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Value Assignment of Standard Reference Material® 1849b Infant/Adult Nutritional Formula I

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National Institute of Standards and Technology
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Abstract

The National Institute of Standards and Technology (NIST) Standard Reference Material® (SRM®) 1849b Infant/Adult Nutritional Formula I delivers certified or non-certified values for over 100 analytes. This material is intended to be used for the evaluation of methods for the determination of elements, amino acids, fatty acids, nucleotides, proximates, sugars, and vitamins and in this and similar matrices. The material was purchased pre-packaged from a commercial vendor. The measurement results used to assign values to the measurands were provided by NIST staff, the manufacturer, and/or interlaboratory comparison exercises. This document describes the material, measurement processes and results, and data analysis used to produce SRM 1849b.

Keywords

Amino Acids; Elements; Fatty Acids; Nucleotides; Proximates; Reference Material; Sugars; Vitamins.

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Fig. 105. Cytidine monophosphate (*CMP*) mass fraction as a function of production sequence 131
Fig. 106. Guanosine monophosphate (*GMP*) mass fraction as a function of production sequence 132
Fig. 107. Uridine monophosphate (*UMP*) mass fraction as a function of production sequence 133
Fig. 108. Total nucleotide equivalents mass fraction as a function of production sequence 134
Fig. 109. Galactooligosaccharides (GOS) mass fraction as a function of production sequence 135
Fig. 110. Lactose mass fraction as a function of production sequence 136
Fig. 111. Glucose mass fraction as a function of production sequence 137
Fig. 112. Total sugars mass fraction as a function of production sequence 138

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1. Introduction

Infant formula is one of the most highly regulated foods in the United States and around the world. The Infant Formula Act of 1980 (Public Law 96-359) requires that manufacturers test their products to make sure that nutrients fall within specified limits [1]. Beginning in 1996 with Standard Reference Material® (SRM®) 1846 Infant Formula, the National Institute of Standards and Technology (NIST) has provided reference materials for use in assuring the quality of infant formula-related nutrient measurements [2,3]. SRM 1846 was replaced with SRM 1849 Infant/Adult Nutritional Formula in July 2009 [4], followed by SRM 1849a Infant/Adult Nutritional Formula I (milk-based) in Dec 2011 [5]. A soy-containing material, SRM 1869 Infant/Adult Nutritional Formula II (milk/whey/soy-based), was introduced in 2018 [6]. In 2021, two materials were released to represent additional matrix types, including Reference Material (RM) 8260 Infant Nutritional Formula (hydrolyzed milk-based) [7] and RM 8261 Adult Nutritional Formula (high-protein) [8].

The SRM 1849 series of certified reference materials is located in sector 6 of the AOAC INTERNATIONAL food composition triangle [9,10] (Fig. 1). The information provided in this diagram is used by measurement laboratories in support of nutrition labeling. Laboratories needing an infant formula or similar material for the demonstration of method validity and accuracy when analyzing food products to generate data for nutrition labels can use the currently available edition of SRM 1849b. The SRM can also be used to test methodologies for food safety.

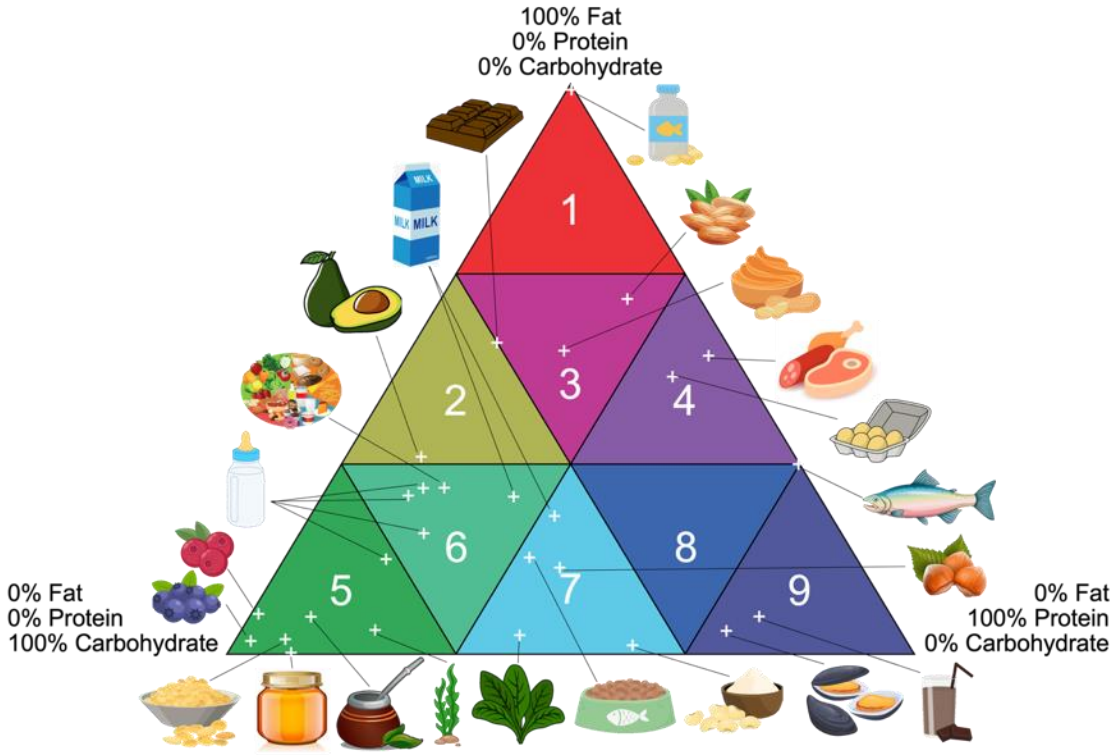

Fig. 1. NIST adaptation of the AOAC INTERNATIONAL food composition triangle. The white "+" depict the location of available food-matrix reference materials.

Fig. 2 displays the sales history of the milk-based infant formula-related materials from the first sale of SRM 1846 in May 1996 to the sale of the last SRM 1849a unit in January 2020.

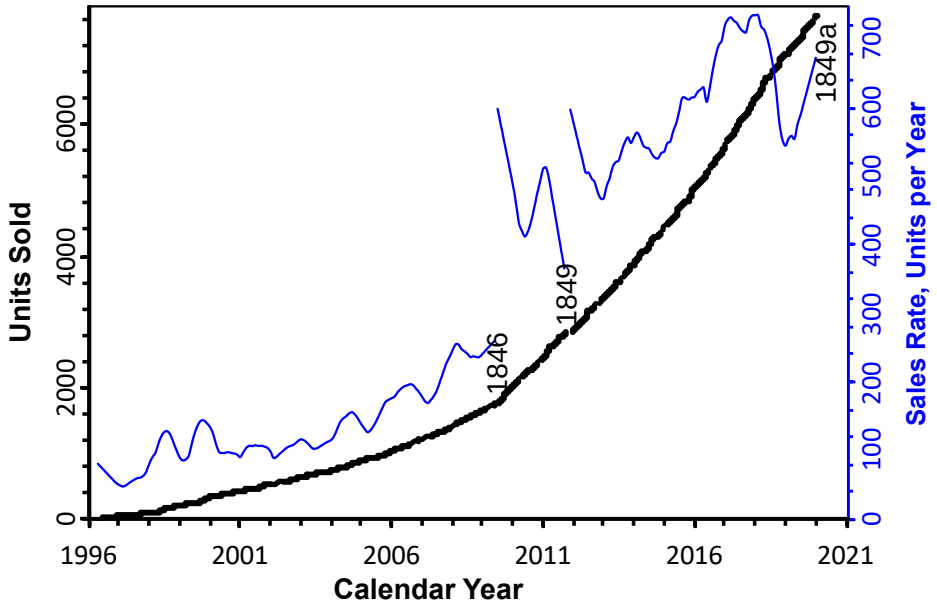

Fig. 2. Sales history of infant formula-related SRMs. The thick black line depicts the cumulative distribution of sales as a function of the order date, plotted using the "Units Sold" axis at the left of the plot. The thin blue line depicts the sales rate (the first derivative of the cumulative distribution), plotted using the "Sales Rate, Units per Year" axis to the right of the graph.

Fig. 3 displays the proportion of sales to various countries or geographical regions over the past 24 years. Over the entire period, nearly 45 % of all sales have been within the USA and Canada, 24% to Europe, 23 % to Asia, 3 % to New Zealand, and the rest scattered elsewhere around the globe. The large proportion of sales to Germany since 2013 probably reflects European sales handled by a German-based NIST-licensed distributor.

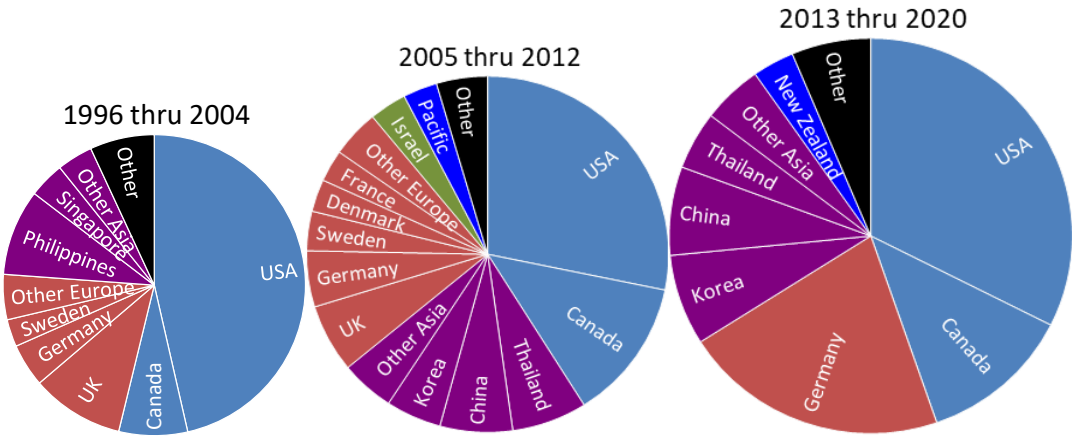

Fig. 3. Location of customers for the infant formula-related materials. From left to right, the three charts display the proportion of sales to various countries or geographic regions from the first sale in 1996 through 12/31/2004, 1/1/2005 through 12/31/2012, and 1/1/2013 through the date of the last unit sold in 2020. Slices are shown for individual countries only when they purchased at least 2.5 % of the units sold during that interval. The area of the circle is proportional to the number of units sold during the interval.

SRM 1849b Infant/Adult Nutritional Formula I (milk-based) is NIST's latest addition to the infant formula-related family of materials. As is true for all NIST food-matrix SRMs, SRM 1849b is intended for use as a primary control material when assigning values to in-house control materials and for validation of analytical methods for the measurement of nutrients in similar matrixes. SRM 1849b contains some nutrients at levels higher than compliant with Public Law 96-359 to provide broader material applicability while retaining its utility for use in measurement assurance within the infant formula industry.

2. Material

2.1. Acquisition & Packaging

SRM 1849b is a milk-based hybrid infant/adult nutritional powder, prepared by a manufacturer of infant formula and adult nutritional products. A base liquid containing all constituents was conventionally heat processed, homogenized, and then spray-dried. The product was packaged into single-use nitrogen-flushed pouches, each containing 10 g of powder. Each unit of SRM 1849b contains 10 pouches. The inventory of the SRM is stored at NIST at –30 °C.

A total of 290 boxes (numbered 1 to 290) were produced and were assembled onto eight pallets (numbered 1 to 8) in approximate packaging order. Samples used to characterize the SRM were identified by pallet and box number.

2.2. Ingredients

Table 1 lists the ingredients used to prepare SRM 1849b and the approximate amounts of each per 1000 kg dry mass. All dairy and dairy-derived ingredients were certified by their providers as suitable for food grade use.

Table 1. Ingredients used to prepare SRM 1849b

Ingredient	Amount per 1000 kg		Ingredient	Amount per 1000 kg	
Lactose	365	kg	Guanosine monophosphate	148	g
Nonfat dry milk	206	kg	Inosine monophosphate	148	g
High oleic sunflower oil	120	kg	Mixed tocopherols	127	g
Soybean oil	84.6	kg	Uridine monophosphate	125	g
Coconut oil	83.6	kg	Adenosine monophosphate	110	g
Galactooligosaccharide powder	55.6	kg	L-Carnitine	100	g
Whey protein concentrate	33.3	kg	Niacinamide	98.9	g
Sodium caseinate	27.5	kg	Cupric sulfate	75.9	g
Magnesium sulfate	11.1	kg	D-Calcium pantothenate	63.9	g
Potassium citrate	9	kg	Vitamin A palmitate	18	g
Sodium chloride	6.9	kg	Thiamine hydrochloride	16.3	g
Tricalcium phosphate	5.9	kg	Pyridoxine hydrochloride	15.7	g
Arachidonic acid	4.5	kg	Riboflavin	12.8	g
Docosahexaenoic acid	2.3	kg	Vitamin A acetate	11.3	g
L-Methionine	1.5	kg	Chromium chloride	4.7	g
Magnesium phosphate dibasic	1	kg	Sodium molybdate	3.7	g
Ascorbic acid	1	kg	Lutein	2.8	g
Choline chloride	646	g	Lycopene	2.8	g
Ferrous sulfate	497	g	Sodium fluoride	2.4	g
Choline bitartrate	490	g	Folic acid	2.2	g
Ascorbyl palmitate	400	g	Biotin	1.9	g
Potassium hydroxide	265.5	g	Sodium selenate	1.6	g
Zinc sulfate	379	g	Potassium iodide	1.6	g
Taurine	364	g	Phylloquinone	1	g
myo-inositol	350	g	β-Carotene	610	mg
Cytidine monophosphate	289	g	Vitamin D_2 (ergocalciferol)	126	mg
D-α-Tocopheryl acetate	174	g	Vitamin D_3 (cholecalciferol)	126	mg
Manganese sulfate	147	g	Cyanocobalamin	43.6	mg

2.3. Homogeneity

Homogeneity of the material was determined based on measurement data provided by the manufacturer. Eleven pouches were selected for analysis, two each from the boxes on pallets 1, 5, and 8 and one each from pallets 2, 3, 4, 6, and 7. The pouches from pallet 1, samples 1:1 and 1:2, represent the same approximate time point in the filling process as do those from pallet 8, samples 8:1 and 8:2. The pouches from pallet 5, samples 5:1 and 5:2, represent time points separated by about 20 min. Sample 5:2 represents the middle time point in the overall filling process.

The material manufacturer provided results for 129 measurands for all eleven pouches. These results confirmed that SRM 1849b is adequately homogenous across the production run and have been used to help assign quantitative values to many SRM 1849b components.

3. NIST Analyses

3.1. NIST WDXRF Analysis of Al, Ca, Cl, Cu, Fe, K, Mg, Mn, Na, P, Rb, S, and Zn

Mass fractions of aluminum (Al), calcium (Ca), chlorine (Cl), copper (Cu), iron (Fe), potassium (K), magnesium (Mg), manganese (Mn), sodium (Na), phosphorus (P), sulfur (S), rubidium (Rb), and zinc (Zn) were determined in SRM 1849b using WDXRF.

3.1.1. Materials

Six pouches of SRM 1849b, one each from boxes 1, 7, 109, 146, 182, and 290, were analyzed in duplicate. The pouches of SRM 1849b were selected using a stratified random sampling scheme based on packaging order. A single pouch of SRM 1849a was used as the quality assurance material, and four samples were prepared using the same procedure as for SRM 1849b. Table 2 lists the materials chosen from among the available powdered food and agricultural material SRMs for use as calibrants. These materials were prepared in the same way as the samples of SRM 1849a and SRM 1849b.

Table 2. SRMs used as WDXRF calibrants[a]

SRM 1515 Apple Leaves	SRM 1575a Trace Elements in Pine Needles
SRM 1547 Peach Leaves	SRM 1577b Bovine Liver[b]
SRM 1549 Non-Fat Milk Powder[b]	SRM 1577c Bovine Liver
SRM 1566b Oyster Tissue	SRM 1869 Infant/Adult Nutritional Formula II
SRM 1568b Rice Flour	SRM 3233 Fortified Breakfast Cereal
SRM 1570a Trace Elements in Spinach Leaves	SRM 3252 Protein Drink Mix
SRM 1571 Orchard Leaves[b]	SRM 3254 Green Tea Leaves
SRM 1572 Citrus Leaves[b]	SRM 3281 Cranberry (Fruit)
SRM 1573a Tomato Leaves	SRM 3287 Blueberry Powder
SRM 1575 Trace Elements in Pine Needles[b]	SRM 3290 Dry Cat Food

[a] COAs for these SRMs can be accessed at https://shop.nist.gov.
[b] Expired and/or unavailable SRMs were internally verified and found to be fit-for-purpose for this analysis.

Blanks were prepared from microcrystalline cellulose from Alfa Aesar (Cat. No. A17730, lot 10200883; Ward Hill, MA, USA) and Whatman (Cat. No. CF11; Maidstone, UK). The cellulose powders were pressed into briquettes after drying with the food powders.

No chemical reagents are necessary for this analytical method.

3.1.2. Equipment

A Sartorius model LP1200S balance (Göttingen, DE) was used to weigh each specimen of powder prior to pressing to ensure approximately equivalent sample quantities among the various

materials. Exact weighing was not required since sample mass is not used in calculations of results. Samples are not diluted and minor variations in briquette thickness can be moderated by use of ratioing analyte signal to background intensity or Compton scatter.

Samples were pressed using a SPEX X-PRESS hydraulic press (model 3630; Metuchen, NJ, USA), operated in manual mode, with painted aluminum pressing caps (Somar, Cat. No. 3130; Reno, NV, USA). Briquettes were placed in double open-ended polyethylene sample cups (Cat. No. SC-4240, 40 mm; Premier Lab Supply, Port St. Lucie, FL, USA) fitted with Spectrolene polypropylene plastic film (Somar, 6 μm thickness, Part No. 3506-33).

An Ultimate wavelength dispersive X-ray fluorescence spectrometer using the SuperQ 6.2b operating system (Malvern Panalytical, Model ZETIUM; Malvern, UK) was used to evaluate the samples.

3.1.3. Sample Preparation

Portions (4.5 g) of the sample, calibrant, and blank materials were weighed into glass containers (e.g., weighing bottles, Petri dishes and beakers) and stored in a desiccator over freshly regenerated silica gel for at least 21 d. To press briquettes, approximately 4.0 g of each dried powder was weighed onto weighing paper and poured into a 31 mm diameter steel pressing die containing an aluminum cap. A polished steel die pellet was inserted, along with the ram cylinder, and the die was pressed at 2 tons (2 000 kg) for 20 s under house vacuum using manual control of the press.

Briquettes were immediately placed into liquid sample cells fitted with 6 μm polypropylene film, and the sample cells were placed into a second desiccator. The plastic film prevents loose powder from dropping into the spectrometer. When all briquettes had been prepared, the samples were loaded into the X-ray fluorescence spectrometer and measured.

3.1.4. Measurement Process

The X-ray spectrometer was used to measure the K-L2,3 characteristic X-ray lines of all elements in a helium environment with the generator operated at 4.0 kW of X-ray power. Background measurement and subtraction were made for Al, Ca, Cl, Cu, Fe, Mg, Mn, Na, Rb, and Zn. Liquid cell sample holders with a 31 mm inner diameter aperture were used to carry all briquettes in liquid cells into the spectrometer and to hold them in the measurement position. The mask between the sample and the collimator was set to view a 29 mm circular area of the sample, and the spinner was used. Counting times were chosen to obtain relative counting statistical errors ranging from 0.1 % to 1.0 % depending on the X ray count rate for each element and the number of measurements of background. The maximum measurement time per sample was approximately 14 min to avoid melting the polymer X-ray foil supporting the briquette and thermally induced swelling of the pressed briquettes. The WDXRF measurements were made in a pseudo-random order with unknown specimens interspersed with calibration standards.

Calibration curves were calculated in the SuperQ Analytical XRF software package (Malvern Panalytical). The generalized calibration algorithm is denoted in Equation 1

$$C_i = D_i - L_{ik} \cdot C_k + E_i \frac{R_i}{R_{\mathrm{Rh}}}\left(1 + \sum_j (\alpha_{ij} \cdot C_j)\right) \quad (1)$$

where *C* denotes mass fraction, *D* is the intercept, *L* is the line overlap factor, *E* is the inverse sensitivity, *R* is the gross or net WDXRF count rate, R_{Rh} is the gross count rate for Rh Compton scatter, and α is the absorption correction factor, with *i* indexing the analyte element, *k* the interfering element by line overlap, and *j* the interfering element by absorption. The Rh Compton scatter was used as an internal reference for Zn and Rb calibrations to compensate for effects of finite sample thickness on more energetic X-rays and some variations in matrix absorption effects. In a few cases (i.e., Ca, Cu, Fe, and Mn), the background intensity for Fe was used as an internal reference because the energy more closely matches that of these analyte lines and would be substituted for R_{Rh} in Equation 1.

The term containing the α factor for X-ray absorption was used for some elements to correct for variations in matrix X-ray absorption and enhancement effects (Table 3). These α factors were calculated empirically based on the measured count rates of the X-ray lines from the interfering elements in cases where no mass fraction values reported for some analytes in the SRMs although some measurable peak was present. Selection of these absorption corrections factors was based on basic X-ray fluorescence theory of matrix effects and how well these factors improved the fit of the calibration points to the modeling equation.

Table 3. Absorption corrections for WDXRF measurements

Element	Self-Absorption Correction	Other Correction
Mg	No	Mg
P	No	Cl, Ca
S	No	Ca
Cl	Yes	Cl
Mn	No	Ca
Zn	No	Ca

A line overlap correction is used when the analyte line of interest significantly overlaps with some other first order fluoresced X-ray signal or higher order diffraction lines. In this analysis, an intensity-based line overlap correction was used to adjust the Na signal for the overlap of the Zn L-series lines.

For some analytes, selected calibrants were observed to be outliers and were excluded from the calibration (Table 4). In some instances, exclusions were made automatically by the system software because the value of the analyte was provided at an informational level only. Other standards were excluded in instances where no value was provided on the COA. Calibrations for Cl, K, Mg, P and S were forced through the origin which effectively negates the influence of the blanks.

Table 4. SRMs excluded from use as WDXRF calibrants for selected analytes

	Na	Mg	Al	P	S	Cl	K	Ca	Fe	Cu	Zn	Rb
SRM 1515					X							
SRM 1547							X					
SRM 1549			X				X					X
SRM 1566b				X								
SRM 1568b		X										
SRM 1570a		X			X	X	X		X			
SRM 1571			X		X	X	X		X			
SRM 1572		X				X	X		X			X
SRM 1573a	X	X		X	X	X	X	X	X	X		
SRM 1575	X	X			X	X					X	
SRM 1575a					X							
SRM 1577b			X									
SRM 1577c			X		X							
SRM 1869	X	X	X	X	X	X		X	X	X		X
SRM 3233	X	X	X	X	X	X	X	X	X			X
SRM 3252	X	X		X	X	X	X	X	X			X
SRM 3254	X	X		X	X	X	X	X	X			X
SRM 3281	X	X		X	X	X	X	X	X			X
SRM 3287	X	X	X	X	X	X	X	X	X			X
SRM 3290	X	X	X	X	X	X	X	X	X		X	X

3.1.5. Measurement Uncertainties

The standard uncertainty for each of the measurements, u_c, combines three uncertainty components as described in Equation 2 and Table 5.

$$u_c = \sqrt{\frac{s^2}{n} + \frac{u_{\mathrm{m}}^2}{3} + (\bar{x}^2 \cdot \frac{u_{\mathrm{m}}^2}{3M^2})}. \quad (2)$$

Table 5. Uncertainty budget for WDXRF analysis

Component	Basis	Type	DF
Variability of Sample Preparation and Measurement, *s*	Standard deviation of the calculated mass fractions for *n* specimens.	A	*n* - 1
Uncertainty of Calibrant Scatter about the Empirical Model Fit, u_m	Estimated from fit of Eq. 1 to calibration data; accounts for repeatability of briquette preparation and measurement and for biases among the calibration standards; calculated from the "goodness of fit" factor determined in system software (Eq. 3). Assumes scatter about the calibration curve in a non-random fashion will be similar for natural materials used as calibrants and unknowns. This uncertainty is assumed to have a uniform distribution	B	∞
Uncertainty of Selection of Empirical Model Parameters	Intended to cover the variations in analyst's inclusion or exclusion of empirical model fitting parameters such as absorption correction factors or calibrants; estimated as a relative uncertainty with u_m relative to the mid-point of the calibration range, *M*, so that the absolute uncertainty from this component depends on where the measurand value falls within the calibration range (i.e., higher absolute uncertainty on the high end of the calibration range and lower absolute uncertainty on the lower end of the calibration range). This uncertainty component is multiplied by the square of the mean of the measured analyte mass fractions, $\bar{x}$, and is assumed to have a uniform distribution	B	∞
Combined Standard Uncertainty, u_c	See Equation 2.		
$U_{k=2}$	Expanded uncertainty estimate with expansion factor, *k*, set equal to 2, i.e., $U_{k=2} = 2u_c$.		
U_{rel}	Relative expanded uncertainty in %. $U_{rel} = U_{k=2}$ / Mean.		

$$\text{No error weighting:} \quad u_{\mathrm{m}} \approx |\, C_{\mathrm{chem}} - C_{\mathrm{calc}} \,| = RMS$$

$$\text{Linear error weighting:} \quad u_{\mathrm{m}} \approx |\, C_{\mathrm{chem}} - C_{\mathrm{calc}} \,| = \left| \frac{RE \cdot W_0 + C_{\mathrm{calc}}}{1 - RE} - C_{\mathrm{calc}} \right| \qquad (3)$$

$$\text{Square root error weighting:} \quad u_{\mathrm{m}} \approx |\, C_{\mathrm{chem}} - C_{\mathrm{calc}} \,| \cong K \cdot \sqrt{C_{\mathrm{calc}} + W_0}$$

where *RMS*, *RE*, and *K* are the regression minimization factors for the respective weighting function and W_0 is a weighting constant. Weighting functions are employed to place higher significance within the curve fitting to different regions of the calibration curve (e.g., the low mass fraction extreme of the calibration versus the high mass fraction extreme). All calibrations used a square root error weighting except for S, K, and Rb which used no error weighting. The calibration process and determination of regression minimization factors are done in SuperQ software.

3.2. NIST ICP-MS Analysis of Cr, Mo, Se, and I

Mass fractions of chromium (Cr), molybdenum (Mo), selenium (Se), and iodine (I) were determined in SRM 1849b using ICP-MS.

3.2.1. Materials

Ten pouches of SRM 1849b, one each from boxes 1, 27, 58, 62, 104, 144, 166, 202, 260, and 290, were analyzed in duplicate. The pouches of SRM 1849b were selected using a stratified random sampling scheme based on packaging order. A single pouch of SRM 1849a was used as the quality assurance material, and four samples were prepared using the same procedure as for SRM 1849b.

Calibration solutions were prepared from SRM 3112a Chromium (Cr) standard Solution Lot No. 170630, SRM 3134 Molybdenum (Mo) Standard Solution Lot No. 130418, SRM 3140 Selenium (Se) Standard Solution Lot No. 100901, and SRM 3180 Iodide Anion (I^-) Standard Solution Lot No. 110530. The internal standard was prepared from SRM 3102a Antimony (Sb) Standard Solution (Lot No. 149011). COAs for these SRMs are available at https://shop.nist.gov.

All samples were prepared using Optima grade acids from Fisher Scientific (Waltham, MA, USA). The iodine analyses also used scintillation grade Triton X-100 from Acros Organics (Waltham, MA, USA) and Optima grade NH_4OH from Fisher Scientific. All dilute acid or base concentrations are expressed in volume fractions with respect to the concentrated acid or base.

3.2.2. Equipment

An Agilent Technologies 7500cs ICP-MS (Santa Clara, CA, USA), equipped with a Peltier-cooled, inert sample introduction system, was used to measure Cr, Mo, Se, I, and Sb in all solutions with read times per mass of 3 s and integration time of 0.1 s per point. Cr, Mo, and Se were measured in two runs; I was measured in three runs. For the Cr, Mo, and Se measurements H_2 was used as the collision gas to minimize polyatomic interferences. For the I measurements, the reaction cell mode was off.

A Mettler AT261 Delta Range analytical balance was used for weighing in the preparation of samples and standards. The balance is serviced and calibrated annually by Mettler. Prior to use, calibration is verified using standard masses ranging from 0.5 g to 20 g that are traceable to the SI.

3.2.3. Sample Preparation

Two 0.5 g aliquots were taken from each pouch of SRM 1849b and four 0.5 g aliquots were taken from one pouch of SRM 1849a and placed in Teflon microwave vessels. Ten procedural reagent blanks were also prepared along with the samples. To each vessel, 10 mL of concentrated HNO_3 and an aliquot of the Sb internal standard solution were added before microwave digestion. The

Sb internal standard solution was prepared at 1000 ng/g Sb from SRM 3102a Antimony (Sb) Standard Solution with a final acid concentration of 1.5% HNO_3 (volume fraction).

3.2.3.1. Chromium, Molybdenum, and Selenium

To each vessel, 0.5 mL of the Sb internal standard solution was added prior to digestion. Samples were digested using MARSXpress vessels in a MARS5 Microwave Digestion (CEM Corporation, Matthews, NC, USA) system using 1600 W power at 85 %, a 20 min ramp time, temperature of 195 °C, and a 20 min hold time. After microwave digestion, solutions were transferred to PTFE beakers and heated on a hot plate with a surface temperature of approximately 180 °C until the volume was reduced to near dryness. Samples were transferred to polyethylene bottles and diluted to 30 g using 1.5 % HNO_3 (volume fraction).

The first aliquot of the pouch from box 290 provided no usable result due to a laboratory error. All results for Cr, Mo, and Se in SRM 1849b are based on the analysis of 19 validly prepared samples.

3.2.3.2. Iodine

To avoid loss of iodine in acidic solutions, a published digestion procedure was used [11]. To the SRM 1849a samples and each blank, 0.5 mL of the Sb solution was added; to the SRM 1849b samples, 0.8 mL was added. To all vessels, 0.4 mL of a 5 % volume fraction Triton X-100 solution was added as a surfactant. Samples were digested using CEM MARSXpress vessels in a CEM MARS5 Microwave Digestion system using the program described in Table 6.

After microwave digestion, sample solutions were transferred to polyethylene bottles and diluted to 60 g by first adding 18 g of 18 MΩ·cm water followed by 30 g of a 7.5 % NH_4OH solution (volume fraction). The diluted samples were tested to ensure that the pH was at or above pH 8. Ammonium hydroxide was used to immediately neutralize the acidic solutions.

Table 6. Microwave settings for digestion of iodine

Step	Power, W	Power Setting, %	Ramp Time, min	Temperature, °C	Hold Time, min
1	800	100	20:00	120	10:00
2	1600	85	20:00	150	10:00
3	1600	85	25:00	190	15:00

3.2.4. Measurement Process

For determination of Cr, Mo, and Se, the method of standard additions was used for calibration. Iodine was determined using an external standard calibration approach.

3.2.4.1. Standard Additions: Cr, Mo, and Se

From each sample dilution, two aliquots were taken, with a spike added to one as described in Table 7 to the approximate analyte mass fractions.

Table 7. Standards and approximate mass fractions for ICP-MS analysis, ng/g

Element	Standard	In Solution	Added as Spike	Total in Spiked Aliquot
Chromium (Cr)	SRM 3112a	5	6	11
Molybdenum (Mo)	SRM 3134	10	11	22
Selenium (Se)	SRM 3149	5	6.5	11.5

Ten instrumental measurements were recorded and averaged for each sample aliquot and each spiked aliquot. Final mass fractions in SRM 1849b were calculated using the method of standard additions in which the analytical instrument is calibrated by measuring the increase in the analytical signal that occurs when a known amount of the analyte is added to the sample. This approach avoids multiplicative types of matrix interferences (enhancements or suppressions) since the calibrant is present within the same matrix as the sample. The method of standard additions can be used in any situation where an analyte and an internal standard can be quantitatively and homogeneously spiked into the sample.

The mass fraction of the analyte in the analyte in the sample, F_{sample}, is calculated using Equation 4:

$$F_{\text{sample}} = R_{\text{u}}\left(\frac{\left(m_{\text{sp}}F_{\text{sp}}/m_{\text{spsolu}}\right)}{R_{\text{sp}}-R_{\text{u}}}\right)\left(\frac{m_{\text{solu}}}{m_{\text{sample}}}\right) \quad (4)$$

where: F_{sp} mass fraction of the analyte in the spiking solution
m_{sample} mass of sample that is present in the solution to be analyzed
m_{solu} total mass of the sample solution after addition of the IS spike
m_{sp} mass of the analyte spiking solution delivered to the solution
m_{spsolu} mass of the solution that will be spiked
R_{sp} analyte/IS signal ratios for the spiked solution
R_{u} analyte/IS signal ratios for the unspiked solution.

3.2.4.2. Standard Additions with External Calibration: I

SRM 3180 *Iodide Anion (I-) Standard Solution* was used to prepare the iodine (I) spike and the standards for the external calibration curve for this analysis. From each sample dilution, two aliquots were taken, with a spike added to one as described in Table 8 to the approximate analyte mass fraction.

Table 8. Standards and approximate mass fractions for ICP-MS analysis, ng/g

Element	Standard	In Solution	Added as Spike	Total in Spiked Aliquot
Iodine (I)	SRM 3180	3	4	7

The iodine mass fraction in SRM 1849b was calculated using external standard calibration since the Sb internal standard instrumental counts were often higher in the spiked sample than the unspiked sample. Limited laboratory access during the time of these experiments prevented prompt analysis immediately after sample preparation, which may have led to such an anomaly if Sb concentrations are more sensitive to the variations of pH over time than iodine concentrations. Ten instrumental measurements were recorded and averaged for each sample aliquot.

External calibration is accomplished by observing the instrument's responses, y, to different levels of the analyte, x. For iodine, the relationship was linear

$$y = b_1 x + b_0, \tag{5}$$

where y is the measured response, x is the mass of iodine (in ng/g) added to each point, b_1 is the sensitivity (slope), and b_0 is the value of y when x = 0 ng/g. This calibration curve is then used to obtain the mass, x_{pred}, of the analyte from a sample which produces an observed response y_{obs} from Equation 6:

$$x_{\text{pred}} = (y_{\text{obs}} - b_0)/b_1 \tag{6}$$

The constants b_1 and b_0 are determined by least squares regression on a set of n pairs of values $\{x_i, y_i\}$, and the result for the analysis is the value calculated for x_{pred}.

3.2.5. Measurement Uncertainties

The standard uncertainty associated with the mean value of each element was estimated as summarized in Equation 7 and Table 9.

$$u = \sqrt{s_{\text{sample}}^2 + s_{\text{blank}}^2 + u_{\text{s}}^2 + u_{\text{b1}}^2 + u_{\text{b2}}^2} \tag{7}$$

Table 9. Uncertainty budget for ICP-MS analysis

Component	Basis	Type	DF
Sample Replication, s_{sample}	The uncertainty due to sample preparation and measurement is estimated by calculating the standard deviation of the mean. (n = 4 or 19; SRM 1849a and SRM 1849b respectively)	A	3 (1849a) 18 (1849b)
Blank Replication, s_{blank}	The uncertainty due to blank preparation and measurement is estimated by calculating the standard deviation of the mean. (n = 10)	A	9
Primary Standard, u_s	The uncertainty associated with the primary standards is calculated to be the expanded uncertainty divided by the expansion factor, k, obtained from the Certificate of Analysis for each SRM used as the standard addition spike.	B	> 60
Weighing of Standards, u_{b1}	The uncertainty for each weighing of the standard is ± 0.01 mg based on the certificate of calibration for the balance. This uncertainty is normalized by division by √3.	B	> 60
Weighing of Samples, u_{b2}	The uncertainty for each weighing of the sample is ± 0.01 mg based on the certificate of calibration for the balance. This uncertainty is normalized by division by √3.	B	> 60

Approximate 95 % level of confidence expanded uncertainties for the mean values were estimated using Equation 8,

$$U_{95\%} = k_{95\%}u \tag{8}$$

where $k_{95\%}$ is the Student's t 95th percentile two-tailed expansion factor appropriate to the df associated with the standard uncertainty, estimated using the Welch-Satterthwaite formula [12, Section G.4.1].

3.3. NIST ICP-OES Analysis of Cu, Fe, Mg, and P

Mass fractions of magnesium (Mg), phosphorus (P), iron (Fe), and copper (Cu) were determined in SRM 1849b using ICP-OES.

3.3.1. Materials

The samples used for ICP-OES were those prepared and digested for ICP-MS measurements as described in Section 5.2.3.1.

Internal standard solutions were prepared from SRM 3148a Scandium (Sc) Standard Solution Lot No. 10070 and SRM 3124a Indium (In) Standard Solution Lot No. 110516. COAs for these SRMs are available at https://shop.nist.gov.

All samples were prepared using Optima grade acids from Fisher Scientific. Samples and acids were diluted using 1.5 % HNO_3 (volume fraction). All dilute acid concentrations are expressed in volume fractions with respect to the concentrated acid.

3.3.2. Equipment

A Perkin-Elmer Optima 8300 Dual View ICP-OES (Waltham, MA, USA) was used to measure Cu, Fe, Mg, and P in all solutions as described in Table 10.

Table 10. Parameters used for ICP-OES analysis

Element	Wavelength, nm	Plasma View	Integration Time, s	Read Time, s	Number of Runs
Cu	324.752	Axial	0.10	1.000	2
Fe	259.939	Axial	0.10	1.000	2
Mg	285.213	Axial	0.10	1.000	2
P	213.617	Radial	0.10	1.000	2
In	230.606	Axial	0.10	1.000	2
Sc	361.383	Axial	0.10	1.000	2
Sc	357.253	Radial	0.10	1.000	2

A Mettler AT261 Delta Range analytical balance was used for weighing in the preparation of samples and standards. The balance is serviced and calibrated annually by Mettler. Prior to use, calibration is verified using standard masses ranging from 0.5 g to 20 g that are traceable to the SI.

3.3.3. Sample Preparation

The solutions used were those used for ICP-MS measurements described in Section 5.2.3.1. To each prepared solution, weighed aliquots containing 0.25 mL of a solution containing 100 µg/g In and 1 mL of a solution containing 100 µg/g of Sc were added as internal standards to improve the precision of the instrumental measurements. The concentration of Sb in these solutions, approximately 16 ng/g as added for ICP-MS analysis, was below the estimated detection limit for ICP-OES and therefore Sb was not used as an internal standard.

3.3.4. Measurement Process

For determination of Cu, Fe, Mg, and P, the method of standard additions was used for calibration. Solutions were diluted and spiked to yield the approximate mass fractions listed in Table 11. Analyte mass fractions were quantified by the method of standard additions (see Section 3.2.4.1).

Table 11. Standards and approximate mass fractions for ICP-OES analysis, mg/kg

Element	Standard	In Solution	Added as Spike	Total in Spiked Aliquot
Cu	SRM 3114	0.3	0.5	0.8
Fe	SRM 3126a	0.25	0.30	0.55
Mg	SRM 3131a	0.3	0.4	0.7
P	SRM 3139a	6	7	13

3.3.5. Measurement Uncertainties

See Section 3.2.5.

3.4. NIST LC-ICP-MS Analysis of Vitamin B_{12} (Cyanocobalamin)

The mass fraction of CNCbl in SRM 1849b was determined using LC-ICP-MS [13] and single-point standard addition with use of an internal standard. Vitamin B_{12} exists in four forms: hydroxo-, methyl-, 5-deoxyadenosyl-, and cyano-cobalamin (CNCbl). In this work, all forms of vitamin B_{12} were converted to CNCbl before the measurement to increase the detection limit and permit SI traceability. By convention, all forms of the vitamin B_{12} cobalamins were reported as mass fraction of CNCbl.

3.4.1. Materials

Eight pouches of SRM 1849b, one each from boxes 1, 41, 157, and 290 and two each from boxes 76 and 182 were analyzed. The pouches of SRM 1849b were selected using a stratified random sampling scheme based on packaging order. Two pouches of SRM 1869 Infant/Adult Nutritional Formula II [6] were used as the quality assurance material, and five samples were prepared using the same procedure as for SRM 1849b.

SRM 3113 Cobalt (Co) Standard Solution was used to prepare internal standards and to establish SI traceability of the USP Reference Standard Cyanocobalamin (Cat. 1152011, Lot F07440, Rockville, MD, USA).

HPLC grade methanol, HPLC grade acetonitrile, and ACS grade ethylenediamine tetraacetic acid (EDTA) diammonium salt were obtained from Fisher Scientific. Taka-diastase was obtained from Accurate Chemical (Carle Place, NY, USA). All other chemicals were of ACS grade, including sodium acetate trihydrate (NaOAc), glacial acetic acid (HOAc), and potassium cyanide (KCN ≥ 95 % purity). Locally prepared sub-boiling distilled water was used as a solvent.

3.4.2. Equipment

A Perkin-Elmer LC system equipped with a Peltier-cooled Series 200 autosampler and a Series 200 quaternary pump was coupled to a Perkin-Elmer Elan DRC II ICP-MS was used to measure

CNCbl. Separation of CNCbl from cobalt (Co) and subsequent detection were accomplished using instrument parameters described in Table 12.

Table 12. LC-ICP-MS parameters for the determination of cyanocobalamin

System	Component	Description
Perkin Elmer Series 200 LC	Column	Waters Atlantis T3 150 mm x 2.1 mm i.d.
	Mobile phase	20 mmol/L EDTA in 25:75 methanol:water (volume fraction)
	Flow rate	200 µL/min, isocratic
	Injection volume	25 µL
Perkin Elmer DRCII ICP-MS	RF power	1300 W
	Nebulizer Gas Flow	1.04 mL/min
	Sample introduction	Concentric nebulizer/cyclonic spray chamber

A Mettler model AT261 Delta Range analytical balance was used for weighing during the preparation of samples and standards. The balance is serviced and calibrated annually by Mettler. Prior to use, calibration of the balance was verified using standard masses ranging from 0.5 g to 50 g that are traceable to the SI. A Jouan model C312 centrifuge (Thermo Fisher Scientific, Waltham, MA, USA) was used to separate particulate matter from the aqueous phase of the samples. A Fisher Scientific Isotemp oven, model number 737F, was used for sample preparation. An Anton-Paar model DMA 35 density meter (Graz, AT) was used for density measurements.

3.4.3. Sample Preparation

Single 3 g aliquots from each pouch of SRM 1849b and five 3 g aliquots from the two pouches of SRM 1869 were accurately weighed into Kimax brand Class A 100 mL volumetric flasks. The samples from boxes 1 and 157 were prepared on day 1, boxes 41 and 290 on day 2, and boxes 76 and 182 on day 3. Three procedural blanks were processed similarly, with a procedural blank and at least one sample of SRM 1869 included on each day of SRM 1849b sample preparation. To each flask, an aliquot of 25 mL of de-ionized water was added and the contents were swirled until the sample powder was dissolved. A 1 mL aliquot of 6 % (mass fraction) taka-diastase was added to each flask, and the contents were mixed by vortexing. The flask was placed in a box devoid of light for 40 min to allow the reaction of the contents to reach completion [14].

At the completion of the reaction, a 30 mL aliquot of 0.25 mol/L NaOAc buffer at pH 4.5 was added to each flask, and the contents were mixed by swirling. A 1 mL aliquot of 1 % (mass fraction) KCN was added to each flask, and samples were heated at 105 °C in an oven for 70 min. After heating, the samples were immediately cooled in an ice bath to near room temperature. The contents of each flask were diluted to volume with water and transferred to two 50 mL polyethylene tubes. Tubes were centrifuged for 30 min at 367 rad/s (3500 rpm) in a Jouan centrifuge. After the centrifugation, the supernatants from each tube were filtered through Whatman 2V filter paper and combined into a 125 mL Erlenmeyer flask.

A Maxi-Clean C18 900 mg SPE cartridge from S*PURE (Part No. 20942/5122344; Singapore) was attached to a 20 mL syringe. Before use, the SPE cartridges were conditioned with 20 mL acetonitrile and rinsed with 10 mL water by gently pressing the piston of the syringe. The

conditioned SPE cartridges with the 20 mL syringe barrels were inserted onto the stopcock of the vacuum manifold. An 80 mL aliquot of each sample filtrate was passed through the cartridge. The exact volume of each aliquot was determined by the mass of the aliquot and the density of the solution measured with a separate portion of the filtrate. The effluent was monitored to exit the SPE cartridges at approximately 40 drops/min to 100 drops/min. After all the sample filtrate passed through the cartridges, the cartridges were air-dried by pulling vacuum until no more effluent was observed, and the stopcock was closed. A pre-weighed 15 mL Falcon tube was placed under the cartridge, and a 5 mL aliquot of 30 % (volume fraction) acetonitrile in water was added to the syringe. Vitamin B_{12} (as CNCbl) in the SPE cartridges was eluted into the Falcon tubes, assisted by gently pressing the piston. An aliquot of 0.2 g solution containing 90 ng/g Co was added to each eluate as an internal standard, and the mass of each tube was weighed again to determine the mass of the contents by the difference. Each sample was filtered through a 0.45 µm Nylon filter. A 0.6 g aliquot of each subsample and a 0.1 g aliquot of a solution containing 15 µg/kg Co as CNCbl were transferred into a 0.75 mL polypropylene vial to constitute a spiked sample for the purpose of quantification by the method of standard addition. A 0.6 g aliquot of each subsample and 0.1 g water were transferred into a separate 0.75 mL polypropylene vial to constitute an unspiked sample for the measurement.

3.4.4. Measurement Process

The mass fractions of CNCbl in the spiked and the unspiked samples were measured with LC--ICP-MS using the separation and spectrometric parameters listed in Table 12 by the method of standard additions (see Section 3.2.4.1). Free Co from the internal standard and Co from CNCbl were measured at 59 Da in the standard mode of the DRCII ICP-MS instrument. All samples were measured in a continuous run spanning two days. Two analytical runs were conducted on the samples with 10 µL injection volume per sample in the first run and 25 µL injection volume per sample in the second run. The results for 10 µL and 25 µL injection volumes per sample were found statistically equivalent; however, the results using the 25 µL injection volume per sample have a better signal-to-background ratio and were used for the quantitative analysis. Fig. 4 displays a typical chromatogram of an SRM 1849b sample spiked with Co as the internal standard.

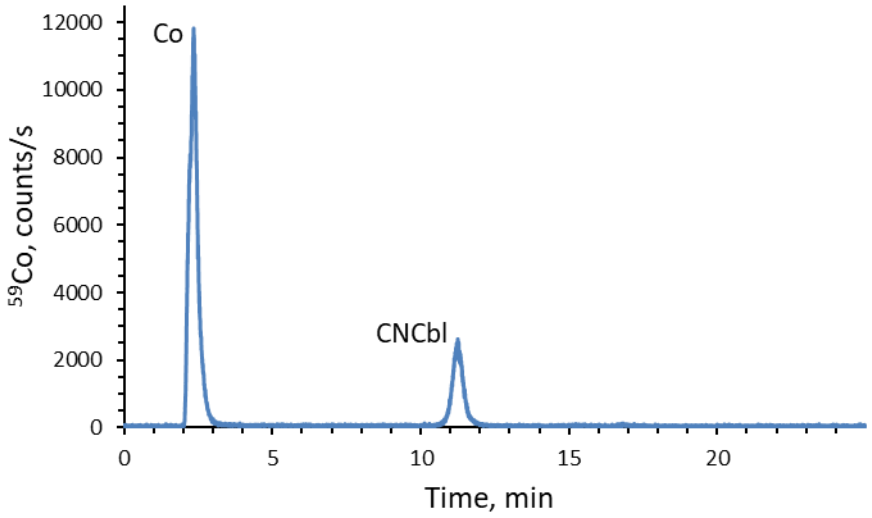

Fig. 4. Exemplar LC-ICP-MS chromatogram for cyanocobalamin in SRM 1849b

3.4.5. Measurement Uncertainty

The standard uncertainty of these LC-ICP-MS measurements is estimated using Equation 9, with the components and their estimated values as described in Table 13.

$$u = \sqrt{u_{\text{rep}}^2 + B_1^2 + B_2^2 + B_3^2 + B_4^2 + B_5^2}\,. \tag{9}$$

Table 13. Cyanocobalamin uncertainty components and estimated values

Component	Description	Degree of Freedom	Value
u_{rep}	Standard uncertainty of replicate LC-ICP-MS measurements	7	0.057
B_1, Calibrant	Experiment-based CNCbl characterization standard uncertainty	large	0.025
B_2, Weighing	Balance calibration specification, converted to standard uncertainty	large	0.049
B_3 Volumetry	Volumetric flask and syringe specifications, converted to standard uncertainty	large	0.024
B_4, Density	Density meter readability, converted to standard uncertainty	large	0.064
B_5, Recovery	Experiment-based method recovery standard uncertainty	large	0.055
u	Combined standard uncertainty:	32	0.083

3.5. NIST ID LC-MS Analysis of Free Carnitine and Total Choline

The mass fractions of free carnitine and total choline in SRM 1849b were determined using microwave-assisted hydrolysis and ID LC-MS [15].

3.5.1. Materials

Six pouches of SRM 1849b, one each from boxes 1, 36, 61, 149, 282, and 290 were analyzed. The pouches of SRM 1849b were selected using a stratified random sampling scheme based on packaging order. Two pouches of SRM 1849a Infant/Adult Nutritional Formula I (milk-based) [5] were used as the quality assurance material, and six samples were prepared using the same procedure as for SRM 1849b.

Carnitine hydrochloride (Cat. C9500-5g, Lot 000133675) and choline bitartrate (Cat. C1629-100g, Lot 011M2016V) were obtained from Sigma-Aldrich (St. Louis, MO, USA) and were used in the preparation of calibration solutions. Purity of the calibrant materials was assessed using quantitative proton NMR (q^1H-NMR) to provide metrological SI-traceability of the measured values. The internal standards, choline chloride trimethyl-d_9 (Cat. D-2142, Lot M280P1) and DL-carnitine HCl trimethyl-d_9 (Cat. D-5780, Lot #DE-245), were obtained from C/D/N Isotopes (Pointe-Claire, QC, Canada).

Hydrochloric acid, ACS grade was purchased from Taylor Scientific (Cat. H2620P-10, Lot 2016031714, St. Louis, MO, USA), ammonium formate (Cat. 70221, Lot BCCB6321) was purchased from Sigma-Aldrich, and sodium hydroxide pellets (Cat. S318-500, Lot 172340) were purchased from Fisher Chemical. HPLC grade water and acetonitrile were used for calibrant and sample preparation, as well as for preparation of LC-MS mobile phases.

3.5.2. Equipment

An Agilent 1200 Series LC system equipped with a binary pump, degasser, autosampler, and column compartment and an Agilent 6130 Quadrupole LC-MS were used to measure carnitine and choline. Separation of carnitine and choline from matrix components and subsequent detection were accomplished using instrument parameters described in Table 14.

Table 14. LC-MS parameters for the determination of carnitine and choline

System	Component	Description	
Agilent Series 1200 LC	Column	Imtakt Scherzo SM-C18, 250 mm x 4.6 mm i.d., 3 µm particle size	
	Mobile phase A	3 mmol/L ammonium formate in water	
	Mobile phase B	10 mmol/L ammonium formate in 80:20 water:acetonitrile (volume fraction)	
	Flow rate	0.5 mL/min	
	Gradient program	Time (min)	%B
		0.0	0
		15.0	100
		25.0	100
		25.1	0
		45.0	0
	Column temperature	25 °C	
	Injection volume	5 µL	
Agilent 6130 Quadrupole MS	Ionization	Electrospray, 1000 V, positive mode	
	Drying gas	12 L/min, 350 °C	
	Nebulizer	60 psig	
	Detection	Analyte	*m/z*
		choline	104
		choline-d_9	113
		carnitine	162
		carnitine-d_9	171

A Mettler model XPR205 analytical balance was used for weighing during the preparation of samples and standards. The balance is serviced and calibrated annually by Mettler. A Microwave Assisted Reaction System (MARS) from CEM Corporation (Matthews, NC, USA) was used for sample preparation. A Beckman Coulter Allegra model X-14R centrifuge (Brea, CA, USA) was used to separate particulate matter from the aqueous phase of the samples.

3.5.3. Sample Preparation

Triplicate 1 g aliquots from each pouch of SRM 1849b and SRM 1849a were accurately weighed into 50 mL polyethylene centrifuge tubes. One sample from each box was prepared on each of 3 days. To each tube, aliquots of 660 µL of carnitine-*d9* solution and 574 µL of choline-*d9* solution, as well as 30 mL of 1 mol/L hydrochloric acid solution. The contents of each tube were mixed by vortexing and transferred into a Teflon Xpress vessel (CEM Corporation, Matthews, NC, USA) designed for microwave extraction. Samples were heated to 110 °C over 15 min and held at 110 °C for 30 min. After cooling to room temperature, samples were individually transferred to clean 50 mL polyethylene tubes. The pH of each sample was adjusted to pH 3.8 to pH 4.0 using 50 % (w/v) sodium hydroxide and confirmed with litmus paper. Samples were centrifuged at 3000 rpm for 15 min and approximately 2 mL to 4 mL of the supernatant was filtered through a 0.45 µm regenerated cellulose syringe filter. Filtered samples were diluted 200-fold by combining

50 µL of supernatant with 10 mL water. Approximately 1 mL of the final diluted sample was transferred to an HLPC vial and stored under refrigeration (2 °C to 8 °C) until analysis by LC-MS.

3.5.4. Measurement Process

The mass fractions of carnitine and choline in the prepared samples were measured with LC-MS using the separation and spectrometric parameters listed in Table 14 through the method of isotope dilution. The MS peak area was manually integrated and recorded for each chromatographic peak, and a response factor for each the calibrants was calculated as described in equation 10.

$$response\ factor = \frac{peak\ area\ (analyte)}{peak\ area\ (internal\ standard)} \times \frac{mass\ (internal\ standard)}{mass\ (analyte)} \quad (10)$$

The average response factors were calculated across independently prepared calibrants injected on the three different days. The average response factor was then used to calculate the mass fraction of carnitine and choline in each sample, as described in equation 11.

$$analyte\ mass\ fraction = \frac{peak\ area\ (analyte)}{peak\ area\ (internal\ standard)} \times \frac{mass\ (internal\ standard)}{response\ factor} \times \frac{1}{mass\ (sample)} \quad (11)$$

Fig. 5 displays a typical chromatogram of an SRM 1849b sample spiked with isotopically labeled carnitine and choline as the internal standards.

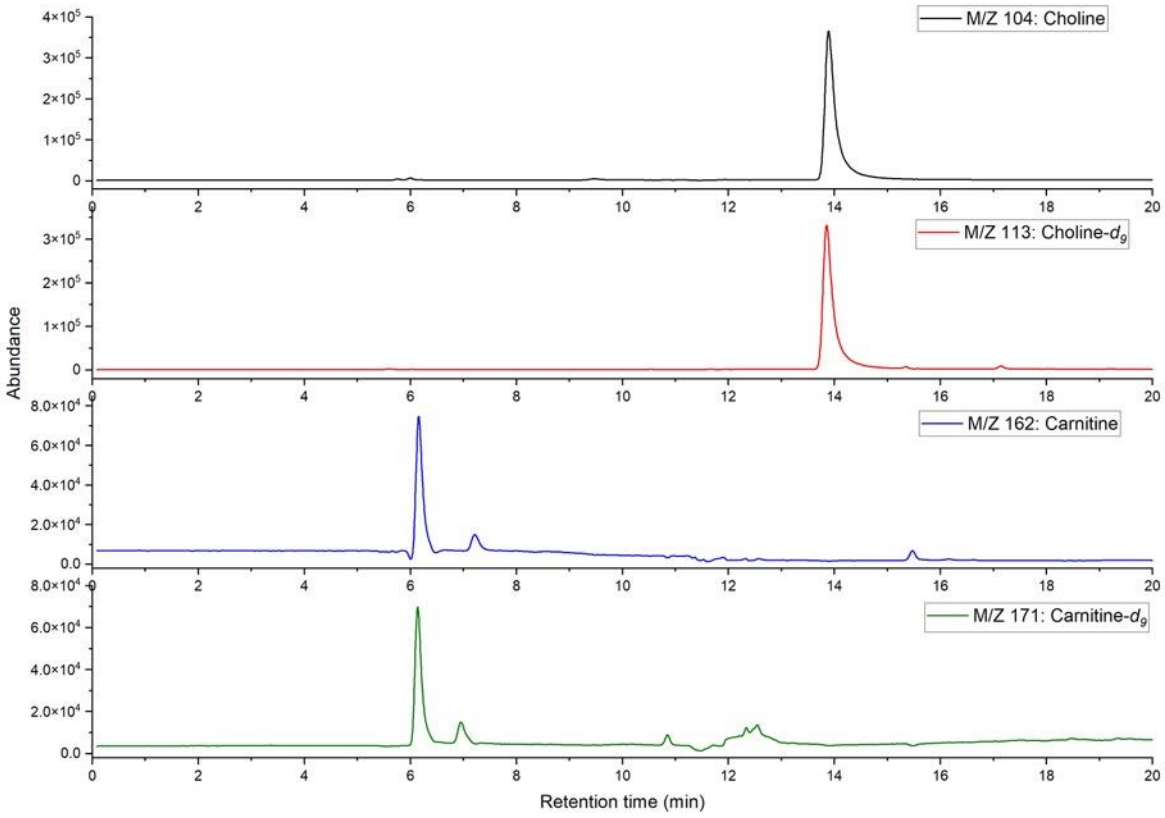

Fig. 5. Exemplar ID LC-MS chromatogram for carnitine and choline in SRM 1849b

4. Interlaboratory Studies

Results from four NIST-coordinated ILS were used to help assign quantitative values to many SRM 1849b components: HAMQAP Exercises 5 and 6 and FNSQAP Exercises 1 and 2. HAMQAP was established in collaboration with the NIH ODS in 2017 to enable laboratories to improve the accuracy of measurements in samples that represent human intake (e.g., foods, dietary supplements, tobacco) and samples that represent human metabolism (e.g., blood, serum, plasma, urine) for demonstration of proficiency and/or compliance with various regulations. FNSQAP was launched in 2021 following reorganization of NIST QAPs to better serve various stakeholder groups, but with the same intention as HAMQAP, to help laboratories improve the accuracy of measurements in food samples. Participation in HAMQAP and FNSQAP is voluntary and anonymous.

Samples of SRM 1849b were tested for the following measurands via HAMQAP:

- Exercise 5, Spring 2020, prefix "E": thiamine (vitamin B_1), riboflavin (vitamin B_2), niacinamide (vitamin B_3), pantothenic acid (vitamin B_5), pyridoxine (vitamin B_6), ergocalciferol (vitamin D_2), and cholecalciferol (vitamin D_3) [16].
- Exercise 6, Spring 2021, prefix "F": chromium, chlorine, iodine, molybdenum, selenium, retinyl acetate (vitamin A), retinyl palmitate (vitamin A), total retinol (vitamin A), biotin (vitamin B7), ascorbic acid (vitamin C), α-tocopherol (vitamin E), total α-tocopherol (vitamin E), α-tocopherol acetate (vitamin E), δ-tocopherol (vitamin E), γ-tocopherol (vitamin E), ash, calories, carbohydrates, fat, protein, and solids [17].

Samples of SRM 1849b were tested for the following measurands via FNSQAP:

- Exercise 1, Summer 2021, prefix "A": calcium, sodium, iron, potassium, folic acid (vitamin B_9), and vitamin K (phylloquinone) [18].
- Exercise 2, Winter 2022, prefix "B": choline, carnitine, β-carotene, lutein, lycopene, arachidonic acid (ARA), docosahexaenoic acid (DHA) [19].

Results from laboratory participants in each ILS are anonymized and are identified with a unique code consisting of an alphabetic study-specific prefix and an arbitrary numeric index ranging from one to a maximum equal to the number of participating laboratories in the exercise.

While all the results reported to the HAMQAP and FNSQAP ILS are presented in the following sections, results identified as non-representative using standard outlier detection methods are not used in value assignment. Such values are identified with a corresponding footnote in each table.

5. Results and Discussion

Results provided by the manufacturer, NIST, and/or by collaborating laboratories participating in one of the HAMQAP or FNSQAP ILS will be presented in a single table that displays all relevant results using the format outlined in Table 15.

Table 15. Exemplar mass fraction results, mg/kg

Manufacturer		Other Results						
ID[a]	Value	Box	Rep_1	…	Rep_m	Mean[b]	SD[c]	
1:1	x_1	1	x_{11}	…	x_{1m}	$\bar{x}_1$	s_1	
…	…	…	…	…	…	…	…	
8:2	x_{11}	109	x_{n1}	…	x_{nm}	$\bar{x}_n$	s_n	
N:	d				*N*:	d		
Mean:	e				Mean, Pooled SD:	e	g	
SD:	f				SD:	f		
					U:	h		

[a] The sample identifier for the manufacturer-supplied results concatenates the pallet and sample indices.
[b] The arithmetic average of the sample replicates.
[c] The standard deviation of the sample replicates.
[d] The number of quantitative results available, either single values (for the manufacturer) or sample means (of two replicates for NIST results or three replicates for ILS results).
[e] The arithmetic average of the (single or sample mean) quantitative results.
[f] The standard deviation of the (single or sample mean) quantitative results.
[g] The pooled sample replicate standard deviation (effectively the average sample replicate SD).
[h] The measurement uncertainty reported by the NIST analyst, where available.

When sufficient quantitative results are available, the data are also presented graphically as exemplified in Fig. 6. The manufacturer's results are solid black circles, NIST results are various symbols with error bars representing one standard deviation above and below the mean, and ILS results are presented in boxplot format. The width of the box is proportional to the square root of the number of quantitative values; top, middle, and bottom lines of the box represent the 75th, 50th, and 25th percentiles; and the whiskers span from the 2.5th to the 97.5th percentile. Results are ordered by the production sequence determined by the pallet and box index of the sampled pouch.

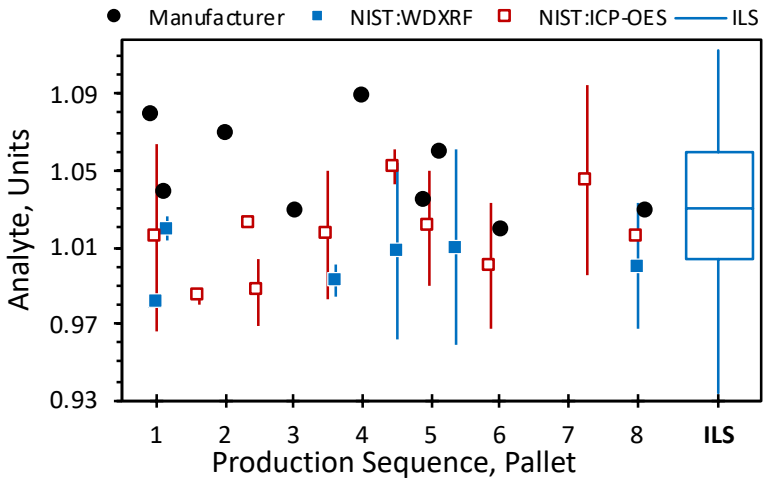

Fig. 6. Exemplar multi-source mass fraction as a function of production sequence

When only the manufacturer-provided results are available for a family of related measurands (e.g., amino acids), results are presented in tabular form using the format in Table 16.

Table 16. Exemplar manufacturer-only mass fraction results, mg/kg

	Manufacturer Results				
Assay	1:1 [a]	…	8:2 [a]	Mean [b]	SD [c]
Measurand$_i$	x_{i1}	…	x_{i11}	$\bar{x}_i$	s_1
…	…	…	…	…	…
Measurand$_m$	x_{m1}	…	x_{m11}	$\bar{x}_m$	s_m

[a] The sample identifier for the manufacturer-supplied results concatenates the pallet and sample indices. The sample results are presented in the order of analysis.
[b] The arithmetic average of the eleven results, one per pouch analyzed.
[c] The standard deviation of the eleven results.

5.1. Elements

5.1.1. Aluminum (Al)

The manufacturer electrothermal AAS [20] and NIST WDXRF measurement results for aluminum (Al) are summarized in Table 17. The Al mass fraction is close to or below the limit of quantification for both methods.

Table 17. Summary of results for aluminum (Al), mg/kg

Manufacturer	
ID	Value
1:1	0.519
2:1	0.693
3:1	<0.455[a]
4:1	<0.468[a]
5:1	0.524
5:2	<0.473[a]
6:1	<0.476[a]
7:1	0.527
8:1	0.570
1:2	0.521
8:2	<0.467[a]
N:	6
Mean:	0.559
SD:	0.068

NIST WDXRF				
Box	Rep_1	Rep_2	Mean	SD
1	4.58	-0.30[a]	4.58	
7	0.82	2.13	1.48	0.93
109	4.50	0.87	2.69	2.57
146	4.15	-0.36[a]	4.15	
182	2.07	-1.70[a]	2.07	
290	1.55	0.71	1.13	0.59
		N:	6	
		Mean, Pooled SD:	2.68	1.61
		SD:	1.41	

[a] Identified as a non-representative value; excluded from statistical analysis.

5.1.2. Calcium (Ca)

The manufacturer ICP-OES [26], NIST WDXRF, and FNSQAP [18] measurement results for calcium (Ca) are summarized in Table 18. Fig. 7 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 18. Summary of results for calcium (Ca), mg/kg

Manufacturer

ID	Value
1:1	4890
2:1	4740
3:1	5000
4:1	4920
5:1	4940
5:2	4790
6:1	5340
7:1	4910
8:1	4880
1:2	4790
8:2	4890
N:	11
Mean:	4917
SD:	159

NIST WDXRF

Box	Rep_1	Rep_2	Mean	SD
1	5223	5203	5213	14
7	5226	5174	5200	37
109	5226	5253	5240	19
146	5243	5237	5240	4
182	5174	5207	5191	23
290	5208	5206	5207	1
		N:	6	
		Mean, Pooled SD:	5215	20
		SD:	21	
		U:	795	

FNSQAP Exercise 1

Code	Rep_1	Rep_2	Rep_3	Mean	SD	Method
A001	5006	5034	5010	5017	15	ICP-MS
A002	5140	5140	5040	5107	58	NAA
A006	5832	5226	5098	5385	392	ICP-MS
A010	4955	4955	4955	4955	0	ICP-OES
A012	4964	4918	4829	4903	69	ICP-OES
A015	5169	5184	5186	5180	9	ICP-OES
A017	5167	5187	5292	5215	67	ICP-MS
A019	4440	4470	4790	4567	194	ICP-MS
A020	4760	4710	4840	4770	66	ICP-OES
A021	4685	4595	4482	4587	102	ICP-MS KED Mode
A023	4840	4840	4820	4833	12	ICP-MS
A027	5386	5570	5701	5552	158	ICP-OES
A028	5144	5228	5100	5157	65	ICP-OES
A030	4920	5070	4990	4993	75	ICP-MS
A031	5308	5485	5430	5408	91	ICP-MS
A032	4600	4750	4700	4683	76	ICP-OES
A034	5124	5100	5127	5117	15	ICP-OES
A035	4972	5124	4882	4993	122	ICP-OES
A038	5040	4930	4960	4977	57	ICP-OES
A039	6743	4948	4682	5458	1121	ICP-MS
A040	4620	4740	4690	4683	60	ICP-MS
A041	4610	4592	4486	4563	68	ICP-MS
A042	5071	4840	5233	5048	198	ICP-MS KED Mode
A045	937[a]	1144[a]	1114[a]			ICP-MS
A046	5160	5150	5140	5150	10	ICP-OES
A049	5263	5431	5354	5349	84	ICP-MS
A050	5020	5065	4725	4937	185	TXRF
A057	5021	5002	5035	5019	17	ICP-OES
			N:	27		
			Mean, Pooled SD:	5022	246	
			SD:	273		

[a] Identified as a non-representative value; excluded from statistical analysis.

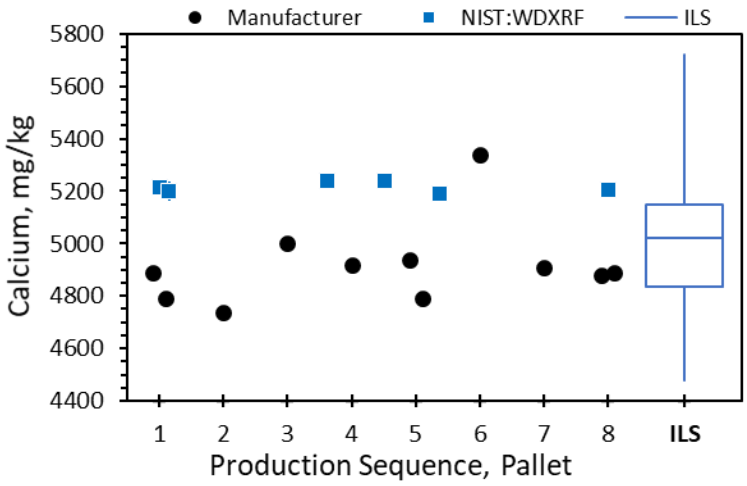

Fig. 7. Calcium (Ca) mass fraction as a function of production sequence

5.1.3. Chlorine (Cl)

The manufacturer potentiometric [21-23], NIST WDXRF, and HAMQAP [17] measurement results for chlorine (Cl) or, equivalently, chloride (Cl^-), are summarized in Table 19. Fig. 8 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 19. Summary of results for chlorine (Cl), mg/kg

Manufacturer

ID	Value
1:1	6600
2:1	6600
3:1	6610
4:1	6610
5:1	6610
5:2	6610
6:1	6620
7:1	6610
8:1	6610
1:2	6610
8:2	6610
N:	11
Mean:	6609
SD:	5

NIST WDXRF

Box	Rep_1	Rep_2	Mean	SD
1	6533	6419	6476	81
7	6506	6437	6472	49
109	6453	6552	6503	70
146	6444	6514	6479	49
182	6504	6551	6528	33
290	6453	6521	6487	34
		N:	6	
		Mean, Pooled SD:	6491	57
		SD:	21	
		U:	1331	

HAMQAP Exercise 6

Code	Rep_1	Rep_2	Rep_3	Mean	SD	Method
F030	6640	6650	6620	6637	15	ISE
F034	6520	6560	6300	6460	140	Other
F039	6640	6650	6640	6643	6	ISE
F061	6540	6550	661[a]	6545	7	Titration
F067	6651	6843	6952	6815	152	IC-CD
			N:	5		
			Mean, Pooled SD:	6620	93	
			SD:	132		

[a] Identified as a non-representative value; excluded from statistical analysis.

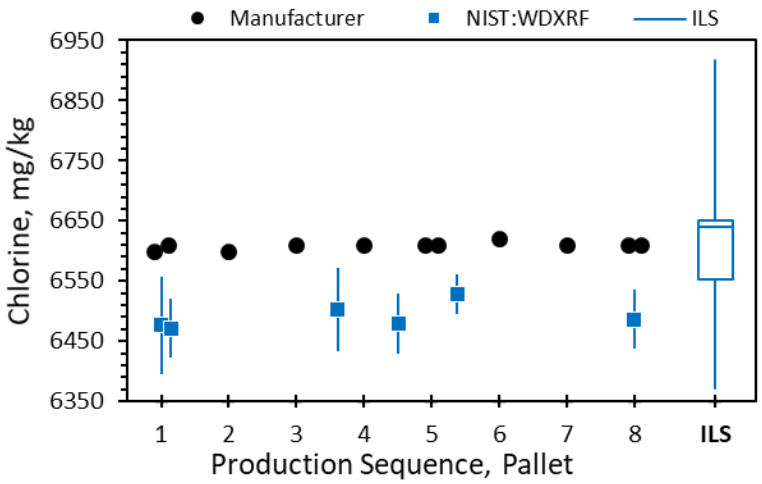

Fig. 8. Chlorine (Cl) mass fraction as a function of production sequence

5.1.4. Chromium (Cr)

The manufacturer ICP-MS [24], NIST ICP-MS, and HAMQAP [17] measurement results for chromium (Cr) are summarized in Table 20. Fig. 9 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 20. Summary of results for chromium (Cr), mg/kg

Manufacturer

ID	Value
1:1	1.08
2:1	1.04
3:1	1.04
4:1	1.03
5:1	1.09
5:2	1.03
6:1	1.03
7:1	1.02
8:1	1.03
1:2	1.03
8:2	1.07
N:	11
Mean:	1.044
SD:	0.024

NIST ICP-MS

Box	Rep_1	Rep_2	Mean	SD
1	0.981	1.050	1.016	0.049
27	0.987	0.981	0.984	0.004
58	1.021	1.024	1.023	0.002
62	0.974	0.999	0.987	0.018
104	0.993	1.040	1.017	0.033
144	1.058	1.046	1.052	0.008
166	0.954	1.046	1.000	0.065
202	0.977	1.023	1.000	0.033
260	0.975	1.154	1.065	0.127
290	1.015		1.015	
		N:	10	
		Mean, Pooled SD:	1.016	0.053
		SD:	0.026	
		U:	31	

HAMQAP Exercise 6

Code	Rep_1	Rep_2	Rep_3	Mean	SD	Method
F005	0.620[a]	0.629[a]	0.582[a]			ICP-MS
F011	1.040	1.060	1.060	1.053	0.012	ICP-MS
F017	1.08	1.07	0.994	1.048	0.047	ICP-MS
F020	1.050	1.010	1.000	1.020	0.026	ICP-MS
F022	1.100	1.180	1.060	1.113	0.061	ICP-MS
F026	1.033	1.015	1.003	1.017	0.015	ICP-MS
F030	1.050	1.070	1.100	1.073	0.025	ICP-MS
F031	1.080	1.020	1.020	1.040	0.035	ID ICP-MS
F033	1.040	1.030	1.050	1.040	0.010	ICP-MS
F034	1.019	1.015	1.008	1.014	0.006	ICP-MS
F039	1.060	1.090	1.040	1.063	0.025	ICP-MS
F041	0.960	0.930	0.970	0.953	0.021	ICP-MS
F042	0.933	0.974	0.970	0.959	0.023	ID ICP-MS
F046	1.007	1.069	0.056	1.069	0.056	ICP-MS
F061	80.5[a]	80.6[a]	83.5[a]			ICP-MS
F062	0.967	0.945	0.942	0.951	0.014	ID ICP-MS
F069	1.5[a]	1.5[a]	1.5[a]			ICP OES
F070	1.070	1.000	1.020	1.030	0.036	ICP-MS
F074	1.058[a]	0.715[a]	0.633[a]			ID ICP-MS
F079	1.040	1.080	1.030	1.050	0.026	ICP-MS
F085	1.004	1.030	1.040	1.025	0.019	ICP-MS
			N:	17		
			Mean, Pooled SD	1.031	0.031	
			SD:	0.044		

[a] Identified as a non-representative value; excluded from statistical analysis.

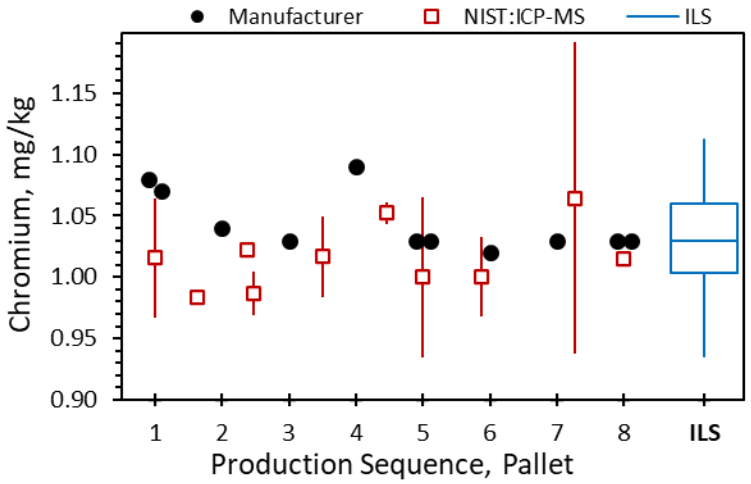

Fig. 9. Chromium (Cr) mass fraction as a function of production sequence

5.1.5. Copper (Cu)

The manufacturer ICP-OES [26], NIST WDXRF, and NIST ICP-OES measurement results for copper (Cu) are summarized in Table 21. Fig. 10 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 21. Summary of results for copper (Cu), mg/kg

Manufacturer	
ID	Value
1:1	18.6
2:1	18.1
3:1	18.6
4:1	18.8
5:1	19.1
5:2	18.2
6:1	18.8
7:1	18.9
8:1	18.8
1:2	18.3
8:2	18.7
N:	11
Mean:	18.63
SD:	0.31

NIST WDXRF				
Box	Rep_1	Rep_2	Mean	SD
1	18.94	18.91	18.93	0.02
7	19.15	18.93	19.04	0.16
109	18.94	19.12	19.03	0.13
146	19.07	19.39	19.23	0.23
182	18.79	18.90	18.85	0.08
290	18.98	18.89	18.94	0.06
		N:	6	
		Mean, Pooled SD:	19.00	0.13
		SD:	0.13	
		U:	0.89	

NIST ICP-OES				
Box	Rep_1	Rep_2	Mean	SD
1	19.55	19.15	19.35	0.28
27	19.31	18.91	19.11	0.28
58	19.03	18.66	18.85	0.26
62	19.40	19.37	19.39	0.02
104	19.73	18.96	19.35	0.54
144	19.13	19.50	19.32	0.26
166	19.60	19.68	19.64	0.06
202	19.24	19.11	19.18	0.09
260	19.48	19.15	19.32	0.23
290	18.95		18.95	
		N:	10	
		Mean, Pooled SD:	19.24	0.27
		SD:	0.23	
		U:	0.15	

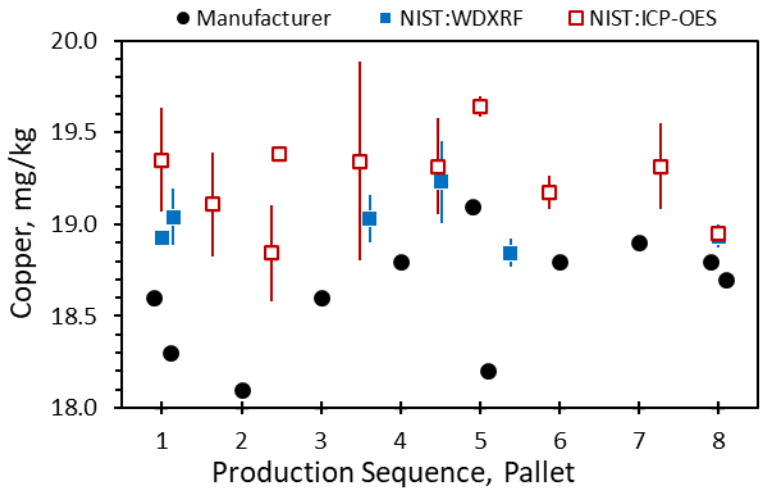

Fig. 10. Copper (Cu) mass fraction as a function of production sequence

5.1.6. Fluorine (F)

The manufacturer ion-selective electrode measurement results for fluoride (F^-), reported as fluorine (F), are summarized in Table 22. Fig. 11 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 22. Summary of Results for Fluorine (F), mg/kg

Manufacturer	
ID	Value
1:1	1.64
2:1	1.60
3:1	1.64
4:1	1.65
5:1	1.80
5:2	1.64
6:1	1.57
7:1	1.59
8:1	1.45
1:2	1.66
8:2	1.47
N:	11
Mean:	1.610
SD:	0.095

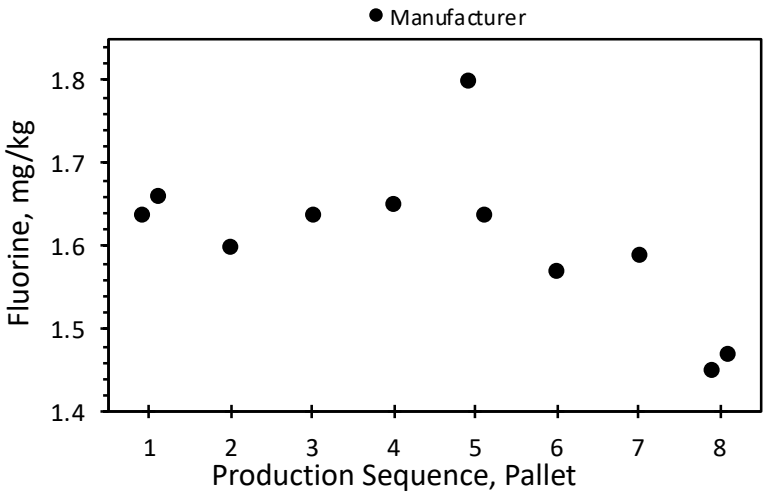

Fig. 11. Fluorine (F) mass fraction as a function of production sequence

5.1.7. Iodine (I)

The manufacturer ICP-MS [25], NIST ICP-MS, and HAMQAP [17] measurement results for iodine (I) are summarized in Table 23. Fig. 12 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 23. Summary of results for iodine (I), mg/kg

Manufacturer

ID	Value
1:1	2.12
2:1	2.07
3:1	2.10
4:1	2.09
5:1	2.05
5:2	2.10
6:1	2.09
7:1	2.13
8:1	2.17
1:2	2.10
8:2	2.03
N:	11
Mean:	2.095
SD:	0.038

NIST ICP-MS

Box	Rep$_1$	Rep$_2$	Mean	SD
1	2.634	1.771	2.203	0.610
27	2.451	1.823	2.137	0.444
58	2.050	2.097	2.074	0.033
62	1.757	2.066	1.912	0.218
104	2.291	1.946	2.119	0.244
144	1.720	1.882	1.801	0.115
166	2.416	1.783	2.100	0.448
202	1.859	1.850	1.855	0.006
260	2.729	2.039	2.384	0.488
290	1.856	2.354	2.105	0.352
		N:	10	
		Mean, Pooled SD:	2.069	0.354
		SD:	0.173	
		U:	180	

HAMQAP Exercise 6

Code	Rep$_1$	Rep$_2$	Rep$_3$	Mean	SD	Method
F005	3.550	3.560	3.580	3.563	0.015	ISE
F017	2.520	2.630	2.530	2.560	0.061	ICP-OES
F026	1.872	1.950	1.900	1.907	0.040	ICP-MS
F031	1.620	1.550	1.550	1.573	0.040	ID ICP-MS
F033	2.260	2.290	2.320	2.290	0.030	ICP-MS
F034	1.42	1.45		1.44	0.021	ICP-MS
F062	1.575	1.610	1.655	1.613	0.040	ID ICP-MS
F067	1.840	1.940	2.020	1.933	0.090	IC-ECD
F070	13.52[a]	10.3[a]	6.61[a]			ICP-MS
			N:	8		
			Mean, Pooled SD:	2.109	0.048	
			SD:	0.698		

[a] Identified as a non-representative value; excluded from statistical analysis.

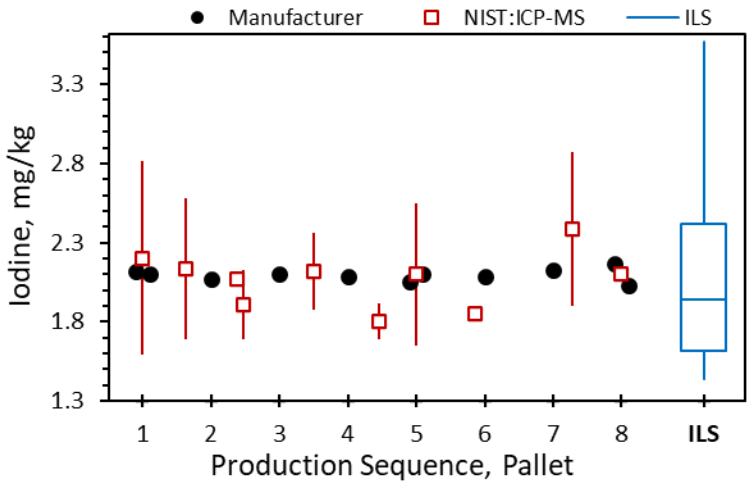

Fig. 12. Iodine (I) mass fraction as a function of production sequence

5.1.8. Iron (Fe)

The manufacturer ICP-OES [26], NIST WDXRF, NIST ICP-OES, and FNSQAP [18] measurement results for iron (Fe) are summarized in Table 24. Fig. 13 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 24. Summary of results for iron (Fe), mg/kg

Manufacturer	
ID	Value
1:1	162
2:1	158
3:1	161
4:1	163
5:1	162
5:2	159
6:1	162
7:1	164
8:1	161
1:2	160
8:2	161
N:	11
Mean:	161.2
SD:	1.7

NIST WDXRF					
Box	Rep_1	Rep_2	Mean	SD	
1	171.7	171.1	171.4	0.4	
7	173.0	173.2	173.1	0.2	
109	171.7	172.0	171.8	0.2	
146	171.5	176.0	173.7	3.2	
182	170.3	170.9	170.6	0.5	
290	170.9	170.7	170.8	0.2	
		N:	6		
		Mean, Pooled SD:	171.9	1.3	
		SD:	1.3		
		U:	16		

NIST ICP-OES				
Box	Rep_1	Rep_2	Mean	SD
1	163.9	163.9	163.9	0.0
27	163.5	166.3	164.9	2.0
58	170.2	168.2	169.2	1.4
62	164.1	171.2	167.7	5.0
104	167.1	178.3	172.7	7.9
144	201.9[a]	177.4	189.7	17.3
166	165.8	166.7	166.3	0.6
202	164.6	165.8	164.6	0.8
260	167.7	173.6	170.7	4.2
290	163.0		163.0	
		N:	10	
		Mean, Pooled SD:	169.3	6.8
		SD:	7.8	
		U:	4.3	

FNSQAP Exercise 1						
Code	Rep_1	Rep_2	Rep_3	Mean	SD	Method
A001	169	167	169	168	0.8	ICP-MS
A002	186	188	203	192	9.3	NAA
A006	354	215	213	261	80	ICP-MS
A010	187	187	187	187	0	ICP-OES
A012	161	160	158	160	1.6	ICP-OES
A015	161	162	163	162	0.9	ICP-OES
A017	174	158	176	170	10	ICP-MS
A019	157	161	164	161	3.5	ICP-MS
A020	153	154	157	155	2.1	ICP-OES
A021	187	181	175	181	6.0	ICP-MS KED Mode
A023	175	171	172	173	2.1	ICP-MS KED Mode
A027	179	189	198	189	10	ICP-OES
A028	169	172	167	169	2.5	ICP-OES
A030	170	172	170	171	1.2	ICP-MS
A031	175	183	179	179	4.2	ICP-MS
A032	140	146	147	144	3.7	ICP-OES
A034	170	168	172	170	1.9	ICP-OES
A035	183	180	184	182	2.1	ICP-OES
A038	163	160	165	163	2.5	ICP-OES
A039	252	184	163	200	47	ICP-MS
A040	161	154	157	157	3.5	ICP-MS
A041	149	141	140	143	4.7	ICP-MS
A042	152	150	150	151	1.3	ICP-MS KED Mode
A045	174	177	196	182	12	ICP-MS
A046	156	157	157	157	0.6	ICP-OES
A049	164	171	168	168	3.3	ICP-MS
A050	180	182	166	176	8.7	TRXF
A057	164	165	165	165	0.6	ICP-OES
A061	160	160	162	161	0.9	UV Spectroscopy
			N:	28		
			Mean, Pooled SD:	173	18	
			SD:	22		

[a] The suspected reason for this higher data point is failure of proper washout as this sample was run directly after the sample calibration curve. Limited laboratory access prevented rerunning of particular sample to verify that this was the cause of the higher value.

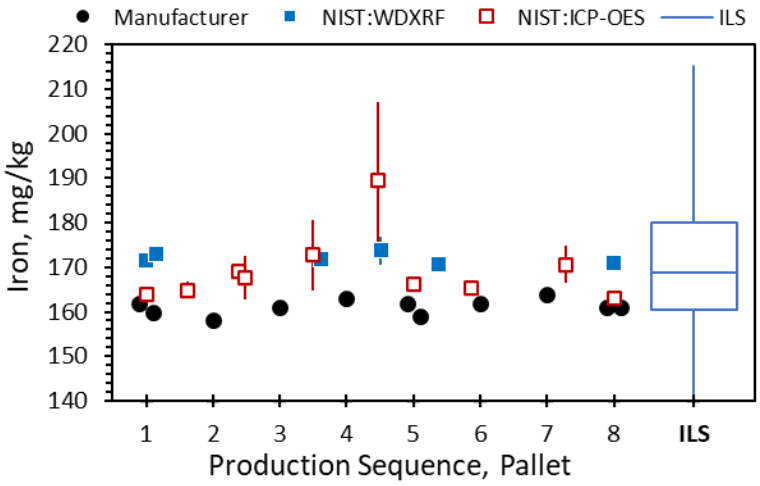

Fig. 13. Iron (Fe) mass fraction as a function of production sequence

5.1.9. Magnesium (Mg)

The manufacturer ICP-OES [26], NIST WDXRF, and NIST ICP-OES measurement results for magnesium (Mg) are summarized in Table 25. Fig. 14 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 25. Summary of results for magnesium (Mg), mg/kg

Manufacturer

ID	Value
1:1	1530
2:1	1490
3:1	1520
4:1	1550
5:1	1600
5:2	1520
6:1	1550
7:1	1540
8:1	1530
1:2	1510
8:2	1550
N:	11
Mean:	1535
SD:	28

NIST WDXRF

Box	Rep$_1$	Rep$_2$	Mean	SD
1	1647	1539	1593	76
7	1598	1563	1580	25
109	1582	1705	1644	87
146	1571	1609	1590	27
182	1617	1638	1628	15
290	1526	1633	1579	76
		N:	6	
		Mean, Pooled SD:	1602	59
		U:	134	

NIST ICP-OES

Box	Rep$_1$	Rep$_2$	Mean	SD
1	1550	1574	1562	17
27	1571	1558	1565	9
58	1554	1580	1567	18
62	1582	1567	1575	11
104	1560	1564	1562	3
144	1571	1563	1567	6
166	1578	1571	1575	5
202	1567	1554	1561	9
260	1573	1584	1579	8
290	1566		1566	
		N:	10	
		Mean, Pooled SD:	1568	11
		SD:	6	
		U:	5.3	

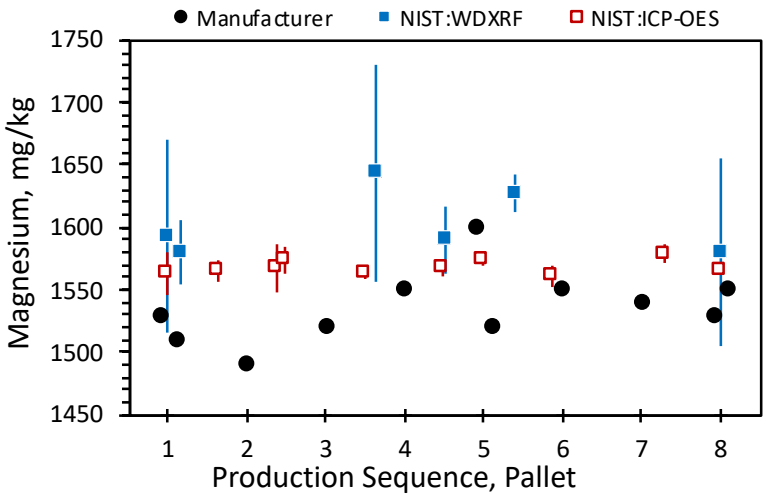

Fig. 14. Magnesium (Mg) mass fraction as a function of production sequence

5.1.10. Manganese (Mn)

The manufacturer ICP-OES [26] and NIST WDXRF measurement results for manganese (Mn) are summarized in Table 26. Fig. 15 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 26. Summary of results for manganese (Mn), mg/kg

Manufacturer	
ID	Value
1:1	45.6
2:1	44.2
3:1	45.2
4:1	45.7
5:1	45.6
5:2	44.7
6:1	45.7
7:1	45.9
8:1	45.4
1:2	44.7
8:2	45.5
N:	11
Mean:	45.29
SD:	0.53

NIST WDXRF				
Box	Rep_1	Rep_2	Mean	SD
1	47.34	47.05	47.20	0.21
7	48.58	45.85	47.22	1.93
109	47.90	47.78	47.84	0.08
146	47.34	47.19	47.27	0.11
182	47.23	47.78	47.51	0.39
290	48.10	46.82	47.46	0.91
		N:	6	
		Mean, Pooled SD:	47.41	0.89
		SD:	0.25	
		U:	16	

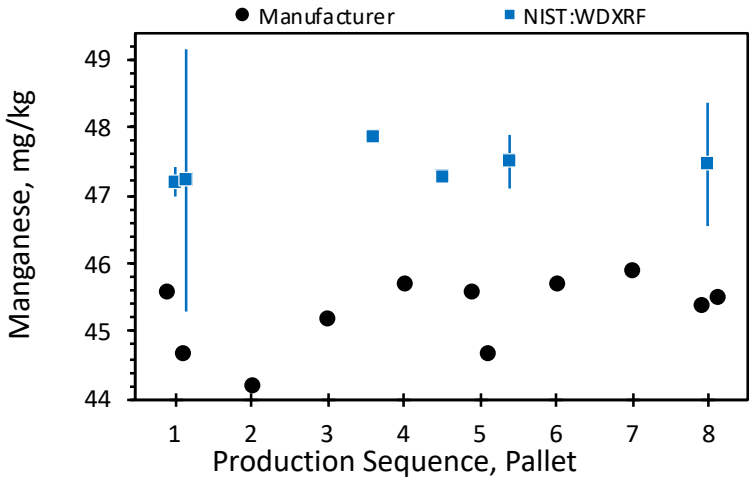

Fig. 15. Manganese (Mn) mass fraction as a function of production sequence

5.1.11. Molybdenum (Mo)

The manufacturer ICP-MS [24], NIST ICP-MS, and HAMQAP [17] measurement results for molybdenum (Mo) are summarized in Table 27. Fig. 16 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 27. Summary of results for molybdenum (Mo), mg/kg

Manufacturer

ID	Value
1:1	1.82
2:1	1.78
3:1	1.77
4:1	1.90
5:1	1.79
5:2	1.78
6:1	1.77
7:1	1.79
8:1	1.78
1:2	1.79
8:2	1.78
N:	11
Mean:	1.795
SD:	0.037

NIST ICP-MS

Box	Rep_1	Rep_2	Mean	SD
1	1.699	1.724	1.712	0.018
27	1.742	1.718	1.730	0.017
58	1.715	1.729	1.722	0.010
62	1.723	1.729	1.726	0.004
104	1.703	1.746	1.725	0.030
144	1.720	1.727	1.724	0.005
166	1.709	1.738	1.724	0.021
202	1.728	1.735	1.732	0.005
260	1.724	1.692	1.708	0.023
290	1.714		1.714	
		N:	10	
		Mean, Pooled SD:	1.772	0.017
	0.037	SD:	0.014	
		U:	0.008	

HAMQAP Exercise 6

Code	Rep_1	Rep_2	Rep_3	Mean	SD	Method
F005	5.265[a]	4.673[a]	4.445[a]			ICP-MS
F011	1.540	1.520	1.500	1.520	0.020	ICP-MS
F017	1.770	1.870	1.820	1.820	0.050	LC MS
F020	1.310	1.329	1.309	1.316	0.011	ICP-MS
F026	1.506	1.530	1.388	1.475	0.076	ICP-MS
F030	1.920	1.960	1.930	1.937	0.021	ICP-MS
F031	1.720	1.790	1.730	1.747	0.038	ID ICP-MS
F033	1.780	1.760	1.800	1.780	0.020	ICP-MS
F034	1.727	1.710	1.721	1.719	0.009	ICP-MS
F039	1.820	1.850	1.780	1.817	0.035	ICP-MS
F042	1.670	1.710	1.670	1.683	0.023	ID ICP-MS
F046	2.774[a]	2.135[a]	2.014[a]			ICP-MS
F061	1.419	1.430	1.470	1.440	0.027	ICP-MS
F062	1.695	1.719	1.706	1.707	0.012	ID ICP-MS
F069	0.5[a]	0.5[a]	0.5[a]			ICP-OES
F070	1.700	1.710	1.670	1.693	0.021	ICP-MS
F073	1.679	1.673	1.656	1.669	0.012	ICP-OES
F074	1.857	1.738	1.529	1.708	0.166	ID ICP-MS
F079	1.800	1.750	1.740	1.763	0.032	ICP-MS
F085	1.619	1.671	1.700	1.663	0.041	ICP-MS
			N:	17		
			Mean, Pooled SD:	1.674	0.051	
			SD:	0.155		

[a] Identified as a non-representative value; excluded from statistical analysis.

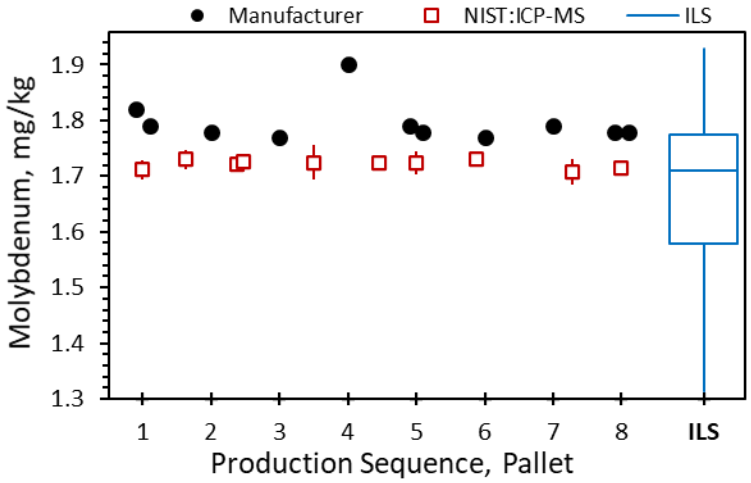

Fig. 16. Molybdenum (Mo) mass fraction as a function of production sequence

5.1.12. Phosphorus (P)

The manufacturer ICP-OES [26] and NIST WDXRF and ICP-OES measurement results for phosphorus (P) are summarized in Table 28. Fig. 17 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 28. Summary of results for phosphorus (P), mg/kg

Manufacturer	
ID	Value
1:1	3620
2:1	3500
3:1	3610
4:1	3650
5:1	3760
5:2	3560
6:1	3660
7:1	3630
8:1	3650
1:2	3580
8:2	3670
N:	11
Mean:	3626
SD:	67

NIST WDXRF				
Box	Rep_1	Rep_2	Mean	SD
1	3840	3745	3792	67
7	3805	3752	3778	37
109	3791	3864	3827	52
146	3773	3808	3790	25
182	3796	3833	3814	26
290	3748	3855	3802	76
		N:	6	
		Mean, Pooled SD:	3801	51
		SD:	18	
		U:	171	

NIST ICP-OES				
Box	Rep_1	Rep_2	Mean	SD
1	4002	3587	3795	293
27	3931	3794	3863	97
58	3897	3792	3845	74
62	3970	3885	3928	60
104	3773	3627	3700	103
144	3733	3627	3680	75
166	3896	3854	3875	30
202	3868	3723	3796	103
260	3768	3977	3873	148
290	3993		3993	
		N:	10	
		Mean, Pooled SD:	3821	130
		SD:	135	
		U:	108	

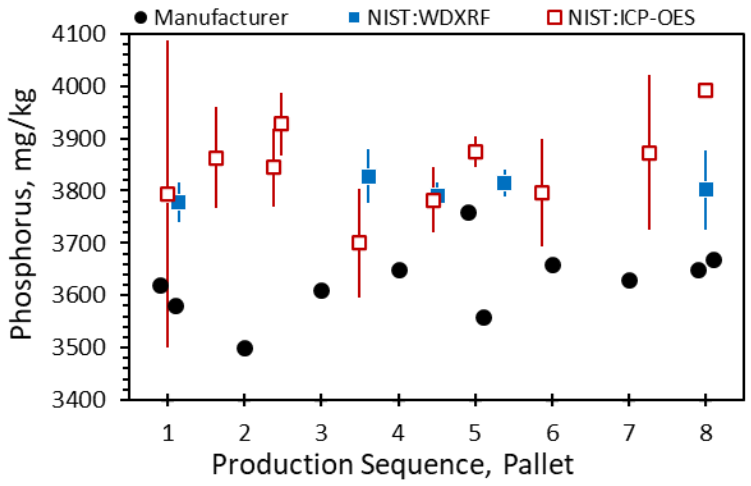

Fig. 17. Phosphorus (P) mass fraction as a function of production sequence

5.1.13. Potassium (K)

The manufacturer ICP-OES [26], NIST WDXRF, and FNSQAP [18] measurement results for potassium (K) are summarized in Table 29. Fig. 18 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 29. Summary of results for potassium (K), mg/kg

Manufacturer

ID	Value
1:1	9030
2:1	8740
3:1	8980
4:1	9080
5:1	8900
5:2	8840
6:1	8980
7:1	9060
8:1	8930
1:2	8850
8:2	9000
N:	11
Mean:	8945
SD:	104

NIST WDXRF

Box	Rep_1	Rep_2	Mean	SD
1	9013	8869	8941	101
7	9000	8920	8960	56
109	8924	8993	8959	49
146	8937	8982	8960	32
182	8938	8985	8961	33
290	8958	8947	8952	8
		N:	6	
		Mean, Pooled SD:	8955	55
		SD:	8	
		U:	485	

FNSQAP Exercise 1

Code	Rep_1	Rep_2	Rep_3	Mean	SD	Method
A001	8893	8895	8935	8908	24	ICP-MS
A002	8900	9640	8850	9130	442	NAA
A006	9827	10274	9985	10029	227	ICP-MS
A010	9757	9749	9889	9799	79	ICP-OES
A012	8650	8659	8480	8596	101	ICP-OES
A015	9172	9295	9317	9261	78	ICP-OES
A017	9347	9550	9602	9500	135	ICP-MS
A019	8500	8630	9190	8773	367	ICP-MS
A020	9170	9240	9140	9183	51	ICP-OES
A021	9438	9027	8991	9152	248	ICP-MS KED Mode
A023	10300	9810	9830	9980	277	ICP-MS
A027	7542	7830	8045	9980	277	ICP-OES
A028	9346	9553	9265	9388	149	ICP-OES
A030	9450	9410	9390	9417	31	CVAAS
A031	10319	10480	9982	10261	254	ICP-MS
A032	7990	8080	7990	8020	52	ICP-OES
A034	9393	9451	9523	9456	65	ICP-OES
A035	9284	9592	9397	9424	156	ICP-OES
A038	9080	8900	8970	8983	91	ICP-OES
A039	9864	9046	8377	9096	745	ICP-MS
A040	8610	8850	8630	8697	133	ICP-MS
A041	8053	7919	7678	7884	190	ICP-MS
A042	8499	8253	8679	8477	214	ICP-MS KED Mode
A045	8976	10159	10356	9830	746	ICP-MS
A046	8740	8670	8650	8687	47	ICP-OES
A049	8673	9403	9184	9087	375	ICP-MS
A050	8865	8985	8465	8772	272	TXRF
A057	9815	9694	9788	9766	64	ICP-OES
			N:	28		
			Mean, Pooled SD:	9120	279	
			SD:	627		

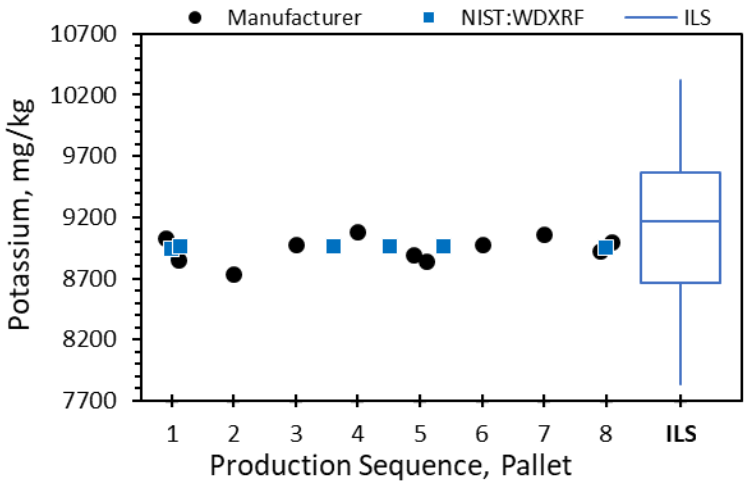

Fig. 18. Potassium (K) mass fraction as a function of production sequence

5.1.14. Rubidium (Rb)

The NIST WDXRF measurement results for rubidium (Rb) are summarized in Table 30. Fig. 19 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 30. Summary of results for rubidium (Rb), mg/kg

	NIST WDXRF			
Box	Rep_1	Rep_2	Mean	SD
1	4.474	4.011	4.243	0.327
7	4.367	4.230	4.299	0.097
109	4.335	4.371	4.353	0.025
146	4.120	4.271	4.196	0.107
182	4.233	4.442	4.338	0.148
290	4.266	4.369	4.318	0.073
		N:	6	
		Mean, Pooled SD:	4.291	0.161
		SD:	0.060	
		U:	4.5	

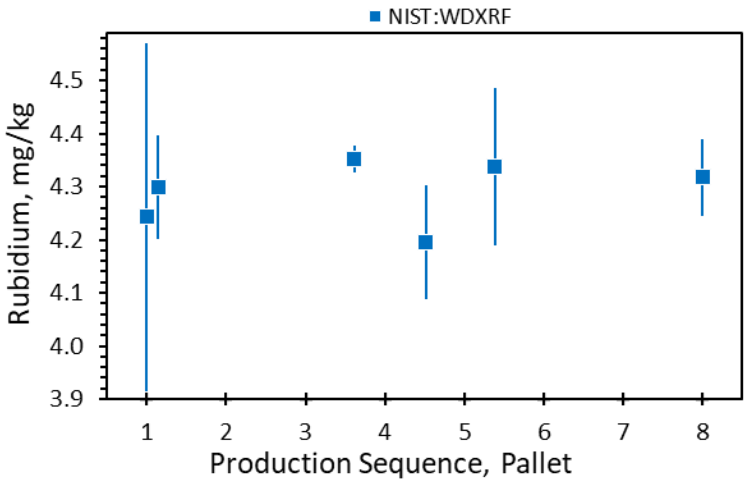

Fig. 19. Rubidium (Rb) mass fraction as a function of production sequence

5.1.15. Selenium (Se)

The manufacturer ICP-MS [24], NIST ICP-MS, and HAMQAP [17] measurement results for selenium (Se) are summarized in Table 31. Fig. 20 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 31. Summary of results for selenium (Se), mg/kg

Manufacturer

ID	Value
1:1	0.841
2:1	0.829
3:1	0.859
4:1	0.877
5:1	0.839
5:2	0.828
6:1	0.820
7:1	0.827
8:1	0.818
1:2	0.831
8:2	0.823
N:	11
Mean:	0.836
SD:	0.018

NIST ICP-MS

Box	Rep$_1$	Rep$_2$	Mean	SD
1	0.781	0.836	0.809	0.039
27	0.826	0.800	0.813	0.018
58	0.898	0.825	0.862	0.052
62	0.769	0.795	0.782	0.018
104	0.777	0.770	0.774	0.005
144	0.885	0.820	0.853	0.046
166	0.813	0.851	0.832	0.027
202	0.747	0.864	0.806	0.083
260	0.785	0.783	0.784	0.001
290	0.786		0.786	
N:			10	
Mean, Pooled SD:			0.810	0.040
SD:			0.030	
U:			0.020	

HAMQAP Exercise 6

Code	Rep$_1$	Rep$_2$	Rep$_3$	Mean	SD	Method
F005	0.984	1.077	0.964	1.008	0.060	ICP-MS
F011	0.600	0.620	0.650	0.623	0.025	ICP-MS
F017	0.774	0.754	0.790	0.773	0.018	ICP-OES
F020	0.737	0.778	0.719	0.745	0.030	ICP-MS
F022	0.770	0.780	0.890	0.813	0.067	ICP-MS
F026	0.828	0.840	0.810	0.826	0.015	ICP-MS
F030	0.860	0.910	0.900	0.890	0.026	ICP-MS
F031	0.840	0.920	0.800	0.853	0.061	ID ICP-MS
F033	0.770	0.766	0.779	0.772	0.007	ICP-MS
F034	0.605	0.606	0.607	0.606	0.001	ICP-MS
F039	0.832	0.874	0.858	0.953	0.021	ICP-MS
F042	0.831	0.783	0.791	0.959	0.023	ID ICP-MS
F046	1.007	1.069	0.056	1.069	0.056	ICP-MS
F061	0.864	0.869	0.872	0.868	0.004	ICP-MS
F062	0.894	0.867	0.906	0.889	0.020	ID ICP-MS
F069	0.810	0.720	0.780	0.770	0.046	ID ICP-MS
F070	0.820	0.690	0.980	0.830	0.145	ICP-MS
F074	1.86[a]	1.391[a]	1.487[a]			ID ICP-MS
F079	0.820	0.700	0.680	0.733	0.076	ICP-MS
F085	0.746	0.787	0.765	0.766	0.021	ICP-MS
N:				19		
Mean, Pooled SD:				0.795	0.063	
SD:				0.096		

[a] Identified as a non-representative value; excluded from statistical analysis.

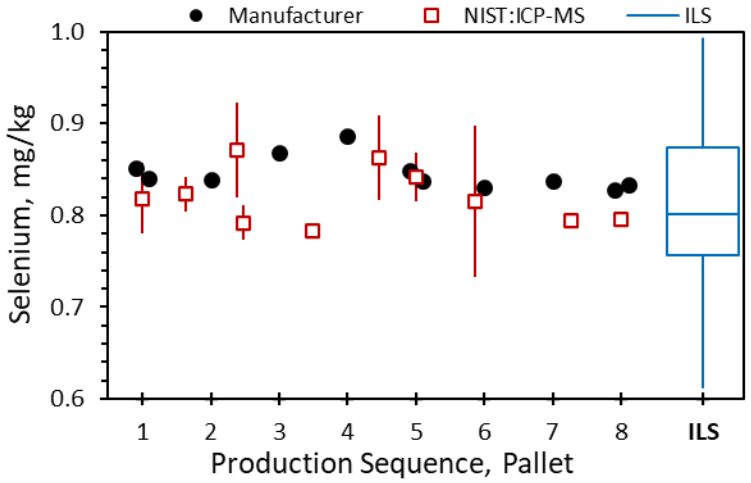

Fig. 20. Selenium (Se) mass fraction as a function of production sequence

5.1.16. Sodium (Na)

The manufacturer ICP-OES [26-28], NIST WDXRF, and FNSQAP [18] measurement results for sodium (Na) are summarized in Table 32. Fig. 21 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 32. Summary of results for sodium (Na), mg/kg

Manufacturer

ID	Value
1:1	4090
2:1	4000
3:1	4070
4:1	4140
5:1	4080
5:2	4010
6:1	4120
7:1	4120
8:1	4100
1:2	4030
8:2	4100
N:	11
Mean:	4078
SD:	46

NIST WDXRF

Box	Rep_1	Rep_2	Mean	SD
1	4307	3973	4140	236
7	4215	4021	4118	137
109	4126	4576	4351	318
146	4004	4148	4076	102
182	4128	4366	4247	168
290	3843	4345	4094	355
		N:	6	
		Mean, Pooled SD:	4171	238
		SD:	107	
		U:	351	

FNSQAP Exercise 1

Code	Rep_1	Rep_2	Rep_3	Mean	SD	Method
A001	4109	4122	4139	4124	15	ICP-MS
A002	4240	4300	4310	4283	38	NAA
A006	4509	4684	4540	4578	94	ICP-MS
A010	4314	4304	4350	4323	24	ICP-OES
A012	4227	4252	4173	4217	40	ICP-OES
A015	4373	4399	4457	4410	43	ICP-OES
A017	4170	4168	4322	4220	88	ICP-MS
A019	4060	3950	4280	4097	168	ICP-MS
A020	4060	3960	4080	4033	64	ICP-OES
A021	4259	4159	4122	4180	71	ICP-MS KED Mode
A023	4630	4470	4480	4527	90	ICP-MS
A027	3853	3999	4107	3986	127	ICP-OES
A028	4131	4221	4086	4146	69	ICP-OES
A031	4507	4548	4438	4498	55	ICP-MS
A032	3670	3710	3700	3693	21	ICP-OES
A034	4337	4306	4321	4321	16	ICP-OES
A035	4308	4276	4347	4310	36	ICP-OES
A038	4240	4170	4210	4207	35	ICP-OES
A039	6104	4357	4063	4841	1103	ICP-MS
A040	3840	3940	3900	3893	50	ICP-MS
A041	3925	3901	3772	3866	82	ICP-MS
A042	3549	3968	3731	3749	210	ICP-MS KED Mode
A045	5348[a]	4794[a]	4916[a]			ICP-MS
A046	4330	4360	4370	4353	21	ICP-OES
A049	4652	4639	4548	4613	57	ICP-MS
A057	3712	3810	3743	3755	50	ICP-OES
			N:	25		
			Mean, Pooled SD:	4209	234	
			SD:	288		

[a] Identified as a non-representative value; excluded from statistical analysis.

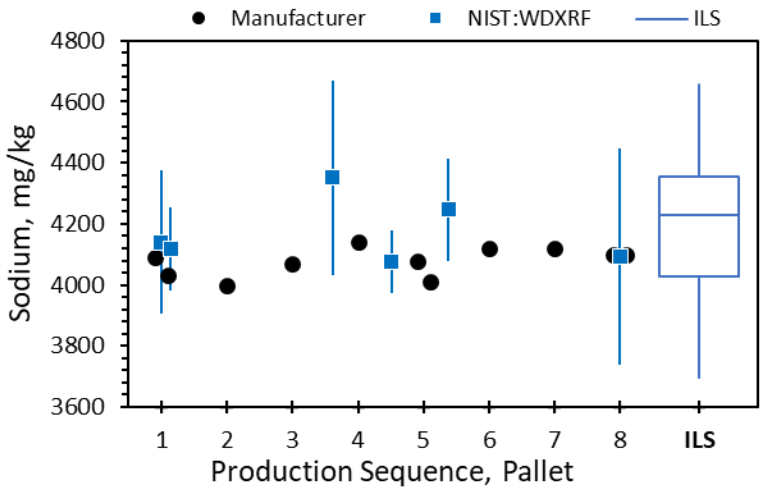

Fig. 21. Sodium (Na) mass fraction as a function of production sequence

5.1.17. Sulfur (S)

The NIST WDXRF measurement results for sulfur (S) are summarized in Table 33. Fig. 22 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 33. Summary of results for sulfur (S), mg/kg

	NIST WDXRF			
Box	Rep_1	Rep_2	Mean	SD
1	3531	3464	3498	47
7	3516	3471	3494	32
109	3492	3551	3522	42
146	3493	3514	3504	15
182	3513	3535	3524	16
290	3470	3542	3506	51
		N:	6	
		Mean, Pooled SD:	3508	37
		SD:	13	
		U:	123	

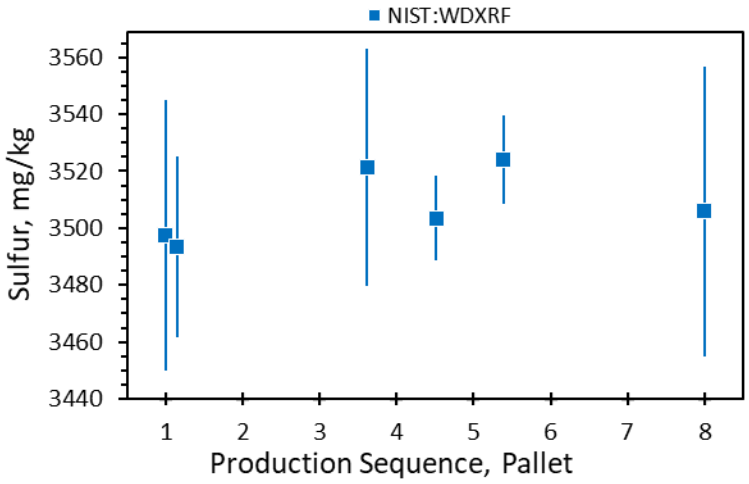

Fig. 22. Sulfur (S) mass fraction as a function of production sequence

5.1.18. Zinc (Zn)

The manufacturer ICP-OES [26] and NIST WDXRF measurement results for zinc (Zn) are summarized in Table 34. Fig. 23 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 34. Summary of results for zinc (Zn), mg/kg

Manufacturer

ID	Value
1:1	140
2:1	136
3:1	140
4:1	142
5:1	142
5:2	138
6:1	141
7:1	141
8:1	140
1:2	138
8:2	141
N:	11
Mean:	139.9
SD:	1.9

NIST WDXRF

Box	Rep_1	Rep_2	Mean	SD
1	144.9	143.2	144.02	1.20
7	144.0	143.8	143.89	0.09
109	144.2	143.1	143.66	0.78
146	143.9	144.4	144.12	0.35
182	144.5	144.5	144.46	0.01
290	143.4	145.1	144.24	1.15
		N:	6	
		Mean, Pooled SD:	144.06	0.77
		SD:	0.28	
		U:	5.7	

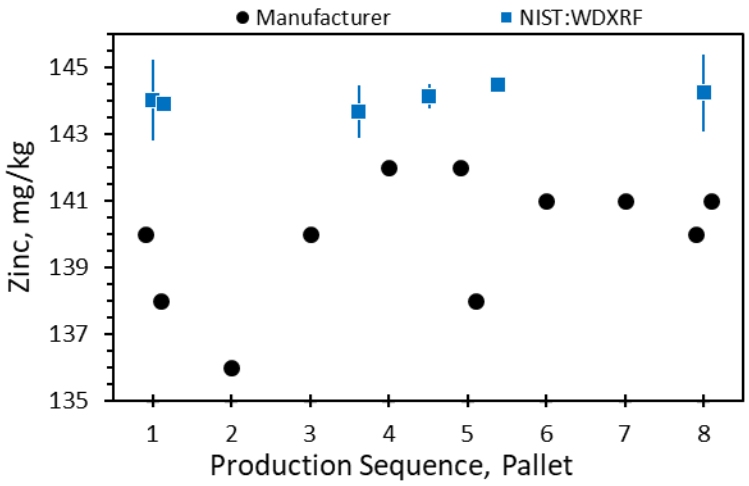

Fig. 23. Zinc (Zn) mass fraction as a function of production sequence

5.2. Vitamins and Related Measurands

5.2.1. Vitamin A Acetate (Retinyl Acetate)

The manufacturer LC-Abs [29] and HAMQAP [17] measurement results for vitamin A acetate (retinyl acetate) are summarized in Table 35. Fig. 24 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 35. Summary of results for vitamin A acetate (retinyl acetate), mg/kg

Manufacturer

ID	Value
1:1	6.90
2:1	6.69
3:1	6.72
4:1	6.81
5:1	6.87
5:2	6.75
6:1	6.75
7:1	6.57
8:1	6.60
1:2	6.78
8:2	6.57
N:	11
Mean:	6.73
SD:	0.11

HAMQAP Exercise 6

Code	Rep_1	Rep_2	Rep_3	Mean	SD	Method
F005	7.30	6.78	7.18	7.09	0.27	LC-Abs or PDA
F039	15.9[a]	15.9[a]	15.6[a]			LC-FL
F041	6.12	5.65	5.92	5.90	0.24	LC-Abs or PDA
F046	10.68	12.30	12.34	11.77	0.95	LC-Abs or PDA
F069	7.00	8.00	13.00	9.33	3.21	LC-Abs or PDA
F075	7.86	7.70	7.66	7.74	0.11	LC-Abs or PDA
F088	5.79	4.70	6.02	5.50	0.71	LC-Abs
			N:	6		
			Mean, Pooled SD:	7.89	1.41	
			SD:	2.35		

[a] Identified as a non-representative value; excluded from statistical analysis.

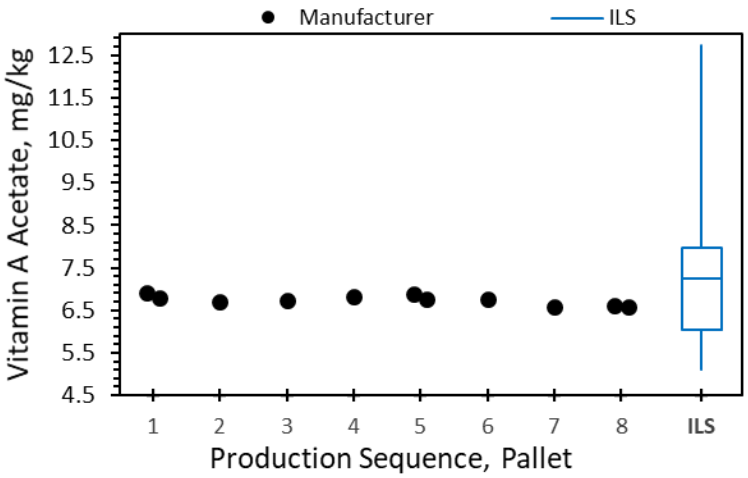

Fig. 24. Vitamin A acetate (retinyl acetate) mass fraction as a function of production sequence

5.2.2. Vitamin A Palmitate (Retinyl Palmitate)

The manufacturer LC-Abs [29] and HAMQAP [17] measurement results for vitamin A palmitate (retinyl palmitate) are summarized in Table 36. Fig. 25 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 36. Summary of results for vitamin A palmitate (retinyl palmitate), mg/kg

Manufacturer

ID	Value
1:1	7.83
2:1	7.71
3:1	7.83
4:1	7.92
5:1	7.92
5:2	7.80
6:1	7.98
7:1	7.65
8:1	7.68
1:2	7.86
8:2	7.65
N:	11
Mean:	7.80
SD:	0.12

HAMQAP Exercise 6

Code	Rep_1	Rep_2	Rep_3	Mean	SD	Method
F005	12.03	9.94	10.55	10.84	1.07	LC-Abs or PDA
F026	13.01			13.01		LC-Abs or PDA
F039	25.4	25.3	25	25.23	0.21	LC-FL
F041	14.46	13.83	14.12	14.14	0.32	LC-Abs or PDA
F046	11.74	17.23	16.62	15.20	3.01	LC-Abs or PDA
F069	36[a]	44[a]	72[a]			LC-Abs or PDA
F075	14.1	14.2	14.2	14.17	0.06	LC-Abs or PDA
F079	6.25	4.8	4.57	5.21	0.91	LC-MS/MS
F088	7.312	7.119	6.13	6.85	0.63	LC-Abs
			N:	8		
			Mean, Pooled SD:	13.08	1.20	
			SD:	6.10		

[a] Identified as a non-representative value; excluded from statistical analysis.

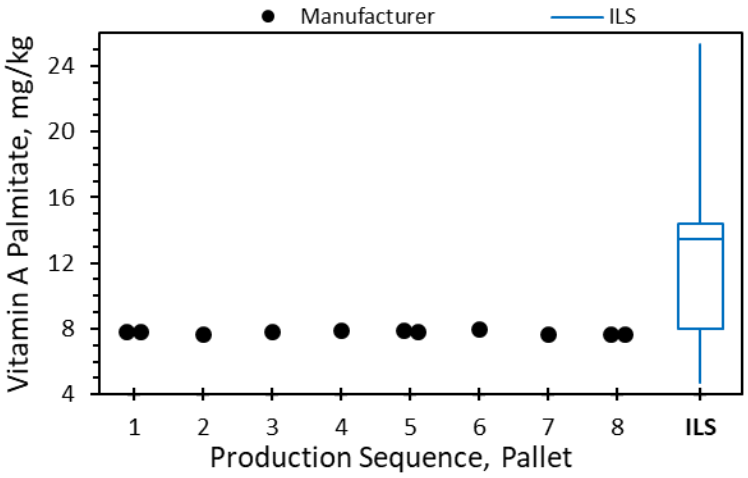

Fig. 25. Vitamin A palmitate (retinyl palmitate) mass fraction as a function of production sequence

5.2.3. Total Vitamin A (Retinol)

The manufacturer LC-Abs [29] and HAMQAP [17] measurement results for total vitamin A (retinol) are summarized in Table 37. Fig. 26 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 37. Summary of results for total vitamin A (retinol), mg/kg

Manufacturer		HAMQAP Exercise 6						
ID	Value	Code	Rep$_1$	Rep$_2$	Rep$_3$	Mean	SD	Method
1:1	10.29	F005	19.33	16.72	17.73	17.93	1.32	LC-Abs or PDA
2:1	10.04	F013	15.10	14.70	16.10	15.30	0.72	LC-Abs or PDA
3:1	10.13	F014	14.00	14.00	13.60	13.87	0.23	LC-FL
4:1	10.26	F020	11.50	12.10	12.10	11.90	0.35	LC-FL
5:1	10.31	F030	14.50	13.40	13.30	13.73	0.67	LC-FL
5:2	10.14	F031	13.02	13.43	15.91	14.12	1.56	LC-Abs or PDA
6:1	10.24	F033	13.10	12.90	13.30	13.10	0.20	LC-Abs or PDA
7:1	9.90	F034	13.08	13.29	13.16	13.18	0.11	LC-MS
8:1	9.95	F039	13.90	13.80	13.60	13.77	0.15	LC-FL
1:2	10.20	F041	13.23	12.47	12.87	12.85	0.38	LC-Abs or PDA
8:2	9.90	F046	18.66[a]	23.51[a]	23.22[a]			LC-Abs or PDA
N:	11	F059	15.10	15.10	13.90	14.70	0.69	LC-Abs or PDA
Mean:	10.13	F061	15.52	15.53	15.83	15.63	0.18	LC-Abs
SD:	0.15	F062	10.07	10.05	9.91	10.01	0.08	LC-Abs or PDA
		F069	0[a]	0[a]	0[a]			LC-Abs or PDA
		F075	14.60	14.40	14.40	14.47	0.12	LC-Abs or PDA
		F088	14.56	16.65	15.15	15.45	1.08	LC-Abs
					N:	15		
					Mean, Pooled SD:	14.00	0.69	
					SD:	1.81		

[a] Identified as a non-representative or non-quantitative value; excluded from statistical analysis.

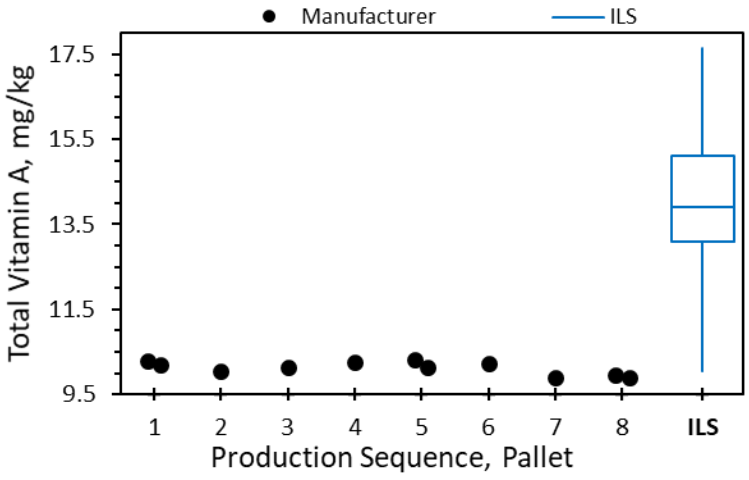

Fig. 26. Total vitamin A (retinol) mass fraction as a function of production sequence

5.2.4. Vitamin B_1 (Thiamine)

The manufacturer LC-FL and HAMQAP [16] measurement results for vitamin B_1 (thiamine) are summarized in Table 38. Fig. 27 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 38. Summary of results for vitamin B_1 (thiamine), mg/kg

Manufacturer		HAMQAP Exercise 5						
ID	Value	Code	Rep$_1$	Rep$_2$	Rep$_3$	Mean	SD	Method
1:1	14.48	E002	16.22	16.19	16.21	16.21	0.02	LC-MS
2:1	14.40	E005	14.00	14.00	14.00	14.00	0.00	LC-Abs or PDA
3:1	14.48	E007	14.00	14.00	14.00	14.00	0.00	LC-Abs or PDA
4:1	14.48	E010	18.66	18.24	18.44	18.45	0.21	LC-Abs or PDA
5:1	14.48	E012	3.75[a]	3.68[a]	3.4[a]			LC-Abs or PDA
5:2	14.48	E023	13.30	13.60	13.90	13.60	0.30	LC-MS/MS
6:1	14.48	E030	16.80	18.70	22.80	19.43	3.07	LC-Abs or PDA
7:1	14.48	E040	9.90	10.40	7.70	9.33	1.44	LC-MS
8:1	14.56	E041	14.30	15.20	14.50	14.67	0.47	LC-Abs or PDA
1:2	14.48	E042	12.53	12.73	12.50	12.59	0.12	LC-MS
8:2	14.56				*N*:	9		
N:	11				Mean, Pooled SD:	14.70	1.14	
Mean:	14.48				SD:	3.05		
SD:	0.04							

[a] Identified as a non-representative value; excluded from statistical analysis.

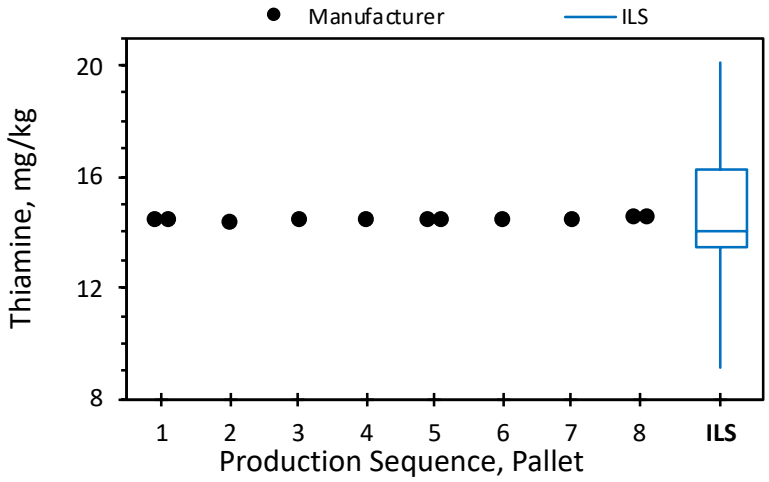

Fig. 27. Vitamin B_1 (thiamine) mass fraction as a function of production sequence

5.2.5. Vitamin B_2 (Riboflavin)

The manufacturer LC-FL and HAMQAP [16] measurement results for vitamin B_2 (riboflavin) are summarized in Table 39. Fig. 28 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 39. Summary of results for vitamin B_2 (riboflavin), mg/kg

Manufacturer		HAMQAP Exercise 5						
ID	Value	Code	Rep$_1$	Rep$_2$	Rep$_3$	Mean	SD	Methods
1:1	16.9	E002	16.37	16.96	16.39	16.57	0.33	LC-MS
2:1	16.9	E005	20.00	20.00	20.00	20.00	0.00	LC-Abs or PDA
3:1	17.0	E010	17.86	17.79	17.99	17.88	0.10	LC-Abs or PDA
4:1	17.0	E012	3.66[a]	3.45[a]	3.39[a]			LC-Abs or PDA
5:1	16.9	E013	<20[a]	<20[a]	<20[a]			LC-Abs or PDA
5:2	16.7	E023	15.20	15.30	15.40	15.30	0.10	LC-MS/MS
6:1	17.0	E030	16.30	18.70	16.90	17.30	1.25	LC-Abs or PDA
7:1	16.9	E040	19.70	15.50	18.00	17.73	2.11	LC-MS
8:1	16.9	E041	12.60	12.00	10.80	11.80	0.92	LC-Abs or PDA
1:2	17.0	E042	5.345[a]	5.117[a]	5.245[a]			LC-MS
8:2	17.0	E047	15.00	15.00	15.00	15.00	0.00	LC-Abs or PDA
N:	11				*N*:	8		
Mean:	16.93				Mean, Pooled SD:	16.45	0.94	
SD:	0.09				SD:	2.45		

[a] Identified as a non-representative or non-quantitative value; excluded from statistical analysis.

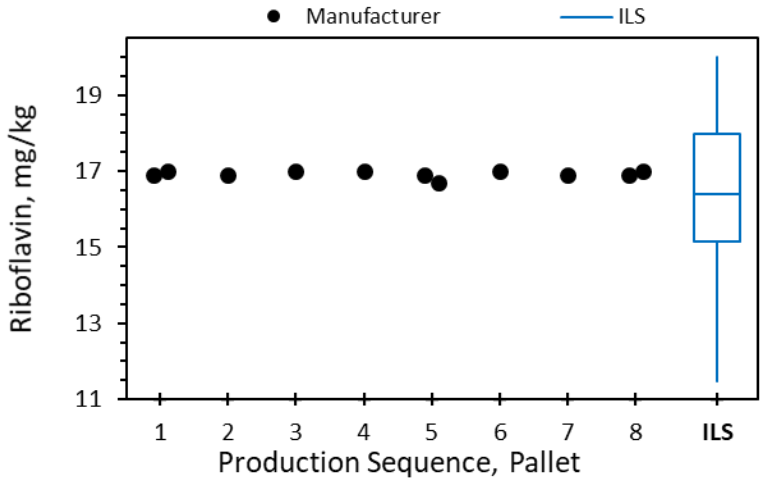

Fig. 28. Vitamin B_2 (riboflavin) mass fraction as a function of production sequence

5.2.6. Vitamin B_3 (Niacinamide)

The manufacturer LC-MS/MS and HAMQAP [16] measurement results for vitamin B_3 (niacinamide) are summarized in Table 40. Fig. 29 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 40. Summary of results for vitamin B_3 (niacinamide), mg/kg

Manufacturer		HAMQAP Exercise 5						
ID	Value	Code	Rep$_1$	Rep$_2$	Rep$_3$	Mean	SD	Methods
1:1	112.0	E002	112.03	110.65	111.99	111.56	0.79	LC-MS
2:1	102.0	E005	100.00	101.00	101.00	100.67	0.58	LC-Abs or PDA
3:1	108.0	E007	99.95	97.79	97.89	98.54	1.22	LC-Abs or PDA
4:1	103.0	E010	111.32	111.09	110.88	111.10	0.22	LC-Abs or PDA
5:1	111.0	E013	<200[a]	<200[a]	<200[a]			LC-Abs or PDA
5:2	112.0	E023	112.00	112.00	110.00	111.33	1.15	LC-MS/MS
6:1	109.0	E030	126.00	139.00	127.00	130.67	7.23	LC-Abs or PDA
7:1	111.0	E040	165.00	140.40	168.30	157.90	15.24	LC-MS
8:1	105.0	E042	110.20	114.80	114.60	113.20	2.60	LC-MS
1:2	115.0	E047	340[a]	340[a]	350[a]			LC-Abs or PDA
8:2	99.1				*N*:	8		
N:	11				Mean, Pooled SD:	116.87	6.08	
Mean:	107.9				SD:	19.19		
SD:	5.0							

[a] Identified as a non-representative or non-quantitative value; excluded from statistical analysis.

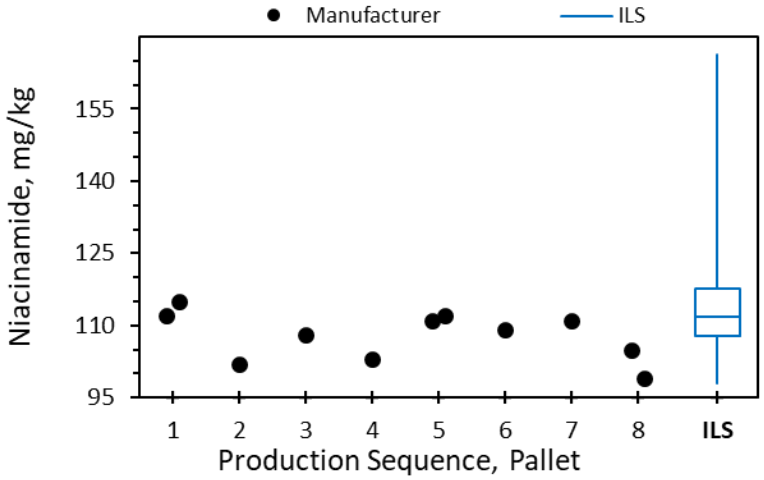

Fig. 29. Vitamin B_3 (niacinamide) mass fraction as a function of production sequence

5.2.7. Vitamin B_5 (Pantothenic Acid)

The manufacturer LC-MS/MS and HAMQAP [16] measurement results for vitamin B_5 (pantothenic acid) are summarized in Table 41. Fig. 30 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 41. Summary of results for vitamin B_5 (pantothenic acid), mg/kg

Manufacturer		HAMQAP Exercise 5						
ID	Value	Code	Rep1	Rep2	Rep3	Mean	SD	Methods
1:1	77.0	E002	75.62	74.12	75.30	75.01	0.79	LC-MS/MS
2:1	70.8	E005	61.00	60.00	60.00	60.33	0.58	LC-Abs or PDA
3:1	74.7	E007	75.60	72.90	73.90	74.13	1.37	LC-Abs or PDA
4:1	70.9	E010	71.07	75.98	78.10	75.05	3.60	LC-Abs or PDA
5:1	74.1	E012	3.34[a]	3.5[a]	3.2[a]			LC-Abs or PDA
5:2	74.2	E013	<200[a]	<200[a]	<200[a]			LC-Abs or PDA
6:1	77.5	E023	69.00	69.10	68.70	68.93	0.21	LC-MS/MS
7:1	71.7	E030	78.10	73.20	75.30	75.53	2.46	LC-Abs or PDA
8:1	69.1	E040	100[a]	69.1[a]	103.7[a]			LC-MS
1:2	74.0	E042	72.73	69.58	70.62	70.98	1.60	LC-MS
8:2	65.0	E047	70.00	70.00	70.00	70.00	0.00	LC-Abs or PDA
N:	11				*N*:	8		
Mean:	72.6				Mean, Pooled SD:	71.25	1.75	
SD:	3.6				SD:	5.09		

[a] Identified as a non-representative or non-quantitative value; excluded from statistical analysis.

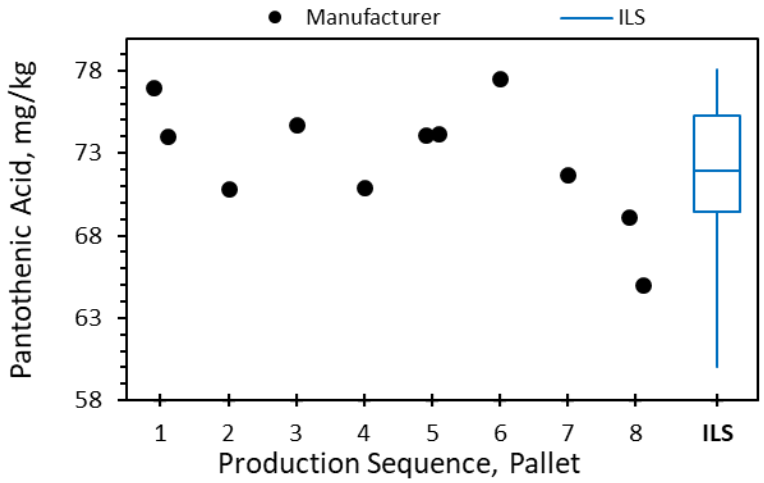

Fig. 30. Vitamin B_5 (pantothenic acid) mass fraction as a function of production sequence

5.2.8. Vitamin B_6 (Pyridoxine)

The manufacturer LC-FL and HAMQAP [16] measurement results for vitamin B_6 (pyridoxine) are summarized in Table 42. Fig. 31 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 42. Summary of results for vitamin B_6 (pyridoxine), mg/kg

Manufacturer

ID	Value
1:1	14.5
2:1	14.5
3:1	15.0
4:1	14.4
5:1	14.5
5:2	15.2
6:1	14.7
7:1	14.3
8:1	14.6
1:2	14.5
8:2	14.4
N:	11
Mean:	14.60
SD:	0.27

HAMQAP Exercise 5

Code	Rep$_1$	Rep$_2$	Rep$_3$	Mean	SD	Methods
E002	15.21	15.22	14.74	15.05	0.27	LC-MS
E005	13.00	12.00	12.00	12.33	0.58	LC-Abs or PDA
E007	18.68	17.74	17.60	18.01	0.59	LC-Abs or PDA
E010	13.34	13.46	13.54	13.45	0.10	LC-Abs or PDA
E013	<30[a]	<30[a]	<30[a]			LC-Abs or PDA
E023	14.30	14.00	14.10	14.13	0.15	LC-MS/MS
E030	18.40	16.40	17.90	17.57	1.04	LC-Abs or PDA
E040	22.90	20.90	20.50	21.43	1.29	LC-MS
E041	15.10	15.10	15.20	15.13	0.06	LC-Abs or PDA
			N:	8		
			Mean, Pooled SD:	15.89	0.66	
			SD:	2.95		

[a] Identified as a non-representative or non-quantitative value; excluded from statistical analysis.

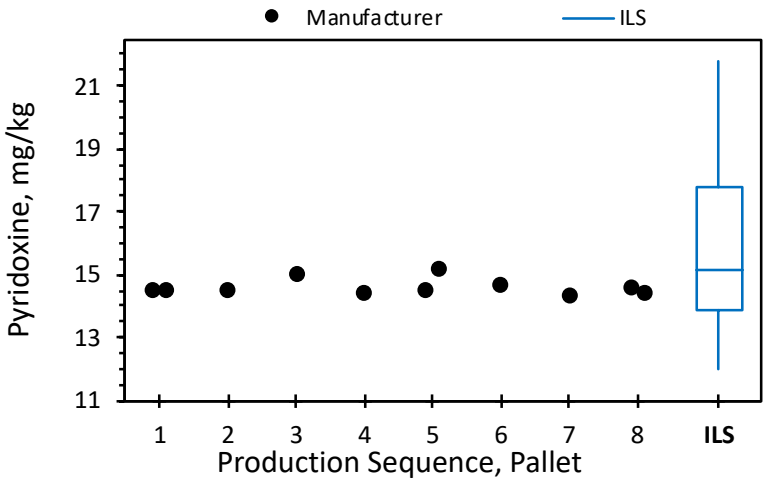

Fig. 31. Vitamin B_6 (pyridoxine) mass fraction as a function of production sequence

5.2.9. Biotin

The manufacturer LC-MS/MS and HAMQAP [17] measurement results for biotin are summarized in Table 43. Fig. 32 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 43. Summary of results for biotin, mg/kg

Manufacturer		HAMQAP Exercise 6						
ID	Value	Code	Rep$_1$	Rep$_2$	Rep$_3$	Mean	SD	Methods
1:1	2.14	F005	1.89	1.96	2.04	1.96	0.08	LC-MS
2:1	2.07	F017	<135[a]	<135[a]	<135[a]			LC-FL
3:1	2.20	F026	2.34			2.34		LC-MS/MS
4:1	2.17	F030	2.06	1.94	2.07	2.02	0.07	Microbiological Assay
5:1	2.16	F031	2.49	2.17	2.11	2.26	0.20	LC-Abs or PDA
5:2	2.16	F034	2.23	2.19	2.19	2.20	0.02	LC-MS
6:1	2.06	F036	1.66	1.87	2.05	1.86	0.20	LC-MS/MS
7:1	2.21	F039	1.72	1.67	1.68	1.69	0.03	Microbiological Assay
8:1	2.01	F061	2.23	2.24	2.24	2.23	0.01	LC-FL
1:2	2.23	F062	2.06	2.08	2.13	2.09	0.04	Microbiological Assay
8:2	2.08	F069	61[a]	57[a]	62[a]			LC-MS
N:	11	F073	1.95	2.04	2.11	2.03	0.08	LC-MS
Mean:	2.14	F074	78426[a]	79938[a]	75709[a]			LC-MS
SD:	0.07	F075	2.00	1.96	1.99	1.98	0.02	LC-MS/MS
		F080	2.10	1.94	2.00	2.01	0.08	LC-Abs
					N:	12		
					Mean, Pooled SD:	2.06	0.09	
					SD:	0.18		

[a] Identified as a non-representative or non-quantitative value; excluded from statistical analysis.

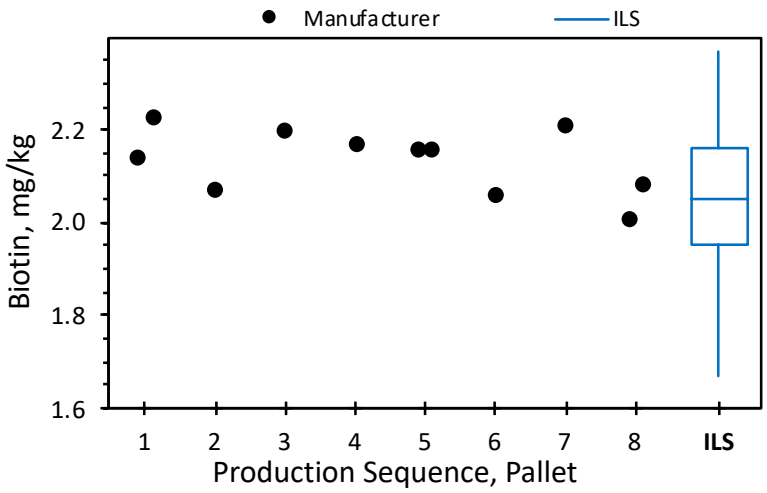

Fig. 32. Biotin mass fraction as a function of production sequence

5.2.10. Folic Acid

The manufacturer LC-MS/MS and FNSQAP [18] measurement results for folic acid are summarized in Table 44. Fig. 33 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 44. Summary of results for folic acid, mg/kg

Manufacturer		FNSQAP Exercise 1						
ID	Value	Code	Rep_1	Rep_2	Rep_3	Mean	SD	Method
1:1	2.44	A001	2.03	1.73	1.32	1.69	0.36	LC-MS
2:1	2.42	A006	2.44	2.73	2.86	2.68	0.22	LC-Abs or PDA
3:1	2.47	A010	2.29	2.53	2.47	2.43	0.12	LC-MS
4:1	2.41	A012	2.49	2.46	2.21	2.39	0.15	LC-Abs or PDA
5:1	2.40	A015	2.382	2.431	2.623	2.48	0.13	LC-MS
5:2	2.55	A020	2.8	2.98	2.78	2.85	0.11	Microbiological Assay
6:1	2.49	A027	3.06	3.05	3.08	3.06	0.02	Not specified or Other
7:1	2.50	A034	2.87	2.94	2.71	2.84	0.12	LC-MS/MS
8:1	2.23	A038	2.3	2.27	2.37	2.31	0.05	Microbiological Assay
1:2	2.64	A040	704[a]	708[a]	692[a]			Not specified or Other
8:2	2.02	A041	3.87	3.64	3.778	3.76	0.12	LC-Abs or PDA
N:	11	A044	2.81	2.85	2.98	2.88	0.09	ELISA
Mean:	2.42	A045	278[a]	301[a]	285[a]			LC-MS/MS
SD:	0.17				*N*:	11		
					Mean, Pooled SD:	2.67	0.16	
					SD:	0.52		

[a] Identified as a non-representative value; excluded from statistical analysis.

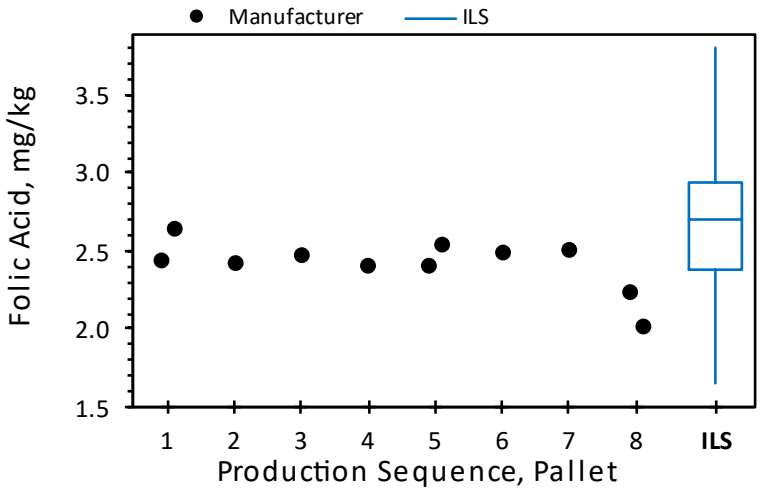

Fig. 33. Folic acid mass fraction as a function of production sequence

5.2.11. Vitamin B_{12} (Cyanocobalamin)

The manufacturer LC-ICP-MS [14] and NIST LC-ICP-MS measurement results for vitamin B_{12} (cyanocobalamin) are summarized in Table 45. Fig. 34 displays these results as an approximate function of the production sequence, as determined by the pallet and box number. The NIST measurement results for CNCbl in the SRM 1869 control material agreed well with the non-certified value stated in its COA and with those in previous LC-ICP-MS measurements.

Table 45. Summary of results for vitamin B_{12} (cyanocobalamin), µg/kg

Manufacturer	
ID	Value
1:1	44.8
2:1	47.6
3:1	68.9
4:1	41.6
5:1	42.9
5:2	42.0
6:1	57.8
7:1	44.7
8:1	43.0
1:2	42.0
8:2	46.0
N:	11
Mean:	47.4
SD:	8.5

NIST LC-ICP-MS	
Box:Pouch	Value
1	51.7
41	52.3
76:1	51.4
76:2	54.8
157	51.4
182:1	52.7
182:2	52.2
290	49.0
N:	8
Mean:	51.9
SD:	1.6

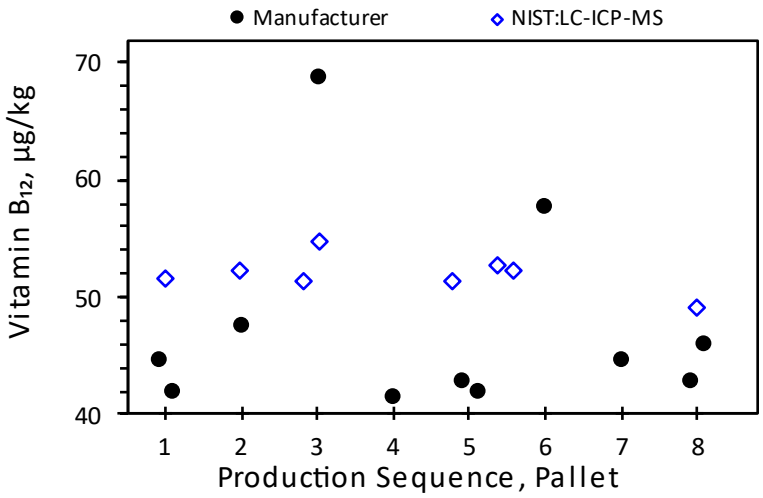

Fig. 34. Vitamin B_{12} (cyanocobalamin) mass fraction as a function of production sequence

5.2.12. Vitamin C (Ascorbic Acid)

The manufacturer LC-Abs [30-32] and HAMQAP [17] measurement results for vitamin C (ascorbic acid) are summarized in Table 46. Fig. 35 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 46. Summary of results for vitamin C (ascorbic acid), mg/kg

Manufacturer		HAMQAP Exercise 6						
ID	Value	Code	Rep$_1$	Rep$_2$	Rep$_3$	Mean	SD	Methods
1:1	946	F005	342.83[a]	342.8[a]	374.77[a]			LC-Abs or PDA
2:1	913	F013	1182.0	1104.2	1123.4	1136.5	40.5	LC-Abs or PDA
3:1	940	F014	872.0	869.0	875.0	872.0	3.0	LC-Abs or PDA
4:1	954	F017	1300.0	1300.0	1300.0	1300.0	0.0	LC-Abs or PDA
5:1	959	F022	991.0	1050.0	1017.0	1019.3	29.6	LC-Abs or PDA
5:2	979	F026	1023.7			1023.7		LC-Abs or PDA
6:1	950	F030	1080.0	935.0	905.0	973.3	93.6	LC-Abs or PDA
7:1	930	F031	865.8	834.0	834.9	844.9	18.1	LC-Abs or PDA
8:1	950	F034	935.0	930.0	937.0	934.0	3.6	LC-Abs or PDA
1:2	929	F036	1127.0	1116.0	1103.0	1115.3	12.0	LC-Abs or PDA
8:2	954	F039	883.0	892.0	872.0	882.3	10.0	Spectrophotometry
N:	11	F046	1075.5	969.2	1063.1	1035.9	58.1	LC-Abs or PDA
Mean:	946	F059	1030.0	1000.0	1000.0	1010.0	17.3	LC-Abs or PDA
SD:	18	F061	930.0	930.0	930.0	930.0	0.0	LC-Abs
		F062	934.3	939.0	914.7	929.3	12.9	LC-Abs or PDA
		F069	1060.0	1030.0	1040.0	1043.3	15.3	LC-Abs or PDA
		F070	514[a]	734[a]	575[a]			LC-Abs or PDA
		F074	1099.0	1099.0	1099.0	1099.0	0.0	Other
		F075	968.6	940.7	936.1	948.5	17.6	LC-Abs or PDA
		F079	335[a]	< 97.0[a]	97.5[a]			Iodine Titration
		F080	853.1	836.2	826.3	838.6	13.6	LC-Abs
					N:	18		
					Mean, Pooled SD:	996	30	
					SD:	117		

[a] Identified as a non-representative or non-quantitative value; excluded from statistical analysis.

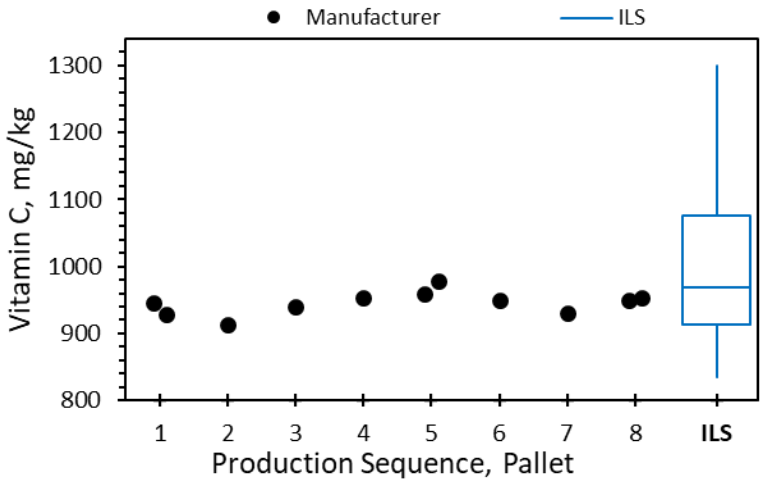

Fig. 35. Vitamin C (ascorbic acid) mass fraction as a function of production sequence

5.2.13. Vitamin D_2 (Ergocalciferol)

The manufacturer LC-MS/MS [33] and HAMQAP [16] measurement results for vitamin D_2 (ergocalciferol) are summarized in Table 47. Fig. 36 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 47. Summary of results for vitamin D_2 (ergocalciferol), mg/kg

Manufacturer		HAMQAP Exercise 5						
ID	Value	Code	Rep$_1$	Rep$_2$	Rep$_3$	Mean	SD	Methods
1:1	0.1140	E002	0.95[a]	1.1[a]	1.29[a]			LC-MS
2:1	0.1143	E003	<0.200[a]	<0.200[a]	<0.200[a]			LC-MS/MS
3:1	0.1168	E005	0.0525	0.0516	0.0533	0.0525	0.0009	LC-MS
4:1	0.1203	E012	3[a]	2.9[a]	2.6[a]			LC-Abs or PDA
5:1	0.1178	E014	0.0660	0.0560	0.0580	0.0600	0.0053	LC-Abs or PDA
5:2	0.1193	E015	2.28[a]	1.39[a]	2.19[a]			LC-Abs or PDA
6:1	0.1173	E023	0.1090	0.1070	0.1080	0.1080	0.0010	LC-MS/MS
7:1	0.1145	E030	<0.100[a]	<0.100[a]	<0.100[a]			LC-MS/MS
8:1	0.1133	E047	<0.200[a]	<0.200[a]	<0.200[a]			LC-Abs or PDA
1:2	0.1123	E057	0.1140	0.1140	0.1220	0.1167	0.0046	LC-MS/MS
8:2	0.1183				*N*:	4		
N:	11				Mean, Pooled SD:	0.0843	0.0036	
Mean:	0.1162				SD:	0.0327		
SD:	0.0026							

[a] Identified as a non-representative value; excluded from statistical analysis.

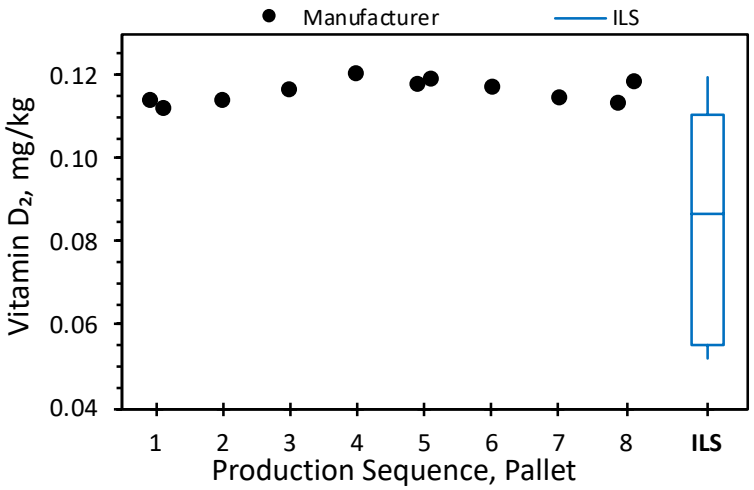

Fig. 36. Vitamin D_2 (ergocalciferol) mass fraction as a function of production sequence

5.2.14. Vitamin D_3 (Cholecalciferol)

The manufacturer LC-MS/MS [33] and HAMQAP [16] measurement results for vitamin D_3 (cholecalciferol) are summarized in Table 48. Fig. 37 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 48. Summary of results for vitamin D_3 (cholecalciferol), mg/kg

Manufacturer		HAMQAP Exercise 5						
ID	Value	Code	Rep$_1$	Rep$_2$	Rep$_3$	Mean	SD	Methods
1:1	0.1058	E002	0.0600	0.0700	0.0800	0.0700	0.0100	LC-MS
2:1	0.1063	E003	<0.200[a]	<0.200[a]	<0.200[a]			LC-MS/MS
3:1	0.1055	E005	0.0523	0.0511	0.0522	0.0519	0.0007	LC-MS
4:1	0.1060	E007	1.15[a]	1.19[a]	1.1[a]			LC-Abs or PDA
5:1	0.1048	E012	2[a]	2[a]	2.2[a]			LC-Abs or PDA
5:2	0.1050	E014	0.0470	0.0470	0.0520	0.0487	0.0029	LC-Abs or PDA
6:1	0.1030	E015	1.5[a]	2.12[a]	1.48[a]			LC-Abs or PDA
7:1	0.1033	E023	0.1020	0.0998	0.1030	0.1016	0.0016	LC-MS/MS
8:1	0.1065	E030	<0.100[a]	<0.100[a]	<0.100[a]			LC-MS/MS
1:2	0.1073	E041	10.9[a]	12.1[a]	12.5[a]			LC-Abs or PDA
8:2	0.1088	E047	0.1000	0.1000	0.1000	0.1000	0.0000	LC-Abs or PDA
N:	11	E057	0.1020	0.1090	0.1000	0.1037	0.0047	LC-MS/MS
Mean:	0.1056				*N*:	6		
SD:	0.0017				Mean, Pooled SD:	0.0793	0.0047	
					SD:	0.0257		

[a] Identified as a non-representative value; excluded from statistical analysis.

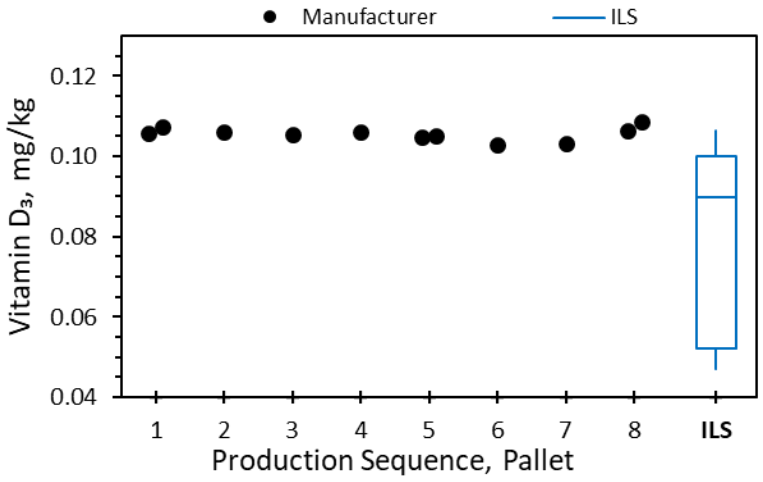

Fig. 37. Vitamin D_3 (cholecalciferol) mass fraction as a function of production sequence

5.2.15. Vitamin E Acetate (α-Tocopheryl Acetate)

The manufacturer LC-Abs [29] and HAMQAP [17] measurement results for vitamin E acetate (α-tocopheryl acetate) are summarized in Table 49. Fig. 38 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 49. Summary of results for vitamin E acetate (α-tocopheryl acetate), mg/kg

Manufacturer

ID	Value
1:1	164
2:1	158
3:1	162
4:1	164
5:1	164
5:2	162
6:1	165
7:1	158
8:1	160
1:2	161
8:2	160
N:	11
Mean:	161.6
SD:	2.5

HAMQAP Exercise 6

Code	Rep_1	Rep_2	Rep_3	Mean	SD	Methods
F005	137.06	143.24	149.76	143.35	6.35	LC-Abs or PDA
F017	71.35	77.54	92.74	80.54	11.01	LC-Abs or PDA
F034	179.00	192.00	195.00	188.67	8.50	LC-Abs or PDA
F039	232.00	243.00	249.00	241.33	8.62	LC-FL
F046	138.54	110.76	113.96	121.09	15.20	LC-Abs or PDA
F074	13.28[a]	13.79[a]	16.9[a]			LC-Abs or PDA
F075	151.00	156.00	153.00	153.33	2.52	LC-FL
F079	132.00	111.00	50.00	97.67	42.59	LC-MS/MS
F088	81.43	56.39	80.20	72.67	14.12	LC-Abs
			N:	8		
			Mean, Pooled SD:	137.3	17.9	
			SD:	57.3		

[a] Identified as a non-representative value; excluded from statistical analysis.

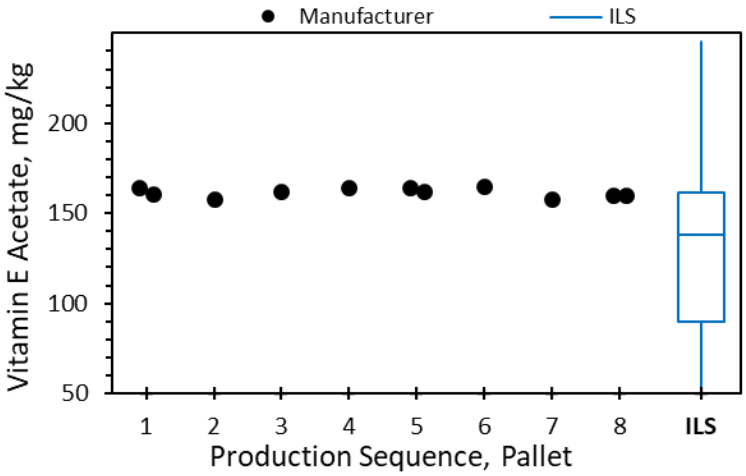

Fig. 38. Vitamin E acetate (α-tocopheryl acetate) mass fraction as a function of production sequence

5.2.16. Free Vitamin E (Free α-Tocopherol)

The manufacturer LC-FL [29] and HAMQAP [17] measurement results for free vitamin E (free α-tocopherol) are summarized in Table 50. Fig. 39 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 50. Summary of results for free vitamin E (free α-tocopherol), mg/kg

Manufacturer		HAMQAP Exercise 6						
ID	Value	Code	Rep_1	Rep_2	Rep_3	Mean	SD	Methods
1:1	61.0	F005	46.24	52.60	60.09	52.98	6.93	LC-Abs or PDA
2:1	59.4	F013	191[a]	175[a]	180[a]			LC-FL
3:1	58.4	F017	26.36	26.81	29.86	27.68	1.90	LC-Abs or PDA
4:1	59.0	F030	188[a]	185[a]	185[a]			LC-FL
5:1	59.8	F033	203[a]	200[a]	199[a]			LC-Abs or PDA
5:2	58.5	F034	71.40	66.30	71.20	69.63	2.89	LC-Abs or PDA
6:1	58.4	F039	211[a]	221[a]	227[a]			LC-FL
7:1	56.3	F046	23.36	22.94	22.66	22.99	0.35	LC-Abs or PDA
8:1	56.5	F061	197.6[a]	202.1[a]	213.1[a]			LC-Abs
1:2	60.5	F062	160.2[a]	147.3[a]	140.5[a]			LC-FL
8:2	54.5	F075	59.40	58.50	59.30	59.07	0.49	LC-FL
N:	11	F088	52.80	43.77	53.93	50.17	5.57	LC-Abs
Mean:	58.4				*N*:	6		
SD:	1.9				Mean, Pooled SD:	47.08	3.90	
					SD:	18.19		

[a] Identified as a non-representative value; excluded from statistical analysis.

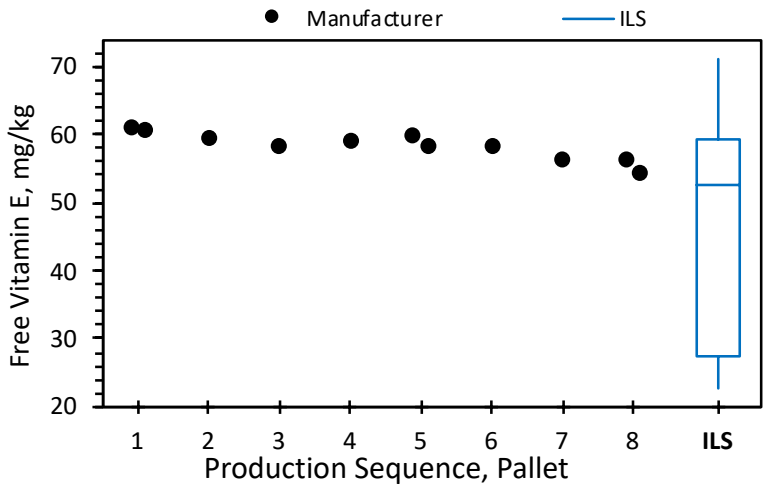

Fig. 39. Free vitamin E (free α-tocopherol) mass fraction as a function of production sequence

5.2.17. Total Vitamin E (Total α-Tocopherol)

The manufacturer LC-Abs/FL [29] and HAMQAP [17] measurement results for total vitamin E (total α-tocopherol) are summarized in Table 51. Fig. 40 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 51. Summary of results for total vitamin E (total α-tocopherol), mg/kg

Manufacturer		HAMQAP Exercise 6						
ID	Value	Code	Rep_1	Rep_2	Rep_3	Mean	SD	Methods
1:1	210.4	F005	183.30	195.84	209.85	196.33	13.28	LC-Abs or PDA
2:1	203.4	F014	211.00	196.00	190.00	199.00	10.82	LC-FL
3:1	206.0	F017	26.36	26.81	29.86	27.68	1.90	LC-Abs or PDA
4:1	208.4	F020	193.00	206.00	204.00	201.00	7.00	LC-FL
5:1	209.2	F022	435[a]	437[a]	426[a]			LC-Abs or PDA
5:2	206.1	F031	212.92	215.66	225.79	218.12	6.78	LC-Abs or PDA
6:1	208.7	F033	364.00	361.00	356.00	360.33	4.04	LC-Abs or PDA
7:1	200.3	F046	149.59	123.86	126.50	133.32	14.15	LC-Abs or PDA
8:1	202.3	F069	0[a]	0[a]	0[a]			LC-Abs or PDA
1:2	207.2	F075	197.00	200.00	199.00	198.67	1.53	LC-FL
8:2	200.3	F079	96.00	71.00	46.00	71.00	25.00	LC-MS/MS
N:	11	F088	217.97	247.01	210.25	225.08	19.38	LC-Abs
Mean:	205.7				*N*:	10		
SD:	3.6				Mean, Pooled SD:	183.1	12.7	
					SD:	91.0		

[a] Identified as a non-representative value; excluded from statistical analysis.

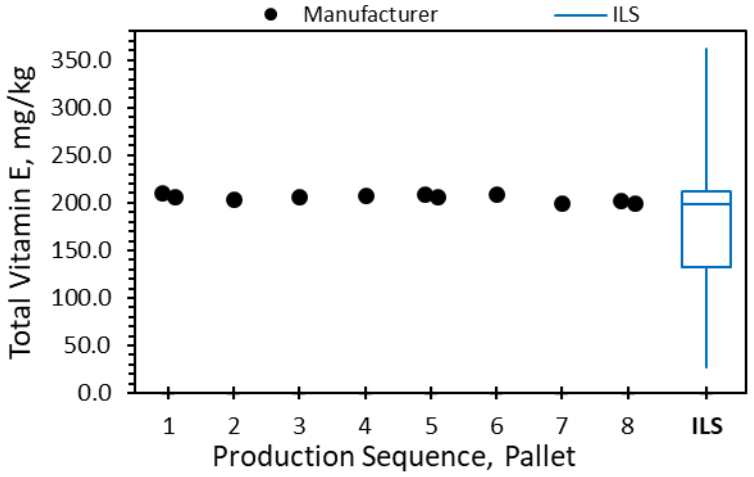

Fig. 40. Total vitamin E (total α-tocopherol) mass fraction as a function of production sequence

5.2.18. β-Tocopherol

The manufacturer LC-FL [34-36] and HAMQAP [17] measurement results for β-tocopherol are summarized in Table 52. Fig. 41 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 52. Summary of results for β-tocopherol, mg/kg

Manufacturer

ID	Value
1:1	4.95
2:1	4.93
3:1	4.93
4:1	5.04
5:1	5.00
5:2	4.95
6:1	4.95
7:1	4.92
8:1	4.95
1:2	4.86
8:2	4.87
N:	11
Mean:	4.94
SD:	0.05

HAMQAP Exercise 6

Code	Rep_1	Rep_2	Rep_3	Mean	SD	Methods
F005	0[a]	0[a]	0[a]			LC-Abs or PDA
F013	2.79	3.15	2.92	2.95	0.18	LC-FL
F014	5.08	4.78	4.81	4.89	0.17	LC-FL
F030	<5[a]	<5[a]	<5[a]			LC-FL
F033	4.60	4.30	4.30	4.40	0.17	LC-Abs or PDA
F039	5.01	5.26	5.31	5.19	0.16	LC-FL
F062	<0.2[a]	<0.2[a]	<0.2[a]			LC-FL
			N:	4		
			Mean, Pooled SD:	4.36	0.17	
			SD:	0.99		

[a] Identified as a non-representative or non-quantitative value; excluded from statistical analysis.

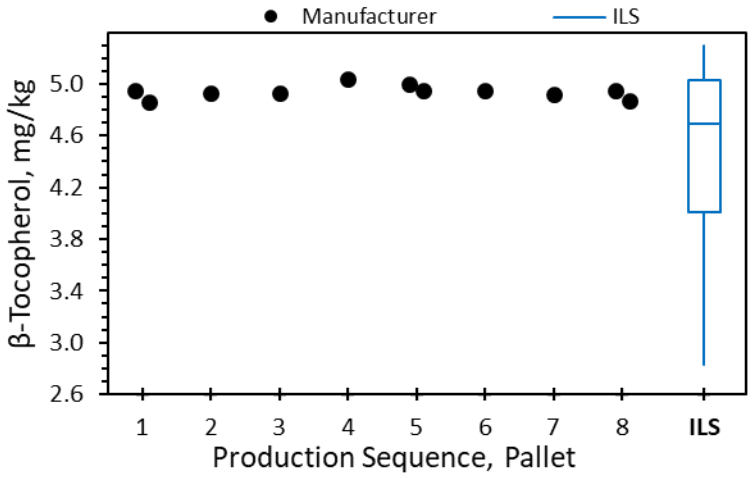

Fig. 41. β-Tocopherol mass fraction as a function of production sequence

5.2.19. γ-Tocopherol

The manufacturer LC-FL [34-36] and HAMQAP [17] measurement results for γ-tocopherol are summarized in Table 53. Fig. 42 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 53. Summary of results for γ-tocopherol, mg/kg

Manufacturer

ID	Value
1:1	114
2:1	114
3:1	114
4:1	115
5:1	115
5:2	115
6:1	114
7:1	114
8:1	114
1:2	114
8:2	111
N:	11
Mean:	114.0
SD:	1.1

HAMQAP Exercise 6

Code	Rep_1	Rep_2	Rep_3	Mean	SD	Methods
F005	117.72	149.49	159.00	142.07	21.62	LC-Abs or PDA
F013	128.00	118.00	122.00	122.67	5.03	LC-FL
F014	126.00	117.00	118.00	120.33	4.93	LC-FL
F030	93.20	94.30	97.30	94.93	2.12	LC-FL
F033	116.00	115.00	112.00	114.33	2.08	LC-Abs or PDA
F039	135.00	141.00	146.00	140.67	5.51	LC-FL
F062	114.20	104.70	98.50	105.80	7.91	LC-FL
F075	112.00	107.00	110.00	109.67	2.52	LC-FL
			N:	8		
			Mean, Pooled SD:	118.81	8.84	
			SD:	16.38		

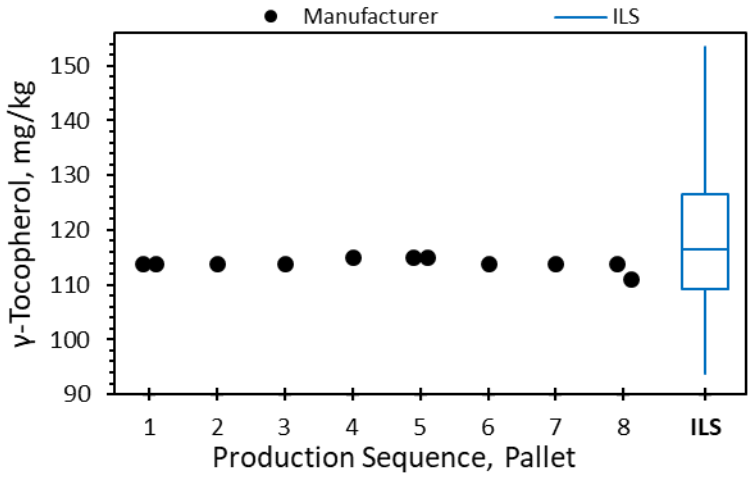

Fig. 42. γ-Tocopherol mass fraction as a function of production sequence

5.2.20. δ-Tocopherol

The manufacturer LC-FL [34-36] and HAMQAP [17] measurement results for δ-tocopherol are summarized in Table 54. Fig. 43 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 54. Summary of results for δ-tocopherol, mg/kg

Manufacturer

ID	Value
1:1	37.2
2:1	38.3
3:1	37.7
4:1	38.3
5:1	37.5
5:2	38.4
6:1	38.2
7:1	38.5
8:1	38.1
1:2	37.3
8:2	37.2
N:	11
Mean:	37.88
SD:	0.51

HAMQAP Exercise 6

Code	Rep_1	Rep_2	Rep_3	Mean	SD	Methods
F005	39.88	52.26	55.37	49.17	8.19	LC-Abs or PDA
F013	28.60	27.70	27.50	27.93	0.59	LC-FL
F014	36.60	33.00	34.90	34.83	1.80	LC-FL
F017	25.29	21.60	19.75	22.21	2.82	LC-Abs or PDA
F030	18.00	18.20	19.80	18.67	0.99	LC-FL
F033	40.30	41.80	40.70	40.93	0.78	LC-Abs or PDA
F039	43.20	45.80	47.30	45.43	2.07	LC-FL
F046	11.77	11.76	11.41	11.65	0.21	LC-Abs or PDA
F062	31.50	31.10	29.30	30.63	1.17	LC-FL
F075	34.30	32.90	34.10	33.77	0.76	LC-FL
			N:	10		
			Mean, Pooled SD:	31.52	2.94	
			SD:	11.88		

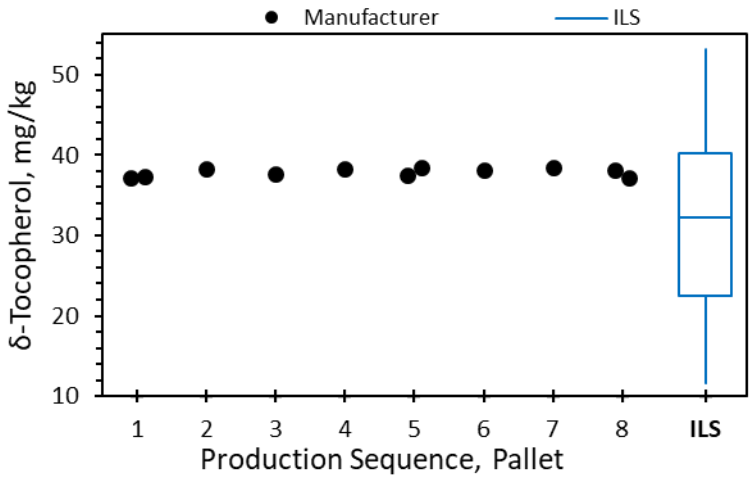

Fig. 43. δ-Tocopherol mass fraction as a function of production sequence

5.2.21. Vitamin K (Phylloquinone)

The manufacturer LC-FL [37] and FNSQAP [18] measurement results for vitamin K (phylloquinone) are summarized in Table 55. Fig. 44 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 55. Summary of results for vitamin K (phylloquinone), mg/kg

Manufacturer

ID	Value
1:1	1.04
2:1	1.03
3:1	1.02
4:1	1.03
5:1	1.04
5:2	1.04
6:1	1.03
7:1	1.03
8:1	1.04
1:2	1.05
8:2	1.03
N:	11
Mean:	1.035
SD:	0.008

FNSQAP Exercise 1

Code	Rep_1	Rep_2	Rep_3	Mean	SD	Methods
A001	< 0.372[a]	< 0.372[a]	< 0.372[a]			LC-MS
A006	15.58[a]	4.34[a]	13.33[a]			LC-Abs or PDA
A010	3[a]	4[a]	4[a]			LC-Abs or PDA
A015	1.179	1.189	1.191	1.19	0.01	LC-FL
A016	0.966	0.916	0.757	0.88	0.11	LC-FL
A027	0.879	0.878	0.846	0.87	0.02	Not specified or Other
			N:	3		
			Mean, Pooled SD:	0.98	0.06	
			SD:	0.18		

[a] Identified as a non-representative or non-quantitative value; excluded from statistical analysis.

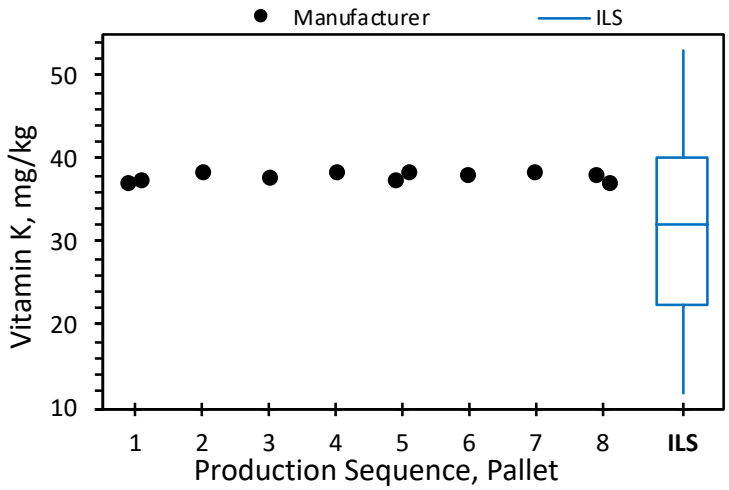

Fig. 44. Vitamin K (phylloquinone) mass fraction as a function of production sequence

5.2.22. β-Carotene

The manufacturer LC-Abs and FNSQAP [19] measurement results for β-carotene are summarized in Table 56. Fig. 45 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 56. Summary of results for β-carotene, mg/kg

Manufacturer

ID	Value
1:1	0.540
2:1	0.542
3:1	0.556
4:1	0.553
5:1	0.554
5:2	0.547
6:1	0.546
7:1	0.534
8:1	0.533
1:2	0.561
8:2	0.531
N:	11
Mean:	0.545
SD:	0.010

FNSQAP Exercise 2

Code	Rep_1	Rep_2	Rep_3	Mean	SD	Methods
B001	0.21	0.23	0.18	0.21	0.02	LC-Abs or PDA
B009	4.99[a]	5.31[a]	5.33[a]			LC
B021	< 0.630[a]	< 0.630[a]	< 0.630[a]			LC-Abs or PDA
B031	< 0.600[a]	< 0.600[a]	< 0.600[a]			LC-Abs or PDA
B035	0.58	0.62	0.59	0.60	0.02	LC-Abs or PDA
B044	0.50	0.50	0.50	0.50	0.00	LC-Abs or PDA
B052	< 0.280[a]	< 0.280[a]	< 0.280[a]			LC-Abs or PDA
B055	< 10.00[a]	< 10.00[a]	< 10.00[a]			LC-Abs or PDA
			N:	3		
			Mean, Pooled SD:	0.43	0.02	
				0.20		

[a] Identified as a non-representative or non-quantitative value; excluded from statistical analysis.

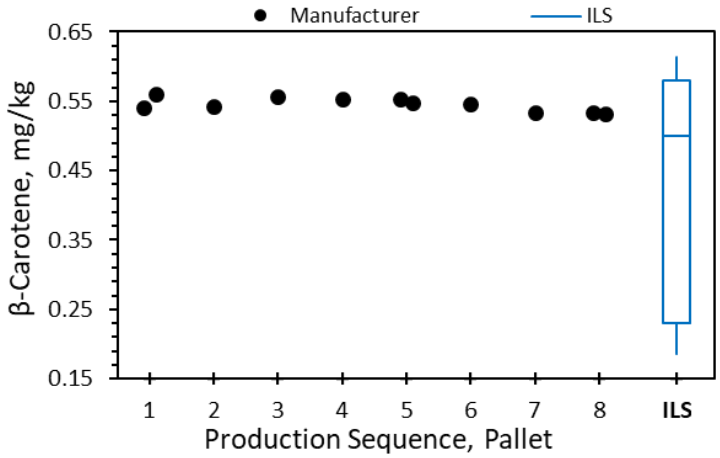

Fig. 45. β-Carotene mass fraction as a function of production sequence

5.2.23. Lycopene

The manufacturer LC-Abs and FNSQAP [19] measurement results for lycopene are summarized in Table 57. Fig. 46 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 57. Summary of results for lycopene, mg/kg

Manufacturer

ID	Value
1:1	1.72
2:1	1.77
3:1	1.75
4:1	1.72
5:1	1.77
5:2	1.73
6:1	1.70
7:1	1.72
8:1	1.71
1:2	1.78
8:2	1.69
N:	11
Mean:	1.733
SD:	0.030

FNSQAP Exercise 2

Code	Rep_1	Rep_2	Rep_3	Mean	SD	Methods
B001	0.178	0.191	0.166	0.178	0.012	LC-Abs or PDA
B021	< 2.470[a]	< 2.470[a]	< 2.470[a]			LC-Abs or PDA
B035	2.21	2.04	2.04	2.097	0.098	LC-Abs or PDA
B055	< 10.00[a]	< 10.00[a]	< 10.00[a]			LC-Abs or PDA
			N:	2		
			Mean, Pooled SD:	1.14	0.07	
			SD:	1.36		

[a] Identified as a non-representative or non-quantitative value; excluded from statistical analysis.

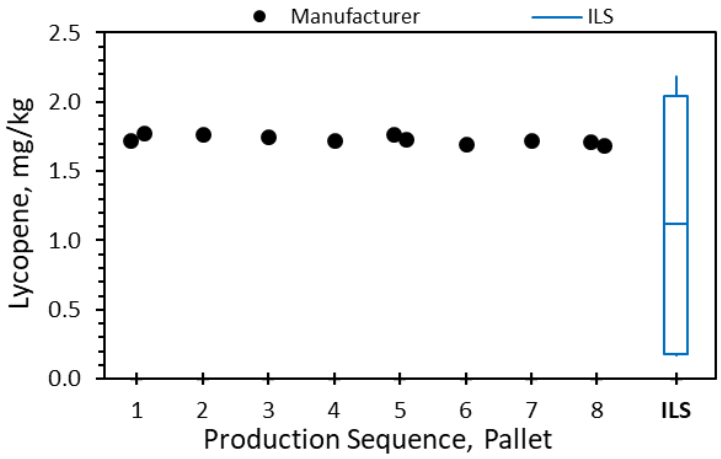

Fig. 46. Lycopene mass fraction as a function of production sequence

5.2.24. Lutein

The manufacturer LC-Abs and FNSQAP [19] measurement results for lutein are summarized in Table 58. Fig. 47 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 58. Summary of results for lutein, mg/kg

Manufacturer

ID	Value
1:1	2.47
2:1	2.50
3:1	2.49
4:1	2.48
5:1	2.48
5:2	2.50
6:1	2.45
7:1	2.50
8:1	2.44
1:2	2.49
8:2	2.45
N:	11
Mean:	2.477
SD:	0.022

FNSQAP Exercise 2

Code	Rep_1	Rep_2	Rep_3	Mean	SD	Methods
B001	1.07	1.123	0.946	1.046	0.091	LC-Abs or PDA
B005	< 0.010[a]	< 0.010[a]	< 0.010[a]			LC-Abs or PDA
B009	2.53	2.46	2.5	2.497	0.035	LC
B021	< 1.650[a]	< 1.650[a]	< 1.650[a]			LC-Abs or PDA
B035	1.87	1.83	1.81	1.837	0.031	LC-Abs or PDA
B052	< 0.040[a]	< 0.040[a]	< 0.040[a]			LC-Abs or PDA
B055	1.9	2.09	2.1	2.03	0.113	LC-Abs or PDA
			N:	4		
			Mean, Pooled SD:	1.85	0.08	
			SD:	0.60		

[a] Identified as a non-representative or non-quantitative value; excluded from statistical analysis.

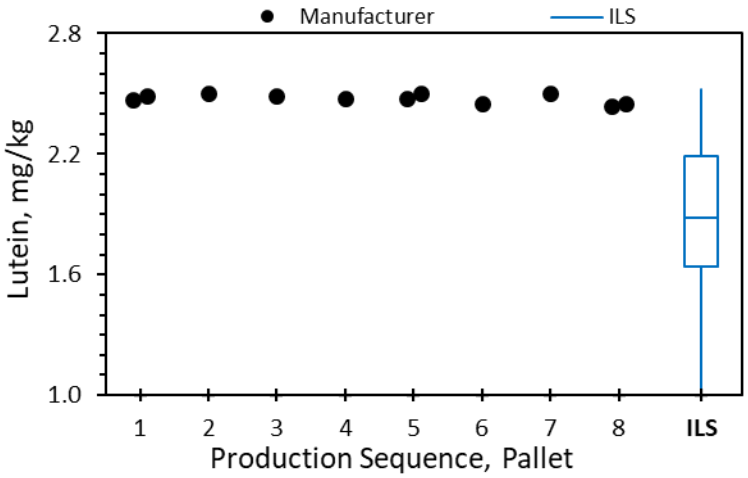

Fig. 47. Lutein mass fraction as a function of production sequence

5.2.25. Carnitine

The manufacturer LC-MS/MS [38], NIST ID LC-MS [15], and FNSQAP [19] measurement results for carnitine are summarized in Table 59. Fig. 48 displays these results as an approximate function of the production sequence, as determined by the pallet and box number. The NIST measurement results for carnitine in the SRM 1849b control material agreed well with the non-certified value stated in its COA.

Table 59. Summary of results for carnitine, mg/kg

Manufacturer

ID	Value
1:1	164
2:1	157
3:1	166
4:1	159
5:1	157
5:2	159
6:1	160
7:1	163
8:1	154
1:2	163
8:2	159
N:	11
Mean:	160.1
SD:	3.6

NIST ID LC-MS

Box	Rep_1	Rep_2	Rep_3	Mean	SD
1	142.1	142.4	136.9	140.5	3.1
36	146.7	143.9	143.7	144.8	1.7
61	150.4	145.1	148.5	148.0	2.7
149	151.5	142.5	145.1	146.4	4.6
282	138.8	143.0	143.4	141.8	2.5
290	144.1	144.5	141.9	143.5	1.4
			N:	6	
			Mean, Pooled SD:	144.2	4.5
			SD:	6.1	

FNSQAP Exercise 2

Code	Rep_1	Rep_2	Rep_3	Mean	SD	Method
B015	152	142	145	146.3	5.1	LC-MS/MS
B019	155	156	154	155.0	1.0	LC-MS/MS
B031	146	141	140	142.3	3.2	LC-MS/MS
B043	47.5[a]	46.6[a]	48.1[a]			LC-MS/MS
B044	158.3	157.3	155.7	157.1	1.3	LC-MS/MS
B055	146	159	145	150.0	7.8	LC-MS/MS
			N:	5		
			Mean, Pooled SD:	150.2	4.5	
			SD:	6.1		

[a] Identified as a non-representative or non-quantitative value; excluded from statistical analysis.

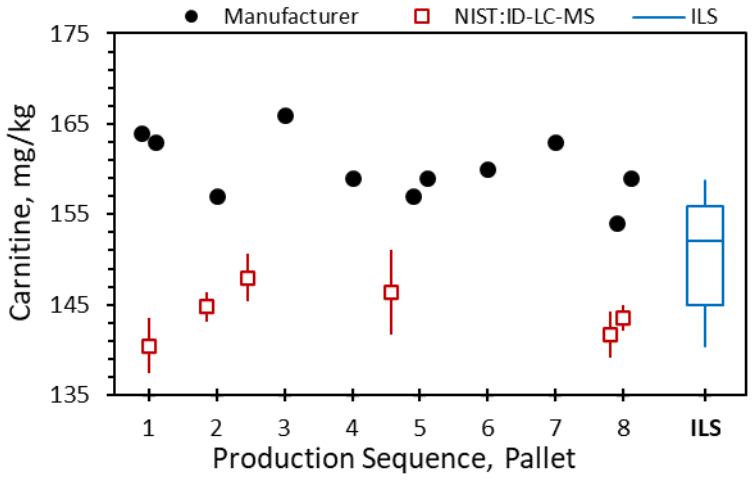

Fig. 48. Carnitine mass fraction as a function of production sequence

5.2.26. Choline

The manufacturer LC-MS/MS [38], NIST ID LC-MS [15], and FNSQAP [19] measurement results for choline are summarized in Table 60. Fig. 49 displays these results as an approximate function of the production sequence, as determined by the pallet and box number. The NIST measurement results for choline in the SRM 1849b control material agreed well with the non-certified value stated in its COA.

Table 60. Summary of results for choline, mg/kg

Manufacturer

ID	Value
1:1	978
2:1	964
3:1	1040
4:1	1010
5:1	1100
5:2	1040
6:1	976
7:1	971
8:1	1000
1:2	990
8:2	1090
N:	11
Mean:	1014
SD:	47

NIST ID LC-MS

Box	Rep$_1$	Rep$_2$	Rep$_3$	Mean	SD
1	1018	1077	998	1031	41
36	1010	1085	982	1025	53
61	1012	1080	987	1026	49
149	1019	1095	994	1036	53
282	1000	1081	987	1023	51
290	1027	1092	987	1035	53
			N:	6	
			Mean, Pooled SD:	1030	50
			SD:	5	

FNSQAP Exercise 2

Code	Rep$_1$	Rep$_2$	Rep$_3$	Mean	SD	Method
B004	7538[a]	7477[a]	7507[a]			LC-MS/MS
B005	704	744	709	719	22	LC-MS/MS
B015	985	970	977	977	8	LC-MS/MS
B019	967	997	984	983	15	LC-MS/MS
B031	1030	1120	1130	1093	55	
B044	1030	1020	1010	1020	10	IC-CD
B055	750	750	750	750	0	LC-MS/MS
			N:	6		
			Mean, Pooled SD:	924	153	
			SD:	24		

[a] Identified as a non-representative or non-quantitative value; excluded from statistical analysis.

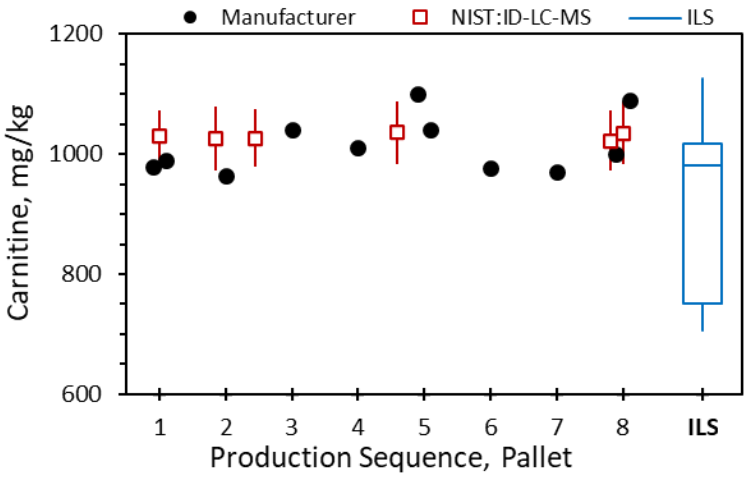

Fig. 49. Choline mass fraction as a function of production sequence

5.2.27. *myo*-Inositol

The manufacturer LC-PAD [39] results for *myo*-inositol are summarized in Table 61. Fig. 50 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 61. Summary of results *myo*-inositol, mg/kg

	Manufacturer
ID	Value
1:1	452
2:1	451
3:1	454
4:1	452
5:1	454
5:2	453
6:1	455
7:1	446
8:1	431
1:2	452
8:2	438
N:	11
Mean:	448.9
SD:	7.7

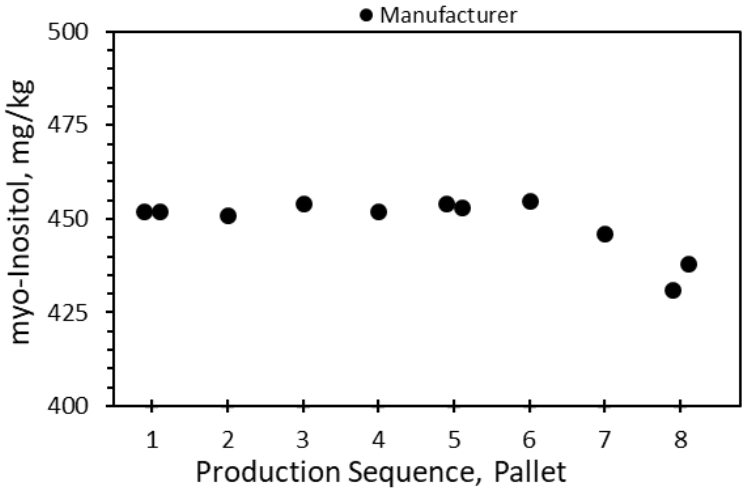

Fig. 50. *myo*-Inositol mass fraction as a function of production sequence

5.3. Proximates

5.3.1. Fat

The manufacturer base hydrolysis [40-43] and HAMQAP [17] measurement results for fat are summarized in Table 62. Fig. 51 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 62. Summary of results for fat, g/100 g

Manufacturer

ID	Value
1:1	28.13
2:1	27.99
3:1	28.37
4:1	27.88
5:1	28.11
5:2	28.21
6:1	28.23
7:1	28.06
8:1	28.14
1:2	27.90
8:2	28.05
N:	11
Mean:	28.10
SD:	0.14

HAMQAP Exercise 6

Code	Rep_1	Rep_2	Rep_3	Mean	SD	Methods
F005	21.78	20.08	21.64	21.167	0.944	Not specified or Other
F009	28.00	26.40	28.10	27.500	0.954	Alkaline Digestion with Ether Extraction
F017	27.74	27.73	27.94	27.803	0.118	RG/M Acid Digestion
F020	27.50	27.40	27.10	27.333	0.208	Acid Hydrolysis
F030	27.35	27.58	27.70	27.543	0.178	RG/M Acid Digestion
F031	28.21	27.93	28.14	28.093	0.146	Acid Hydrolysis
F039	27.64	27.78	27.78	27.733	0.081	Alkaline Digestion with Ether Extraction
F059	27.95	27.96	27.90	27.937	0.032	Oven Drying
F079	27.81	27.95	27.87	27.877	0.070	Acid Hydrolysis
F080	28.01	28.06	27.90	27.990	0.082	RG/M Acid Digestion
			N:	10		
			Mean, Pooled SD:	27.10	0.44	
			SD:	2.10		

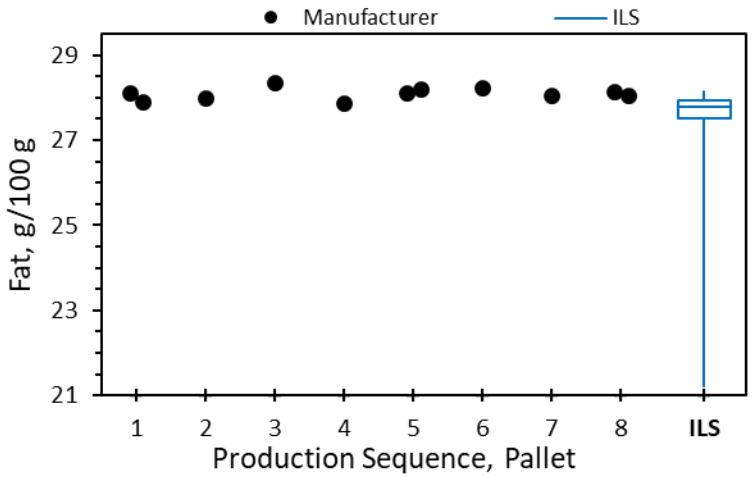

Fig. 51. Fat mass fraction as a function of production sequence

5.3.2. Protein

The manufacturer Kjeldahl [44] and HAMQAP [17] measurement results for protein are summarized in Table 63. Fig. 52 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 63. Summary of results for protein, g/100 g

Manufacturer		HAMQAP Exercise 6						
ID	Value	Code	Rep_1	Rep_2	Rep_3	Mean	SD	Methods
1:1	12.96	F002	75.3[a]	75.6[a]	75.5[a]			Combustion
2:1	12.64	F005	0.0379[a]	0.0128[a]	0.0877[a]			Combustion
3:1	12.95	F009	12.60	12.00	13.10	12.567	0.551	Kjeldahl
4:1	12.99	F017	13.10	13.20	13.30	13.200	0.100	Combustion
5:1	12.96	F020	12.99	13.00	12.95	12.980	0.026	Combustion
5:2	13.21	F030	12.49	12.48	12.57	12.513	0.049	Kjeldahl
6:1	12.83	F031	13.07	13.07	13.05	13.063	0.012	Kjeldahl
7:1	12.74	F039	12.70	12.76	13.02	12.827	0.170	Kjeldahl
8:1	12.75	F059	12.87	12.69	12.74	12.767	0.093	Kjeldahl
1:2	13.00	F079	12.58	12.61	12.43	12.540	0.096	Kjeldahl
8:2	12.81	F080	13.23	13.18	13.25	13.220	0.036	Kjeldahl
N:	11				*N*:	9		
Mean:	12.89				Mean, Pooled SD:	12.85	0.20	
SD:	0.16				SD:	0.28		

[a] Identified as a non-representative value; excluded from statistical analysis.

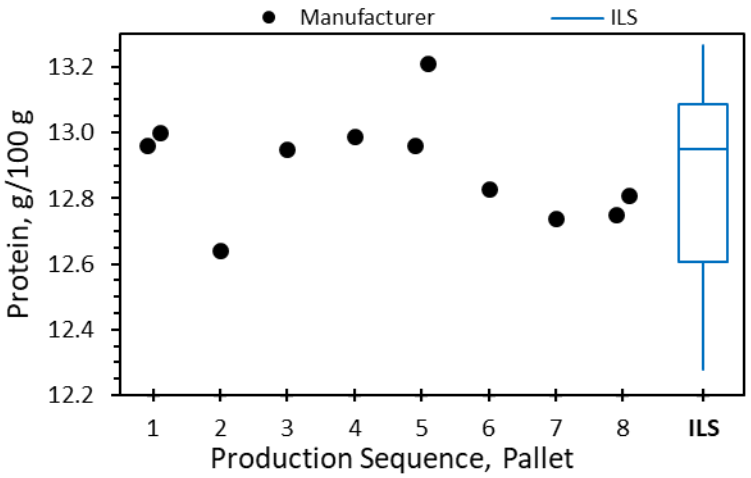

Fig. 52. Protein mass fraction as a function of production sequence

5.3.3. Carbohydrates

The manufacturer calculation and HAMQAP [17] results for carbohydrates are summarized in Table 64. Fig. 53 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 64. Summary of results for carbohydrates, g/100 g

Manufacturer

ID	Value
1:1	54.46
2:1	54.89
3:1	54.20
4:1	54.63
5:1	54.46
5:2	54.11
6:1	54.46
7:1	54.77
8:1	54.69
1:2	54.66
8:2	54.64
N:	11
Mean:	54.54
SD:	0.23

HAMQAP Exercise 6

Code	Rep_1	Rep_2	Rep_3	Mean	SD	Methods
F005	47.45[a]	46.55[a]	43.24[a]			Not specified or Other
F009	52.60	54.90	52.30	53.267	1.422	100-Solids-Protein-Fat-Ash
F017	52.65	52.48	52.17	52.433	0.243	100-Solids-Protein-Fat-Ash
F020	52.40	52.50	52.60	52.500	0.100	100-Solids-Protein-Fat-Ash
F031	52.00	52.10	52.10	52.067	0.058	100-Solids-Protein-Fat-Ash
F039	52.43	52.05	51.77	52.083	0.331	100-Solids-Protein-Fat-Ash
F049	50.40	50.50	50.60	50.500	0.100	Not specified or Other
F079	52.71	53.41	52.58	52.900	0.446	100-Solids-Protein-Fat-Ash
F080	51.93	51.69	51.70	51.773	0.136	100-Solids-Protein-Fat-Ash
			N:	8		
			Mean, Pooled SD:	52.19	0.55	
			SD:	0.83		

[a] Identified as a non-representative value; excluded from statistical analysis.

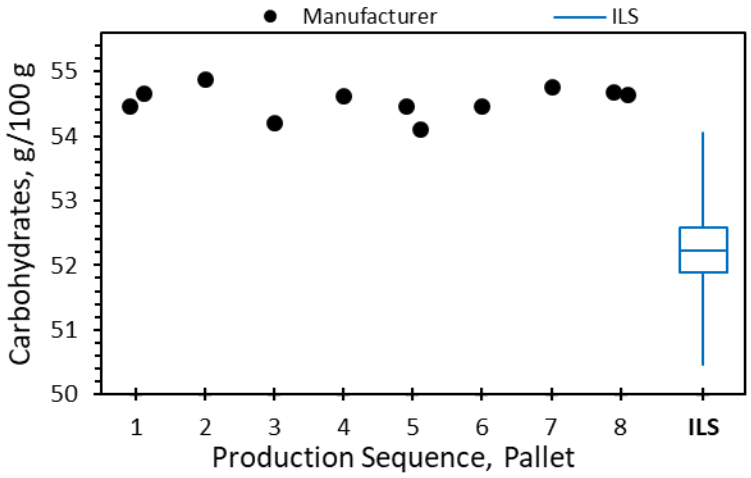

Fig. 53. Carbohydrates mass fraction as a function of production sequence

5.3.4. Solids

The manufacturer vacuum oven drying [45,46] and HAMQAP [17] measurement results for solids are summarized in Table 65. Fig. 54 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 65. Summary of results for solids, g/100 g

Manufacturer

ID	Value
1:1	97.80
2:1	97.79
3:1	97.88
4:1	97.90
5:1	97.83
5:2	97.88
6:1	97.85
7:1	97.79
8:1	97.88
1:2	97.76
8:2	97.77
N:	11
Mean:	97.830
SD:	0.050

HAMQAP Exercise 6

Code	Rep$_1$	Rep$_2$	Rep$_3$	Mean	SD	Methods
F005	84.3[a]	85.2[a]	97.3[a]			Vacuum Oven
F009	97.80	97.80	98.10	97.900	0.173	Forced-Air Oven
F017	98.00	97.99	98.02	98.003	0.015	Forced-Air Oven
F019	97.80	97.72	97.72	97.747	0.046	Forced-Air Oven
F030	97.75	97.71	97.66	97.707	0.045	Thermogravimetric
F031	97.85	97.76	97.89	97.833	0.067	Forced-Air Oven
F039	97.30	97.10	97.10	97.167	0.115	Forced-Air Oven
F059	97.80	97.90	97.80	97.833	0.058	Vacuum Oven
F079	97.49	97.61	98.56	97.887	0.586	Forced-Air Oven
F080	97.63	97.55	97.54	97.573	0.049	Forced-Air Oven
			N:	9		
			Mean, Pooled SD:	97.74	0.21	
			SD:	0.25		

[a] Identified as a non-representative value; excluded from statistical analysis.

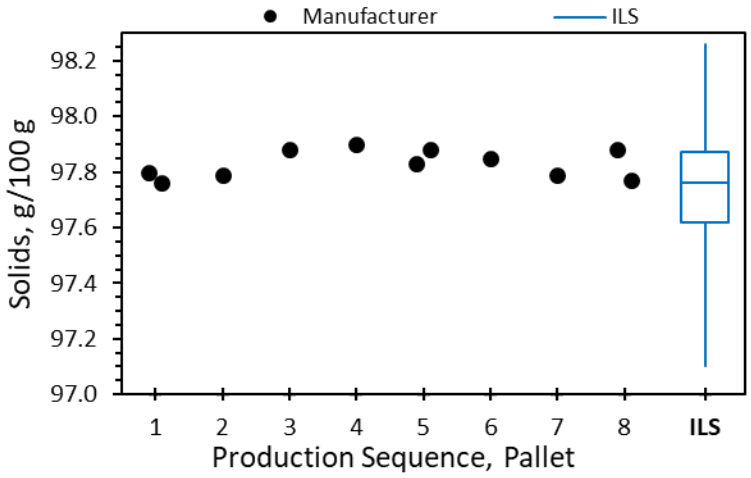

Fig. 54. Solids mass fraction as a function of production sequence

5.3.5. Ash

The manufacturer dry ashing [47] and HAMQAP [17] measurement results for ash are summarized in Table 66. Fig. 55 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 66. Summary of results for ash, g/100 g

Manufacturer		HAMQAP Exercise 6						
ID	Value	Code	Rep_1	Rep_2	Rep_3	Mean	SD	Methods
1:1	4.45	F005	4.29	4.31	4.33	4.310	0.020	Weight loss after ignition
2:1	4.48	F009	4.60	4.60	4.60	4.600	0.000	Weight loss after ignition
3:1	4.48	F011	4.33	4.38	4.40	4.370	0.036	Weight loss after ignition
4:1	4.50	F017	4.51	4.58	4.61	4.567	0.051	Weight loss after ignition
5:1	4.47	F019	4.30	4.49	4.49	4.427	0.110	Dry Ashing
5:2	4.47	F020	4.50	4.60	4.50	4.533	0.058	Weight loss after ignition
6:1	4.48	F021	7.71[a]	8.02[a]	6.89[a]			Dry Ashing
7:1	4.43	F030	4.41	4.45	4.35	4.403	0.050	Thermogravimetric
8:1	4.42	F031	4.58	4.61	4.62	4.603	0.021	Weight loss after ignition
1:2	4.44	F039	4.53	4.51	4.53	4.523	0.012	Weight loss after ignition
8:2	4.50	F059	4.46	4.59	4.42	4.490	0.089	Weight loss after ignition
N:	11	F062	4.48	4.53	4.53	4.513	0.029	Dry Ashing
Mean:	4.47	F079	4.54	4.58	4.59	4.570	0.026	Weight loss after ignition
SD:	0.03	F080	4.46	4.62	4.69	4.590	0.118	Weight loss after ignition
		F088	4.46	4.45	4.45	4.452	0.005	Dry Ashing
					N:	14		
					Mean, Pooled SD:	4.497	0.057	
					SD:	0.092		

[a] Identified as a non-representative value; excluded from statistical analysis.

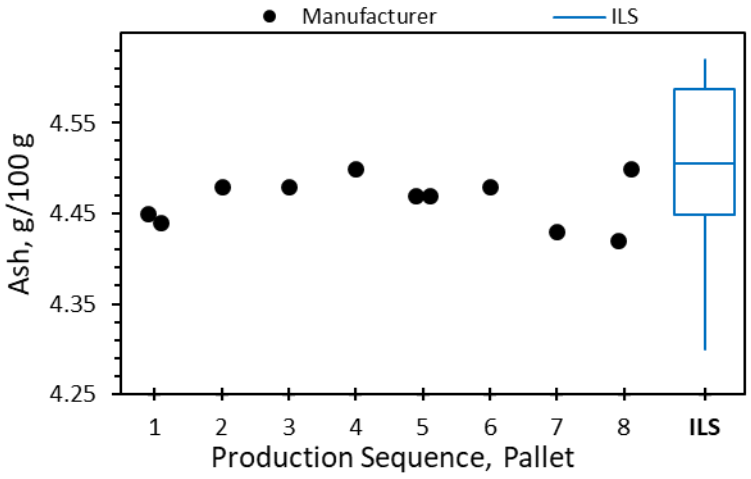

Fig. 55. Ash mass fraction as a function of production sequence

5.3.6. Calories

The manufacturer calculation and HAMQAP [17] results for calories are summarized in Table 67. Fig. 56 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 67. Summary of results for calories, kcal/100 g

Manufacturer

ID	Value
1:1	522.85
2:1	522.03
3:1	523.93
4:1	521.40
5:1	522.67
5:2	523.17
6:1	523.23
7:1	522.58
8:1	523.02
1:2	521.74
8:2	522.25
N:	11
Mean:	522.62
SD:	0.73

HAMQAP Exercise 6

Code	Rep$_1$	Rep$_2$	Rep$_3$	Mean	SD	Methods
F005	385.97[a]	366.97[a]	368.07[a]			9(Fat)+4(Protein)+4(Carbohydrate)
F009	513.00	505.00	514.00	510.7	4.9	9(Fat)+4(Protein)+4(Carbohydrate)
F031	514.00	512.00	514.00	513.3	1.2	9(Fat)+4(Protein)+4(Carbohydrate)
F039	540[a]	536[a]	538[a]			Bomb Calorimetry
F079	511.00	516.00	511.00	512.7	2.9	9(Fat)+4(Protein)+4(Carbohydrate)
F080	512.73	512.02	510.90	511.9	0.9	9(Fat)+4(Protein)+4(Carbohydrate)
			N:	4		
			Mean, Pooled SD:	512.14	2.95	
			SD:	1.15		

[a] Identified as a non-representative value; excluded from statistical analysis.

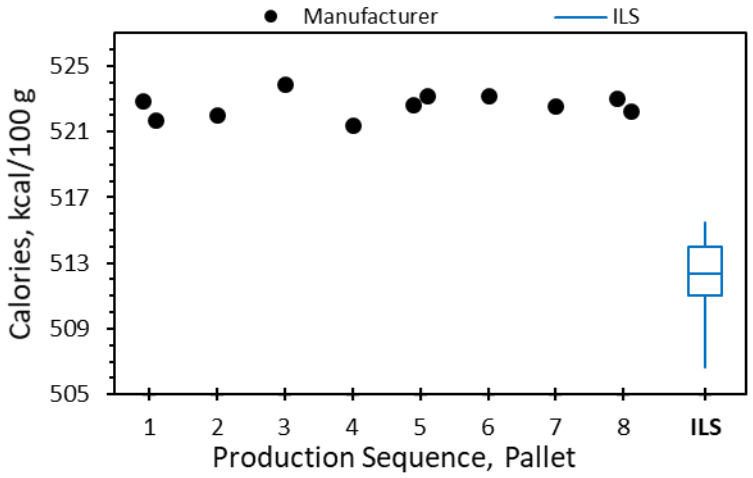

Fig. 56. Calories as a function of production sequence

5.4. Cholesterol

The manufacturer GC-MS [48] measurement results for cholesterol are summarized in Table 68. Fig. 57 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 68. Cholesterol mass fraction results, mg/100 g

Manufacturer	
ID	Value
1:1	13.6
2:1	13.5
3:1	13.3
4:1	13.4
5:1	13.5
5:2	13.4
6:1	13.6
7:1	13.4
8:1	13.7
1:2	13.2
8:2	13.4
N:	11
Mean:	13.45
SD:	0.14

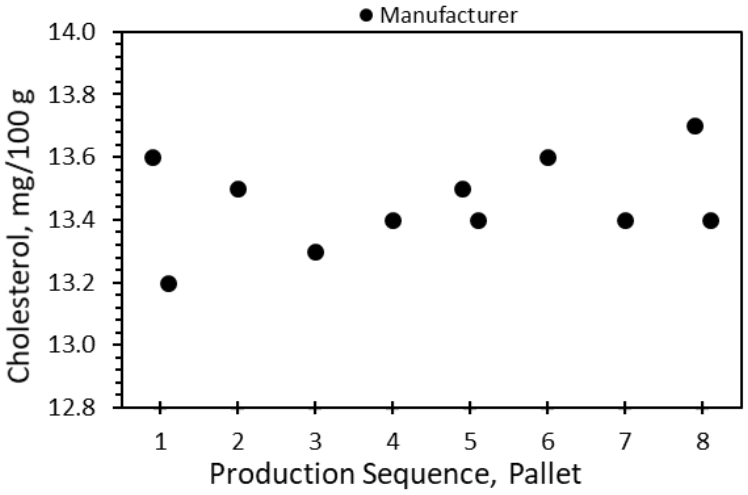

Fig. 57. Cholesterol mass fraction as a function of production sequence

5.5. Fatty Acids

5.5.1. Caproic Acid (C6:0)

The manufacturer GC-FID [49-53] measurement results for caproic acid (C6:0) are summarized in Table 69. Fig. 58 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 69. Caproic acid (C6:0) mass fraction results, %

	Manufacturer
ID	Value
1:1	0.0460
2:1	0.0469
3:1	0.0469
4:1	0.0460
5:1	0.0460
5:2	0.0460
6:1	0.0451
7:1	0.0460
8:1	0.0451
1:2	0.0451
8:2	0.0460
N:	11
Mean:	0.0459
SD:	0.0006

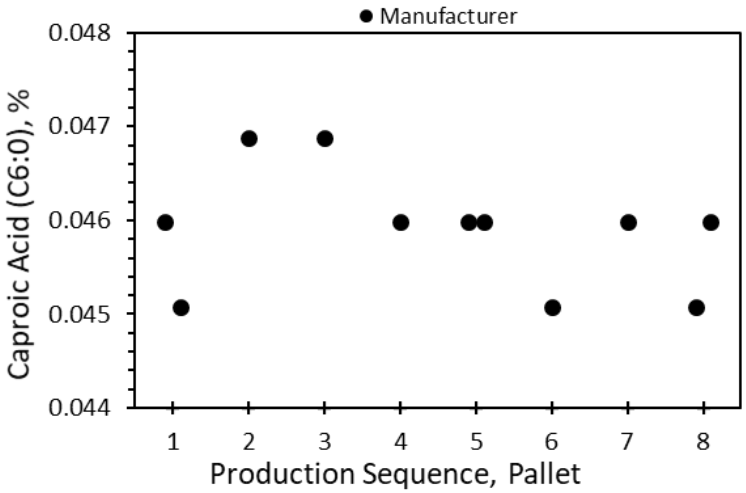

Fig. 58. Caproic acid (C6:0) mass fraction as a function of production sequence

5.5.2. Caprylic Acid (C8:0)

The manufacturer GC-FID [49,50] measurement results for caprylic acid (C8:0) are summarized in Table 70. Fig. 59 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 70. Caprylic acid (C8:0) mass fraction results, %

Manufacturer	
ID	Value
1:1	0.5065
2:1	0.5129
3:1	0.5138
4:1	0.5129
5:1	0.5056
5:2	0.5083
6:1	0.5056
7:1	0.5065
8:1	0.5083
1:2	0.5028
8:2	0.5111
N:	11
Mean:	0.5086
SD:	0.0036

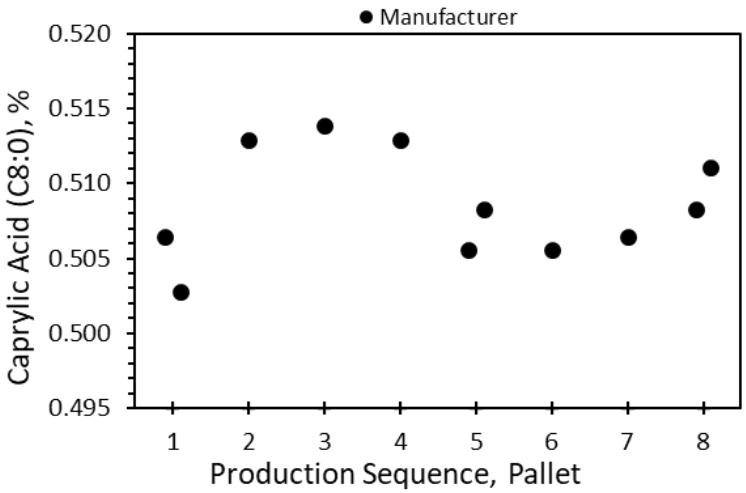

Fig. 59. Caprylic acid (C8:0) mass fraction as a function of production sequence

5.5.3. Capric Acid (C10:0)

The manufacturer GC-FID [49,50] measurement results for capric acid (C10:0) are summarized in Table 71. Fig. 60 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 71. Capric acid (C10:0) mass fraction results, %

	Manufacturer
ID	Value
1:1	0.4089
2:1	0.4117
3:1	0.4126
4:1	0.4117
5:1	0.4089
5:2	0.4098
6:1	0.4089
7:1	0.4089
8:1	0.4098
1:2	0.4080
8:2	0.4117
N:	11
Mean:	0.410
SD:	0.0016

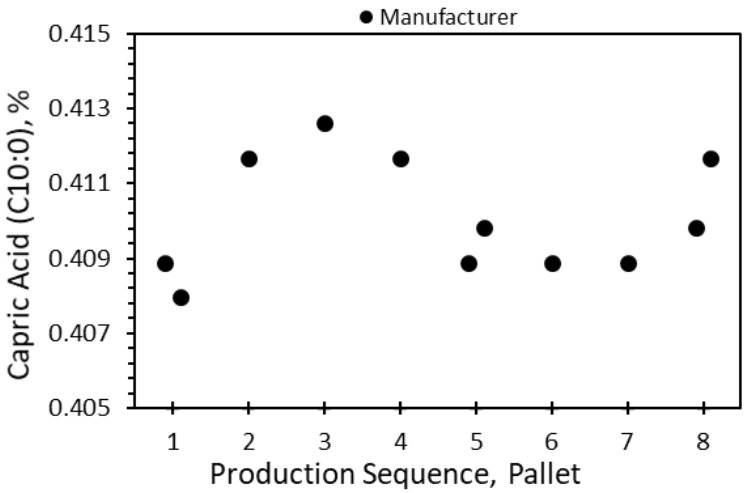

Fig. 60. Capric acid (C10:0) mass fraction as a function of production sequence

5.5.4. Lauric Acid (C12:0)

The manufacturer GC-FID [49,50] measurement results for lauric acid (C12:0) are summarized in Table 72. Fig. 61 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 72. Lauric acid (C12:0) mass fraction results, %

	Manufacturer
ID	Value
1:1	3.1977
2:1	3.1977
3:1	3.2071
4:1	3.2071
5:1	3.1977
5:2	3.1977
6:1	3.1977
7:1	3.1883
8:1	3.1977
1:2	3.1883
8:2	3.1977
N:	11
Mean:	3.1977
SD:	0.0059

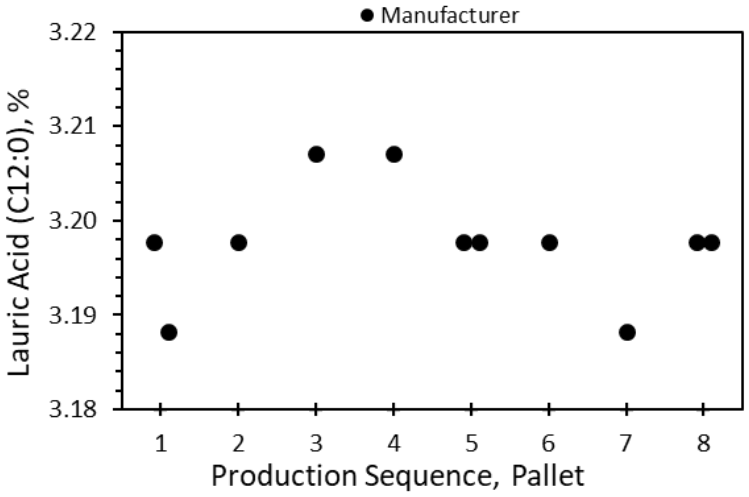

Fig. 61. Lauric acid (C12:0) mass fraction as a function of production sequence

5.5.5. Myristic Acid (C14:0)

The manufacturer GC-FID [49,50] measurement results for myristic acid (C14:0) are summarized in Table 73. Fig. 62 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 73. Myristic acid (C14:0) mass fraction results, %

Manufacturer	
ID	Value
1:1	1.3548
2:1	1.3453
3:1	1.3453
4:1	1.3548
5:1	1.3548
5:2	1.3453
6:1	1.3453
7:1	1.3453
8:1	1.3453
1:2	1.3453
8:2	1.3453
N:	11
Mean:	1.3479
SD:	0.0044

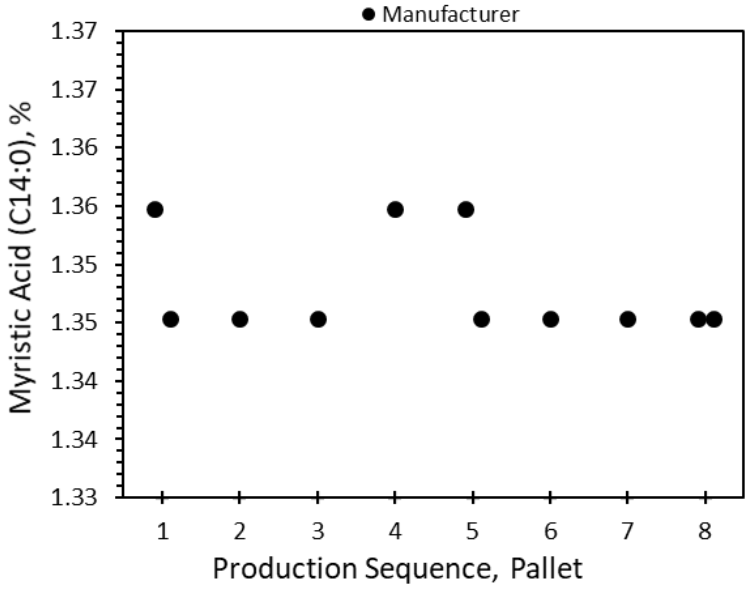

Fig. 62. Myristic acid (C14:0) mass fraction as a function of production sequence

5.5.6. Palmitic Acid (C16:0)

The manufacturer GC-FID [49,50] measurement results for palmitic acid (C16:0) are summarized in Table 74. Fig. 63 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 74. Palmitic acid (C16:0) mass fraction results, %

	Manufacturer
ID	Value
1:1	2.1726
2:1	2.1631
3:1	2.1631
4:1	2.1726
5:1	2.1726
5:2	2.1631
6:1	2.1631
7:1	2.1631
8:1	2.1726
1:2	2.1726
8:2	2.1631
N:	11
Mean:	2.1674
SD:	0.0050

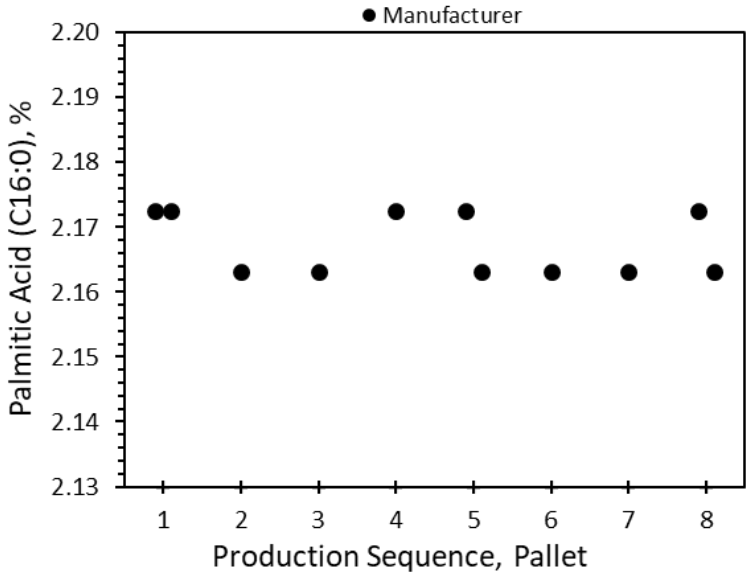

Fig. 63. Palmitic acid (C16:0) mass fraction as a function of production sequence

5.5.7. Palmitoleic Acid (C16:1)

The manufacturer GC-FID [49,50] measurement results for palmitoleic acid (C16:1) are summarized in Table 75. Fig. 64 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 75. Palmitoleic acid (C16:1) mass fraction results, %

Manufacturer	
ID	Value
1:1	0.0229
2:1	0.0229
3:1	0.0229
4:1	0.0229
5:1	0.0229
5:2	0.0229
6:1	0.0229
7:1	0.0229
8:1	0.0229
1:2	0.0229
8:2	0.0229
N:	11
Mean:	0.0229
SD:	0.0000

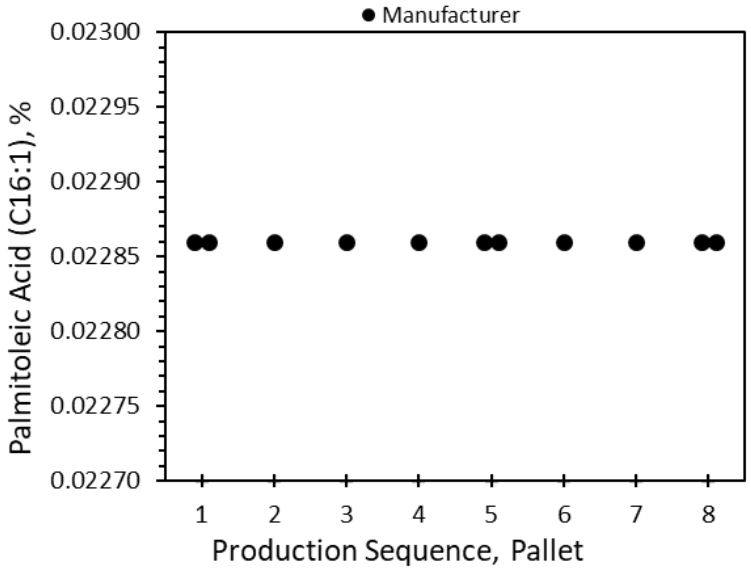

Fig. 64. Palmitoleic acid (C16:1) mass fraction as a function of production sequence

5.5.8. Heptadecanoic Acid (C17:0)

The manufacturer GC-FID [49,50] measurement results for heptadecanoic acid (C17:0) are summarized in Table 76. Fig. 65 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 76. Heptadecanoic acid (C17:0) mass fraction results, %

Manufacturer	
ID	Value
1:1	0.0143
2:1	0.0143
3:1	0.0143
4:1	0.0143
5:1	0.0143
5:2	0.0143
6:1	0.0143
7:1	0.0143
8:1	0.0143
1:2	0.0143
8:2	0.0143
N:	11
Mean:	0.0143
SD:	0.0000

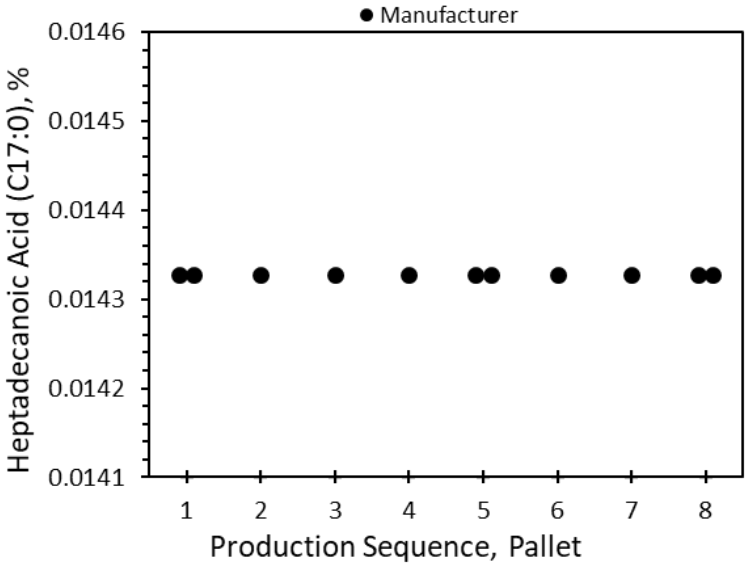

Fig. 65. Heptadecanoic acid (C17:0) mass fraction as a function of production sequence

5.5.9. Stearic Acid (C18:0)

The manufacturer GC-FID [49,50] measurement results for stearic acid (C18:0) are summarized in Table 77. Fig. 66 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 77. Stearic acid (C18:0) mass fraction results, %

Manufacturer	
ID	Value
1:1	0.7859
2:1	0.7812
3:1	0.7792
4:1	0.7850
5:1	0.7821
5:2	0.7812
6:1	0.7802
7:1	0.7812
8:1	0.7812
1:2	0.7831
8:2	0.7773
N:	11
Mean:	0.7816
SD:	0.0024

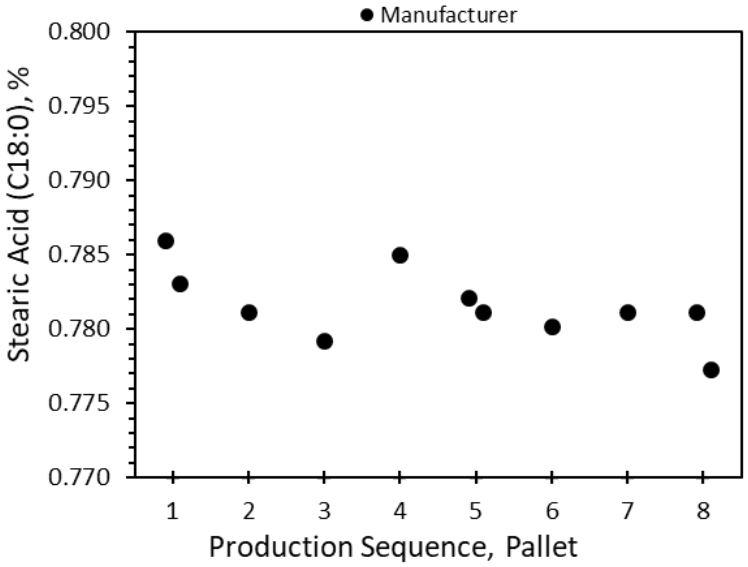

Fig. 66. Stearic acid (C18:0) mass fraction as a function of production sequence

5.5.10. Oleic Acid (C18:1,9c)

The manufacturer GC-FID [49,50] measurement results for oleic acid (C18:1,9c) are summarized in Table 78. Fig. 67 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 78. Oleic acid (C18:1,9c) mass fraction results, %

Manufacturer	
ID	Value
1:1	10.431
2:1	10.336
3:1	10.336
4:1	10.431
5:1	10.431
5:2	10.336
6:1	10.336
7:1	10.336
8:1	10.336
1:2	10.431
8:2	10.336
N:	11
Mean:	10.370
SD:	0.048

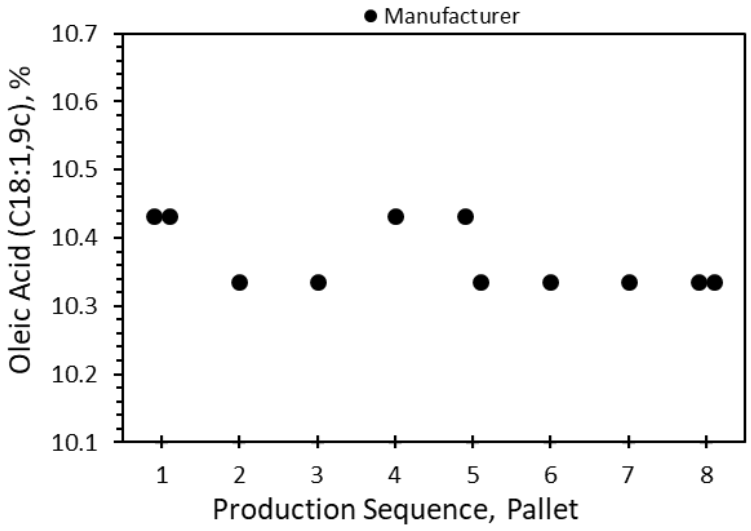

Fig. 67. Oleic acid (C18:1,9c) mass fraction as a function of production sequence

5.5.11. Linoleic Acid (C18:2)

The manufacturer GC-FID [49,50] measurement results for linoleic acid (C18:2) are summarized in Table 79. Fig. 68 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 79. Linoleic acid (C18:2) mass fraction results, %

Manufacturer	
ID	Value
1:1	5.750
2:1	5.721
3:1	5.711
4:1	5.750
5:1	5.731
5:2	5.721
6:1	5.711
7:1	5.721
8:1	5.721
1:2	5.731
8:2	5.692
N:	11
Mean:	5.724
SD:	0.017

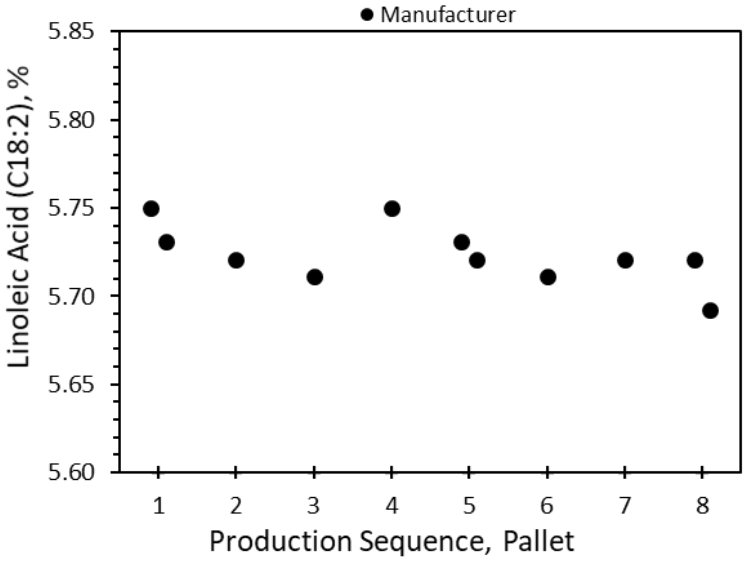

Fig. 68. Linoleic acid (C18:2) mass fraction as a function of production sequence

5.5.12. α-Linolenic Acid (C18:3,9c,12c,15c+9c,12c,15t)

The manufacturer GC-FID [49,50] measurement results for α-linolenic acid (C18:3,9c,12c,15c+9c,12c,15t) are summarized in Table 80. Fig. 69 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 80. α-Linolenic acid (C18:3,9c,12c,15c+9c,12c,15t) mass fraction results, %

Manufacturer	
ID	Value
1:1	0.5576
2:1	0.5538
3:1	0.5538
4:1	0.5566
5:1	0.5557
5:2	0.5547
6:1	0.5528
7:1	0.5538
8:1	0.5538
1:2	0.5566
8:2	0.5595
N:	11
Mean:	0.5553
SD:	0.0021

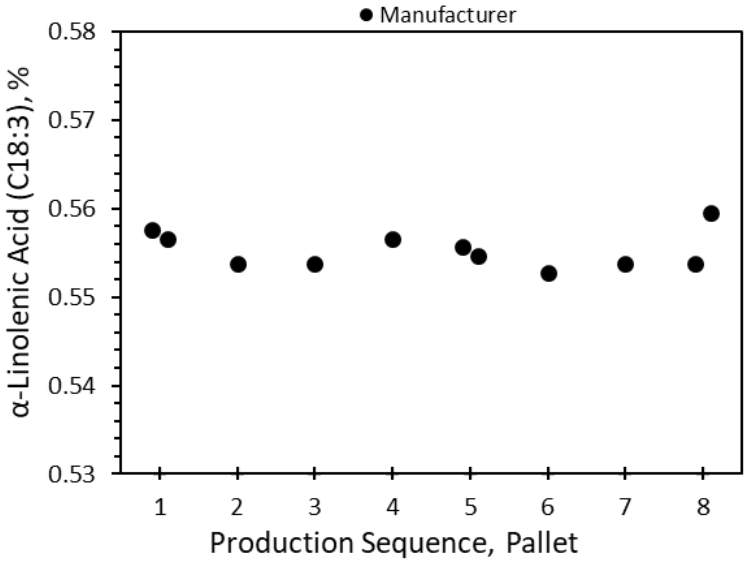

Fig. 69. α-Linolenic acid (C18:3,9c,12c,15c+9c,12c,15t) mass fraction as a function of production sequence

5.5.13. γ-Linolenic Acid (C18:3)

The manufacturer GC-FID [49,50] measurement results for γ-linolenic acid (C18:3) are summarized in Table 81. Fig. 70 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 81. γ-Linolenic acid (C18:3) mass fraction results, %

	Manufacturer
ID	Value
1:1	0.0115
2:1	0.0115
3:1	0.0115
4:1	0.0115
5:1	0.0115
5:2	0.0115
6:1	0.0115
7:1	0.0115
8:1	0.0105
1:2	0.0115
8:2	0.0115
N:	11
Mean:	0.0114
SD:	0.0003

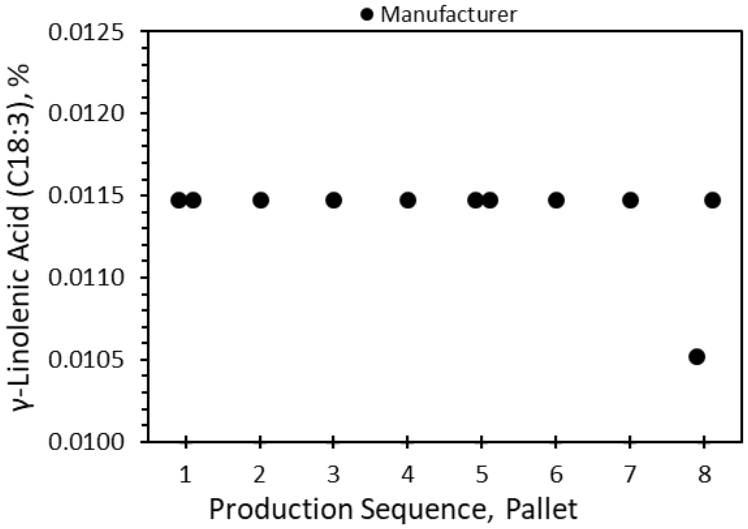

Fig. 70. γ-Linolenic acid (C18:3) mass fraction as a function of production sequence

5.5.14. Arachidic Acid (C20:0)

The manufacturer GC-FID [49,50] measurement results for arachidic acid (C20:0) are summarized in Table 82. Fig. 71 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 82. Arachidic acid (C20:0) mass fraction results, %

Manufacturer	
ID	Value
1:1	0.0778
2:1	0.0769
3:1	0.0769
4:1	0.0769
5:1	0.0769
5:2	0.0769
6:1	0.0769
7:1	0.0769
8:1	0.0769
1:2	0.0769
8:2	0.0759
N:	11
Mean:	0.0769
SD:	0.0004

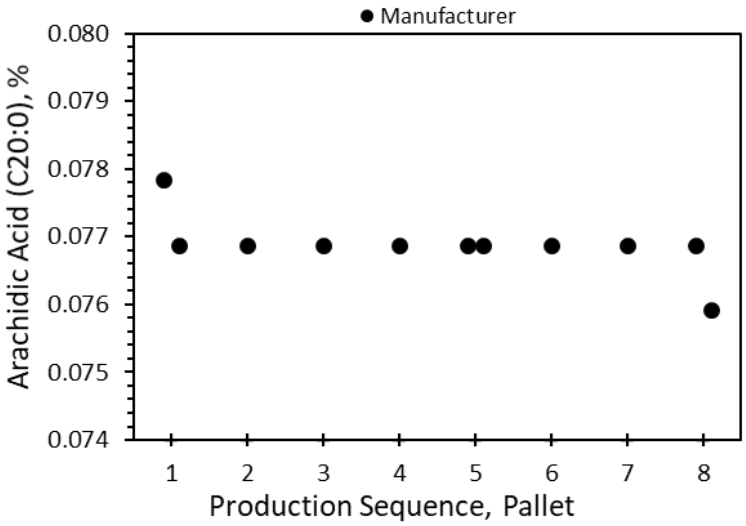

Fig. 71. Arachidic acid (C20:0) mass fraction as a function of production sequence

5.5.15. Eicosenoic Acid (C20:1)

The manufacturer GC-FID [49,50] measurement results for eicosenoic acid (C20:1) are summarized in Table 83. Fig. 72 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 83. Eicosenoic acid (C20:1) mass fraction results, %

Manufacturer	
ID	Value
1:1	0.0749
2:1	0.0740
3:1	0.0740
4:1	0.0749
5:1	0.0740
5:2	0.0740
6:1	0.0740
7:1	0.0740
8:1	0.0740
1:2	0.0749
8:2	0.0740
N:	11
Mean:	0.0742
SD:	0.0004

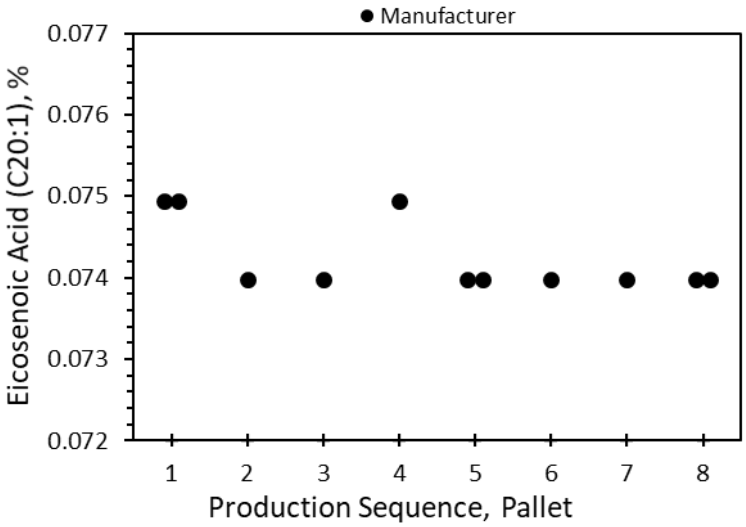

Fig. 72. Eicosenoic acid (C20:1) mass fraction as a function of production sequence

5.5.16. Homo-γ-Linolenic Acid (C20:3,n6)

The manufacturer GC-FID [49,50] measurement results for homo-γ-linolenic acid (C20:3,n6) are summarized in Table 84. Fig. 73 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 84. Homo-γ-linolenic acid (C20:3,n6) mass fraction results, %

Manufacturer	
ID	Value
1:1	0.0144
2:1	0.0144
3:1	0.0144
4:1	0.0154
5:1	0.0154
5:2	0.0154
6:1	0.0144
7:1	0.0144
8:1	0.0144
1:2	0.0144
8:2	0.0144
N:	11
Mean:	0.0147
SD:	0.0004

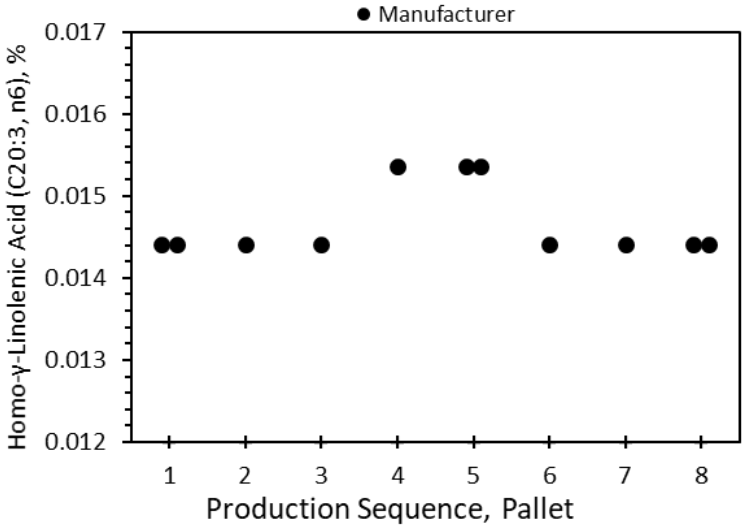

Fig. 73. Homo-γ-linolenic acid (C20:3,n6) mass fraction as a function of production sequence

5.5.17. Arachidonic Acid (C20:4,n6)

The manufacturer GC-FID [49,50] and FNSQAP [19] measurement results for arachidonic acid (C20:4,n6) are summarized in Table 85. Fig. 74 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 85. Arachidonic acid (C20:4,n6) mass fraction results, %

Manufacturer

ID	Value
1:1	0.1632
2:1	0.1632
3:1	0.1632
4:1	0.1642
5:1	0.1632
5:2	0.1632
6:1	0.1622
7:1	0.1632
8:1	0.1632
1:2	0.1632
8:2	0.1622
N:	11
Mean:	0.1631
SD:	0.0005

FNSQAP Exercise 2

Code	Rep_1	Rep_2	Rep_3	Mean	SD	Methods
B002	0.16	0.16		0.16	0	GC-FID
B005	0.16	0.16	0.15	0.157	0.006	GC-FID
B009	0.14	0.14	0.14	0.14	0	GC-FID
B015	0.147	0.178	0.179	0.168	0.018	GC-MS
B019	0.147	0.142	0.144	0.144	0.003	GC-FID
B028	0.19	0.18	0.19	0.187	0.006	GC-MS
B044	0.16	0.16	0.16	0.16	0	GC-FID
			N:	7		
			Mean, Pooled SD:	0.159	0.008	
			SD:	0.015		

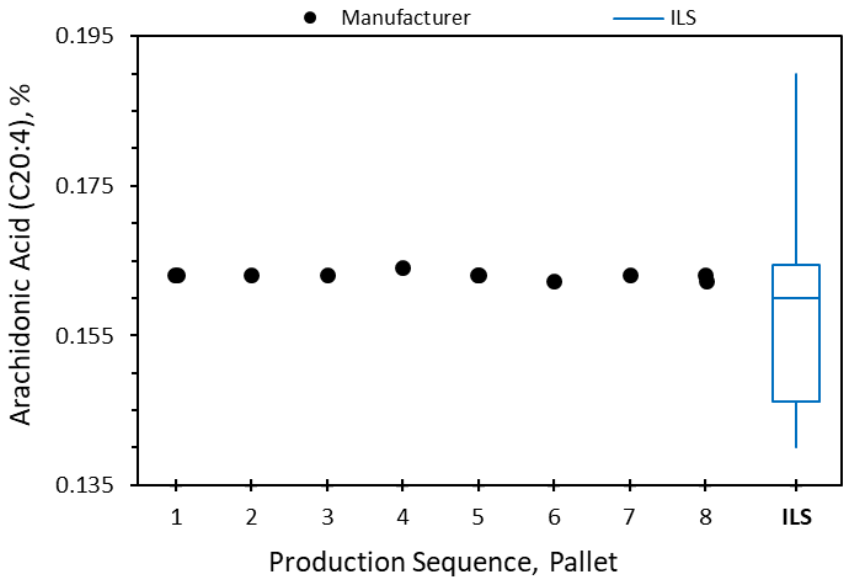

Fig. 74. Arachidonic acid (C20:4,n6) mass fraction as a function of production sequence

5.5.18. Behenic Acid (C22:0)

The manufacturer GC-FID [49,50] measurement results for behenic acid (C22:0) are summarized in Table 86. Fig. 75 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 86. Behenic acid (C22:0) mass fraction results, %

Manufacturer	
ID	Value
1:1	0.0656
2:1	0.0656
3:1	0.0656
4:1	0.0665
5:1	0.0656
5:2	0.0656
6:1	0.0656
7:1	0.0656
8:1	0.0656
1:2	0.0656
8:2	0.0646
N:	11
Mean:	0.0656
SD:	0.0004

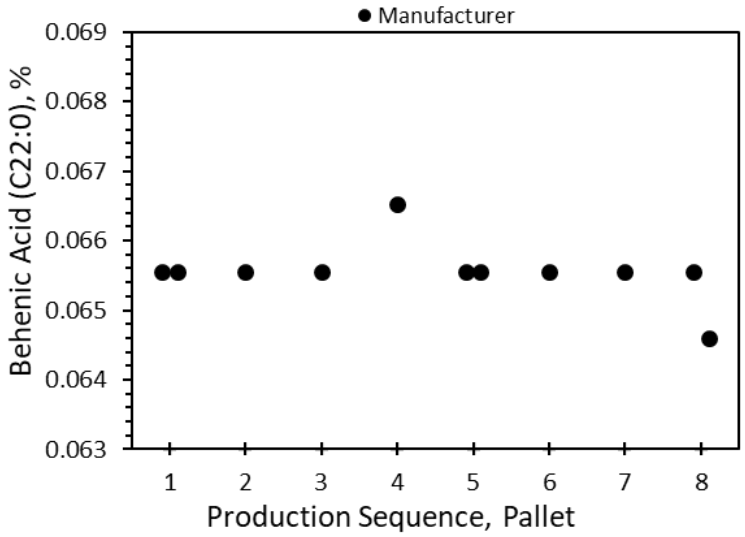

Fig. 75. Behenic acid (C22:0) mass fraction as a function of production sequence

5.5.19. Docosahexaenoic Acid (C22:6, DHA)

The manufacturer GC-FID [49,50] and FNSQAP [19] measurement results for docosahexaenoic acid (C22:6, DHA) are summarized in Table 87. Fig. 76 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 87. Docosahexaenoic acid (C22:6, DHA) mass fraction results, %

Manufacturer

ID	Value
1:1	0.0568
2:1	0.0568
3:1	0.0578
4:1	0.0578
5:1	0.0568
5:2	0.0568
6:1	0.0539
7:1	0.0539
8:1	0.0549
1:2	0.0568
8:2	0.0530
N:	11
Mean:	0.0559
SD:	0.0017

FNSQAP Exercise 2

Code	Rep_1	Rep_2	Rep_3	Mean	SD	Methods
B002	0.05	0.05		0.05	0	GC-FID
B005	0.06	0.06	0.04	0.053	0.012	GC-FID
B009	0.06	0.06	0.05	0.057	0.006	GC-FID
B015	0.043	0.059	0.058	0.053	0.009	GC-MS
B019	0.045	0.042	0.041	0.043	0.002	GC-FID
B028	0.06	0.05	0.06	0.057	0.006	GC-MS
B044	0.05	0.05	0.05	0.05	0	GC-FID
			N:	7		
			Mean, Pooled SD:	0.052	0.006	
			SD:	0.005		

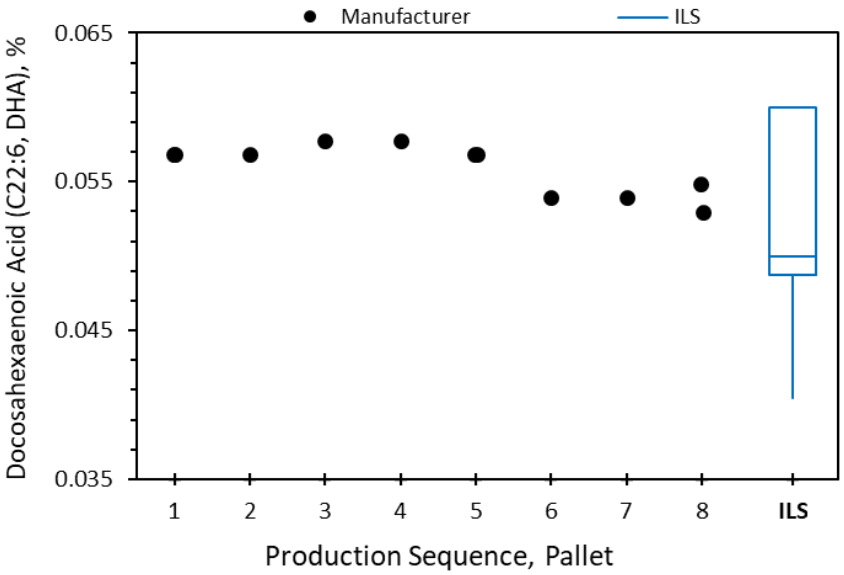

Fig. 76. Docosahexaenoic acid (C22:6, DHA) mass fraction as a function of production sequence

5.5.20. Lignoceric Acid (C24:0)

The manufacturer GC-FID [49,50] measurement results for lignoceric acid (C24:0) are summarized in Table 88. Fig. 77 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 88. Lignoceric acid (C24:0) mass fraction results, %

Manufacturer	
ID	Value
1:1	0.0367
2:1	0.0367
3:1	0.0367
4:1	0.0377
5:1	0.0367
5:2	0.0377
6:1	0.0367
7:1	0.0367
8:1	0.0367
1:2	0.0367
8:2	0.0367
N:	11
Mean:	0.0369
SD:	0.0004

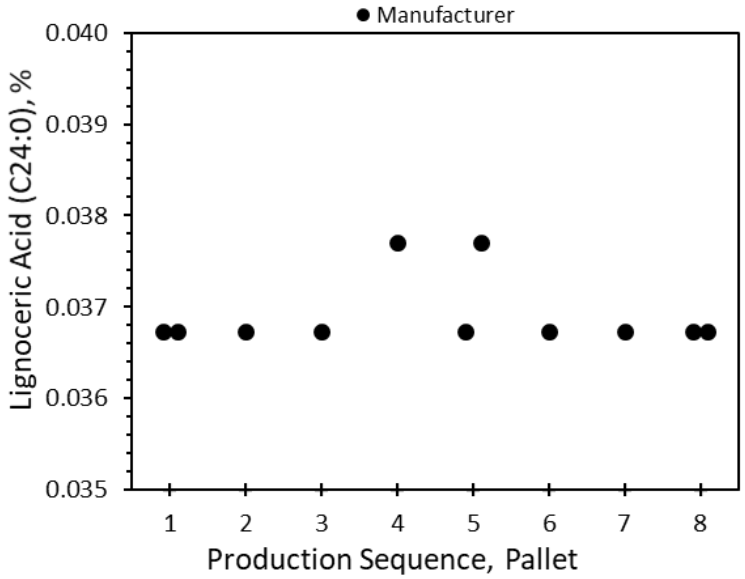

Fig. 77. Lignoceric acid (C24:0) mass fraction as a function of production sequence

5.5.21. Nervonic Acid (C24:1)

The manufacturer GC-FID [49,50] measurement results for nervonic acid (C24:1) are summarized in Table 89. Fig. 78 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 89. Nervonic acid (C24:1) mass fraction results, %

Manufacturer	
ID	Value
1:1	0.0203
2:1	0.0203
3:1	0.0203
4:1	0.0203
5:1	0.0203
5:2	0.0203
6:1	0.0203
7:1	0.0203
8:1	0.0203
1:2	0.0203
8:2	0.0203
N:	11
Mean:	0.0203
SD:	0.0000

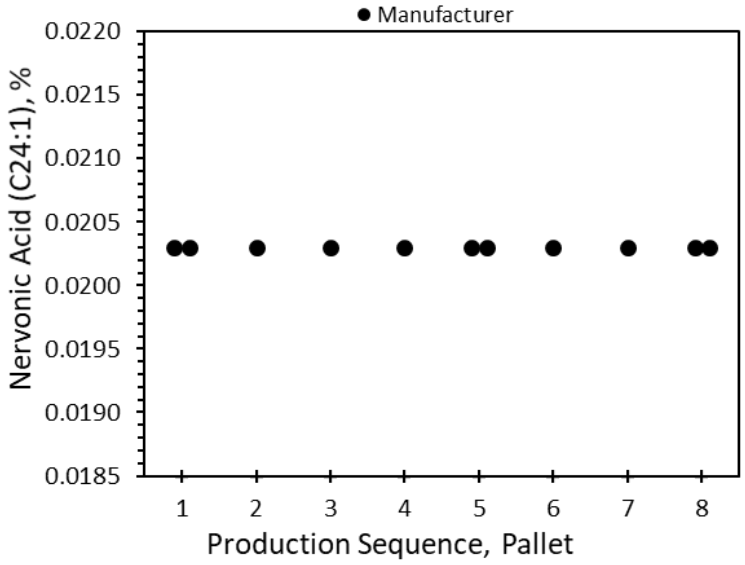

Fig. 78. Nervonic acid (C24:1) mass fraction as a function of production sequence

5.5.22. Monounsaturated Fatty Acids

The manufacturer GC-FID [49,50] measurement results for monounsaturated fatty acids are summarized in Table 90. Fig. 79 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 90. Monounsaturated fatty acids mass fraction results, %

Manufacturer	
ID	Value
1:1	10.7
2:1	10.7
3:1	10.7
4:1	10.8
5:1	10.7
5:2	10.7
6:1	10.7
7:1	10.7
8:1	10.7
1:2	10.7
8:2	10.6
N:	11
Mean:	10.70
SD:	0.04

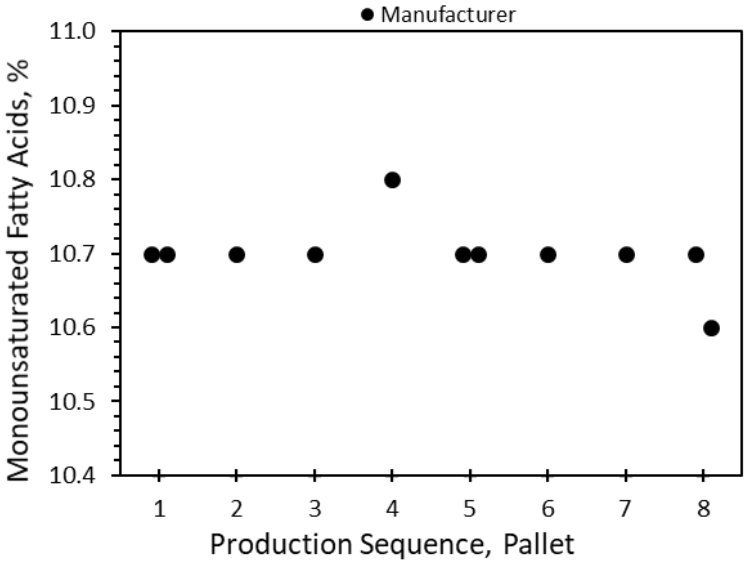

Fig. 79. Monounsaturated fatty acids mass fraction as a function of production sequence

5.5.23. Polyunsaturated Fatty Acids

The manufacturer GC-FID [49,50] measurement results for polyunsaturated fatty acids are summarized in Table 91. Fig. 80 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 91. Polyunsaturated fatty acids mass fraction results, %

	Manufacturer
ID	Value
1:1	6.53
2:1	6.50
3:1	6.49
4:1	6.53
5:1	6.51
5:2	6.50
6:1	6.48
7:1	6.49
8:1	6.50
1:2	6.51
8:2	6.47
N:	11
Mean:	6.501
SD:	0.019

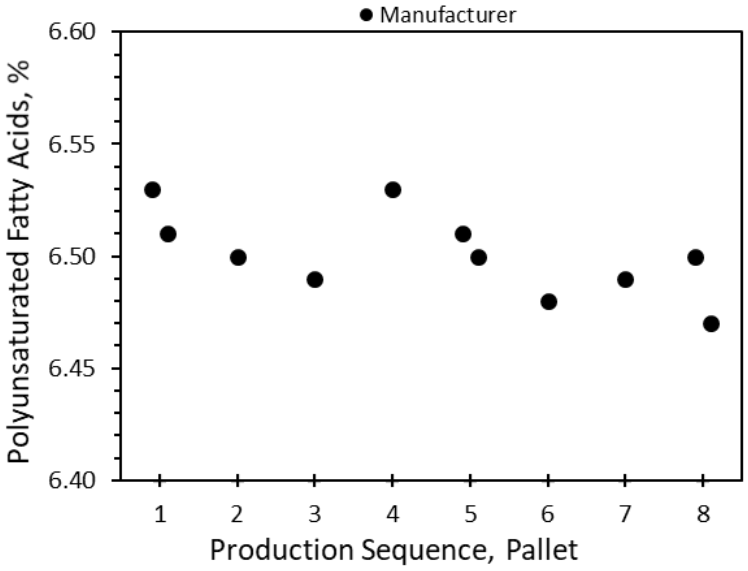

Fig. 80. Polyunsaturated fatty acids mass fraction as a function of production sequence

5.5.24. Saturated Fatty Acids

The manufacturer GC-FID [49,50] measurement results for saturated fatty acids are summarized in Table 92. Fig. 81 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 92. Saturated fatty acids mass fraction results, %

Manufacturer	
ID	Value
1:1	8.67
2:1	8.66
3:1	8.66
4:1	8.69
5:1	8.65
5:2	8.65
6:1	8.64
7:1	8.64
8:1	8.66
1:2	8.64
8:2	8.64
N:	11
Mean:	8.655
SD:	0.016

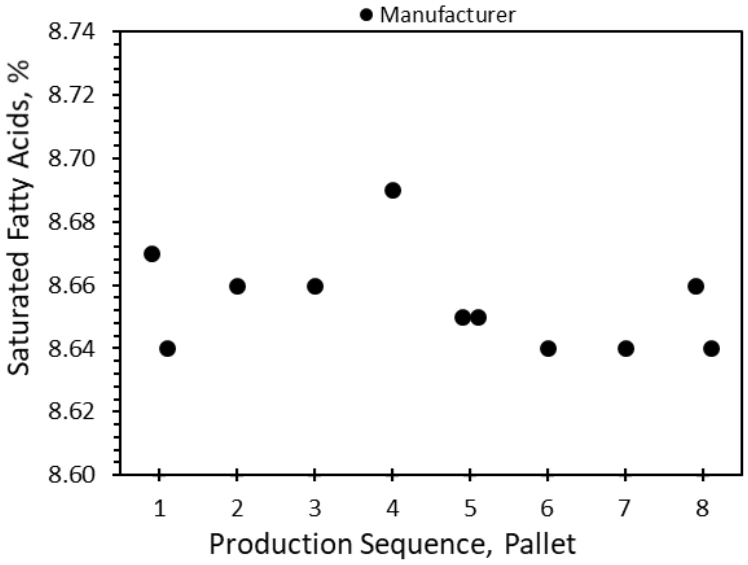

Fig. 81. Saturated fatty acids mass fraction as a function of production sequence

5.5.25. Total *trans*-C18:1 and -C18:2 Fatty Acids

The manufacturer GC-FID [49,50] measurement results for total *trans*-C18:1 and -C18:2 fatty acids are summarized in Table 93. Fig. 82 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 93. Total *trans*-C18:1 and -C18:2 fatty acids mass fraction results, %

Manufacturer	
ID	Value
1:1	0.070
2:1	0.070
3:1	0.069
4:1	0.069
5:1	0.070
5:2	0.069
6:1	0.069
7:1	0.069
8:1	0.069
1:2	0.069
8:2	0.069
N:	11
Mean:	0.0693
SD:	0.0005

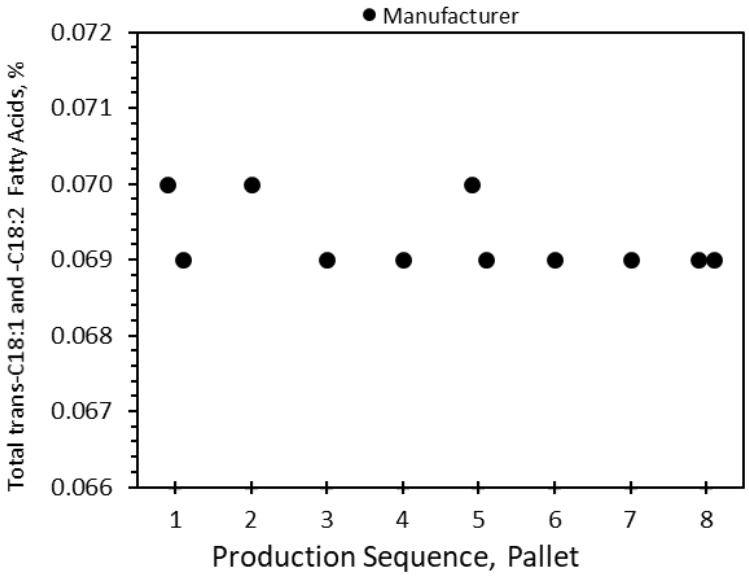

Fig. 82. Total *trans*-C18:1 and -C18:2 fatty acids mass fraction as a function of production sequence

5.5.26. Total Fatty Acids

The manufacturer GC-FID [49,50] measurement results for total fatty acids are summarized in Table 94. Fig. 83 displays these results as an approximate function of the production sequence, as determined by the pallet and box number. In addition to the fatty acids summarized in previous sections, the following fatty acids were assayed but not detected at a level of 0.01 g/100g: myristoleic acid (C14:1), pentadecanoic acid (C15:0), pentadecenoic acid (C15:1), heptadecenoic acid (C17:1), octadecatetraenoic acid (C18:4), eicosadienoic acid (C20:2), eicosatrienoic acid (C20:3,n3), arachidonic acid (C20:4,n3), eicosapentaenoic acid (C20:5), heneicosapentaenoic acid (C21:5), erucic acid (C22:1), docosadienoic acid (C22:2), docosatrienoic acid (C22:3), docosatetraenoic acid (C22:4), docosapentaenoic acid (C22:5,n3), and docosapentaenoic acid (C22:5,n6).

Table 94. Total fatty acids mass fraction results, %

Manufacturer	
ID	Value
1:1	27.3
2:1	27.2
3:1	27.2
4:1	27.4
5:1	27.3
5:2	27.2
6:1	27.2
7:1	27.2
8:1	27.3
1:2	27.3
8:2	27.1
N:	11
Mean:	27.25
SD:	0.08

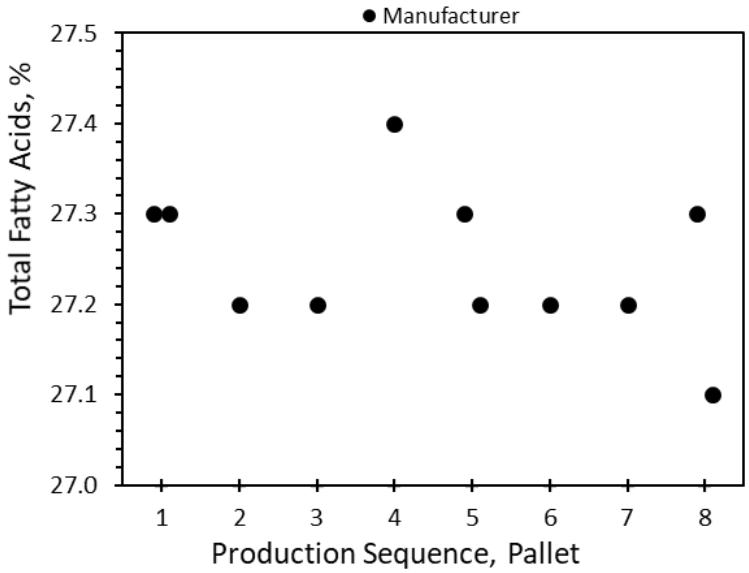

Fig. 83. Total fatty acids mass fraction as a function of production sequence

Amino Acids

5.6.1. Alanine

The manufacturer LC-Abs [54-57] measurement results for alanine are summarized in Table 95. Fig. 84 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 95. Alanine mass fraction results, g/100 g

Manufacturer	
ID	Value
1:1	0.468
2:1	0.472
3:1	0.468
4:1	0.473
5:1	0.478
5:2	0.472
6:1	0.467
7:1	0.459
8:1	0.468
1:2	0.479
8:2	0.467
N:	11
Mean:	0.470
SD:	0.006

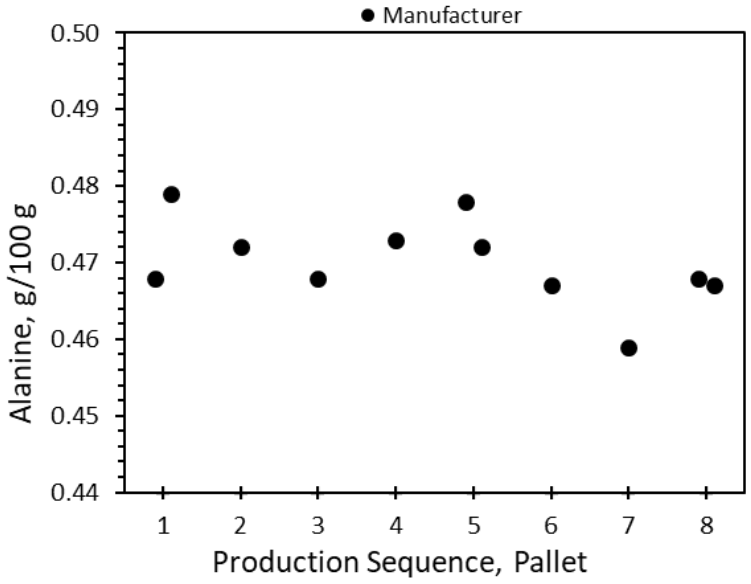

Fig. 84. Alanine mass fraction as a function of production sequence

5.6.2. Arginine

The manufacturer LC-Abs [54-57] measurement results for arginine are summarized in Table 96. Fig. 85 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 96. Arginine mass fraction results, g/100 g

Manufacturer	
ID	Value
1:1	0.440
2:1	0.420
3:1	0.420
4:1	0.426
5:1	0.434
5:2	0.421
6:1	0.423
7:1	0.419
8:1	0.418
1:2	0.466
8:2	0.416
N:	11
Mean:	0.428
SD:	0.015

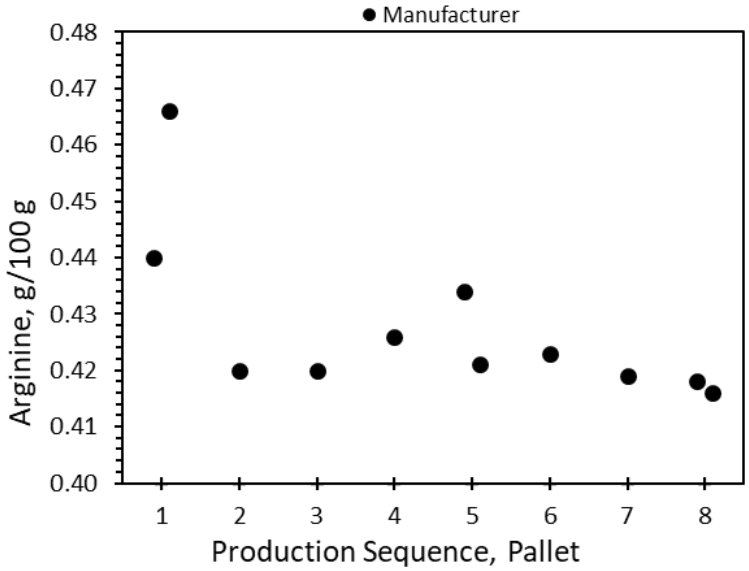

Fig. 85. Arginine mass fraction as a function of production sequence

5.6.3. Aspartic Acid

The manufacturer LC-Abs [54-57] measurement results for aspartic acid are summarized in Table 97. Fig. 86 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 97. Aspartic acid mass fraction results, g/100 g

Manufacturer	
ID	Value
1:1	1.060
2:1	1.050
3:1	1.050
4:1	1.050
5:1	1.070
5:2	1.050
6:1	1.040
7:1	1.050
8:1	1.050
1:2	1.070
8:2	1.050
N:	11
Mean:	1.054
SD:	0.009

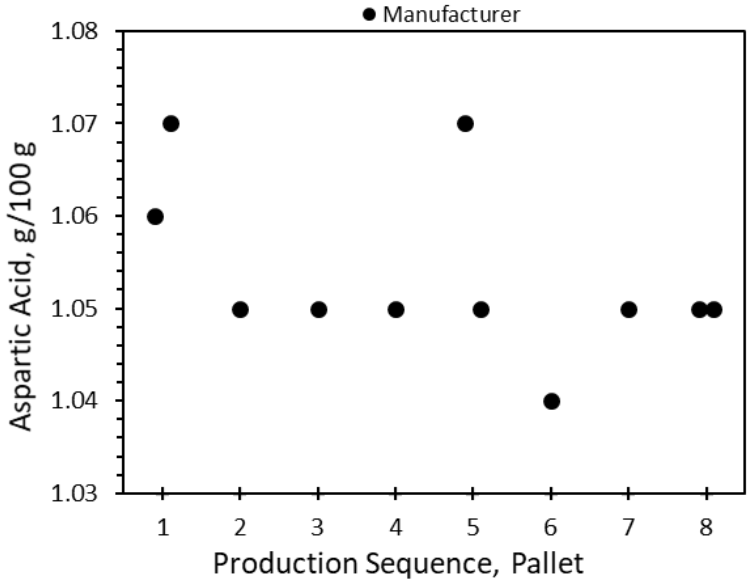

Fig. 86. Aspartic acid mass fraction as a function of production sequence

5.6.4. Cystine

The manufacturer LC-Abs [54-57] measurement results for cystine are summarized in Table 98. Fig. 87 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 98. Cystine mass fraction results, g/100 g

Manufacturer	
ID	Value
1:1	0.115
2:1	0.123
3:1	0.124
4:1	0.128
5:1	0.119
5:2	0.123
6:1	0.125
7:1	0.126
8:1	0.126
1:2	0.201
8:2	0.122
N:	11
Mean:	0.130
SD:	0.024

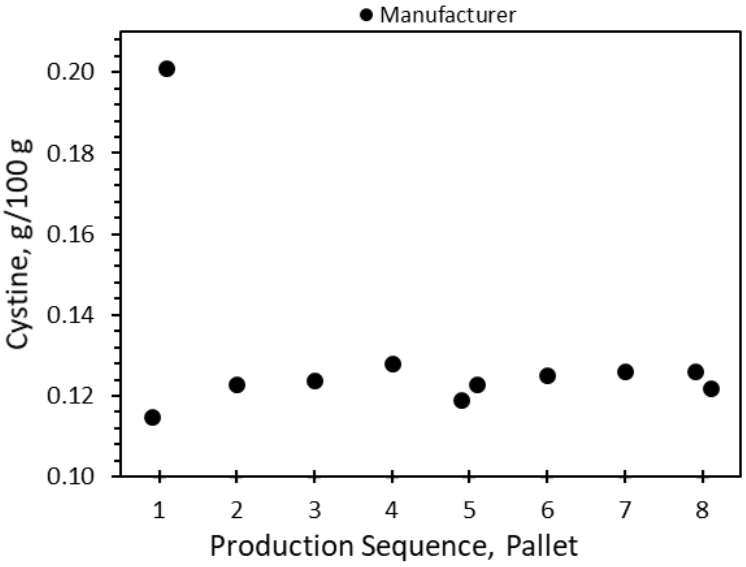

Fig. 87. Cystine mass fraction as a function of production sequence

5.6.5. Glutamic Acid

The manufacturer LC-Abs [54-57] measurement results for glutamic acid are summarized in Table 99. Fig. 88 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 99. Glutamic acid mass fraction results, g/100 g

Manufacturer	
ID	Value
1:1	2.490
2:1	2.490
3:1	2.490
4:1	2.500
5:1	2.540
5:2	2.470
6:1	2.480
7:1	2.490
8:1	2.480
1:2	2.630
8:2	2.470
N:	11
Mean:	2.503
SD:	0.046

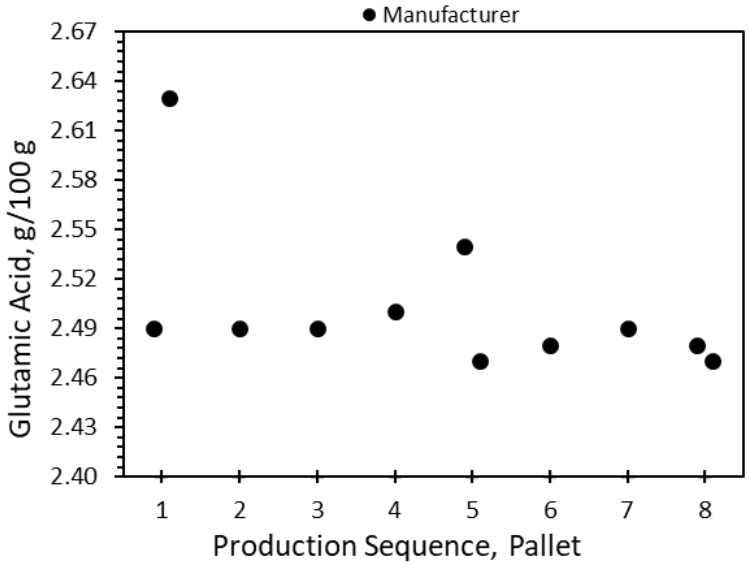

Fig. 88. Glutamic acid mass fraction as a function of production sequence

5.6.6. Glycine

The manufacturer LC-Abs [54-57] measurement results for glycine are summarized in Table 100. Fig. 89 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 100. Glycine mass fraction results, g/100 g

Manufacturer	
ID	Value
1:1	0.247
2:1	0.248
3:1	0.248
4:1	0.248
5:1	0.246
5:2	0.244
6:1	0.245
7:1	0.246
8:1	0.246
1:2	0.245
8:2	0.249
N:	11
Mean:	0.247
SD:	0.002

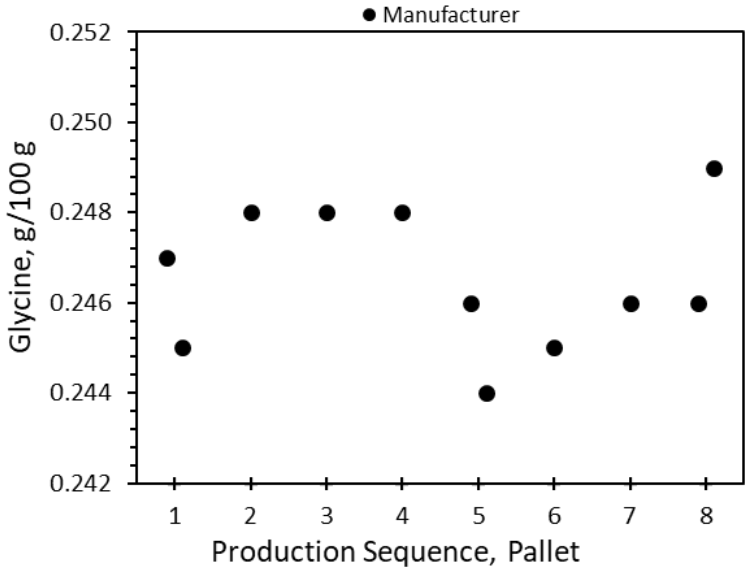

Fig. 89. Glycine mass fraction as a function of production sequence

5.6.7. Histidine

The manufacturer LC-Abs [54-57] measurement results for histidine are summarized in Table 101. Fig. 90 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 101. Histidine mass fraction results, g/100 g

Manufacturer	
ID	Value
1:1	0.307
2:1	0.307
3:1	0.301
4:1	0.304
5:1	0.311
5:2	0.277
6:1	0.292
7:1	0.289
8:1	0.299
1:2	0.305
8:2	0.274
N:	11
Mean:	0.297
SD:	0.012

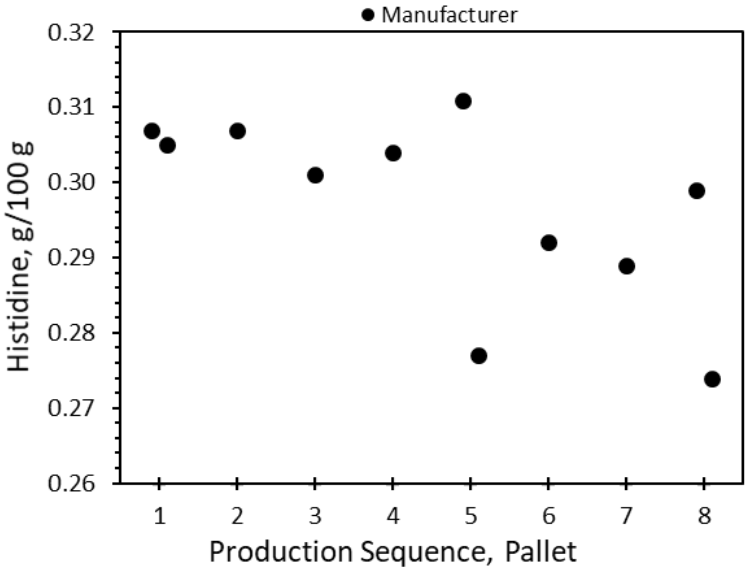

Fig. 90. Histidine mass fraction as a function of production sequence

5.6.8. Isoleucine

The manufacturer LC-Abs [54-57] measurement results for isoleucine are summarized in Table 102. Fig. 91 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 102. Isoleucine mass fraction results, g/100 g

Manufacturer	
ID	Value
1:1	0.701
2:1	0.686
3:1	0.664
4:1	0.692
5:1	0.713
5:2	0.682
6:1	0.679
7:1	0.689
8:1	0.689
1:2	0.699
8:2	0.680
N:	11
Mean:	0.689
SD:	0.013

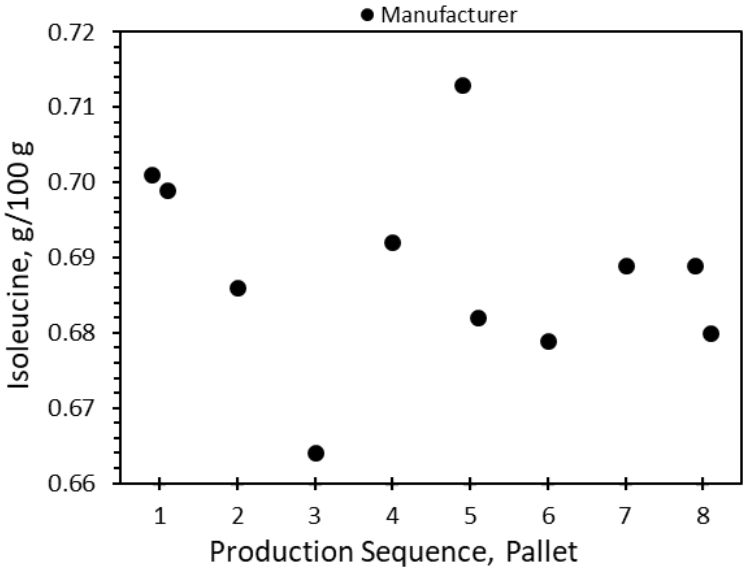

Fig. 91. Isoleucine mass fraction as a function of production sequence

5.6.9. Leucine

The manufacturer LC-Abs [54-57] measurement results for leucine are summarized in Table 103. Fig. 92 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 103. Leucine mass fraction results, g/100 g

Manufacturer	
ID	Value
1:1	1.280
2:1	1.280
3:1	1.270
4:1	1.270
5:1	1.300
5:2	1.270
6:1	1.260
7:1	1.280
8:1	1.280
1:2	1.280
8:2	1.270
N:	11
Mean:	1.276
SD:	0.010

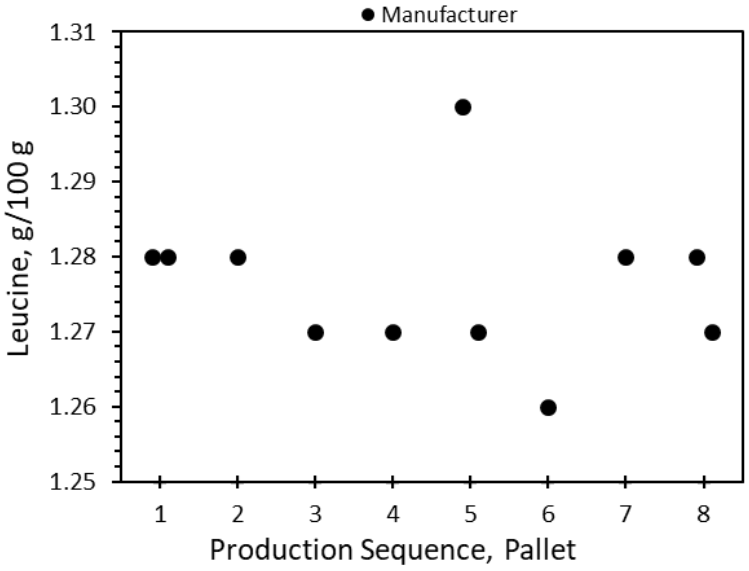

Fig. 92. Leucine mass fraction as a function of production sequence

5.6.10. Lysine

The manufacturer LC-Abs [54-57] measurement results for lysine are summarized in Table 104. Fig. 93 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 104. Lysine mass fraction results, g/100 g

Manufacturer	
ID	Value
1:1	1.010
2:1	0.919
3:1	0.908
4:1	0.942
5:1	1.030
5:2	0.847
6:1	0.869
7:1	0.868
8:1	0.903
1:2	0.947
8:2	0.870
N:	11
Mean:	0.919
SD:	0.059

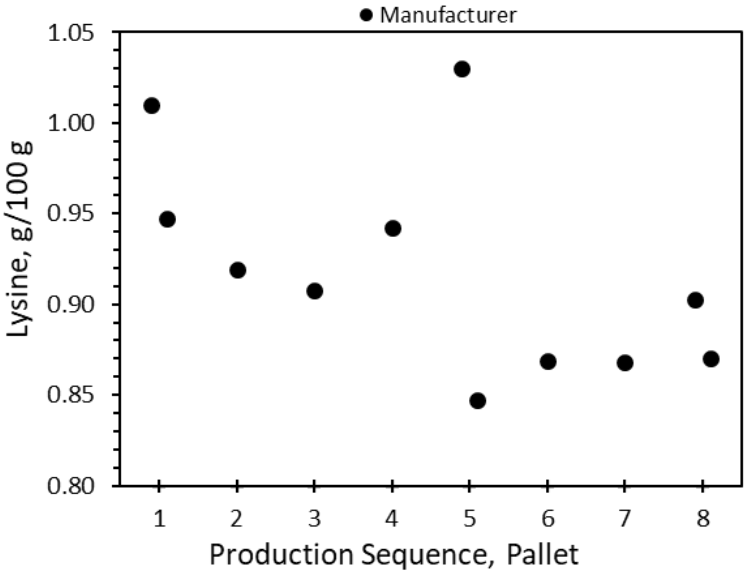

Fig. 93. Lysine mass fraction as a function of production sequence

5.6.11. Methionine (Free)

The manufacturer LC-Abs [54,55] measurement results for methionine (free) are summarized in Table 105. Fig. 94 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 105. Methionine (free) mass fraction results, g/100 g

Manufacturer	
ID	Value
1:1	0.109
2:1	0.110
3:1	0.109
4:1	0.110
5:1	0.111
5:2	0.110
6:1	0.108
7:1	0.114
8:1	0.109
1:2	0.110
8:2	0.110
N:	11
Mean:	0.110
SD:	0.002

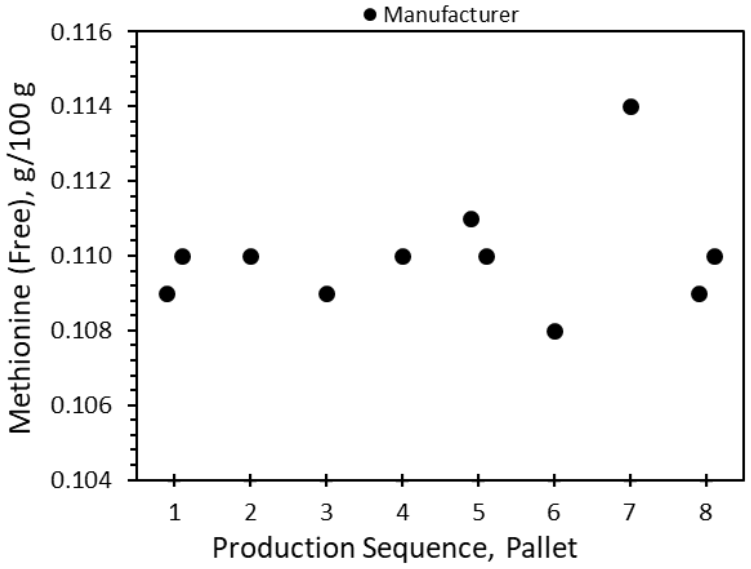

Fig. 94. Methionine (free) mass fraction as a function of production sequence

5.6.12. Methionine (Total)

The manufacturer LC-Abs [54-57] measurement results for methionine (total) are summarized in Table 106. Fig. 95 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 106. Methionine (total) mass fraction results, g/100 g

Manufacturer	
ID	Value
1:1	0.456
2:1	0.454
3:1	0.454
4:1	0.453
5:1	0.452
5:2	0.451
6:1	0.449
7:1	0.451
8:1	0.443
1:2	0.468
8:2	0.444
N:	11
Mean:	0.452
SD:	0.007

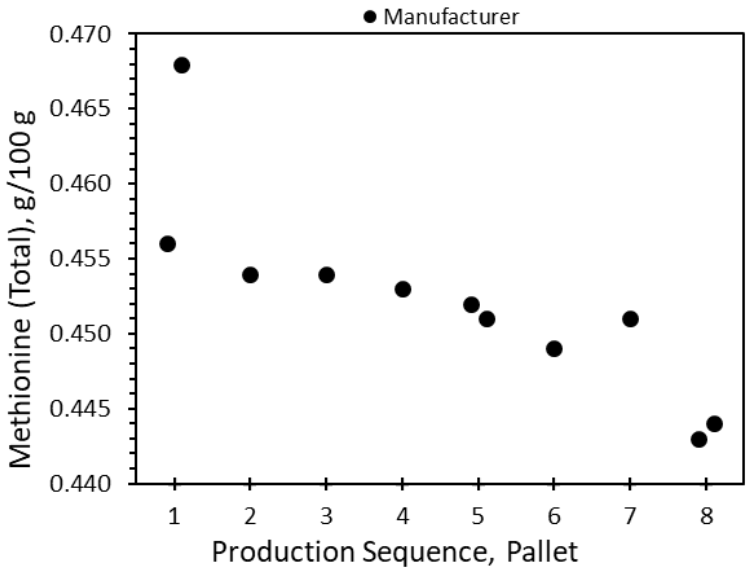

Fig. 95. Methionine (total) mass fraction as a function of production sequence

5.6.13. Phenylalanine

The manufacturer LC-Abs [54-57] measurement results for phenylalanine are summarized in Table 107. Fig. 96 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 107. Phenylalanine mass fraction results, g/100 g

Manufacturer	
ID	Value
1:1	0.582
2:1	0.585
3:1	0.580
4:1	0.570
5:1	0.570
5:2	0.578
6:1	0.571
7:1	0.573
8:1	0.572
1:2	0.580
8:2	0.575
N:	11
Mean:	0.576
SD:	0.005

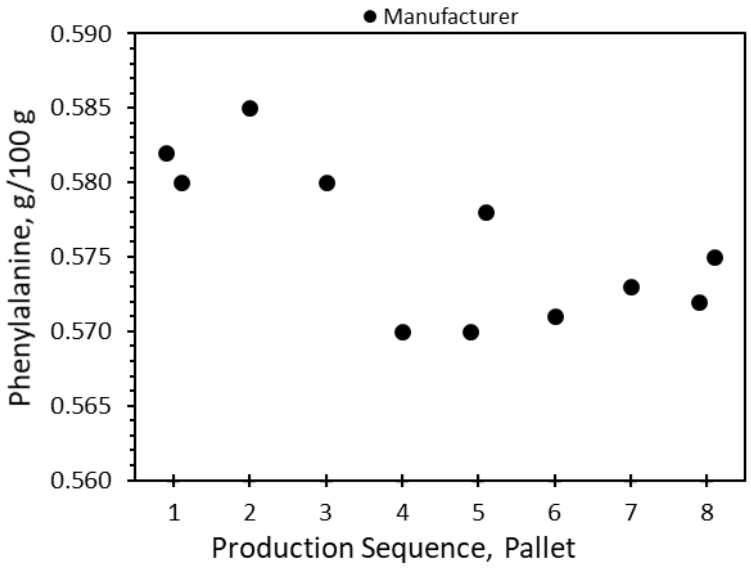

Fig. 96. Phenylalanine mass fraction as a function of production sequence

5.6.14. Proline

The manufacturer LC-Abs [54-57] measurement results for proline are summarized in Table 108. Fig. 97 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 108. Proline mass fraction results, g/100 g

Manufacturer	
ID	Value
1:1	1.190
2:1	1.180
3:1	1.190
4:1	1.190
5:1	1.210
5:2	1.170
6:1	1.190
7:1	1.190
8:1	1.180
1:2	1.230
8:2	1.180
N:	11
Mean:	1.191
SD:	0.016

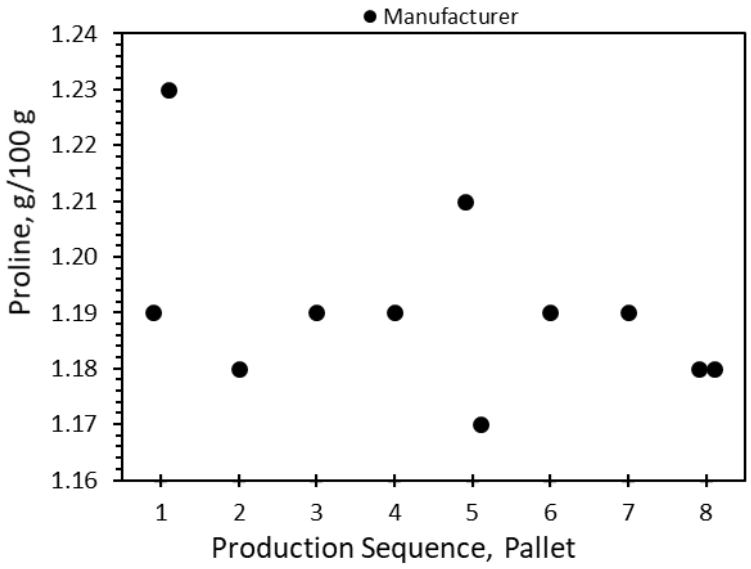

Fig. 97. Proline mass fraction as a function of production sequence

5.6.15. Serine

The manufacturer LC-Abs [54-57] measurement results for serine are summarized in Table 109. Fig. 98 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 109. Serine mass fraction results, g/100 g

Manufacturer	
ID	Value
1:1	0.649
2:1	0.651
3:1	0.659
4:1	0.658
5:1	0.668
5:2	0.648
6:1	0.646
7:1	0.651
8:1	0.653
1:2	0.709
8:2	0.646
N:	11
Mean:	0.658
SD:	0.018

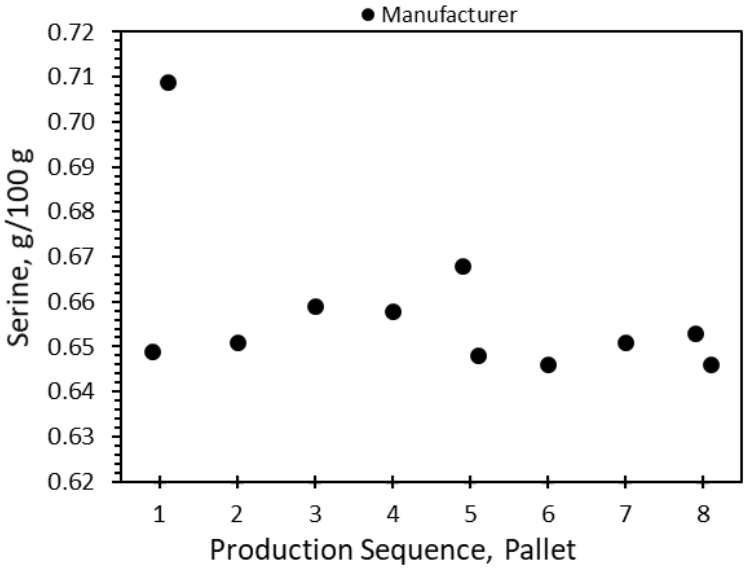

Fig. 98. Serine mass fraction as a function of production sequence

5.6.16. Taurine

The manufacturer LC-Abs [54,55,57,58] measurement results for taurine are summarized in Table 110. Fig. 99 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 110. Taurine mass fraction results, g/100 g

Manufacturer	
ID	Value
1:1	0.0354
2:1	0.0358
3:1	0.0360
4:1	0.0355
5:1	0.0368
5:2	0.0358
6:1	0.0355
7:1	0.0362
8:1	0.0364
1:2	0.0357
8:2	0.0365
N:	11
Mean:	0.0360
SD:	0.0005

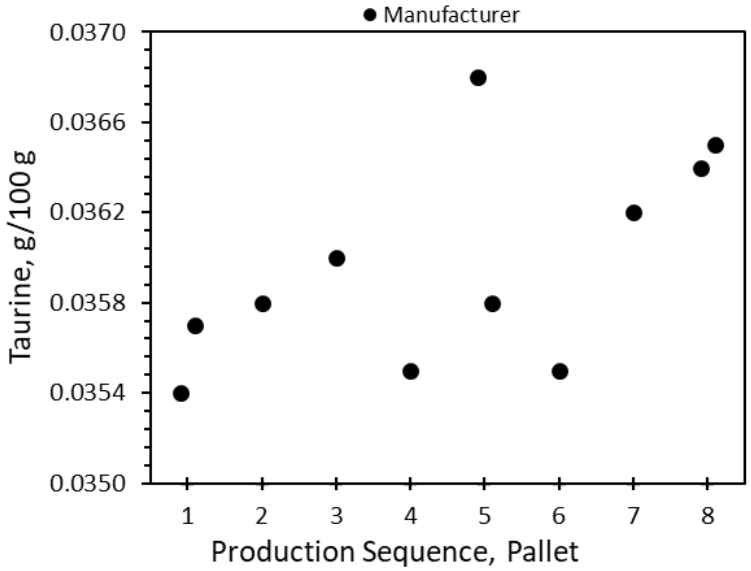

Fig. 99. Taurine mass fraction as a function of production sequence

5.6.17. Threonine

The manufacturer LC-Abs [54-57] measurement results for threonine are summarized in Table 111. Fig. 98 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 111. Threonine mass fraction results, g/100 g

Manufacturer	
ID	Value
1:1	0.634
2:1	0.635
3:1	0.641
4:1	0.638
5:1	0.640
5:2	0.625
6:1	0.629
7:1	0.632
8:1	0.631
1:2	0.639
8:2	0.628
N:	11
Mean:	0.634
SD:	0.005

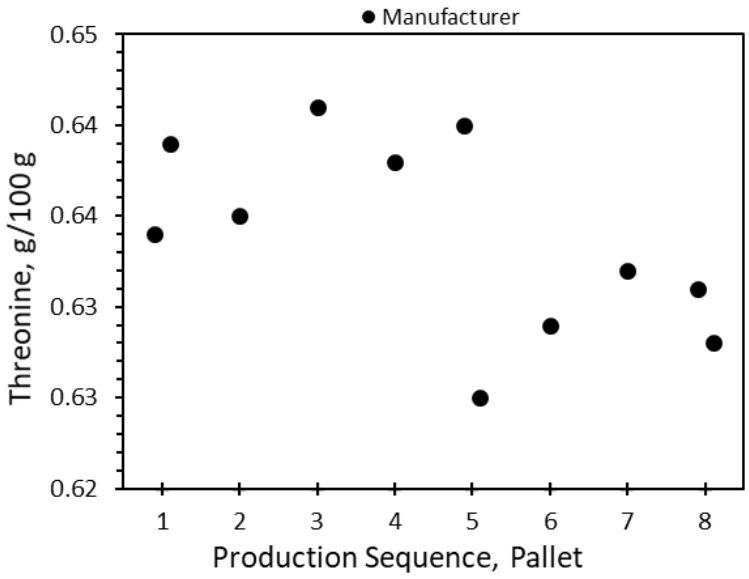

Fig. 100. Threonine mass fraction as a function of production sequence

5.6.18. Tryptophan

The manufacturer LC-Abs [54,55,57,59] measurement results for tryptophan are summarized in Table 112. Fig. 101 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 112. Tryptophan mass fraction results, g/100 g

Manufacturer	
ID	Value
1:1	0.166
2:1	0.179
3:1	0.180
4:1	0.184
5:1	0.171
5:2	0.181
6:1	0.183
7:1	0.174
8:1	0.174
1:2	0.179
8:2	0.180
N:	11
Mean:	0.177
SD:	0.005

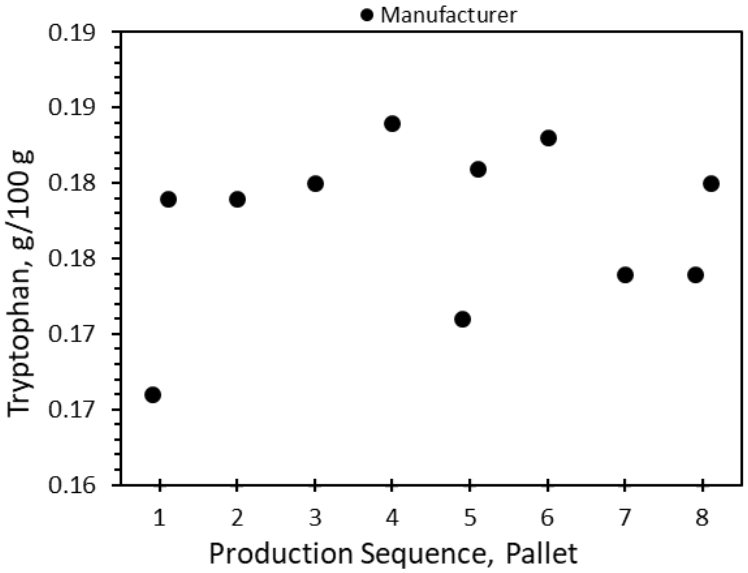

Fig. 101. Tryptophan mass fraction as a function of production sequence

5.6.19. Tyrosine

The manufacturer LC-Abs [54-57] measurement results for tyrosine are summarized in Table 113. Fig. 102 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 113. Tyrosine mass fraction results, g/100 g

Manufacturer	
ID	Value
1:1	0.581
2:1	0.585
3:1	0.585
4:1	0.584
5:1	0.585
5:2	0.582
6:1	0.577
7:1	0.584
8:1	0.582
1:2	0.605
8:2	0.579
N:	11
Mean:	0.584
SD:	0.007

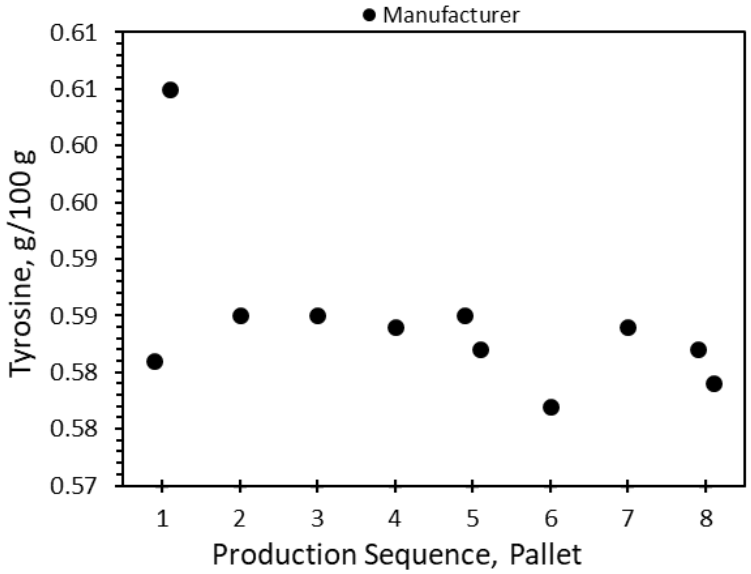

Fig. 102. Tyrosine mass fraction as a function of production sequence

5.6.20. Valine

The manufacturer LC-Abs [54-57] measurement results for valine are summarized in Table 114. Fig. 103 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 114. Valine mass fraction results, g/100 g

Manufacturer	
ID	Value
1:1	0.748
2:1	0.746
3:1	0.727
4:1	0.742
5:1	0.752
5:2	0.743
6:1	0.737
7:1	0.740
8:1	0.746
1:2	0.772
8:2	0.741
N:	11
Mean:	0.745
SD:	0.011

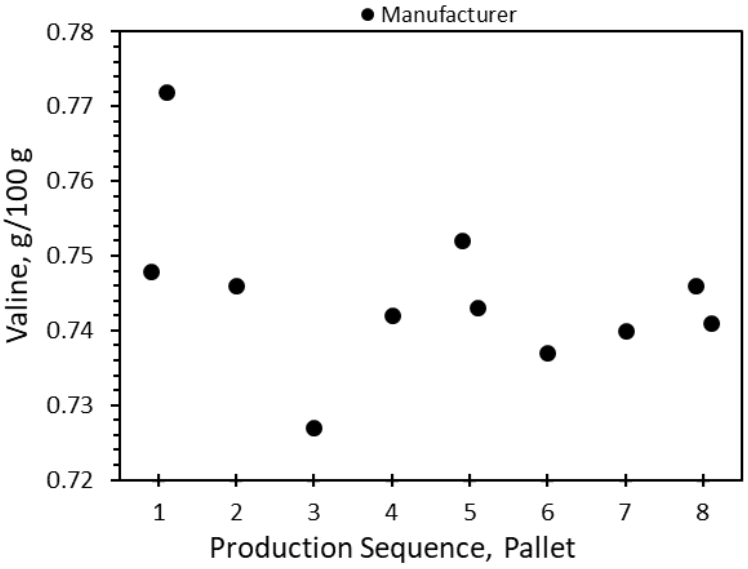

Fig. 103. Valine mass fraction as a function of production sequence

5.7. Nucleotide Equivalents

5.7.1. Adenosine Monophosphate (*AMP*)

The manufacturer LC-Abs measurement results for adenosine monophosphate (AMP) are summarized in Table 115. Fig. 104 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 115. Adenosine monophosphate (*AMP*) mass fraction results, mg/kg

Manufacturer	
ID	Value
1:1	128
2:1	128
3:1	131
4:1	131
5:1	127
5:2	131
6:1	131
7:1	127
8:1	128
1:2	127
8:2	128
N:	11
Mean:	128.8
SD:	1.8

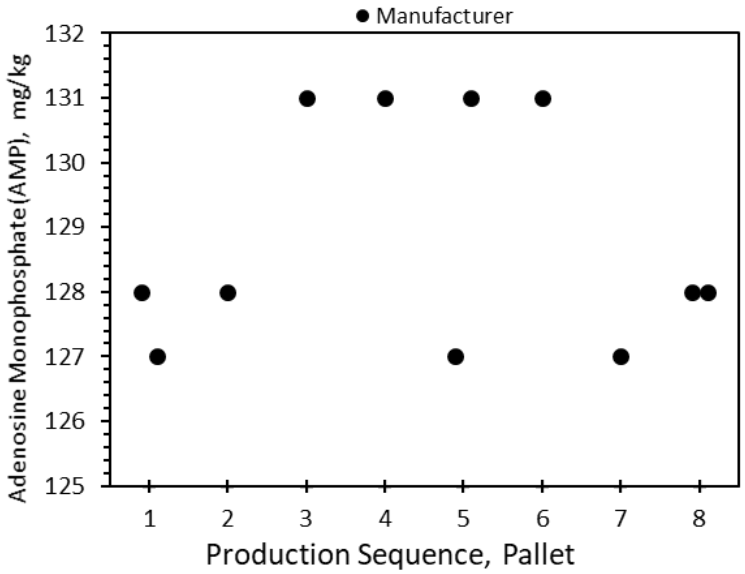

Fig. 104. Adenosine monophosphate (*AMP*) mass fraction as a function of production sequence

5.7.2. Cytidine Monophosphate (*CMP*)

The manufacturer LC-Abs measurement results for cytidine monophosphate (CMP) are summarized in Table 116. Fig. 105 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 116. Cytidine monophosphate (*CMP*) mass fraction results, mg/kg

Manufacturer	
ID	Value
1:1	352
2:1	353
3:1	351
4:1	352
5:1	351
5:2	349
6:1	350
7:1	348
8:1	353
1:2	350
8:2	351
N:	11
Mean:	350.9
SD:	1.6

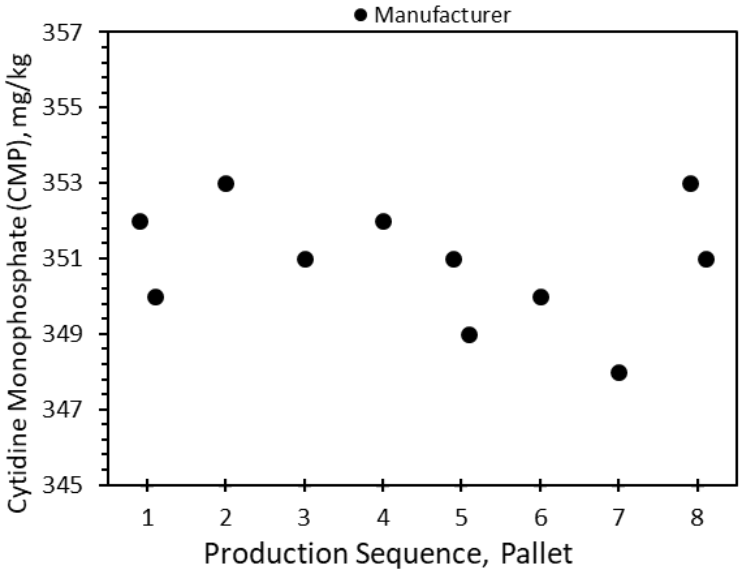

Fig. 105. Cytidine monophosphate (*CMP*) mass fraction as a function of production sequence

5.7.3. Guanosine Monophosphate (*GMP*)

The manufacturer LC-Abs measurement results for guanosine monophosphate (GMP) are summarized in Table 117. Fig. 106 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 117. Guanosine monophosphate (*GMP*) mass fraction results, mg/kg

Manufacturer	
ID	Value
1:1	191
2:1	191
3:1	190
4:1	190
5:1	192
5:2	191
6:1	190
7:1	190
8:1	190
1:2	189
8:2	190
N:	11
Mean:	190.36
SD:	0.81

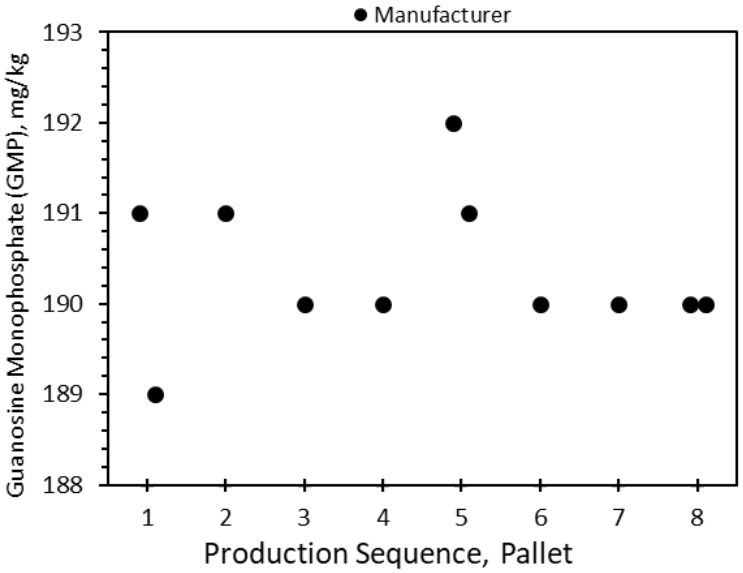

Fig. 106. Guanosine monophosphate (*GMP*) mass fraction as a function of production sequence

5.7.4. Uridine Monophosphate (*UMP*)

The manufacturer LC-Abs measurement results for uridine monophosphate (*UMP*) are summarized in Table 118. Fig. 107 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 118. Uridine monophosphate (*UMP*) mass fraction results, mg/kg

Manufacturer	
ID	Value
1:1	178
2:1	178
3:1	177
4:1	177
5:1	177
5:2	175
6:1	176
7:1	176
8:1	178
1:2	178
8:2	177
N:	11
Mean:	177.0
SD:	1.0

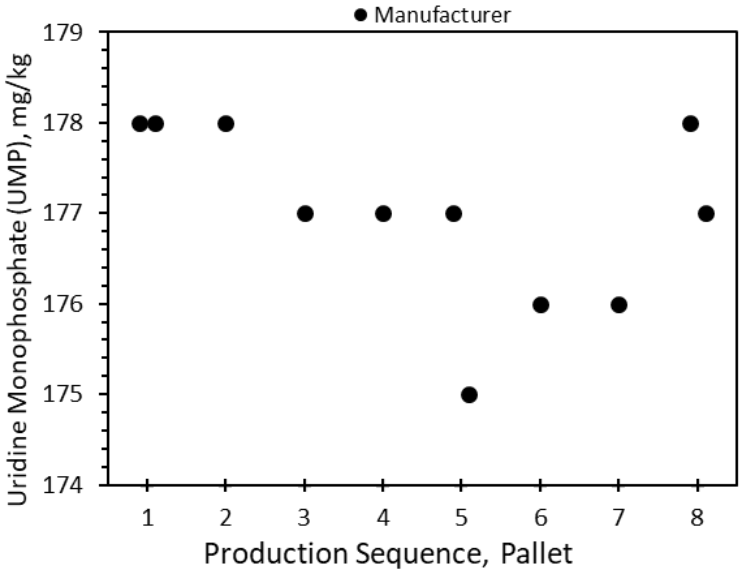

Fig. 107. Uridine monophosphate (*UMP*) mass fraction as a function of production sequence

5.7.5. Total Nucleotide Equivalents

The manufacturer LC-Abs measurement results for total nucleotide equivalents (without inosine monophosphate) are summarized in Table 119. Fig. 108 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 119. Total nucleotide equivalents[a] mass fraction results, mg/kg

	Manufacturer
ID	Value
1:1	849
2:1	850
3:1	849
4:1	850
5:1	847
5:2	846
6:1	847
7:1	840
8:1	849
1:2	844
8:2	846
N:	11
Mean:	847.0
SD:	3.0

[a] Without inosine monophosphate (IMP)

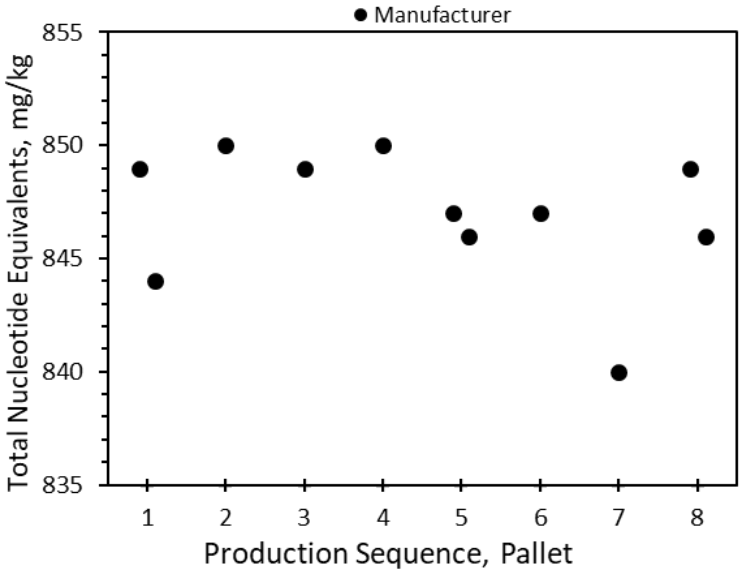

Fig. 108. Total nucleotide equivalents mass fraction as a function of production sequence[a]

5.8. Sugars

5.8.1. Galactooligosaccharides (GOS)

The manufacturer HPAEC-PAD measurement results for galactooligosaccharides (GOS) are summarized in Table 120. Fig. 109 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 120. Galactooligosaccharides (GOS) mass fraction results, g/100 g

	Manufacturer
ID	Value
1:1	2.44
2:1	2.56
3:1	2.59
4:1	2.63
5:1	2.63
5:2	2.64
6:1	2.42
7:1	2.53
8:1	2.65
1:2	2.65
8:2	2.58
N:	11
Mean:	2.575
SD:	0.081

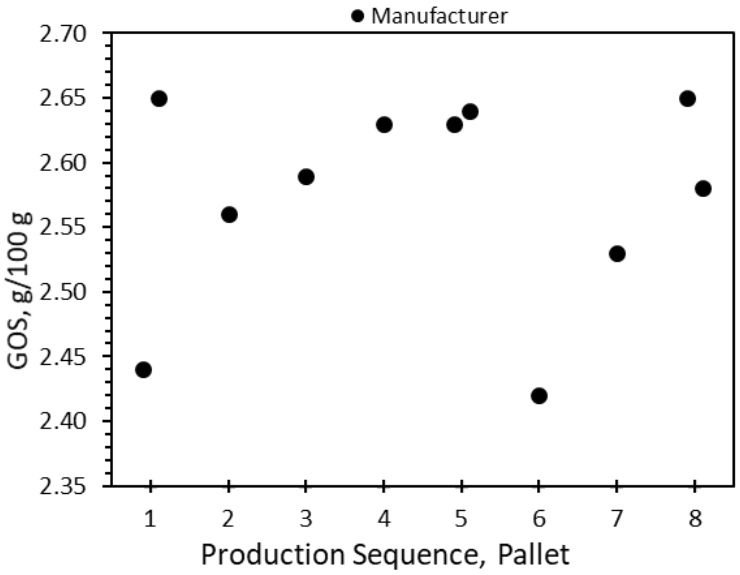

Fig. 109. Galactooligosaccharides (GOS) mass fraction as a function of production sequence

5.8.2. Lactose

The manufacturer GC-FID [60,61] measurement results for lactose are summarized in Table 121. Fig. 110 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 121. Lactose mass fraction results, %

Manufacturer	
ID	Value
1:1	47.89
2:1	47.46
3:1	46.44
4:1	48.05
5:1	47.60
5:2	46.35
6:1	47.15
7:1	47.32
8:1	47.64
1:2	46.93
8:2	47.89
N:	11
Mean:	47.34
SD:	0.57

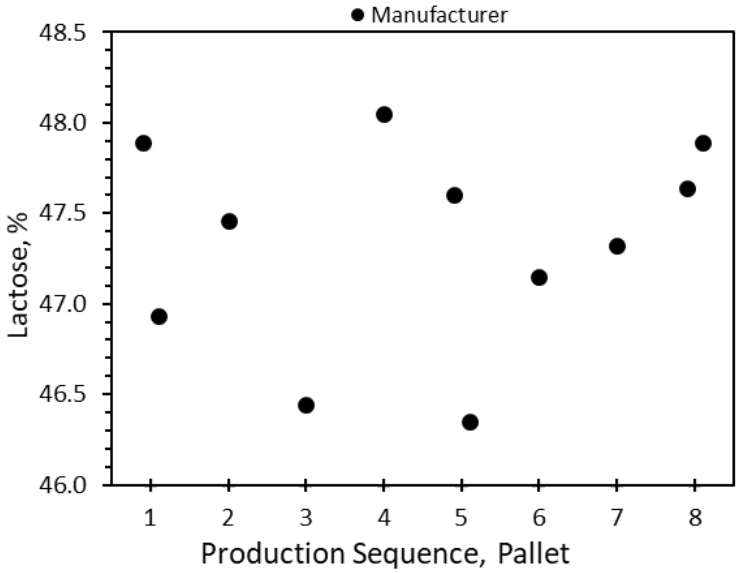

Fig. 110. Lactose mass fraction as a function of production sequence

5.8.3. Glucose

The manufacturer GC-FID [60,61] measurement results for glucose are summarized in Table 122. Fig. 111 displays these results as an approximate function of the production sequence, as determined by the pallet and box number.

Table 122. Glucose Mass Fraction Results, %

Manufacturer	
ID	Value
1:1	0.88
2:1	0.86
3:1	0.84
4:1	0.86
5:1	0.88
5:2	0.84
6:1	0.85
7:1	0.85
8:1	0.88
1:2	0.87
8:2	0.88
N:	11
Mean:	0.863
SD:	0.016

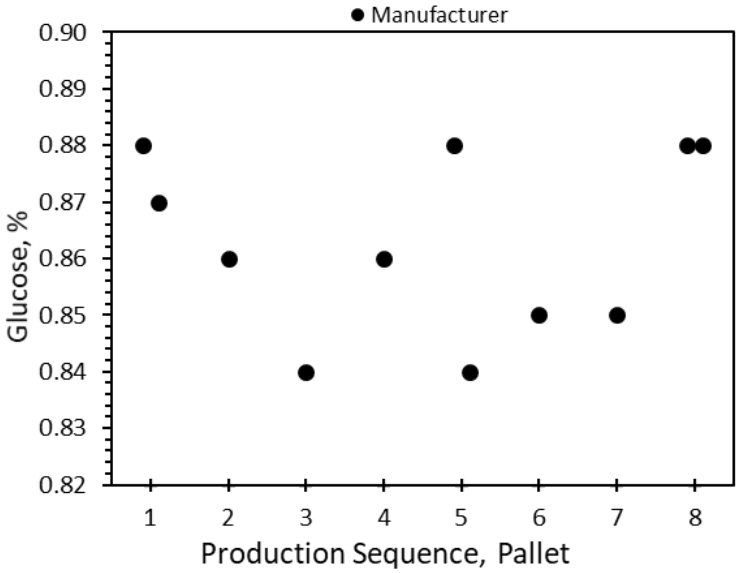

Fig. 111. Glucose mass fraction as a function of production sequence

5.8.4. Total Sugars

The manufacturer GC-FID [60,61] measurement results for total sugars are summarized in Table 123. Fig. 112 displays these results as an approximate function of the production sequence, as determined by the pallet and box number. In addition to the sugars summarized in the previous sections, fructose, galactose, maltose, and sucrose were assayed but not detected at a level of 0.10 %.

Table 123. Total sugars mass fraction results, %

Manufacturer	
ID	Value
1:1	48.76
2:1	48.32
3:1	47.28
4:1	48.91
5:1	48.48
5:2	47.19
6:1	48
7:1	48.17
8:1	48.52
1:2	47.81
8:2	48.77
N:	11
Mean:	48.20
SD:	0.58

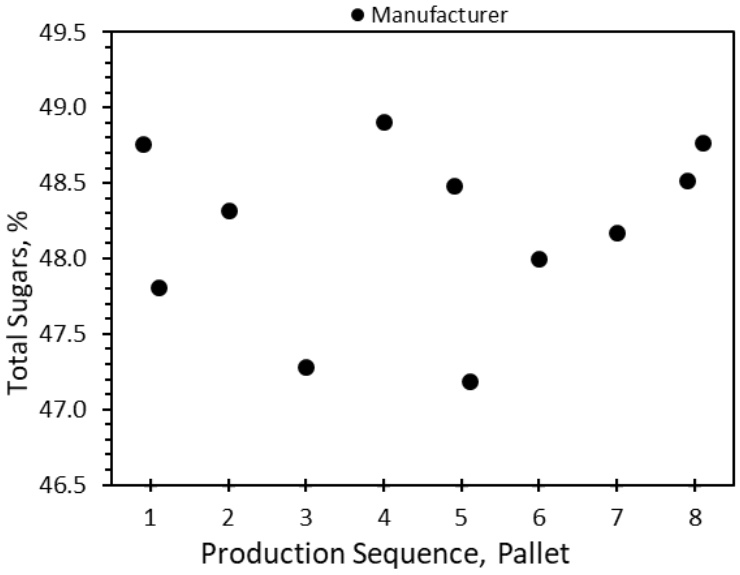

Fig. 112. Total sugars mass fraction as a function of production sequence

6. Value Assignment

6.1. Statistical Approaches

Statistical analysis of the data collected for the characterization of SRM 1849b was provided by the NIST Statistical Engineering Division.

6.1.1. Determination of Method Means and Uncertainties

Data for most analytes was provided by the material manufacturer. Data for some analytes was also collected using one or more NIST methods. For each analyte, the method mean for each method is the mean of the measurements available for that analyte using that method. The uncertainty of each such mean is the standard error of that mean.

For some fatty acids, all measurement data reported by the material manufacturer were identical. For these analytes, a Type B uncertainty related to instrument resolution [12] was incorporated into the uncertainties for the method means.

6.1.2. Determination of Interlaboratory Study Means and Uncertainties

There are often very marked differences between the results from the different collaborative laboratories. The method estimate for each analyte is the weighted median of the individual laboratory means for that analyte, with weights determined using a Laplace random effects model [62]. For most analytes in this SRM, the weighted median is equal to or very close to the unweighted median of laboratory means. The uncertainty of the weighted median is estimated using a bootstrap procedure based on a Laplace random effects model for the between-laboratory and within-laboratory effects [12,62–65].

6.1.3. Assignment of Values and Uncertainties

For each analyte, the assigned value is the unweighted mean of the method estimates available for that analyte. When the value is based on more than one method, the uncertainty of the combined mean is estimated using a bootstrap procedure based on a Gaussian random effects model for the between-method effects [12,63–65]. If only one method is available for an analyte, then that method estimate is the analyte estimate, with its corresponding expanded uncertainty.

Graphical analyses and analyses of variance with 5 % significance level were utilized for datasets that included box information to address issues of possible inhomogeneity of the SRM. No evidence of significant box effects was observed.

A number of outlier measurements were flagged by the analysts and omitted from the calculations. The deviance of these measurements from the others exceeded the usual variation, often differing by an order of magnitude or more. Other measurements may be questionable but

could not be determined to be unrepresentative extreme outliers because of the sparseness and variation of the rest of the data.

For some analytes, interlaboratory comparison data was not included in the final value assignment (e.g., vitamin A palmitate, total vitamin A, vitamin D_2, vitamin D_3). In these cases, analysts suspect bias in the data as a result of calibration, chromatographic peak identification, or final result calculation.

6.2. Certificate of Analysis

The results of the statistical analysis for all analytes are presented in the COA for SRM 1849b Infant/Adult Nutritional Formula I (milk-based). The most current version of the COA for SRM 1849b Infant/Adult Nutritional Formula I (milk-based) is available at https://shop.nist.gov/ccrz__ProductDetails?sku=1849b.

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Appendix A. List of Abbreviations and Acronyms

AAS
atomic absorption spectrometry

Abs
absorbance spectroscopy

ANOVA
analysis of variance

CNCbl
cyanocobalamin (vitamin B_{12})

COA
Certificate of Analysis

CVAAS
cold vapor atomic absorption spectroscopy

df
degrees of freedom

FL
fluorescence spectroscopy

FNSQAP
Food Nutrition and Safety Measurements Quality Assurance Program

GC-FID
gas chromatography with flame ionization detection

HAMQAP
Health Assessment Measurements Quality Assurance Program

HPAEC
high-performance anion-exchange chromatography

ICP
inductively coupled plasma

ILS
interlaboratory study

IC-ECD
ion chromatography with electrical conductivity detection

ISE
ion selective electrode

LC
liquid chromatography

MS
mass spectrometry

MS/MS
tandem mass spectrometry

NAA
neutron activation analysis

NIST
National Institute of Standards and Technology

NMR
nuclear magnetic resonance spectroscopy

OES
optical emission spectrometry

PAD
pulsed amperometric detection

PTFE
polytetrafluoroethylene

q^1-NMR
quantitative proton nuclear magnetic resonance spectroscopy

RG/M
Roese-Gottlieb/Mojonnier. Acid Digestion with Ether Extraction (AOAC 986.25 & 945.48, 989.05)

RM
reference material

SI
International System of Units

SPE
solid phase extraction

SRM
Standard Reference Material

TXRF
total reflection X-ray fluorescence spectrometry

WDXRF
wavelength dispersive X-ray fluorescence spectrometry

Appendix B. Change Log

Date	Type of Edit	Change	Location
Feb 2024	Authorship	Addition of Elena S.C. Wood	Front matter
	Technical	Addition of NIST method for carnitine and choline analysis	Section 3.5
	Technical	Addition of FNSQAP Exercise 2	Section 4
	Editorial	Removal of fatty acids as example text	Section 5
	Technical	Addition of FNSQAP data set for β-carotene	Section 5.2.22
	Technical	Addition of FNSQAP data set for lycopene	Section 5.2.23
	Technical	Addition of FNSQAP data set for lutein	Section 5.2.24
	Technical	Addition of NIST and FNSQAP data set for carnitine	Section 5.2.25
	Technical	Addition of NIST and FNSQAP data set for choline	Section 5.2.26
	Technical	Addition of FNSQAP data set for arachidonic acid	Section 5.5.17
	Technical	Addition of FNSQAP data set for β-carotene	Section 5.5.19
	Editorial	Relocation of list of acronyms	Appendix A
	Technical	Addition of acronyms for NMR and qNMR	Appendix A