text{S}_{\text{V-CDT}} = +8.53\text{‰}$ ) were used as internal reference materials. The analytical precision (SD,  $n = 9$ ) was 0.2‰.

Table 1  
Sources of retail organic and conventional Irish beef samples

Source	Organic ( $n$ )	Conventional ( $n$ )
Supermarket (A)	46	54
Supermarket (B)	35	47
Organic butcher (C)	35	–
Organic butcher (D)	11	–
Conventional butchers (E)	–	14
Total	127	115

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